3M Environmental Laboratory

Final Report- Analytical Study

Single-Dose Dermal Absorption/Toxicity Study of T-6067, T-6068 and T-6069 in Rabbits

In-Vivo Study Reference Number: HWI#6329-152

Study Number: AMDT-011095.1
Test Substance: L-13492 (T-6067, T-6068 and T-6069)

Name and Address of Sponsor: 3M SCD Division
367 Grove Street
St. Paul, MN 55106

Name and Address of Testing Facility:
3M Environmental Technology & Services
935 Bush Avenue
St. Paul, MN 55106

Method Numbers and Revisions:

AMDT-M-1-0,	Thermal Extraction of Fluoride by Means of a Modified Dohrmann DX2000 Organic Halide Analyzer-Liver
AMDT-M-2-0,	Fluoride Measurement by Means of an Orion EA940 Expandable Ion Analyzer
AMDT-M-4-0,	Extraction of Fluorochemicals from Rabbit Liver
AMDT-M-5-0,	Analysis of Rabbit Liver Extract for Fluorochemicals Using Electrospray Mass Spectrometry
AMDT-M-8-0,	Analysis of Fluoride Using the Skalar Segmented Flow Analyzer with Ion Selective Electrode
AMDT-M-14-0,	Thermal Extraction of Fluoride by Means of a Modified Dohrmann DX2000 Organic Halide Analyzer-Serum

Initiation Date: See attached protocol

Author: James D. Johnson

Approved By: James D. Johnson 11/28/95
James D. Johnson, Study Director — Completion Date

1.0 SUMMARY

A t-butyl ammonium salt of perfluorooctanoate in the form of treated fabric and as a liquid formulation was applied dermally to rabbits. Liver samples were analyzed at 28 days post dose by combustion for total organic fluorine. The results from treated animals were the same as control values. All total organic values were below the practical quantitation limit. Serum levels were also below the practical quantitation limits of the analysis for samples collected at day 1 and 2 after administration of the mixture or the treated fabric. From the pharmacokinetic study (HWI#6329-151), it would be unlikely that any extent of absorption could have been detected in this study.

2.0 INTRODUCTION

This study was designed to provide information as to assess the systemic absorption and toxicity and relative skin irritancy of L-13492 (T-6067, T-6068 and T-6069) when applied to the skin of rabbits.

In a pharmacokinetic study (HWI#6329-151), this compound showed little accumulation at 48 hours in liver. The biological half-life determined from serum levels in female rabbits was about 4 hours after an intravenous dose. A rabbit injected with 40 mg/kg died 5 minutes after injection. With this rapid half-life of disappearance, there is almost no chance of being able to see the material after a dermal dose when the first sampling interval is 24 hours for serum and 28 days for liver. However, if there is a sex difference and absorption it is possible that there will be some appearance of the material in male rabbits.

3.0 TEST MATERIALS

3.1 Test, Control, and Reference Substances and Matrices

3.1.1 Analytical Reference Substance: FC-95, lot 161 or 171. They are equivalent.

3.1.2 Analytical Reference Matrix: Bovine liver, bovine serum and rabbit serum

3.1.3 Analytical Control Substance: None

3.1.4 Analytical Control Matrix: Bovine liver, bovine serum and rabbit serum

3.2 Source of Materials: 3M ICP/PCP Division for FC-95, bovine liver from grocery store, bovine serum from Sigma Chemical Company, rabbit serum from AMDT-110394.1 control animals

3.3. Purity and Strength of Reference Substance: Responsibility of Sponsor.

3.4 Stability of Reference Substance: To be determined by Sponsor.

3.5 Storage Conditions for Test Materials: Room temperature for FC-95. For biological samples the storage is -20 ± 10^{0} C.

3.6 Disposition of Specimens: Biological tissues and fluids will be retained per GLP Regulation for the time period required for studies longer than 28 days.

4.0 EXPERIMENTAL - Overview

The tissues from animals dosed as described (HWI#6329-152), were available for analysis for fluorine compounds. At the discretion of the Study Director, a series of analytical tests could be performed. The screening for fluoride in liver via combustion was the most likely analysis to present definitive data for absorption if the pharmacokinetic test (intravenous administration) was positive for fluorine in the liver. Other available tests were electrospray mass spectroscopy and gas chromatography/mass spectrometry. However, if the definitive results could be obtained with combustion analysis alone, only the liver samples would be analyzed and any other tests would be for confirmation. And, if no organic fluorine increase was detectable even just using meter readings below the practical quantitation limit of the method, then further analysis of liver for specific fluorinated compounds would not be performed.

5.0 EXPERIMENTAL - METHODS

5.1 AMDT-M-1-0, Thermal Extraction of Fluoride by Means of a Modified Dohrmann DX2000 Organic Halide Analyzer-Liver

5.2 AMDT-M-2-0, Fluoride Measurement by Means of an Orion EA940 Expandable Ion Analyzer

5.3 AMDT-M-4-0, Extraction of Fluorochemicals from Rabbit Liver

5.4 AMDT-M-5-0, Analysis of Rabbit Liver Extract for Fluorochemicals Using Electrospray Mass Spectrometry

5.5 AMDT-M-8-0, Analysis of Fluoride Using the Skalar Segmented Flow Analyzer with Ion Selective Electrode

5.6 AMDT-M-14-0, Thermal Extraction of Fluoride by Means of a Modified Dohrmann DX2000 Organic Halide Analyzer-Serum

6.0 DATA ANALYSIS

The data are attached. There appears to no difference in total organic fluorine in whole liver between the controls and the 4, 16, 40 mg/kg groups. The dose was with respect to the T-numbered compound. In addition, there appears to be no difference between either fabric treatment and the controls. The values are all below the practical limit of quantitation and the comparisons are relative and based on meter reading. Very little total organic fluorine was observed in serum at 1 and 2 days post dose in either sex. The method did not work because the time interval of sampling was too late. The material if present would have been cleared before the samples were collected.

Other data was collected using electrospray mass spectrometry (see appendices). This data, although supportive, in the opinion of the Study Director is not required to reach the conclusion stated here and therefore is not discussed in detail.

6.1 Circumstances that May Affect the Quality of the Data: The possible problem with this analysis is that there is not a good marker at the times samples were collected to measure dermal absorption if it did occur. No conclusion can be drawn from this data as to the extent of dermal absorption.

7.0 CONCLUSION

In this study, no conclusion can be drawn from this data as to the extent of dermal absorption.

8.0 MAINTENANCE OF RAW DATA AND RECORDS

8.1 Raw Data and Data: Raw data, approved protocol, approved final report, appropriate specimens, and electronic data will be maintained in the AMDT archives.

9.0 APPENDICES

9.1 Protocol and Amendments

9.1.1 Protocol and Final Report HWI#6329-152 "Single-Dose Dermal Absorption/Toxicity Study of T-6067, T-6068 and T-6069 in Rabbits" (Protocol type TP3016.AB for dosing of animals, tissue collection, etc.)

9.1.2 Analytical protocol AMDT-011095.1

9.1.3 Amendment to analytical protocol AMDT-011095.1

9.2 Signed Reports from Individual Scientists: None

9.3 Quality Assurance Unit Statement: See attached

9.4 Key Personnel Involved in the Study: See attached

9.5 Materials and Equipment: See methods

9.6 Solutions, Reagents, and Standards: See methods

9.7 Sample Preparation: See methods

9.8 Quality Control Practices: See methods

9.9 Test Methods: See Protocol AMDT-011095.1

9.10 Instrument Settings: See methods

9.11 Data: See attached.

9.11.1 Summary and raw data; ug F^- in whole liver as determined by thermal extraction followed by analysis using Orion ion analyzer.

9.11.2 Summary and raw data; analysis of liver extracts using electrospray mass spectrometry.

9.11.3 Summary and raw data; ppm F^- in serum as determined by thermal extraction followed by analysis using Skalar segmented flow analyzer with ion selective electrode.

9.11.4 Summary and raw data; ppm F^- in serum as determined by thermal extraction followed by analysis using Orion ion analyzer.

9.1.1 Protocol and Final Report HWI#6329-152 "Single-Dose Dermal Absorption/Toxicity Study of T-6067, T-6068 and T-6069 in Rabbits" (Protocol type TP-3016.AB for dosing of animals, tissue collection, etc.)

HAZLETON
WISCONSIN
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CORNING

Sponsor:

3M
St. Paul, Minnesota

FINAL REPORT

Study Title:

Single-Dose Dermal Absorption/Toxicity
Study of T-6067, T-6068, and T-6069 in Rabbits

Author:

Steven M. Glaza

Study Completion Date:

May 26, 1995

Performing Laboratory:

Hazleton Wisconsin, Inc.
3301 Kinsman Boulevard
Madison, Wisconsin 53704

Laboratory Project Identification:

HWI 6329-152

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HWI 6329-152

QUALITY ASSURANCE STATEMENT

This report has been reviewed by the Quality Assurance Unit of Hazleton Wisconsin, Inc., in accordance with the Food and Drug Administration (FDA) Good Laboratory Practice Regulations, 21 CFR 58.35 (b) (6) (7). The following inspections were conducted and findings reported to the Study Director and management. Written status reports of inspections and findings are issued to Hazleton management monthly according to standard operating procedures.

Inspection Dates			Date Reported to	Date to
From	To	Phase	Study Director	Management
11/28/94	11/28/94	Protocol Review	11/28/94	12/10/94
12/08/94	12/08/94	Dose Preparation	12/08/94	01/10/95
02/20/95	02/22/95	Data/Report Review	02/22/95	03/10/95
05/24/95	05/24/95	Report Rereview	05/24/95	06/10/95
05/25/95	05/25/95	Report Rereview	05/25/95	06/10/95

Tracy Hrach for
Cecilia M. Danner
Representative, Quality Assurance Unit

5·26·95
Date

STUDY IDENTIFICATION

Single-Dose Dermal Absorption/Toxicity Study of
Study of T-6067, T-6068, and T-6069 in Rabbits

Test Materials	1. T-6067 2. T-6068 3. T-6069
Sponsor	3M Toxicology Service Medical Department 3M Center, Bldg. 220-2E-02 P.O. Box 33220 St. Paul, MN 55133-3220
Sponsor's Representative	John L. Butenhoff, PhD 3M Toxicology Service Medical Department 3M Center, Bldg. 220-2E-02 P.O. Box 33220 St. Paul, MN 55133-3220 (612) 733-1962
Study Director	Steven M. Glaza Hazleton Wisconsin, Inc. P.O. Box 7545 Madison, WI 53707-7545 (608) 241-7292
Study Location	Hazleton Wisconsin, Inc. Building No. 3 3802 Packers Avenue Madison, WI 53704
Study Timetable	
Study Initiation Date	December 6, 1994
Experimental (In-life) Start Date	December 8, 1994
In-life End Date	January 5, 1995
Experimental Termination Date	May 26, 1995
Study Completion Date	May 26, 1995

KEY PERSONNEL

Acute Toxicology

Steven M. Glaza
Study Director
Manager

Francis (Bud) W. McDonald
Study Coordinator

Patricia Padgham
In-life Supervisor

Rose M. Bridge
Report Supervisor

Quality Assurance

Sherry R. W. Petsel
Manager

Laboratory Animal Medicine

Cindy J. Cary, DVM
Diplomate, ACLAM
Supervisor

Anatomical Pathology

Thomas E. Palmer, PhD
Anatomical Pathologist

Jack Serfort/
Deborah L. Pirkel
Supervisors
Necropsy

Anne Mosher
Supervisor
Pathology Data

CONTENTS

SUMMARY

This study was done to assess the systemic absorption/toxicity and relative skin irritancy of T-6067, T-6068, and T-6069 when applied to the skin of rabbits.

The study was conducted using three male and three female acclimated rabbits of the Hra:(NZW)SPF strain for each treatment group.

Group	Test Material	Dose Level (mg/kg)	Number of Animals Males	Females
1 (Control)	Distilled water	0[a]	3	3
2	T-6067	4	3	3
3	T-6067	16	3	3
4	T-6067	40	3	3
5	T-6068	[b]	3	3
5	T-6069	[b]	3	3

[a] Administered at a dose volume of 2.0 mL/kg.
[b] Administered as a 10-cm x 10-cm piece of test material (fabric).

The back of each rabbit was clipped free of hair and a single dose of the respective material at the indicated dose level was administered to the skin of the rabbits. The treatment sites remained intact. The area of application was covered with a gauze bandage secured with paper tape around all edges and overwrapped with Saran Wrap® and Elastoplast® tape to provide an occlusive dressing for a 24-hour exposure period.

Clinical observations were conducted predose and at approximately 1, 2.5, and 4 hours after test or control material administration. Additional clinical observations and twice a day mortality checks were conducted daily thereafter for 28 days. Body weights were determined on Day -8 or Day -7 for randomization purposes, before test or control material administration (Day 1), and at in-life termination (Day 29). The initial dermal irritation reading was made before test or control material administration (recorded as the Day 1 reading). Subsequent readings of dermal irritation were made approximately 30 minutes after bandage removal (Day 2) and on Days 4 and 8. Blood samples were collected from a marginal ear vein of the animals before in-life initiation (Day 1), approximately 24-hours postdose (Day 2), on Days 4, 8, 15, and 22. In addition, at the time of necropsy on Day 29, approximately 20 mL of blood was obtained from each animal. All samples were centrifuged and separated into serum and cellular fractions. All animals were euthanized at termination of the in-life phase and necropsied. The whole liver, bile, an approximate 1-cm x 1-cm section of the dermal application site from all animals, and both kidneys from one male and one female in each group were collected at necropsy. The blood samples (serum and cellular fractions), livers, bile, dermal application sites, and kidneys were sent frozen to the Sponsor after termination of the in-life phase.

Application of T-6067, T-6068, and T-6031 did not result in any test material-related changes in body weight gain or macroscopic findings at necropsy. All animals appeared clinically normal throughout the study. The control material did not produce any dermal irritation. Test material T-6067 produced very slight irritation in a few of the animals at all three dose levels (4, 16, and 40 mg/kg). Both fabric test materials, T-6068 and T-6069, produced slight to moderate erythema and slight edema and/or desquamation reactions.

OBJECTIVE

The objective of this study was to assess the systemic toxicity/absorption and relative skin irritancy of test materials when applied to the skin of rabbits.

REGULATORY COMPLIANCE

This study was conducted in accordance with the U.S. Food and Drug Administration's Good Laboratory Practice Regulations for Nonclinical Laboratory Studies, 21 CFR 58. All procedures used in this study are in compliance with the Animal Welfare Act Regulations. In the opinion of the Sponsor and study director, the study did not unnecessarily duplicate any previous work.

TEST AND CONTROL MATERIALS

Identification

The test materials were identified and described as follows:

Identification	Physical Description
T-6067	Clear, colorless liquid
T-6068	White web cloth
T-6069	White web cloth

The control material was distilled water and was described as a clear, colorless liquid.

Purity and Stability

The Sponsor assumes responsibility for test material purity and stability determinations (including under test conditions). The purity and stability of the control material were considered to be adequate for the purposes of this study.

Storage and Retention

The test and control materials were stored at room temperature. A reserve sample of each test and control material was taken and will be retained in a freezer set to maintain a temperature of -20°C ±10°C for 10 years in accordance with Hazleton Wisconsin (HWI) Standard Operating Procedure (SOP). Any unused test material was returned to the Sponsor after completion of all in-life testing according to HWI SOP. Any remaining control material is retained for other testing and will not be discarded after issuance of the final report.

Safety Precautions

The test and control material handling procedures were according to HWI SOPs and policies.

TEST SYSTEM

Test Animal

Adult albino rabbits of the Hra:(NZW)SPF strain were procured from HRP, Inc., Kalamazoo, MI, on November 16, 1994 and maintained at the Hazleton Wisconsin facility at 3802 Packers Avenue, Madison, Wisconsin.

Housing

After receipt, the animals were acclimated for a period of at least 7 days. During acclimation and throughout the study, the animals were individually housed in screen-bottom stainless steel cages in temperature- and humidity-controlled quarters. Environmental controls for the animal room were set to maintain a temperature of 19° to 23°C, a relative humidity of 50% ±20%, and a 12-hour light/12-hour dark lighting cycle. In cases where variations from these conditions existed, they were documented and considered to have had no adverse effect on the study outcome.

Animal Diet

The animals were provided access to water *ad libitum* and a measured amount of Laboratory Rabbit Diet HF #5326, PMI Feeds, Inc. The feed is routinely analyzed by the manufacturer for nutritional components and environmental contaminants. Samples of the water are periodically analyzed by HWI. There were no known contaminants in the feed or water at levels that would have interfered with or affected the results of the study.

Selection of Test Animals

The animals were identified by animal number and corresponding ear tag and were placed into study groups using a stratified body weight randomization program. The randomization body weights were determined on Day -8. The weight variation of the animals for each group of each sex selected for the study did not exceed ±2 standard deviations of the mean weight, and the mean body weights for each group of each sex were not statistically different at the 5% probability level.

Study Design

Animals weighing from 2,377 to 2,796 g at initiation of treatment were placed into the following study groups:

Group	Test Material	Dose Level (mg/kg)	Number of Animals Males	Number of Animals Females
1 (Control)	Distilled water	0[a]	3	3
2	T-6067	4	3	3
3	T-6067	16	3	3
4	T-6067	40	3	3
5	T-6068	[b]	3	3
6	T-6069	[b]	3	3

a Administered at a dose volume of 2.0 mL/kg.
b Administered as a 10-cm x 10-cm section of test material (fabric).

Justification for Species Selection

Historically, the New Zealand White albino rabbit has been the animal of choice because of the large amount of background information on this species.

PROCEDURES

Preparation of Exposure Area

On the day before test material application, the back and, if necessary (to obtain unblemished skin), the flanks of each rabbit was clipped free of hair. The clipped area made up approximately 20% of the total body surface area. The test sites (intact skin) were inspected for interfering lesions, irritation, or defects that would preclude the use of any of the animals. The animals were clipped on Days 8 and 29 to aid in visualizing the application sites.

Dose Administration

All animals received a single administration of the respective test or control material. The day of treatment was designated as Day 1.

Group 1. An individual dose (2.0 mL/kg) was calculated and measured based on each animal's body weight on the day of treatment. The control material (distilled water) was applied evenly to the test site at a rate of approximately 0.05 mL/cm^2.

Groups 2, 3, and 4. The test material (T-6067) was administered undiluted to the test sites of the Groups 2, 3, and 4 animals (4, 16, or 40 mg/kg, respectively) using the average bulk density of 1.04 g/mL to determine the dose volume for each dose level. An individual dose of the test material was calculated for each animal based on its body weight on the day of treatment. The area of exposure for the 4, 16, and 40 mg/kg dose levels was 9, 9, and 16 cm^2, respectively. The approximate rate of application ranged from 0.001 to 0.006 mL/cm^2.

Groups 5 and 6. The test material (T-6068 or T-6069) was applied to the respective test site as a 10-cm x 10-cm section of material that was moistened with distilled water.

Each area of application was covered with a 10-cm x 10-cm gauze bandage secured with paper tape around all edges and overwrapped with Saran Wrap® and Elastoplast® tape to provide an occlusive dressing. Collars were used to restrain the animals during the 24-hour exposure period.

Approximately 24 hours after test or control material application, the restraining collars and bandages were removed and the residual test material was removed with tap water and disposable paper towels.

Reason for Route of Administration

The dermal route is a potential route of exposure in humans.

Observations of Animals

Clinical observations were conducted predose and at approximately 1, 2.5, and 4 hours after test or control material administration. Additional clinical observations and twice a day mortality checks (morning and afternoon) were conducted daily thereafter for 28 days.

Body weights were determined for randomization purposes on Day -8 or Day -7, before test material administration (Day 1), and at in-life termination (Day 29).

The initial dermal irritation reading was made before test or control material administration according to the Draize[1] technique (recorded as the Day 1 reading). Subsequent readings of dermal irritation were made approximately 30 minutes after bandage removal (Day 2) and on Days 4 and 8.

Sample Collections

Blood samples (approximately 4 mL) were collected from a marginal ear vein of all animals before experimental initiation (Day 1). Subsequent collection of blood was conducted 24-hours postdose (Day 2), and on Days 4, 8, 15, and 22. In addition, at the time of necropsy on Day 29, approximately 20 mL of blood was obtained from the posterior vena cava of each animal. All samples were centrifuged and separated into serum and cellular fractions. These samples were then stored in a freezer set to maintain a temperature of -20°C ±10°C until shipped to the Sponsor.

Pathology

At termination of the experimental phase (Day 29), animals were anesthetized with sodium pentobarbital, bled via the posterior vena cava, exsanguinated, and necropsied in random order. The sites of test and control material application were washed with lukewarm tap water before the necropsy procedure. All animals were subjected to an abbreviated gross necropsy examination and any abnormalities were recorded. The whole liver, bile, an approximate 1-cm x 1-cm section of the dermal application site from all animals, and both kidneys from the first male and female in each group were collected and immediately placed on dry ice, then placed in a freezer set to maintain a temperature of -20°C ±10°C. After necropsy, the animals were discarded.

Shipment of Blood, Bile, and Tissues

After experimental termination, the blood samples (serum and cellular fractions), livers, bile, dermal application sites, and kidneys were sent frozen (on dry ice) to the Sponsor (James D. Johnson, 3M E.E. & P.C., Bldg. 2-3E-09, 935 Bush Avenue, St. Paul, MN, 55106). The Sponsor is responsible for the retention and disposition of the samples. HWI does not accept any responsibility for the analysis of the tissue samples collected in this study nor are these results presented in this report.

Statistical Analyses

No statistical analyses were required by the protocol.

Location of Raw Data, Records, and Final Report

The raw data, records, and an original signed copy of the final report will be retained in the archives of HWI in accordance with HWI SOP.

RESULTS

Body Weights

Individual and mean body weights are in Table 1. All animals exhibited body weight gains from Day 1 to Day 29.

Clinical Observations

Individual clinical signs are in Table 2. All animals appeared normal throughout the study.

Dermal Irritation

Individual dermal irritation scores are in Table 3. The control material produced no dermal irritation. Test material T-6067 produced slight erythema reactions in one animal treated at 4 mg/kg, in two animals treated at 16 mg/kg, and in two animals treated at 40 mg/kg. Both fabric test materials, T-6068 and T-6069, produced slight to moderate erythema and slight edema and desquamation reactions.

Pathology

Individual animal pathology comments are presented in Table 4. There were no lesions observed in any of the animals.

Page 15 contains a pathology report by the study pathologist.

DISCUSSION

The acute systemic absorption/toxicity and relative skin irritancy of T-6067, T-6068, and T-6069 were evaluated in male and female albino rabbits when administered as a single dermal application. Application of the these materials did not result in any effects on body weight gain or macroscopic findings at necropsy. All animals appeared normal throughout the study. No dermal irritation was observed as a result of the application of the control material, very slight irritation was seen in a few of the animals treated with T-6067, and slight to moderate irritation was seen in the animals treated with T-6068 or T-6069.

SIGNATURE

Steven M. Glaza
Study Director
Acute Toxicology

5-26-95
Date

REFERENCE

1. Draize, J. H., "Acute Dermal Toxicity (Single Exposure)," In: *Appraisal of the Safety of Chemicals in Foods, Drugs and Cosmetics - Dermal Toxicity*, Association of Food and Drug Officials of the U.S., pp. 54-56 (1959).

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PATHOLOGY REPORT

There were six rabbits (three males and three females) each from six dose levels euthanized and necropsied at the termination of the study. The test material, dose level, day of death, and gross observations recorded for each animal are in the Individual Pathology Comments that follow this report.

At necropsy, there were no visible lesions in any of the animals. The liver, bile, an approximate 1-cm x 1-cm section of the dermal application site from all animals, and both kidneys from one male and one female in each group were collected, frozen, and sent to the Sponsor. After necropsy, the animals were discarded.

Thomas E. Palmer, PhD
Pathologist

5-26-95
Date

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Table 1
Individual and Mean Body Weights (g)

Male				Female			
Animal Number	Randomization Day -8	Day 1	Day 29	Animal Number	Randomization Day -8	Day 1	Day 29
Group 1 (Control) - Distilled Water (0 mg/kg)							
F52725	2,699	2,796	3,135	F52721	2,463	2,651	2,963
F52711	2,592	2,726	3,085	F52806	2,524	2,627	3,086
F52719	2,456	2,494	2,671	F52727	2,639	2,713	3,103
Mean	2,582	2,672	2,964		2,542	2,664	3,051
Group 2 - T-6067 (4 mg/kg)							
F52718	2,380	2,450	2,795	F52715	2,363	2,377	2,752
F52723	2,482	2,778	3,181	F52809	2,498	2,511	2,909
F52713	2,602	2,527	2,980	F52738	2,489	2,619	2,946
Mean	2,488	2,585	2,985		2,450	2,502	2,869
Group 3 - T-6067 (16 mg/kg)							
F52737	2,631	2,634	2,878	F52733	2,543	2,629	3,017
F52801	2,611	2,699	3,113	F52788	2,575*	2,429	2,994
F52724	2,634	2,616	2,933	F52720	2,560	2,627	3,028
Mean	2,625	2,650	2,975		2,559	2,562	3,013
Group 4 - T-6067 (40 mg/kg)							
F52802	2,543	2,785	3,088	F52714	2,523	2,578	2,893
F52730	2,408	2,520	2,766	F52734	2,557	2,660	2,839
F52731	2,339	2,489	2,715	F52774	2,364*	2,556	2,930
Mean	2,430	2,598	2,856		2,481	2,598	2,887

* Day -7 body weight.

Table 1 (Continued)
Individual and Mean Body Weights (g)

Male				Female			
Animal Number	Randomization Day -8	Day 1	Day 29	Animal Number	Randomization Day -8	Day 1	Day 29
		Group 5 - T-6068 (10 cm x 10 cm Section)					
F52729	2,431	2,545	2,893	F52716	2,652	2,778	3,211
F52803	2,442	2,596	2,920	F52787	2,494*	2,544	3,017
F52712	2,631	2,645	2,886	F52728	2,495	2,619	3,009
Mean	2,501	2,595	2,900		2,547	2,647	3,079
		Group 6 - T-6069 (10 cm x 10 cm Section)					
F52736	2,681	2,745	2,996	F52739	2,442	2,480	2,957
F52717	2,405	2,538	3,025	F52732	2,477	2,525	2,930
F52735	2,570	2,606	2,875	F52726	2,591	2,768	3,110
Mean	2,552	2,630	2,965		2,503	2,591	2,999

* Day -7 body weight.

Table 2
Individual Clinical Signs

Sex	Animal Number	Observation	1-4 Hours (Day 1)	Day 2 through 29
		Group 1 (Control) - Distilled Water (0 mg/kg)		
Male	F52725	Appeared normal	✓	✓
	F52711	Appeared normal	✓	✓
	F52719	Appeared normal	✓	✓
Female	F52721	Appeared normal	✓	✓
	F52806	Appeared normal	✓	✓
	F52727	Appeared normal	✓	✓
		Group 2 - T-6067 (4 mg/kg)		
Male	F52718	Appeared normal	✓	✓
	F52723	Appeared normal	✓	✓
	F52713	Appeared normal	✓	✓
Female	F52715	Appeared normal	✓	✓
	F52809	Appeared normal	✓	✓
	F52738	Appeared normal	✓	✓
		Group 3 - T-6067 (16 mg/kg)		
Male	F52737	Appeared normal	✓	✓
	F52801	Appeared normal	✓	✓
	F52724	Appeared normal	✓	✓
Female	F52733	Appeared normal	✓	✓
	F52788	Appeared normal	✓	✓
	F52720	Appeared normal	✓	✓

✓ Condition existed.

Table 2 (Continued)
Individual Clinical Signs

Sex	Animal Number	Observation	1-4 Hours (Day 1)	Day 2 through 29
		Group 4 - T-6067 (40 mg/kg)		
Male	F52802	Appeared normal	✓	✓
	F52730	Appeared normal	✓	✓
	F52731	Appeared normal	✓	✓
Female	F52714	Appeared normal	✓	✓
	F52734	Appeared normal	✓	✓
	F52774	Appeared normal	✓	✓
		Group 5 - T-6068 (10 cm x 10 cm Section)		
Male	F52729	Appeared normal	✓	✓
	F52803	Appeared normal	✓	✓
	F52712	Appeared normal	✓	✓
Female	F52716	Appeared normal	✓	✓
	F52787	Appeared normal	✓	✓
	F52728	Appeared normal	✓	✓
		Group 6 - T-6069 (10 cm x 10 cm Section)		
Male	F52736	Appeared normal	✓	✓
	F52717	Appeared normal	✓	✓
	F52735	Appeared normal	✓	✓
Female	F52739	Appeared normal	✓	✓
	F52732	Appeared normal	✓	✓
	F52726	Appeared normal	✓	✓

✓ Condition existed.

Table 3
Individual Dermal Irritation Scores

Group 1 (Control) - Distilled Water (0 mg/kg)

Dermal Reaction	Males Study Day 1	2	4	8	Females Study Day 1	2	4	8
	Animal No. F52725				Animal No. F52721			
Erythema	0	0	0	0	0	0	0	0
Edema	0	0	0	0	0	0	0	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	0	0	0	0	0
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0
	Animal No. F52711				Animal No. F52806			
Erythema	0	0	0	0	0	0	0	0
Edema	0	0	0	0	0	0	0	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	0	0	0	0	0
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0
	Animal No. F52719				Animal No. F52727			
Erythema	0	0	0	0	0	0	0	0
Edema	0	0	0	0	0	0	0	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	0	0	0	0	0
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0

Table 3 (Continued)
Individual Dermal Irritation Scores

Group 2 - T-6067 (4 mg/kg)

Dermal Reaction	Males Study Day 1	2	4	8	Females Study Day 1	2	4	8
	Animal No. F52718				Animal No. F52715			
Erythema	0	0	0	0	0	1	0	0
Edema	0	0	0	0	0	0	0	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	0	0	0	0	0
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0
	Animal No. F52723				Animal No. F52809			
Erythema	0	0	0	0	0	0	0	0
Edema	0	0	0	0	0	0	0	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	0	0	0	0	0
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0
	Animal No. F52713				Animal No. F52738			
Erythema	0	0	0	0	0	0	0	0
Edema	0	0	0	0	0	0	0	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	0	0	0	0	0
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0

Table 3 (Continued)
Individual Dermal Irritation Scores

Group 3 - T-6067 (16 mg/kg)

Dermal Reaction	Males Study Day 1	2	4	8	Females Study Day 1	2	4	8
	Animal No. F52737				Animal No. F52733			
Erythema	0	1	0	0	0	0	0	0
Edema	0	0	0	0	0	0	0	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	0	0	0	0	0
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0
	Animal No. F52801				Animal No. F52788			
Erythema	0	0	0	0	0	0	0	0
Edema	0	0	0	0	0	0	0	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	0	0	0	0	0
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0
	Animal No. F52724				Animal No. F52720			
Erythema	0	1	0	0	0	0	0	0
Edema	0	0	0	0	0	0	0	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	0	0	0	0	0
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0

Table 3 (Continued)
Individual Dermal Irritation Scores

Group 4 - T-6067 (40 mg/kg)

Dermal Reaction	Males Study Day 1	2	4	8	Females Study Day 1	2	4	8
	Animal No. F52802				Animal No. F52714			
Erythema	0	1	0	0	0	0	0	0
Edema	0	0	0	0	0	0	0	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	0	0	0	0	0
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0
	Animal No. F52730				Animal No. F52734			
Erythema	0	0	0	0	0	0	0	0
Edema	0	0	0	0	0	0	0	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	0	0	0	0	0
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0
	Animal No. F52731				Animal No. F52774			
Erythema	0	0	0	0	0	1	0	0
Edema	0	0	0	0	0	0	0	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	0	0	0	0	0
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0

Table 3 (Continued)
Individual Dermal Irritation Scores

Group 5 - T-6068 (10 cm x 10 cm Section)

Dermal Reaction	Males Study Day 1	2	4	8	Females Study Day 1	2	4	8
	Animal No. F52729				Animal No. F52716			
Erythema	0	1	1	1	0	0	0	0
Edema	0	0	0	0	0	0	0	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	1	0	0	0	0
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0
	Animal No. F52803				Animal No. F52787			
Erythema	0	1	0	0	0	1	1	0
Edema	0	0	0	0	0	0	0	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	0	0	0	0	0
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0
	Animal No. F52712				Animal No. F52728			
Erythema	0	1	1	0	0	1	2	1
Edema	0	0	0	0	0	0	0	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	0	0	0	0	1
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0

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Table 3 (Continued)
Individual Dermal Irritation Scores

Group 6 - T-6069 (10 cm x 10 cm Section)

Dermal Reaction	Males Study Day 1	2	4	8	Females Study Day 1	2	4	8
	Animal No. F52736				Animal No. F52739			
Erythema	0	1	2	1	0	1	2	1
Edema	0	0	0	0	0	1	1	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	1	0	0	0	1
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0
	Animal No. F52717				Animal No. F52732			
Erythema	0	1	1	0	0	1	1	0
Edema	0	0	0	0	0	1	1	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	0	0	0	0	0
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0
	Animal No. F52735				Animal No. F52726			
Erythema	0	1	2	1	0	1	2	1
Edema	0	1	1	0	0	0	1	0
Atonia	0	0	0	0	0	0	0	0
Desquamation	0	0	0	1	0	0	0	1
Coriaceousness	0	0	0	0	0	0	0	0
Fissuring	0	0	0	0	0	0	0	0

Table 4
Individual Pathology Comments

Animal Number	Sex	Test Day Died	Test Day Sacrificed	Necropsy Observation
		Group 1 (Control) - Distilled Water (0 mg/kg)		
F52725	M	-	29	No visible lesions.
F52711	M	-	29	No visible lesions.
F52719	M	-	29	No visible lesions.
F52721	F	-	29	No visible lesions.
F52806	F	-	29	No visible lesions.
F52727	F	-	29	No visible lesions.
		Group 2 - T-6067 (4 mg/kg)		
F52718	M	-	29	No visible lesions.
F52723	M	-	29	No visible lesions.
F52713	M	-	29	No visible lesions.
F52715	F	-	29	No visible lesions.
F52809	F	-	29	No visible lesions.
F52738	F	-	29	No visible lesions.
		Group 3 - T-6067 (16 mg/kg)		
F52737	M	-	29	No visible lesions.
F52801	M	-	29	No visible lesions.
F52724	M	-	29	No visible lesions.
F52733	F	-	29	No visible lesions.
F52788	F	-	29	No visible lesions.
F52720	F	-	29	No visible lesions.

- Not applicable.

Table 4 (Continued)
Individual Pathology Comments

Animal Number	Sex	Test Day Died	Test Day Sacrificed	Necropsy Observation
		Group 4 - T-6067 (40 mg/kg)		
F52802	M	-	29	No visible lesions.
F52730	M	-	29	No visible lesions.
F52731	M	-	29	No visible lesions.
F52714	F	-	29	No visible lesions.
F52734	F	-	29	No visible lesions.
F52774	F	-	29	No visible lesions.
		Group 5 - T-6068 (10 cm x 10 cm Section)		
F52729	M	-	29	No visible lesions.
F52803	M	-	29	No visible lesions.
F52712	M	-	29	No visible lesions.
F52716	F	-	29	No visible lesions.
F52787	F	-	29	No visible lesions.
F52728	F	-	29	No visible lesions.
		Group 6 - T-6069 (10 cm x 10 cm Section)		
F52736	M	-	29	No visible lesions.
F52717	M	-	29	No visible lesions.
F52735	M	-	29	No visible lesions.
F52739	F	-	29	No visible lesions.
F52732	F	-	29	No visible lesions.
F52726	F	-	29	No visible lesions.

- Not applicable.

APPENDIX A

Protocol TP3016.AB

HAZLETON
WISCONSIN
POST OFFICE BOX 7545
MADISON WI 53707-7545

CORNING

Sponsor:

3M
St. Paul, Minnesota

PROTOCOL TP3016.AB

Study Title:

Single-Dose Dermal Absorption/Toxicity Study of
T-6067, T-6068, and T-6069 in Rabbits

Date:

December 6, 1994

Performing Laboratory:

Hazleton Wisconsin, Inc.
3301 Kinsman Boulevard
Madison, Wisconsin 53704

Laboratory Project Identification:

HWI 6329-152

Phone 608 241 4471 Fax 608 24
EXPRESS MAIL DELIVERY 3301 KINSMAN BLVD MADISON WI 53704

STUDY IDENTIFICATION

Single-Dose Dermal Absorption/Toxicity Study of
T-6067, T-6068, and T-6069 in Rabbits

HWI No.	6329-152
Test Materials	1. T-6067 2. T-6068 3. T-6069
Sponsor	3M Toxicology Services 220-2E-02 3M Center St. Paul, MN 55144
Sponsor's Representative	John L. Butenhoff, PhD 3M Toxicology Services 220-2E-02 3M Center St. Paul, MN 55144 (612) 733-1962
Study Director	Steven M. Glaza Hazleton Wisconsin, Inc. P.O. Box 7545 Madison, WI 53707-7545 (608) 241-7292
Study Location	Hazleton Wisconsin, Inc. Building No. 3 3802 Packers Avenue Madison, WI 53704
Proposed Study Timetable	
Experimental Start Date	December 8, 1994
Experimental Termination Date	January 5, 1995
Draft Report Date	February 16, 1995

1. Study
Single-Dose Dermal Absorption/Toxicity Study in Rabbits

2. Purpose
To assess the systemic absorption and toxicity and relative skin irritancy of test materials when applied to the skin of rabbits

3. Regulatory Compliance
This study will be conducted in accordance with the following Good Laboratory Practice Regulations/Standards/Guidelines:

[ ] Conduct as a Nonregulated Study
[X] 21 CFR 58 (FDA)
[ ] 40 CFR 160 (EPA-FIFRA)
[ ] 40 CFR 792 (EPA-TSCA)
[ ] C(81)30 (Final) (OECD)
[ ] 59 Nohsan No. 3850 (Japanese MAFF)
[ ] Notification No. 313 (Japanese MOHW)

All procedures in this protocol are in compliance with the Animal Welfare Act Regulations. In the opinion of the Sponsor and study director, the study does not unnecessarily duplicate any previous work.

4. Quality Assurance
The protocol, study conduct, and the final report will be audited by the Quality Assurance Unit in accordance with Hazleton Wisconsin (HWI) Standard Operating Procedures (SOPs) and policies.

5. Test Materials

A. Identification
1. T-6067
2. T-6068
3. T-6069

B. Physical Description
1. (To be documented in the raw data)
2. (To be documented in the raw data)
3. (To be documented in the raw data)

C. Purity and Stability
The Sponsor assumes responsibility for purity and stability determinations (including under test conditions).

D. Storage
Room temperature

E. Reserve Samples
Reserve sample(s) of each batch/lot of test and control materials will be taken for this study.

The test and control material reserve samples will be stored at HWI in a freezer set to maintain a temperature of -20°C ±10°C for 10 years per HWI SOP. The Sponsor will be contacted after 10 years for disposition in accordance with the appropriate regulatory Good Laboratory Practices.

F. Retention
Any unused test materials will be returned to the Sponsor after completion of the in-life phase of the study.

G. Safety Precautions
As required by HWI SOPs and policies

6. Control Material

A. Identification
Distilled water

B. Physical Description
Clear, colorless liquid

C. Purity and Stability
The purity and stability of this manufactured material is considered to be adequate for the purposes of this study.

D. Storage Conditions
Room temperature

E. Reserve Samples
See Section 5. E. Reserve Samples

F. Retention
Any remaining control material may be used for other testing and will not be discarded after issuance of the final report.

G. Safety Precautions
As required by HWI SOPs and policies

7. Experimental Design

A. Animals

(1) Species
Rabbit

(2) Strain/Source
Hra:(NZW)SPF/HRP, Inc.

(3) Age at Initiation
Adult

(4) Weight at Initiation
2.0 to 3.0 kg

(5) Number and Sex
18 males and 18 females

(6) Identification
Individual numbered ear tag

(7) Husbandry

(a) Housing
Individually, in screen-bottom stainless steel cages (heavy gauge)

(b) Food
A measured amount of Laboratory Rabbit Diet HF #5326 (PMI Feeds, Inc.). The food is routinely analyzed by the manufacturer for nutritional components and environmental contaminants.

(c) Water
Ad libitum from an automatic system. Samples of the water are analyzed by HWI for total dissolved solids, hardness, and specified microbiological content and for selected elements, heavy metals, organophosphates, and chlorinated hydrocarbons.

(d) Contaminants
There are no known contaminants in the food or water that would interfere with this study.

(e) Environment
Environmental controls for the animal room will be set to maintain a temperature of 19°C to 23°C, a relative humidity of 50% ±20%, and a 12-hour light/12-hour dark cycle.

(f) Acclimation
At least 7 days

(8) Selection of Test Animals
Based on health and body weight according to HWI SOPs. An adequate number of extra animals will be purchased so that no animal in obviously poor health is placed on test. The animals will be placed into study groups using a stratified body weight randomization program within nine days of study initiation.

(9) Justification for Species Selection
Historically, the New Zealand White albino rabbit has been the animal of choice because of the large amount of background information on this species.

B. Dose Administration

(1) Test Groups

Group	Test Material	Dose Level (mg/kg)	Number of Animals Males	Number of Animals Females
1 (Control)	Distilled water	0*	3	3
2	T-6067	4	3	3
3	T-6067	16	3	3
4	T-6067	40	3	3
5	T-6068	**	3	3
6	T-6069	**	3	3

* To be administered at a dose volume of 2.0 mL/kg
** To be administered as a 10.0-cm x 10.0-cm piece of test material (fabric)

(2) Preparation of Exposure Area
On the day before test material application, the back and, if necessary (to obtain unblemished skin), the flanks of each rabbit will be clipped free of hair with an electric clipper. The shaved area will constitute approximately 20% of the total body surface area. The treatment sites (intact skin) will be inspected for interfering lesions, irritation, or defects that would preclude the use of any of the animals. The animals will be clipped as needed throughout the study.

(3) Dose Administration
All animals will receive a single administration of the respective test or control material. The day of treatment will be designated as Day 1. The respective doses for the animals in Groups 1, 2, 3, and 4 will be based on the animal's body weight just before administration and spread onto the area of exposure in a thin and uniform layer. The Group 1, 2, 3, and 4 materials will be applied undiluted. The Group 5 and 6 materials will be applied as a 10.0-cm x 10.0-cm piece of the respective test material moistened with distilled water. The area of application (Groups 1-6) will be covered with a 10-cm x 10-cm gauze bandage secured with paper tape around all edges and overwrapped with Saran Wrap® and Elastoplast® tape to provide an occlusive dressing. The rabbits will be collared during the 24-hour application period.

(4) Reason for Route of Administration
The dermal route is a potential route of exposure in humans.

(5) Removal of Test Material
Approximately 24 hours after test or control material application the bandages and collars will be removed and the residual test material will be removed using water or an appropriate solvent, if necessary.

C. Observation of Animals

(1) Clinical Observations
For clinical signs before test or control material administration and for clinical signs and mortality at approximately 1, 2.5, and 4 hours after test material administration (Day 1) and daily thereafter for clinical signs, and twice daily (a.m. and p.m.) for mortality for at least 28 days. Observations may be extended when directed by the study director.

(2) Reading of Dermal Irritation
Before test or control material administration the initial dermal irritation reading will be made and recorded as the Day 1 reading (Attachment 1). Additional dermal irritation readings will be made approximately 30 minutes after bandage removal (Day 2) and on Study Days 4 and 8. Individual dermal irritation records will be maintained for each animal.

(3) Body Weights
Just before test or control material application (Day 1), on Day 29, and at unscheduled death (when survival exceeds 1 day)

(4) Sample Collections

(a) Frequency
Before initiation (Day 1), approximately 24 hours post-dose (Day 2), Days 4, 8, 15, 22, and at experimental termination (Day 29)

(b) Number of Animals
All

(c) Method of Collection
Blood samples (approximately 4 mL) will be collected from the marginal ear vein of either ear on Days 1, 2, 4, 8, 15, and 22. Approximately 20 mL of blood (actual volume to be documented in the raw data) will be obtained from the posterior vena cava of each animal sacrificed in a moribund condition or sacrificed at the time of necropsy (Day 29). The samples will be stored at room temperature and then centrifuged, and the separate serum and cellular fractions stored in a freezer set to maintain -20°C ±10°C. The separated serum and cellular fractions will be sent frozen to the Sponsor after experimental termination.

Samples will be shipped to:

James D. Johnson
3M E.E. & P.C.
Bldg. 2-3E-09
935 Bush Avenue
St. Paul, MN 55106

James D. Johnson or alternate will be notified by telephone at (612) 778-5294 prior to the shipment of the samples.

D. Pathology

(1) Unscheduled Sacrifices and Deaths
Any animal dying during the study or sacrificed in a moribund condition will be subjected to an abbreviated gross necropsy examination and all abnormalities will be recorded. Animals in a moribund condition will be anesthetized with sodium pentobarbital (via injection in the marginal ear vein), bled via the vena cava, and exsanguinated. After necropsy, the animals will be discarded.

(2) Scheduled Sacrifice
At termination of the experimental phase (Day 29), surviving animals will be anesthetized with sodium pentobarbital (via injection in the marginal ear vein), bled via the vena cava, exsanguinated, and subjected to an abbreviated gross necropsy examination. The animals will be necropsied in random order and all abnormalities will be recorded.

(a) Sample Collection
The sites of test and control material application will be washed with lukewarm tap water prior to the necropsy procedure. The whole liver, bile, an approximate 1-cm x 1-cm section of the dermal application site from all animals, and both kidneys from the first male and female necropsied in each group will be collected and immediately placed in a freezer set to maintain a temperature of -20°C ±10°C. After necropsy, the animals will be discarded.

The tissues (liver, bile, dermal application site, kidneys) will be sent frozen on dry ice to the Sponsor after experimental termination. The samples will be shipped to the person listed in Section 7.C.(4).(c). The Sponsor is responsible for the retention and disposition of the samples.

E. Statistical Analyses
No statistical analyses are required.

8. Report
A final report including those items listed below will be submitted.

Description of the test and control materials
Description of the test system
Procedures
Dates of experimental initiation and termination
Tabulation of mortality data by sex and dose level
Description of any toxic effects/dermal irritation
Tabulation of mean body weights by sex and dose level
Gross pathology findings/gross pathology report

9. Location of Raw Data, Records, and Final Report

Original data, or copies thereof, will be available at HWI to facilitate auditing the study during its progress and before acceptance of the final report. When the final report is completed, all original paper data, including those item listed below will be retained in the archives of HWI according to HWI SOP.

Protocol and protocol amendments
Dose preparation records
In-life records
  Body weights
  Dose administration
  Observations
Anatomical pathology records
Sample collection records
Shipping records
Study correspondence
Final report (original signed copy)

The following supporting records will be retained at HWI but will not be archived with the study data.

Animal receipt/acclimation records
Water analysis records
Animal room temperature and humidity records
Refrigerator and freezer temperature records
Instrument calibration and maintenance records

PROTOCOL APPROVAL

John L. Butenhoff, PhD
Sponsor's Representative
3M

12-12-94
Date

Steven M. Glaza
Study Director
Acute Toxicology
Hazleton Wisconsin, Inc.

12-6-94
Date

Representative
Quality Assurance Unit
Hazleton Wisconsin, Inc.

12/6/94
Date

(6329-152.protdsk1)

9.1.2 Analytical protocol AMDT-011095.1

3M Environmental Laboratory

Protocol - Analytical Study

Single-Dose Dermal Absorption/Toxicity Study of T-6067, T-6068 and T-6069 in Rabbits

In-Vivo Study Reference Number: HWI#6329-152

Study Number: AMDT-011095.1
Test Substance: L-13492 (T-6067, T-6068 and T-6069)

Name and Address of Sponsor: 3M SCD Division
367 Grove Street
St. Paul, MN 55106

Name and Address of Testing Facility:
3M Environmental Technology and Services
935 Bush Avenue
St. Paul, MN 55106

Proposed Initiation Date: July 25, 1995
Proposed Completion Date: August 25, 1995

Method Numbers and Revisions:

AMDT-M-1-0,	Thermal Extraction of Fluoride by Means of a Modified Dohrmann DX2000 Organic Halide Analyzer-Liver
AMDT-M-2-0,	Fluoride Measurement by Means of an Orion EA940 Expandable Ion Analyzer
AMDT-M-4-0,	Extraction of Fluorochemicals from Rabbit Liver
AMDT-M-5-0,	Analysis of Rabbit Liver Extract for Fluorochemicals Using Electrospray Mass Spectrometry
AMDT-M-8-0,	Analysis of Fluoride Using the Skalar Segmented Flow Analyzer with Ion Selective Electrode
AMDT-M-14-0,	Thermal Extraction of Fluoride by Means of a Modified Dohrmann DX2000 Organic Halide Analyzer-Serum

Author: James D. Johnson

Approved By:

James D. Johnson 11/16/95
James D. Johnson Date
Study Director

John 2. Butenhoff 11/20/95
John Butenhoff, PhD Date
Sponsor Representative

1.0 PURPOSE

This study is designed to provide information as to whether L-13492 (T-6067, T-6068 and T-6069) is dermally absorbed. The analytical aspect of this study is to determine total organic fluorine in the tissue and serum of rabbits at various times post dose dermal application of L-13492.

2.0 TEST MATERIALS

2.1 Test, Control, and Reference Substances and Matrices

2.1.1 Analytical Reference Substance: FC-95, lot 161 or 171. They are equivalent.

2.1.2 Analytical Reference Matrix: Bovine liver and bovine serum

2.1.3 Analytical Control Substance: None

2.1.4 Analytical Control Matrix: Bovine liver and bovine serum

2.2 Source of Materials: 3M ICP/PCP Division (2.1.1), Grocery store (2.1.2, 2.1.4 liver), Sigma Chemical Company (2.1.2, 2.1.4 serum)

2.3 Number of Test and Control Samples: Tissues and fluids from 30 test animals and 6 control animals. Tissues and fluids include liver, kidney, serum, cellular fraction, dermal application site and bile. Analysis of these tissues will be at the discretion of the Study Director.

2.4 Identification of Test and Control Samples: The samples are identified using the HWI animal identification number which consists of a letter and five digit number, plus the tissue identity and day identity (serum).

2.5 Purity and Strength of Reference Substance: To be determined by Sponsor.

2.6 Stability of Reference Substance: To be determined by Sponsor.

2.7 Storage Conditions for Test Materials: Room temperature (2.1.1), -20 ± 10°C (2.1.2, 2.1.4). Test and Control samples will be received according to AMDT-S-10-0.

2.8 Disposition of Specimens: Biological tissues and fluids will be retained per GLP Regulation for the time period required for studies longer than 28 days.

2.9 Safety Precautions: Refer to appropriate MSDS. Wear appropriate laboratory attire. Use caution when handling knives for cutting the samples.

3.0 EXPERIMENTAL - Overview

The tissues from animals dosed as described (HWI#6329-152), are available for analysis for fluorine compounds. At the discretion of the Study Director, a series of analytical tests can be performed. The screening for fluoride in liver via combustion (See Methods--next Section) is the appropriate analysis to present definitive data for fluorine in the liver. Not all of the tissues and fluid samples will be analyzed. When sufficient data has been collected to meet the objectives of the study in the opinion of the Study Director, analysis will cease.

4.0 EXPERIMENTAL - Methods

4.1 Liver and Serum screening methods: (attached)

4.1.1 AMDT-M-1-0, Thermal Extraction of Fluoride by Means of a Modified Dohrmann DX2000 Organic Halide Analyzer-Liver

4.1.2 AMDT-M-2-0, Fluoride Measurement by Means of an Orion EA940 Expandable Ion Analyzer

4.1.3 AMDT-M-4-0, Extraction of Fluorochemicals from Rabbit Liver

4.1.4 AMDT-M-5-0, Analysis of Rabbit Liver Extract for Fluorochemicals using Electrospray Mass Spectrometry

4.1.5 AMDT-M-8-0, Analysis of Fluoride Using the Skalar Segmented Flow Analyzer with Ion Selective Electrode

4.1.6 AMDT-M-14-0, Thermal Extraction of Fluoride by Means of a Modified Dohrmann DX2000 Organic Halide Analyzer-Serum

5.0 DATA ANALYSIS

5.1 Data Reporting: Data will be reported as a concentration (weight/weight) of fluoride per tissue or fluid, or as FC-143 (electrospray mass spectrometry) per unit of tissue or fluid. Statistics used, at the discretion of the Study Director, may include regression analysis of serum concentrations with time and averages and standard deviations of concentrations for different dose groups. If necessary (in the opinion of the Study Director), simple statistical tests such as Student's t test may be applied to determine statistical difference.

6.0 MAINTENANCE OF RAW DATA AND RECORDS

6.1 Raw Data and Records: Raw data, approved protocol, appropriate specimens, approved final report, and electronic data will be maintained in the AMDT archives.

7.0 REFERENCES

7.1 AMDT-S-10-0, Sample Tracking System

8.0 ATTACHMENTS

8.1 AMDT-M-1-0, Thermal Extraction of Fluoride by Means of a Modified Dohrmann DX2000 Organic Halide Analyzer-Liver

8.2 AMDT-M-2-0, Fluoride Measurement by Means of an Orion EA940 Expandable Ion Analyzer

8.3 AMDT-M-4-0, Extraction of Fluorochemicals from Rabbit Liver

8.4 AMDT-M-5-0, Analysis of Rabbit Liver Extract for Fluorochemicals Using Electrospray Mass Spectrometry

8.5 AMDT-M-8-0, Analysis of Fluoride Using the Skalar Segmented Flow Analyzer with Ion Selective Electrode

8.6 AMDT-M-14-0, Thermal Extraction of Fluoride by Means of a Modified Dohrmann DX2000 Organic Halide Analyzer-Serum

3M Environmental Laboratory

Method

Thermal Extraction of Fluoride by Means of a Modified Dohrmann DX2000 Organic Halide Analyzer - Liver

Method Identification Number: AMDT-M-1 **Adoption Date:** 10-4-95

Revision Number: 0 **Revision Date:** None

Author: Rich Youngblom

Approved by:

Group Leader 10/3/95
Date

Quality Assurance 10-4-95
Date

Software: MS Word 5.1a

Affected Documents: AMDT-M-2 Fluoride Measurement by Means of an Orion EA940 Expandable Ion Analyzer
AMDT-EP-3 Routine Maintenance of a Modified Dohrmann DX2000 Organic Halide Analyzer

1.0 SCOPE , APPLICABLE COMPOUNDS, AND MATRICES

1.1 Scope: This method is for the operation of a Dohrmann DX2000 when it is used to extract fluoride from various matrices. The fluoride is typically collected in TISAB solution for analysis with an ion selective electrode.
1.2 Applicable Compounds: Fluorochemicals or other fluorinated compounds.
1.3 Matrices: Biological tissues, particularly liver.

2.0 KEYWORDS

2.1 Fluoride, fluorine, extraction, pyrolysis, ionization, ion selective electrode, Dohrmann, halide, DX2000, fluorochemicals.

3.0 PRECAUTIONS

3.1 Glassware and exhaust gases can be extremely hot.
3.2 Glassware is fragile, broken glass may cause injuries.
3.3 Pressurized gases, proper compressed gas handling practices required.
3.4 Solvent based samples may flash, may need to allow them to dry down before starting run.
3.5 Potential biohazards due to the biological matrices. Use appropriate personal protective equipment.

4.0 SUPPLIES AND MATERIALS

4.1 Compressed Oxygen, Hydrocarbon free, regulated to 30 PSI.
4.2 Compressed Helium, High Purity Grade, regulated to 45 PSI.
4.3 Quartz glass sample boat with Teflon™ tubing, Dohrmann 890-097 or equivalent.
4.4 Quartz glass combustion tube, Reliance Glass G-9405-012 or equivalent.
4.5 Orion 940999 Total Ionic Strength Adjustment Buffer (TISAB II) or equivalent.
4.6 Sample collection vials, HDPE.
4.7 Milli-Q™ water
4.8 Polystyrene pipettes.
4.9 Activated Charcoal, E. Merck 2005 or equivalent.
4.10 Hamilton Syringe or equivalent.
4.11 Miscellaneous laboratory glassware

5.0 EQUIPMENT

5.1 Rosemount Dohrmann DX2000 Organic Halide Analyzer, modified for fluoride extraction.
5.2 IBM compatible 386 or 486 computer.
5.3 DX2000 software, version 1.00, modified for fluoride extraction.
5.4 Excel Spreadsheet, version 5.0 or greater

6.0 INTERFERENCES

6.1 Sample size is limited to approximately 150 mg, depending on sample moisture content. This may vary from matrix to matrix.

7.0 SAMPLE HANDLING

7.1 Samples are not to be handled with bare hands. Fluoride may leach from the skin to the sample. Use forceps or probe to transfer tissues.
7.2 Samples of liver are cut from frozen liver and placed in a tared and labeled weigh boat. Use a clean scalpel and cutting board. The cutting board and scalpel should be cleaned with water, methanol, or methanol-water solution after each liver is cut.

8.0 CALIBRATION AND STANDARDIZATION

8.1 Preparation of Calibration Standards

8.1.1 The standards required for each project will need to be appropriate for that individual project. Refer to protocol for that project.
8.1.2 Typically 50-500 ppm FC-95 in methanol standards are used.
8.1.3 For rabbit liver studies, use beef liver as the matrix. Cut a piece of frozen beef liver (100 - 150 mg) and weigh it in a labeled and tared weigh boat.

8.2 Calibration - Overview

The normal calibration is the fluoride curve (AMDT-M-2). However, if an optional spiked liver curve is required the procedure listed below is used.

8.2.1 A calibration curve for the DX2000 is generated by spiking samples with known standards and combusting them using the same methods and matrix type as the samples to be tested.
8.2.2 Typically, three replicates of each standard and five concentrations of standards will be spiked.
8.2.3 Standard curve will be plotted as Mass Spiked F (ug) on the x-axis and Standard Mass Recovered F (ug) on the y-axis. Generate a regression curve and calculate the equation for the line and the r^2 value.
8.2.4 Mass Spiked F (ug) = (Amount spiked in mL) x (Conc. of standard in ppm) x (0.6004)*
*FC-95 is 60.04% F therefore 0.6004 is the factor used to convert FC-95 to F
8.2.5 Standard Mass Recovered F (ug) = (TISAB volume in mL) x (Orion reading in ppm)

8.3 Calibration - Procedure

8.3.1 Start Up
8.3.1.1 Run 2 or more Clean Cycles when starting instrument each day. More clean cycles may be used if the previous samples contained high concentrations of fluoride.

8.3.2 Blanks
8.3.2.1 Prepare sample using the same methods and type of matrix as the test sample.
8.3.2.2 For rabbit studies, use beef liver as the matrix. Prepare at least 3 samples of beef liver (100 - 150 mg) for blanks.
8.3.2.3 Put sample in Dohrmann boat. Combust each sample as described in section 9.0 and analyze sample according to method AMDT-M-2 for the ion selective electrode analysis.

8.3.2.4 For rabbit studies, the meter reading for a blank sample should be 0.03 ppm or lower before proceeding with the calibration. Burn samples until this limit is reached, or until in the judgement of the operator the reading is stable with respect to historical readings (previous 48 hours).
8.3.2.5 For non-rabbit studies, the blank readings should reach a predetermined ion concentration before proceeding with the calibration.
8.3.2.6 It may be necessary to mix approximately 50 mg of charcoal with the sample to aid combustion.

8.3.3 Standard Curve
8.3.3.1 Weigh out at least 15 matrix samples (5 standards with 3 replicates each) in tared and labeled weigh boats. For rabbit studies, weigh 100-150 mg beef liver samples. Record weights in study data. Store the matrix samples on dry ice or ice packs to keep them frozen until used.
8.3.3.2 Place weighed beef liver sample in Dohrmann sample boat.
8.3.3.3 Start with the lowest standard concentration. Using a Hamilton syringe, eject a fixed quantity of the standard on or in the matrix. For rabbit studies, use 4 uL of standard and eject it on or in the beef liver.
8.3.3.4 At least 3 replicates should be used for the lowest standard concentration; more replicates may be used at the discretion of the analyst.
8.3.3.5 Combust the sample as described in section 9.3 and analyze according to AMDT-M-2.
8.3.3.6 Run all 15 standards. If one replicate is significantly different from the other two replicates, run another sample for that standard. Indicate in data that the new replicate replaces the old replicate and that the new replicate will be used to calculate the regression curve.
8.3.3.7 When all standards have been run, calculate the r^2. r^2 must be at least 0.95. If it is not at least 0.95, consult with supervisor.
8.3.3.8 A new standard curve should be run when the combustion tube or sample matrix is changed. New standard curve may also be run at the discretion of the analyst.

8.4 Storage Conditions for Standards

8.4.1 Storage requirements for standards are dependent on the individual standards used. Typically, standards are stored at room temperature in plastic screw top bottles.
8.4.2 New FC-95 standards should be prepared at least once a month.

9.0 PROCEDURES

9.1 Typical Operating Conditions:
9.1.1 Combustion tube temperature = 950°C.
9.1.2 Oxygen and Helium flow = 50 cc/minute.
9.1.3 Vaporization/Drying time = 240 seconds.
9.1.4 Bake time = 300 seconds.

9.2 Start Up Procedure:
9.2.1 If the program is not started, start the EOX program on the PC.
9.2.2 Open the SYSTEM SETUP window.
9.2.3 Put the furnace module and the cell in the READY mode.
9.2.4 Close the SYSTEM SETUP window.

9.2.5 When the oven has reached the READY temperature, run the CLEAN BOAT program found in the CELL CHECK menu.
9.2.6 See AMDT-EP-3 for details of the Dohrmann software.

9.3 Sample Extraction Procedure:
9.3.1 Open the SAMPLE HATCH and place the sample in the BOAT. It may be necessary to mix approximately 50 mg of charcoal with the sample to aid combustion. If this is done, charcoal should also be mixed in while establishing the baseline and when generating the standard curve.
9.3.2 Close SAMPLE HATCH.
9.3.3 Add appropriate volume of TISAB solution or 1:1 TISAB:Milli-Q™ water mixture to a labeled sample collection vial. Typically 0.6 mL to 15 mL are used. For rabbit studies, use 1.0 or 2.0 mL of 1:1 TISAB:Milli-Q™ water mixture.
9.3.4 Place the vial so that the tip of the COMBUSTION TUBE is in the TISAB at least 0.25 inches. Gases released during pyrolysis must bubble through the TISAB.
9.3.5 Run the EOX-SOLIDS program found in the RUN menu.
9.3.6 When the EOX program is finished, remove the collection vial from the combustion tube.
9.3.7 If undiluted TISAB was used to collect the sample, add an equal volume of Milli-Q™ water to the TISAB to make 1:1 TISAB:Milli-Q™.
9.3.8 Rinse the end of the combustion tube with Milli-Q™ water and wipe with a KIMWIPE to remove any TISAB remaining on the tube.
9.3.9 Open the sample hatch and remove any remaining ash from the boat. Ash can be removed with a cotton tipped applicator or vacuumed out. It may be necessary to scrap particles off the bottom with a spatula or other similar device. A drop of Milli-Q™ water may be added to the boat to aid in the Clean Cycle.
9.3.10 Close the hatch.
9.3.11 Run the CLEAN BOAT program.
9.3.12 Sample is ready for analysis by ion selective electrode (AMDT-M-2).

9.4 Sample Calculations

9.4.1 Use the standard curve to calculate the sample value.
9.4.2 Sample Mass Recovered F (ug) = (TISAB vol in mL) x $\frac{\text{(Orion reading in ppm - intercept)}}{\text{(Slope)}}$

10.0 VALIDATION

10.1 Quality Control
10.1.1 Daily Start Up Check Samples: Once the standard curve is established, each day of analysis is started by analyzing QC samples. The QC samples are to be the same as the lowest concentration spiked samples used to generate the standard curve. Each concentration must be done in triplicate unless the first two replicates are within 20% of the standard curve, then a third replicate is not necessary.

10.2 Precision and Accuracy: See method development analysis and sample analysis in Fluoride Notebooks 2,3, and 5. Precision and accuracy varies when analyzing samples of different matrices and different reference compounds.

10.3 Other Validation Parameters: NA

11.0 DATA ANALYSIS

11.1 Calculations

11.1.1 For the standard curve, use regression analysis in Excel, version 5.0 or greater.
11.1.2 To calculate the fluoride contraction in the sample, see method AMDT-M-2.

11.2 Analyzing the Data

11.2.1 r^2 must be at least 0.95 or greater. "Outliers" may be excluded if two of the three replicates are within 20% of each other and the outlier is greater than 200% of the average of those two or less than 50% of the average of those two. Any such outliers should be pointed out in the data and noted in the Final Report along with the reason it was considered an outlier.

12.0 ATTACHMENTS

None

13.0 REFERENCES

13.1 Rosemount Dohrmann DX2000 Organic Halide Analyzer Operator's Manual (Manual 915-349, revision B, December 1993)
13.2 AMDT-M-2 Fluoride Measurement by Means of an Orion EA940 Expandable Ion Analyzer
13.3 AMDT-EP-3 Routine Maintenance of a Modified Dohrmann DX2000 Organic Halide Analyzer

14.0 REVISIONS

Revision Number	Reason for Change	Revision Date

3M Environmental Laboratory

Method

Fluoride Measurement by Means of an Orion EA940 Expandable Ion Analyzer

Method Identification Number: AMDT-M-2 **Adoption Date:** 10-4-95

Revision Number: 0 **Revision Date:** None

Author: Rich Youngblom

Approved By:

James D. Johnson 10/3/95
Group Leader Date

Carl Van Buskirk 10-4-95
Quality Assurance Date

Software: MS Word 5.1a

Affected Documents: AMDT-M-1 Thermal Extraction of Fluoride by Means of a Modified Dohrmann DX2000 Organic Halide Analyzer

1.0 SCOPE , APPLICABLE COMPOUNDS, AND MATRICES

1.1 SCOPE: This method is for the calibration and operation of an Orion EA940 Expandable Ion Analyzer.

1.2 APPLICABLE COMPOUNDS: Fluoride.

1.3 APPLICABLE MATRICES: Liquid samples in an appropriate buffer solution. Preferred pH of 6.0.

2.0 KEYWORDS

2.1 Fluoride, fluorine, ion selective electrode

3.0 PRECAUTIONS

3.1 No hazards identified with this method.

4.0 SUPPLIES AND MATERIALS

4.1 Orion 940999 Total Ionic Strength Adjustment Buffer II (TISABII) or equivalent.
4.2 Orion Model 900001 electrode filling solution (AgCl) or equivalent.
4.3 Orion 940907 100 ppm fluoride standard or equivalent.
4.4 Milli-Q™ water or equivalent.
4.5 Magnetic stir bars.
4.6 Lab tissues.
4.7 Sample collection vials.
4.8 Plastic 100 mL volumetric flasks.
4.9 Polystyrene pipettes.
4.10 Miscellaneous laboratory glassware.

5.0 EQUIPMENT

5.1 Orion Model EA940 Expandable Ion Analyzer or equivalent.
5.2 Orion Model 960900 Solid State Combination Fluoride electrode or equivalent.
5.3 Magnetic Stir Plate.
5.4 IBM compatible 386 or 486 computer (only needed if using Orion 3E software).
5.5 Orion RS232 interface cable (only needed if using Orion 3E software).
5.6 Microsoft Excel 5.0 (only needed if using Orion 3E software).

6.0 INTERFERENCES

6.1 It is recommended that the pH be at or near 6.0. A 1:1 mixture of TISAB and sample/Milli-Q™ water will generally bring sample to pH of 6.0.
6.2 Sample temperature may effect fluoride measurement. It is recommended that the sample be at room temperature as the standards were when the meter was calibrated.
6.3 The rate the samples are stirred at should be consistent with the rate the standards were stirred.

6.4 Air bubbles trapped under electrode can give erroneous readings. Make sure no air is trapped under electrode.

7.0 SAMPLE HANDLING

7.1 No special handling necessary.

8.0 CALIBRATION AND STANDARDIZATION

8.1 Preparation of Calibration Standards
8.1.1 Measure 50 mL of TISAB II into 5 100 mL plastic volumetric flasks.
8.1.2 Label the flasks as 0.05, 0.1, 0.5, 1.0, and 1.5 ppm F-, along with the date and your initials.
8.1.3 Pipette 0.05, 0.1, 0.5, 1.0, and 1.5 mL of 100 ppm fluoride standard into the appropriately labeled flasks.
8.1.4 Add approximately 30 mL of Milli-Q™ water to each flask.
8.1.5 Shake the flasks to mix the solutions.
8.1.6 Eliminate air bubbles from the flasks by tipping the flasks on their sides and rolling the air in the flasks over the air bubbles.
8.1.7 Bring the volume in the flasks up to the 100 mL mark with Milli-Q™ water.
8.1.8 Invert and shake the flasks for the final mixing.
8.1.9 Record standards in Standards Log Book.

8.2 Calibration
8.2.1 If necessary, remove tape from electrode filling hole.
8.2.2 Invert probe to wet top seal.
8.2.3 Eject a few drops of filling solution from bottom of electrode to wet lower seal.
8.2.4 Fill the electrode with filling solution.
8.2.5 The meter and the F- electrode are typically calibrated by direct measurement with no blank correction, using standards with concentrations of 0.05, 0.1, 0.5, 1.0, and 1.5 ppm F-, following the manufacturer's instructions.
8.2.6 Record the slope in the appropriate log book.
8.2.7 Clean the electrode by rinsing with Milli-Q™ water and wiping the sides down with lab tissues.

8.3 Storage Conditions for Standards
8.3.1 Calibration standards are stored at room temperature.

9.0 PROCEDURES

9.1 Calibration and Measurement, Standard method:
9.1.1 The sample to be measured needs to be mixed with TISAB using the proportions recommended by the TISAB manufacturer.
9.1.2 Place a stir bar in the sample and place the sample on the stir plate.
9.1.3 Allow the sample to mix for a few seconds before inserting the electrode. When the electrode is inserted, make sure there are no air bubbles trapped under the electrode.
9.1.4 The sample should be the same temperature as the calibration standards and stirred at the same rate as the calibration standards.
9.1.5 When the readings have stabilized, record the reading in the appropriate log book.

9.2 Calibration And Measurement, Using Orion 3E Software:

9.2.1 Calibration:
9.2.1.1 Follow steps 8.2.1 to 8.2.4.
9.2.1.2 Press Function Key #8 (F8).
9.2.1.3 The computer screen will ask you to confirm the number of standards to be used, concentration of the standards, and whether or not a blank is to be included in the calibration. Make any necessary changes to the information presented and click on CONTINUE.
9.2.1.4 Place the electrode in the first standard on the stir plate and click on CONTINUE.
9.2.1.5 Observe the readings on the graphic display on the computer. When the readings have stabilized, press ACCEPT READING.
9.2.1.6 Repeat step 9.2.1.4 and 9.2.1.5 for the remaining standards.
9.2.1.7 After the final standard, the computer will display the slope of the curve, as well as the intercept and correlation. Record the slope, intercept, and correlation in the appropriate log book and click on CONTINUE. The calibration data is automatically copied to C:\Orion\Data\Calib.txt.

9.2.2 Data Spreadsheet:
9.2.2.1 Select either NEW or OPEN from the FILE menu to open a new or existing spreadsheet to store data in.
9.2.2.2 Record the name of the spreadsheet used in the appropriate log book.

9.2.3 Fluoride Measurement:
9.2.3.1 Follow steps 9.2.1 through 9.2.4
9.2.3.2 Enter the name of the sample in the appropriate place on the screen.
9.2.3.3 Click on the NEW SAMPLE button
9.2.3.4 When the readings have stabilized, click on the RECORD button and write the result in the appropriate log book.

10.0 VALIDATION

10.1 Quality Control:

10.2 Precision and Accuracy

10.3 Other Validation Parameters According to Reference 13.2, the range of detection is 0.02 ppm fluoride up to a saturated solution of fluoride.

11.0 DATA ANALYSIS

11.1 Calculations None necessary.

11.2 Analyzing the Data None necessary.

12.0 ATTACHMENTS

None

13.0 REFERENCES

13.1 Orion Model EA940 Expandable Ion Analyzer Instruction Manual, Orion Research Incorporated, 1991.
13.2 Orion Model 960900 Solid State Combination Fluoride Electrode Instruction Manual, Orion Research Incorporated, 1991.

14.0 REVISIONS

Revision Number	Reason for Change	Revision Date

3M Environmental Laboratory

Method

Extraction of Fluorochemicals from Rabbit Livers

SOP Identification Number: AMDT-M-4 **Adoption Date:** 10-31-95

Revision Number: 0 **Revision Date:** None

Author: Dave Christenson/Cynthia Weber

Approved By:

James W. Johnson 10-31-95
Group Leader Date

Carl Bryan Burbank 10-31-95
Quality Assurance Date

Software: MS Word, 6.0
Affected Documents: M-5, Analysis of Rabbit Extract for Fluorochemicals Using Electrospray Mass Spectroscopy.

1.0 SCOPE

1.1 **Scope:** This method is for the extraction of fluorochemicals from rabbit livers. Ethyl acetate is used to extract fluorochemicals from the livers for analysis by electrospray mass spectroscopy.

1.2 **Applicable Compounds:** Fluorochemicals or other fluorinated compounds.

1.3 **Matrices:** Rabbit Livers.

2.0 KEYWORDS

2.1 Fluorochemicals, rabbit livers, electrospray mass spectrometer, fluorinated compounds, extraction.

3.0 PRECAUTIONS

3.1 Use gloves when handling the rabbit livers, they may contain pathogens.

4.0 SUPPLIES AND MATERIALS

4.1 **Supplies**

- 4.1.1 Syringe, capable of measuring 100 µL
- 4.1.2 Eppendorf type or disposable pipets
- 4.1.3 Gloves
- 4.1.4 Plastic grinding tubes
- 4.1.5 Plastic centrifuge tubes, 15 mL
- 4.1.6 Labels
- 4.1.7 Nitrogen
- 4.1.8 Timer
- 4.1.9 Filters, Titan nylon syringe filters, 0.2 µm.
- 4.1.10 Analytical pipets: glass volumetric pipets.
- 4.1.11 Disposable plastic 3 cc syringes.
- 4.1.12 Crimp cap autovials.

4.2 **Reagents**

- 4.2.1 Aqueous Ammonium Acetate (Aldrich), approx. 250 ppm: Prepare a 2500 ppm aqueous solution of ammonium acetate by adding 250 mg ammonium acetate to a 100 mL volumetric flask and dilute to volume with Milli-Q water. Dilute this solution 1:10 for a 250 ppm solution.
- 4.2.2 Sodium carbonate/Sodium Bicarbonate Buffer (J.T. Baker), (Na_2CO_3/$NaHCO_3$) 0.25 M: Weigh 26.5 g of sodium carbonate (Na_2CO_3) and 21.0 g of sodium bicarbonate ($NaHCO_3$) into a 1 L volumetric flask and bring to volume with Milli-Q water.
- 4.2.3 Dilute acetonitrile solution, dilute acetonitrile 1:1 with Milli-Q water.
- 4.2.4 Ethyl Acetate
- 4.2.5 Methanol
- 4.2.6 Milli-Q water
- 4.2.7 1H,1H,2H,2H - perfluorooctanesulfonic acid (Aldrich)
- 4.2.8 FC-95 (3M Specialty Chemical Division)

5.0 EQUIPMENT

5.1 Ultra-Turrax T25 Grinder for grinding liver samples.
5.2 Vortex mixer
5.3 Centrifuge
5.4 Shaker
5.5 Analytical Evaporator

6.0 INTERFERENCES

6.1 There are no known interferences at this time.

7.0 SAMPLE HANDLING

7.1 The rabbit livers are received frozen, and must be kept frozen until the extraction is performed.

8.0 CALIBRATION AND STANDARDIZATION

8.1 **Preparation of Internal Standards**

8.1.1 Prepare an internal standard of approximately 12 ppm 1H,1H,2H,2H-perfluorooctanesulphonic acid to be added to each liver sample.

8.1.2 Weigh at least 0.1 g of 1H,1H,2H,2H-perfluorooctanesulphonic acid into a 100 mL volumetric flask. Record the actual weight.

8.1.3 Bring it up to volume with methanol, this is the stock standard.

8.1.4 To a 250 mL volumetric flask, add 3 mLs of the stock standard and bring to volume with Milli-Q water. Calculate the actual concentration of the standard.

$$\frac{\text{actual mg perfluorooctane-sulphonic acid}}{0.1\ \text{L}} \times \frac{3\ \text{mL}}{250\ \text{mL}} = \text{actual concentration, ppm}$$

8.2 **Prepare FC-95 Anion Standards**

8.2.1 Prepare FC-95 standards for the standard curve.

8.2.2 Weigh approximately 100 mg of FC-95 into a 100 mL volumetric flask. Record the actual weight.

8.2.3 Bring up to volume with dilute acetonitrile.

8.2.4 Dilute the solution with dilute acetonitrile 1:10 for a solution of approximately 100 ppm. Dilute this solution 1:10 with dilute acetonitrile for a solution of approx. 10 ppm.

8.2.5 Use the 10 ppm solution to make working standards with values close to 5.0 ppm, 1.0 ppm and 500 ppb.

8.3 **Prepare Beef Liver Homogenate to Use for Standards**

8.3.1 Weigh 40 g of Bovine liver into a 250 mL Nalgene bottle containing 200 mLs Milli-Q water. Grind to a homogenous solution.

8.3.2 Add 1 mL of the solution to a 15 mL centrifuge tube. Prepare a total of eight 1 mL aliquots of the solution in 15 mL centrifuge tubes. Be sure to re-suspend solution by shaking it between aliquots.

8.3.3 Spike seven of the 1 mL aliquots with the following amounts of working standards in step 9.12 of the procedure. One 1 mL aliquot serves as the blank.

Working Standard (Approximate Conc.)	uL	Approximate final concentration of FC-95 in liver
-	-	Blank
500 ppb	100	0.292 ppm
500 ppb	200	0.584 ppm
500 ppb	300	0.877 ppm
500 ppb	400	1.168 ppm
1 ppm	500	2.924 ppm
5 ppm	200	5.848 ppm
5 ppm	300	8.772 ppm

8.4 Calculate the actual value of the standards:

$$\frac{\text{uL of standard} \times \text{concentration (in ppm)}}{\text{171 mg liver}^{*} / \text{1 ml homogenate}} = \text{final concentration (ppm) of FC -95 in liver}$$

*Average weight of bovine liver in solution as determined by weighing 1 mL homogenates of 40 mg liver in 200 mL of Milli-Q water. The amount of FC-95 is reported as equivalents of FC-95 potassium salt.

8.5 **Calibration**

8.5.1 Extract the spiked beef liver homogenate following 9.13 to 9.23 of this method. Use these standards to establish your curve on the mass spectrometer.

8.5.2 Alternatively, a standard curve may be generated using ratios of responses of the perfluorooctansulfonate anion and the internal standard anion versus concentration of the perfluorooctanesulfonate anion.

8.6 **Storage Conditions for Standards**

8.6.1 New standards are prepared with each analysis. Standards are stored in covered plastic centrifuge tubes until the analysis on the mass spectrometer is performed.

8.7 **Storage Conditions for Standards**

8.7.1 Beef liver homogenates may be frozen after preparation.

9.0 PROCEDURES

9.1 Obtain frozen liver samples. In spent tissue, note that the liver has not been packaged with other tissues.

9.2 Use a dissecting scalpel and cut off approximately 1 g of liver.

9.3 Weigh the sample directly into a tared plastic grinding tube.

9.4 Record the liver weight in the study note book.

9.5 Put a label on the vial with the study number, weight, rabbit ID, date and analyst initials.

9.6 Add 2.5 mLs water.
9.7 Grind the sample. Put the grinder probe in the sample and grind for about 2 minutes, until the sample is a homogeneous solution with no large chunks.
9.8 Rinse the probe off into the sample with 2.5 mLs water using a pipet.
9.9 Take the grinder apart and clean it with methanol after each sample. Follow AMDT-EP-22.
9.10 Cap the sample and vortex for 15 seconds.
9.11 Pipet 1 mL into a 15 mL centrifuge tube. Label the centrifuge tube with the identical information as the grinding tube. (See AMDT-M-4 Worksheet for documenting the remaining steps.)
9.12 Spike the beef liver homogenates with the appropriate amount of FC-95 standard as described in **8.3**.
9.13 Spike the samples and beef liver homogenates with 100 uL of internal standard.
9.14 Add 1 mL of the sodium carbonate/sodium bicarbonate buffer and 1 mL ammonium acetate.
9.15 Using an analytical pipet, add 5 mL ethyl acetate.
9.16 Cap the sample and vortex 20 to 30 seconds.
9.17 Put them in the shaker for 20 min.
9.18 Centrifuge for 20 to 25 minutes, until the layers are well separated. Set the power on the centrifuge to 25.
9.19 Remove 4 mLs of the top organic layer to a fresh 15 mL centrifuge tube with a 5 mL graduated glass pipet. Transfer the label to the fresh tube.
9.20 Blow the sample down on the analytical evaporator to near dryness with nitrogen, approximately 30 to 40 minutes.
9.21 Bring the remaining sample up in 1 mL dilute acetonitrile with an analytical pipet.
9.22 Vortex 15 seconds.
9.23 Transfer the sample to a 3 mL syringe. Attach a 0.2 µm nylon mesh filter, and filter the sample into a fresh centrifuge tube or a autovial. Label the tube or vial with the study number and animal number.
9.24 Cap and hold for analysis by electrospray mass spectroscopy.
9.25 Complete AMDT-M-4 worksheet and attach to page of study notebook.

10.0 VALIDATION

10.1 Quality Control - not applicable
10.2 Precision and Accuracy- not applicable
10.3 Other Validation Parameters- not applicable

11.0 DATA ANALYSIS

11.1 None

12.0 ATTACHMENTS

12.1 Worksheet AMDT-M-4

13.0 REFERENCES

13.1 AMDT-EP-22 Routine Maintenance of Ultra-Turrax T-25

14.0 REVISIONS

Revision Number	Reason for Change	Revision Date

Worksheet AMDT-M-4

Study #	Sample Number set #	FC-95 approx 0.5 ppm actual ppm #W_______	FC-95 approx 1 ppm actual ppm #W_______	FC-95 approx. 5 ppm actual ppm #W_______	Date and Initials for Std.
-	Blank Liver	-	-	-	
-		100 uL	-	-	
-		200 uL	-	-	
-		300 uL	-	-	
-		400 uL	-	-	
-		-	500 uL	-	
-		-	-	200 uL	
-		-	-	300 uL	
[1]		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
[1] study number where the original worksheet is located and place a copy.					

Liver Extraction Process:	Date & Initials
Pipet 1 mL of Liver Solution	
Pipet 100 uL of 12 ppm Internal Standard Std. #	
Vortex 15 sec	
Pipet 1 mL of 250 ppm Ammonium Acetate Std. #	
Pipet 1 mL of 0.25 Na_2CO_3/0.25M $NaHCO_3$ Buffer	
Pipet 5 mL of Ethyl Acetate	
Vortex 20-30 sec	
Shake 20 min	
Centrifuge 20-25 min	
Remove a 4 mL aliquot of organic layer	
Blow down to near dryness (≤0.25 mL) with N_2	
Add 1 m: of 1:1 Acetonitrile/H_2O TN#	
Vortex 15 sec	
Filter using a 3cc B-D syringe with a 0.2um SRI filter into a 1.5 mL autosample vial	

3M Environmental Laboratory

Method

Analysis of Rabbit Liver Extract for Fluorochemicals using Electrospray Mass Spectroscopy

SOP Identification Number: AMDT-M-5 **Adoption Date:** 6-6-95

Revision Number: 0 **Revision Date:** None

Author: Dave Christenson/Cynthia Weber

Approved By:

Group Leader — Date: 6/6/95

Quality Assurance — Date: 6/6/95

Software: MS Word, 6.0
Affected Documents: M-4, Extraction of Fluorochemicals from Rabbit Livers

1.0 SCOPE

1.1 **Scope:** This method is for the analysis of extracts of rabbit liver or other tissues or fluids for fluorochemicals using the electrospray mass spectrometer. The analysis is performed by single ion monitoring of FC-95 anion, M/Z= 499, the internal standard M/Z = 427, and other appropriate masses.

1.2 **Applicable Compounds:** Fluorochemicals or other fluorinated compounds.

1.3 **Matrices:** Rabbit Livers (samples), Beef Liver (standards), other tissues and fluids.

2.0 KEYWORDS

2.1 Fluorochemicals, fluorinated compounds, electrospray mass spectroscopy, mass spectrometer, rabbit livers.

3.0 PRECAUTIONS

3.1 Use caution with the voltage cable for the probe. When the voltage cable is plugged into the probe DO NOT TOUCH THE PROBE, there is risk of electrical shock.

3.2 Do not run the pump above it's capacity of 4000 psi. If pressure goes over 4000 psi stop and release pressure. The peak tubing may be plugged. Troubleshoot back to find the plug and replace the plugged tubing. See AMDT-EP-15

3.3 Do not run the pump to dryness.

4.0 SUPPLIES AND MATERIALS

4.1 **Supplies**

4.1.1 Nitrogen gas regulated to 140 psi.

4.1.2 Fluofix column or equivalent.

4.1.3 100 uL or 250 uL flat tip syringe for sample injection.

4.2 **Reagents**

4.2.1 Dilute acetonitrile mobile phase, dilute acetonitrile 1:1 with Milli-Q water.

4.2.2 Milli-Q water, all water used in this method should be Milli-Q water.

5.0 EQUIPMENT

5.1 VG Trio 2000 Electrospray Mass Spectrometer or equivalent.

5.2 ISCO Syringe Pump

5.3 Spectraphysics AS300 Autosampler

5.4 100 uL Assembly

5.5 Autovials or capped centrifuge tubes.

6.0 INTERFERENCES

6.1 There are no known interferences at this time.

7.0 SAMPLE HANDLING

7.1 Keep the extracted samples in capped 15 mL centrifuge tubes or in capped autovials until ready for analysis.

8.0 CALIBRATION AND STANDARDIZATION

8.1 Preparation of Calibration Standards

8.1.1 Seven beef liver standards and one blank beef liver are prepared during the extraction procedure. (See AMDT-M-4, section 8.0)

8.2 Calibration

8.2.1 Run the seven beef liver standards twice, starting with the lowest standard to obtain the standard curve.

8.2.2 Typically one standard is run after each 5 to 7 samples. Choose a standard in the same range of concentration as the samples.

8.3 Storage Conditions for Standards

8.3.1 Fresh standards are prepared with each analysis. Standards are stored in covered plastic centrifuge tubes until the analysis on the mass spectometer is performed. Samples and standards are NOT refrigerated.

8.4 Storage Conditions for Beef Liver Homogenates

8.4.1 Beef liver homogenates may be frozen after preparation.

9.0 PROCEDURE

9.1 Initial Set-up

9.1.1 Set software to "Operate on", Ion Mode ES^-.

9.1.2 Record backing pressure in the instrument log.

9.1.3 Fill the solvent cylinder with mobile phase.

9.1.4 Set the pump to "Run". Set the flow to 1000 uL/min. Observes droplets coming out of the tip of the probe. The pressure should be at 1700 to 1800 psi.

9.1.5 Check the fused silica at the end of the probe. Use an eye piece to check for chips. The tip should be flat with no jagged edges. If any chips are found cut off the tip of the silica with a column cutter and pull the silica through to the appropriate length.

9.1.6 Check your nitrogen supply. Turn on the nitrogen. There should be no nitrogen leaking around the tip of the probe. A fine mist should be coming out of the tip.

9.1.7 Carefully guide the probe into the opening. Insert it until it won't go any further. Connect the voltage cable to the probe.

9.1.8 Go to the "Editor" page, and set Ionization Mode to ES^-, and the appropriate masses to 427 and 499.

9.1.9 If it is not in single ion mode go to "Option" and set SIR.

9.1.10 Start Acquisition. Assign a file name, MO-DAY-YR + letter. Record it in the log book.

9.1.11 Run the beef liver samples first, running each standard twice at the beginning of the run.. Run a QC check by running one standard after every 5 to 7 samples.

9.2 Manual Injection

9.2.1 Draw 150 uL of sample into a syringe. Inject the sample into the rheodyne injection port. Inject slowly. Record the sample ID in the log book.

9.2.2 Turn the valve to "On".

9.2.3 Wait two minutes, and inject the next sample.

9.2.4 Record the scan number for each sample in the logbook.

9.3 Using the Autosampler

9.3.1 Set up sample tray A, B, or C.

9.3.2 Record the samples and their positions in the instrument log book. Up to 17 vials may be in each run.

9.3.3 Set-up the sampler:

- **9.3.3.1** Push the sample button
- **9.3.3.2** Set sample loop size = 100 uL
- **9.3.3.3** Set inject/sample = 2
- **9.3.3.4** Set Cycle time = 0
- **9.3.3.5** Name the file: Livers
- **9.3.3.6** Identify the tray used
- **9.3.3.7** Add the samples to Queue by pressing "Enter"
- **9.3.3.8** Press "Run" to start

10.0 VALIDATION

10.1 Quality Control

10.1.1 Run a standard every 5 to 7 samples. If a significant change(± 50%) in peak height occurs stop the run. Only the samples before the last acceptable standard will be used. The remaining samples will be reanalyzed.

10.2 Precision and Accuracy

10.2.1 See Method Validation Report number AMDT-M-5.0.V1

10.3 Other Validation Parameters

10.4 Refer to Method Validation Report Number AMDT-M-5.0.V1

11.0 DATA ANALYSIS

11.1 Calculations

11.2 Plot the standard curve, using the mean of the two values obtained for each standard.

11.2.1 Read peak heights or areas for the samples from the printout. Use linear regression to determine the sample concentrations.

11.2.2 Calculate the mg of FC-95 anion, or other fluorochemical in the total rabbit liver:

mg FC-95 anion in the total rabbit liver =

$$\frac{\text{mg FC-95 anion from std. curve}}{\text{gms of liver used for analysis}} \times \text{Total mass of liver, gms}$$

11.3 Make a results table and enter it in the study book.

11.4 Print a chromatogram for each sample, with the peaks labeled with the sample or standard ID. Write the study number on the printout, initial, date, and put it in the study folder. Staple all chromatograms together and number pages.

12.0 ATTACHMENTS

None

13.0 REFERENCES

13.1 AMDT-EP-17

14.0 REVISIONS

Revision Number	Reason for change	Revision Date

3M Environmental Laboratory

Method

Analysis of Fluoride Using the Skalar Segmented Flow Analyzer With Ion Selective Electrode

Method Identification Number: AMDT-M-8 **Adoption Date:** 10-5-95

Revision Number: 0 **Revision Date:** None

Author: Deb Wright / Cynthia Weber

Approved By:

James D Johnson 10/5/95
Group Leader Date

Gale Von Burkirk 9-27-95
Quality Assurance Date

Software: IBM MS Word, 6.0
Affected Documents: **AMDT-EP-26,** Operation and Maintenance of the Skalar Segmented Flow Analyzer

1.0 SCOPE

1.1 This method is for the analysis for fluoride, thermally extracted from samples using the Dohrmann DX2000 (AMDT-M-1), and collected in TISAB for analysis with an Ion Selective Electrode (ISE). The analysis is performed using the Skalar Segmented Flow Analyzer with ISE.

1.2 Samples can be tissues, serum, biological material, or other materials extracted on the Dohrmann.

2.0 KEYWORDS

2.1 Skalar, segmented flow, fluoride.

3.0 PRECAUTIONS

3.1 Follow standard laboratory safety practices.

4.0 SUPPLIES AND MATERIALS

4.1 Supplies

4.1.1 Sample cups, 4 mL plastic cups with caps
4.1.2 Autopipets, oxford or equivalent with plastic tips
4.1.3 Polypropylene volumetric flasks, 100 mL
4.1.4 Cartridge components, refer to the Skalar Methods for components and part numbers.
4.1.5 Sample prefilters, Evergreen

4.2 Reagents

4.2.1 Brij 35, 30% S.F.A.S. Detergent
4.2.2 TISAB II buffer solution: Purchase TISAB II from Orion. To 1 liter of TISAB II add 2.5 mL or 100 ppm fluoride solution and 1 mL Brij.
4.2.3 Sampler rinsing solution: Dilute TISAB II 1:1 with Milli-Q water.
4.2.4 Nitric acid solution for decontamination, 1 N (lab grade): Slowly add 64 mLs concentrated nitric acid (HNO_3) to 250 mLs of Milli-Q water. Bring the volume up to 1 L with Milli-Q water.

4.3 Standards

4.3.1 Stock solution, 100 ppm F: purchased from Orion.
4.3.2 Intermediate standard, 10 ppm: Dilute 10 mLs of stock solution to 100 mLs with Milli-Q water. Use polypropylene volumetric flasks.
4.3.3 Working standard: Make up the following working standards by adding the volumes of intermediate or stock standard indicated on the table, using oxford or pumpmate pipets, to 50 mLs of TISAB and diluting to 100 mLs with Milli-Q water.

Working Standard	mLs of Stock Standard	mLs of Intermediate Standard
0.015 ppm	-	0.15
0.03 ppm	-	0.3
0.06 ppm	-	0.6
0.09 ppm	-	0.9
0.12 ppm	-	1.2
0.15 ppm	-	1.5
0.3 ppm	0.3	-
0.6 ppm	0.6	-

1.2 ppm	1.2	-
1.5 ppm	1.5	-

5.0 EQUIPMENT

5.1 Skalar Segmented Flow Auto Analyzer Sans^{Plus} System equipped with ISE

6.0 INTERFERENCES

6.1 High concentrations of alkalinity, chloride, phosphate, sulfate or iron can cause interferences.

7.0 SAMPLE HANDLING

7.1 Samples should be stored in polyethylene bottles. Samples should be analyzed within 30 days.

8.0 CALIBRATION AND STANDARDIZATION

8.1 **Preparation of Calibration Standards**

8.1.1 Prepare calibration standards as in section 4.3.

8.2 **Calibration**

8.2.1 The standards are analyzed at the beginning of the run.

8.3 **Storage Conditions for Standards**

8.3.1 Standards are stored in capped polypropylene volumetric flasks. New standards are prepared at a minimum of every six months, or as necessary.

9.0 PROCEDURE

9.1 **Start Up Procedure**

9.1.1 Clamp down the pumpdecks, air bars and sampler-pump tubing.

9.1.2 Put the fluoride electrodes in the electrode chamber.

9.1.3 Turn on the power of the sampler, pumps, offset potentiometer and heating bath.

9.1.4 Put the reagent-lines in the appropriate bottles.

9.1.5 Turn on the interface, computer, display and printer. **Make sure you turn on the interface before the computer.**

9.1.6 Let the system stabilize for approximately 30 minutes.

9.2 **Starting a Run**

9.2.1 Create a sample table by selecting FILES, TABLE, and CREATE, type in the name of the file, and press ENTER.

9.2.2 Print the sample table, inserted in the system table by pushing ESC, PRINT, GROUP 1. This will print the entire run.

9.2.3 Dial the sampler settings to the appropriate number of samples, number of seconds for sample wash, and number of seconds for the sample.

9.2.4 Fill the sample tray with the standards, samples, washes and drifts. IW and FW/RUNOUT cups on the sampler do not need to be filled.

9.2.5 Set the baseline.

9.2.5.1 Select GRAPHICS, REAL TIME. If you cannot get real-time, you may be in the Data Handling Panel. Switch to the Analysis Panel by selecting CONTROL PANEL and pushing F7.

9.2.5.2 Use the small screwdriver for the offset potentiometer to set the base line. Adjust the baseline until it is approximately 3/4 inch from the bottom of the screen.

9.2.5.3 Check the highest standard and adjust the gain, if necessary, with the interface screw #3.

9.2.6 Go to CONTROL PANEL, and to analysis panel. Deselect the analysis that will not be run. (Select or deselect analysis by pressing ENTER.) Press Tab to return to the Analysis Panel.

9.2.7 Press the spacebar to bring up the local menu.

9.2.8 Select START to start the analysis.

9.2.9 Type your ID (initials), the sample table which you created under **9.2.1** (or press ENTER for choices), choose running with or without the system table and select START ANALYSIS.

9.2.10 After starting the software, start the sampler. Make sure that the sampler is set to the right number of samples and that the sample/wash/air times are OK.

9.2.11 Select GRAPHICS, REAL TIME to view the progress of the analysis.

9.3 Loading and Printing the Data-File

9.3.1 Go to CONTROL PANEL, press the spacebar to bring up the local menu and select LOAD. Select AUTOCALCULATION and enter the filename (or highlight the file to be printed and press ENTER).

9.3.2 To view the calibration curve, go to GRAPHICS, CALIBRATION CURVE.

9.3.3 To print the high level curve, push PRINT SCREEN.

9.3.4 To print the low level screen, push ESC to get out of graphics. Select SETTINGS. Change the max y value to approximately 900. Go to CAL CURVE and press ESC, and Enter. Press PRINT SCREEN.

9.3.5 Return to SETTINGS and change the max value back to 4095, go to EDIT, press ENTER and PRINT SCREEN to print sample peaks.

9.3.6 To print the results go to CONTROL PANEL, SPACEBAR, OUTPUT, OUTPUT. Select PRINTER for the Epson or PRN for the Laser.

9.4 Shutdown

9.4.1 Put all the reagent-lines in Milli-Q water.

9.4.2 Let the system rinse for approximately 30 minutes.

9.4.3 After the system has rinsed completely, turn off the sampler, pump and offset potentiometer. Turn off the heating bath on weekends. Leave liquid in the lines.

9.4.4 Take the electrode out and soak in 100 ppm F overnight.

9.4.5 Release the pump-decks, air bars and sampler pump-tubing.

9.4.6 Select FILES, press ALT F and select QUIT to exit the program.

9.4.7 On Friday, turn off the computer, display and interface for the weekend.

10.0 VALIDATION

10.1 Quality Control

10.1.1 Run a standard (mid to high concentration) every 10 samples. If a significant change in peak height occurs, only the samples before the last acceptable standard will be used. The remaining samples will be reanalyzed.

10.2 Precision and Accuracy

10.2.1 See Method Validation Report number AMDT-M-8.0.V1

10.3 Other Validation Parameters

10.4 Refer to Method Validation Report Number AMDT-M-8.0.V1

11.0 DATA ANALYSIS

11.1 Calculations

11.1.1 The standard curve is plotted by the Skalar software.

11.1.2 All calculations are done by the Skalar software. r^2 should be 0.995 or better.

11.2 Prepare spreadsheets to summarize data. Include sample volume, weights used etc.

11.3 Write the study number on the printouts, initial, date the printout, and bind together with all package documents and place in the study folder. Make a copy of the summary sheet and tape into the study notebook. Back up all data and spreadsheets onto study disk and backup disks.

11.4 Electronic Data

11.4.1 GLP studies: Electronic data is copied onto the Study floppy disk for each study, and also data is copied onto a floppy disk that is stored in the lab.

11.4.2 Other studies: All data is copied onto a floppy disk that is stored in the lab.

12.0 ATTACHMENTS

None

13.0 REFERENCES

13.1 AMDT-M-1, Thermal Extraction of Fluoride by Means of a Modified Dohrmann DX2000 Organic Halide Analyzer-Liver

13.2 Skalar Methods, #335, Skalar Methods Manual

13.3 AMDT-EP-26, Operation and Maintenance of the Skalar Segmented Flow Analyzer

14.0 REVISIONS

Revision Number	Reason for change	Revision Date

3M Environmental Laboratory

Method

Thermal Extraction of Fluoride by Means of a Modified Dohrmann DX2000 Organic Halide Analyzer - Serum

Method Identification Number: AMDT-M-14 **Adoption Date:** 10-3-95

Revision Number: 0 **Revision Date:** None

Author: Rich Youngblom

Approved by:

James D Johnson 10/3/95

Group Leader Date

E.G. Von Benkitz 9-27-95

Quality Assurance Date

Software: MS Word 5.1a

Affected Documents: AMDT-M-2 Fluoride Measurement by Means of an Orion EA940 Expandable Ion Analyzer
AMDT-EP-3 Routine Maintenance of a Modified Dohrmann DX2000 Organic Halide Analyzer

1.0 SCOPE , APPLICABLE COMPOUNDS, AND MATRICES

1.1 Scope: This method is for the operation of a Dohrmann DX2000 when it is used to extract fluoride from various matrices. The fluoride is typically collected in TISAB solution for analysis with an ion selective electrode.
1.2 Applicable Compounds: Fluorochemicals or other fluorinated compounds.
1.3 Matrices: Biological fluids, particularly serum.

2.0 KEYWORDS

2.1 Fluoride, fluorine, extraction, pyrolysis, ionization, ion selective electrode, Dohrmann, halide, DX2000, fluorochemicals.

3.0 PRECAUTIONS

3.1 Glassware and exhaust gases can be extremely hot.
3.2 Glassware is fragile, broken glass may cause injuries.
3.3 Pressurized gases, proper compressed gas handling practices required.
3.4 Solvent based samples may flash, may need to allow them to dry down before starting run.
3.5 Potential biohazards due to the biological matrices. Use appropriate personal protective equipment.

4.0 SUPPLIES AND MATERIALS

4.1 Compressed Oxygen, Hydrocarbon free, regulated to 30 PSI.
4.2 Compressed Helium, High Purity Grade, regulated to 45 PSI.
4.3 Quartz glass sample boat with Teflon™ tubing, Dohrmann 890-097 or equivalent.
4.4 Quartz glass combustion tube, Reliance Glass G-9405-012 or equivalent.
4.5 Orion 940999 Total Ionic Strength Adjustment Buffer (TISAB II) or equivalent.
4.6 Sample collection vials, HDPE.
4.7 Milli-Q™ water
4.8 Polystyrene pipettes.
4.9 Activated Charcoal, E. Merck 2005 or equivalent.
4.10 Hamilton Syringe or equivalent.
4.11 Miscellaneous laboratory glassware

5.0 EQUIPMENT

5.1 Rosemount Dohrmann DX2000 Organic Halide Analyzer, modified for fluoride extraction.
5.2 IBM compatible 386 or 486 computer.
5.3 DX2000 software, version 1.00, modified for fluoride extraction.
5.4 Excel Spreadsheet, version 5.0 or greater

6.0 INTERFERENCES

6.1 Sample size is limited to approximately 100 µl. This may vary from matrix to matrix.

7.0 SAMPLE HANDLING

7.1 Samples are to be handled with plastic pipettes. A new pipette is to be used for each sample.

8.0 CALIBRATION AND STANDARDIZATION

8.1 Preparation of Calibration Standards

8.1.1 The standards required for each project will need to be appropriate for that individual project. Refer to protocol for that project.
8.1.2 Typically 50-500 ppm FC-95 in methanol standards are used.
8.1.3 For rabbit serum studies, use beef serum as the matrix.

8.2 Calibration - Overview

The normal calibration is the fluoride curve (AMDT-M-2). However, if an optional spiked serum curve is required the procedure listed below is used.

8.2.1 A calibration curve for the DX2000 is generated by spiking samples with known standards and combusting them using the same methods and matrix type as the samples to be tested.
8.2.2 Typically, three replicates of each standard and five concentrations of standards will be spiked.
8.2.3 Standard curve will be plotted as Mass Spiked F (ug) on the x-axis and Standard Mass Recovered F (ug) on the y-axis. Generate a regression curve and calculate the equation for the line and the r^2 value.
8.2.4 Mass Spiked F (ug) = (Amount spiked in mL).x (Conc. of standard in ppm) x (0.6004)*
*FC-95 is 60.04% F therefore 0.6004 is the factor used to convert FC-95 to F
8.2.5 Standard Mass Recovered F (ug) = (TISAB volume in mL) x (Orion reading in ppm)

8.3 Calibration - Procedure

8.3.1 Start Up
8.3.1.1 Run 2 or more Clean Cycles when starting instrument each day. More clean cycles may be used if the previous samples contained high concentrations of fluoride.

8.3.2 Blanks
8.3.2.1 Prepare sample using the same methods and type of matrix as the test sample.
8.3.2.2 For rabbit studies, use beef serum as the matrix.
8.3.2.3 Put serum blank in Dohrmann boat. Combust sample as described in section 9.0 and analyze sample according to method AMDT-M-2 for the ion selective electrode analysis.
8.3.2.4 For rabbit studies, the meter reading for a blank sample should be 0.03 ppm or lower before proceeding with the calibration. Burn samples until this limit is reached, or until in the judgement of the operator the reading is stable with respect to historical readings (previous 48 hours).
8.3.2.5 For non-rabbit studies, the blank readings should reach a predetermined ion concentration before proceeding with the calibration.
8.3.2.6 It may be necessary to mix approximately 50 mg of charcoal with the sample to aid combustion.

8.3.3 Standard Curve

8.3.3.1 If beef serum is frozen, thaw at least enough to complete the standard curve analysis for the day (≈30 mL).

8.3.3.2 Pipette 100µL of beef serum into Dohrmann sample boat.

8.3.3.3 Start with the lowest standard concentration. Using a Hamilton syringe, eject a fixed quantity of the standard on or in the matrix. For rabbit studies, use 4 uL of standard and eject it on or in the beef serum.

8.3.3.4 At least 3 replicates should be used for the lowest standard concentration; more replicates may be used at the discretion of the analyst.

8.3.3.5 Combust the sample as described in section 9.3 and analyze according to AMDT-M-2.

8.3.3.6 Run all 15 standards. If one replicate is significantly different from the other two replicates, run another sample for that standard. Indicate in data that the new replicate replaces the old replicate and that the new replicate will be used to calculate the regression curve.

8.3.3.7 When all standards have been run, calculate the r^2. r^2 must be at least 0.95. If it is not at least 0.95, consult with supervisor.

8.3.3.8 A new standard curve should be run when the combustion tube or sample matrix is changed. New standard curve may also be run at the discretion of the analyst.

8.4 Storage Conditions for Standards

8.4.1 Storage requirements for standards are dependent on the individual standards used. Typically, standards are stored at room temperature in plastic screw top bottles.

8.4.2 New FC-95 standards should be prepared at least once a month.

9.0 PROCEDURES

9.1 Typical Operating Conditions:

9.1.1 Combustion tube temperature = 950°C.

9.1.2 Oxygen and Helium flow = 50 cc/minute.

9.1.3 Vaporization/Drying time = 240 seconds.

9.1.4 Bake time = 300 seconds.

9.2 Start Up Procedure:

9.2.1 If the program is not started, start the EOX program on the PC.

9.2.2 Open the SYSTEM SETUP window.

9.2.3 Put the furnace module and the cell in the READY mode.

9.2.4 Close the SYSTEM SETUP window.

9.2.5 When the oven has reached the READY temperature, run the CLEAN BOAT program found in the CELL CHECK menu.

9.2.6 See AMDT-EP-3 for details of the Dohrmann software.

9.3 Sample Extraction Procedure:

9.3.1 Open the SAMPLE HATCH and pipette 100µL of sample into the BOAT. It may be necessary to mix approximately 50 mg of charcoal with the sample to aid combustion. If this is done, charcoal should also be mixed in while establishing the baseline and when generating the standard curve.

9.3.2 Close SAMPLE HATCH.

9.3.3 Add appropriate volume of TISAB solution or 1:1 TISAB:Milli-Q™ water mixture to a labeled sample collection vial. Typically 0.6 mL to 15 mL are used. For rabbit studies, use 1.0 or 2.0 mL of 1:1 TISAB:Milli-Q™ water mixture.
9.3.4 Place the vial so that the tip of the COMBUSTION TUBE is in the TISAB at least 0.25 inches. Gases released during pyrolysis must bubble through the TISAB.
9.3.5 Run the EOX-WATER program found in the RUN menu.
9.3.6 When the EOX program is finished, remove the collection vial from the combustion tube.
9.3.7 If undiluted TISAB was used to collect the sample, add an equal volume of Milli-Q™ water to the TISAB to make 1:1 TISAB:Milli-Q™.
9.3.8 Rinse the end of the combustion tube with Milli-Q™ water and wipe with a KIMWIPE to remove any TISAB remaining on the tube.
9.3.9 Open the sample hatch and remove any remaining ash from the boat. Ash can be removed with a cotton tipped applicator and/or vacuumed out. It may be necessary to scrap particles off the bottom with a spatula or other similar device. A drop of Milli-Q™ water may be added to the boat to aid in the Clean Cycle.
9.3.10 Close the hatch.
9.3.11 Run the CLEAN BOAT program.
9.3.12 Sample is ready for analysis by ion selective electrode (AMDT-M-2).

9.4 Sample Calculations

9.4.1 Use the standard curve to calculate the sample value.
9.4.2 Sample Mass Recovered F (ug) = (TISAB vol in mL) x $\frac{\text{(Orion reading in ppm - intercept)}}{\text{(Slope)}}$

10.0 VALIDATION

10.1 Quality Control

10.1.1 Daily Start Up Check Samples: Once the standard curve is established, each day of analysis is started by analyzing QC samples. The QC samples are to be the same as the lowest concentration spiked samples used to generate the standard curve. Each concentration must be done in triplicate unless the first two replicates are within 20% of the standard curve, then a third replicate is not necessary.

10.2 Precision and Accuracy: See method development analysis and sample analysis in Fluoride Notebooks 2,3, and 5. Precision and accuracy varies when analyzing samples of different matrices and different reference compounds.

10.3 Other Validation Parameters: NA

11.0 DATA ANALYSIS

11.1 Calculations

11.1.1 For the standard curve, use regression analysis in Excel, version 5.0 or greater.
11.1.2 To calculate the fluoride contraction in the sample, see method AMDT-M-2.

11.2 Analyzing the Data

11.2.1 r^2 must be at least 0.95 or greater. "Outliers" may be excluded if two of the three replicates are within 20% of each other and the outlier is greater than 200% of the average of those two or less than 50% of the average of those two. Any such outliers should be pointed out in the data and noted in the Final Report along with the reason it was considered an outlier.

12.0 ATTACHMENTS

None

13.0 REFERENCES

13.1 Rosemount Dohrmann DX2000 Organic Halide Analyzer Operator's Manual (Manual 915-349, revision B, December 1993)
13.2 AMDT-M-2 Fluoride Measurement by Means of an Orion EA940 Expandable Ion Analyzer
13.3 AMDT-EP-3 Routine Maintenance of a Modified Dohrmann DX2000 Organic Halide Analyzer

14.0 REVISIONS

Revision Number	Reason for Change	Revision Date

9.1.3 Amendment to analytical protocol AMDT-011095.1

Attachment I

GLP Study

Protocol Amendment

Study Number: AMDT-011095.1

Study Title: Single Dose Dermal Absoption/Toxicity of T-60676, T-6068 and T-6069 in Rabbits

Study Director: James D. Johnson

Amendment Date: November 10, 1995 Amendment Number: 1

This amendment modifies the following portion of the protocol:

AMDT-M-14-0 specifies using bovine serum for blanks and QC check samples in the thermal extraction. However, rabbit serum from the control animals (AMDT-110394.1) was used was used because the bovine serum blanks were higher than the samples.

Approved by:

James D. Johnson 11/28/95

Study Director Date

9.3 Quality Assurance Unit Statement

Attachment D

GLP Study
Quality Assurance Statement

Completed by: QAU Auditor Original to: Study Director Copies to: QAU Files

Study Title: **Single-dose Dermal Absorption/Toxicity Study of T-6067, T-6068 and T-6069 in Rabbits**

Study Number: AMDT-011095.1 Name of Auditor: Kari Rambo

This study has been inspected by the Quality Assurance Unit as indicated in the following table. The findings were reported to the study director and management.

Inspection Dates From	To	Phase	Date Inspection Reported to Management	Study Director
11-15-95	11-20-95	Final Report	11-20-95	11-20-95

Kari Rambo 11-20-95

QAU Auditor Date

9.4 Key Personnel Involved in the Study

3M Environmental Laboratory

Key Personnel

Thermal extraction followed by analysis using Orion ion analyzer:
Jim Johnson
Deb Wright
Rich Youngblom
Deann Plummer

Analysis of liver extracts using electrospray mass spectrometry:
Jim Johnson
Dave Christenson

Thermal extraction followed by analysis using Skalar segmented flow analyzer with ion selective electrode:
Jim Johnson
Deb Wright
Rich Youngblom
Deann Plummer

Documentation and Reporting:
Jim Johnson
Rich Youngblom

Quality Assurance Unit:
Gale Van Buskirk
Cynthia Weber
Kari Rambo

9.11 Data

9.11.1 Summary and raw data; ug F^- in whole liver as determined by thermal extraction followed by analysis using Orion ion analyzer.

Summary of Combustion Data - Liver
AMDT-011095.1, HWI 6329-152
As Referenced in Final Report section 6.0 *DATA ANALYSIS*

Total ug Fluoride in Whole Liver
Mean per Dose Group*

	µg	Std. Dev.
Control Group	12.5	± 2.1
4.0 mg/kg dose (T6067)	12.5	± 1.6
16 mg/kg dose (T6067)	14.7	± 4.0
40 mg/kg dose (T6067)	16.5	± 5.2
Fabric exposure (T6068)**	15.7	± 4.2
Fabric exposure (T6069)**	12.8	± 3.7

*Calculated as the mean of triplicate samples from each of three male and three female rabbits.
**Administered as a 10 cm x 10 cm piece of test material (fabric).

L13492 AB ID	% rcvry	Actual ppm F- in liver (W/W)	Average ppm F- in liver (W/W)	Liver burned (grams)	Whole liver weight (grams)	Total F- in whole liver (µg)	Dosage (mg/kg)
liver blank-1		0.213		0.125			
liver spk-1	97%	1.10		0.134			
liver spk-2	85%	0.872		0.147			
liver spk-3	85%	0.883		0.147			
liver spk-4	107%	2.63		0.123			
liver spk-5	97%	2.00		0.147			
liver spk-6	95%	2.55		0.112			
liver blank-2		0.159		0.140			
F52711-1		0.167		0.134	66.1		
F52711-2		0.144	0.153	0.141	66.1	10.1	0.0
F52711-3		0.149		0.149	66.1		
F52719-1		0.166		0.137	76.5		
F52719-2		0.157	0.176	0.124	76.5	13.5	0.0
F52719-3		0.205		0.133	76.5		
Liver blk 1		0.250		0.129			
Liver blk 2		0.169		0.142			
Liver spk 1	97%	1.19		0.124			
Liver spk 2	98%	1.37		0.109			
F52725-1		0.228		0.126	69.5		
F52725-2		0.231	0.230	0.116	69.5	16.0	0.0
F52725-3		0.230		0.124	69.5		
F52713-1		0.172		0.154	71.4		
F52713-2		0.152	0.164	0.166	71.4	11.7	4.0
F52713-3		0.170		0.163	71.4		
F52718-1		0.161		0.128	57.7		
F52718-2		0.156	0.183	0.135	57.7	10.6	4.0
F52718-3		0.233		0.126	57.7		
F52723-1		0.175		0.171	72.6		
F52723-2		0.230	0.187	0.150	72.6	13.6	4.0
F52723-3		0.155		0.156	72.6		
F52721-1		0.218		0.101	62.9		
F52721-2		0.191	0.184	0.116	62.9	11.6	0.0
F52721-3		0.142		0.154	62.9		
F52727-1		0.194		0.153	69.2		
F52727-2		0.206	0.187	0.136	69.2	12.9	0.0
F52727-3		0.160		0.134	69.2		
F52806-1		0.158		0.134	69.8		
F52806-2		0.155	0.160	0.137	69.8	11.1	0.0
F52806-3		0.166		0.125	69.8		
F52715-1		0.226		0.127	62.1		
F52715-2		0.255	0.230	0.133	62.1	14.3	4.0
F52715-3		0.211		0.122	62.1		
F52738-1		0.178		0.134	64.6		
F52738-2		0.169	0.171	0.126	64.6	11.0	4.0
F52738-3		0.166		0.131	64.6		

L13492 AB ID	% rcvry	Actual ppm F- in liver (W/W)	Average ppm F- in liver (W/W)	Liver burned (grams)	Whole liver weight (grams)	Total F- in whole liver (µg)	Dosage (mg/kg)
F52809-1		0.432		0.138	55.4		
F52809-2		0.159	0.253	0.128	55.4	14.0	4.0
F52809-3		0.168		0.112	55.4		
F52724-1		0.187		0.119	82.0		
F52724-2		0.235	0.204	0.139	82.0	16.8	16.0
F52724-3		0.192		0.151	82.0		
F52737-1		0.163		0.129	62.5		
F52737-2		0.145	0.154	0.147	62.5	9.6	16.0
F52737-3		0.155		0.142	62.5		
liver blnk-1		0.344		0.134			
liverspk-1	89%	1.10		0.123			
liverspk-2	86%	0.973		0.134			
liverspk-3	91%	0.924		0.149			
F52801-1		0.372		0.112	76.7		
F52801-2		0.278	0.271	0.119	76.7	20.7	16.0
F52801-3		0.161		0.148	76.7		
F52720-1		0.252		0.128	68.7		
F52720-2		0.206	0.228	0.133	68.7	15.7	16.0
F52720-3		0.225		0.130	68.7		
F52733-1		0.224		0.123	67.7		
F52733-2		0.182	0.205	0.139	67.7	13.9	16.0
F52733-3		0.208		0.139	67.7		
F52788-1		0.179		0.143	68.0		
F52788-2		0.154	0.166	0.146	68.0	11.3	16.0
F52788-3		0.165		0.121	68.0		
Liver blk 1		0.760		0.140			
Liver blk 2		0.301		0.116			
Liver spk 1	111%	1.06		0.160			
Liver spk 2	108%	1.02		0.160			
F52730-1		0.339		0.123	57.9		
F52730-2		0.237	0.318	0.113	57.9	18.4	40.0
F52730-3		0.380		0.159	57.9		
F52731-1		0.163		0.158	59.0		
F52731-2		0.202	0.175	0.108	59.0	10.3	40.0
F52731-3		0.160		0.158	59.0		
F52802-1		0.286		0.150	71.9		
F52802-2		0.542	0.357	0.110	71.9	25.7	40.0
F52802-3		0.244		0.122	71.9		
F52714-2		0.247		0.154	45.5		
F52714-1		0.454	0.327	0.152	45.5	14.9	40.0
F52714-3		0.281		0.151	45.5		
F52734-1		0.223		0.111	58.1		
F52734-2		0.297	0.234	0.146	58.1	13.6	40.0
F52734-3		0.183		0.143	58.1		
F52774-1		0.287		0.138	71.1		
F52774-2		0.215	0.226	0.122	71.1	16.0	40.0
F52774-3		0.174		0.137	71.1		

L13492 AB ID	% rcvry	Actual ppm F- in liver (W/W)	Average ppm F- in liver (W/W)	Liver burned (grams)	Whole liver weight (grams)	Total F- in whole liver (µg)	Dosage (mg/kg)
F52712-1		0.442		0.115	72.0		
F52712-2		0.336	0.330	0.134	72.0	23.7	Fabric
F52712-3		0.210		0.111	72.0		
F52729-1		0.291		0.127	63.1		
F52729-2		0.185	0.220	0.135	63.1	13.9	Fabric
F52729-3		0.184		0.132	63.1		
F52803-1		0.294		0.118	71.1		
F52803-2		0.193	0.212	0.115	71.1	15.1	Fabric
F52803-3		0.148		0.140	71.1		
F52716-1		0.179		0.117	67.3		
F52716-2		0.165	0.173	0.137	67.3	11.7	Fabric
F52716-3		0.177		0.121	67.3		
F52728-1		0.192		0.125	79.9		
F52728-2		0.166	0.191	0.126	79.9	15.3	Fabric
F52728-3		0.215		0.106	79.9		
F52787-1		0.177		0.141	71.2		
F52787-2		0.242	0.203	0.110	71.2	14.5	Fabric
F52787-3		0.190		0.128	71.2		
Liver blk 1		0.255		0.147			
Liver blk 2		0.250		0.122			
Liver stnd 1	87%	1.14		0.115			
Liver stnd 2	88%	1.13		0.118			
52717-1		0.360		0.119	73.0		
52717-2		0.202	0.276	0.121	73.0	20.1	Fabric
52717-3		0.265		0.122	73.0		
52735-1		0.206		0.137	53.9		
52735-2		0.203	0.200	0.129	53.9	10.8	Fabric
52735-3		0.191		0.111	53.9		
52736-1		0.158		0.160	72.9		
52736-2		0.168	0.160	0.117	72.9	11.7	Fabric
52736-3		0.155		0.145	72.9		
52726-1		0.135		0.152	74.4		
52726-2		0.143	0.150	0.165	74.4	11.2	Fabric
52726-3		0.173		0.113	74.4		
52732-1		0.162		0.156	66.1		
52732-2		0.148	0.158	0.130	66.1	10.4	Fabric
52732-3		0.163		0.113	66.1		
52739-1		0.313		0.127	64.9		
52739-2		0.127	0.191	0.162	64.9	12.4	Fabric
52739-3		0.133		0.160	64.9		
Liver spk-3	0.69	0.923		0.113			
Liver spk-4	0.8222	0.888		0.140			
Liver spk-5	0.8295	1.11		0.113			
Liver spk-6	1.039	1.23		0.128			
Liver spk-7	0.769	2.29		0.102			
Liver spk-8	0.8718	1.82		0.145			
Liver spk-9	0.9319	2.35		0.120			

9.11.2 Summary and raw data; analysis of liver extracts using electrospray mass spectrometry.

This data, although supportive, in the opinion of the Study Director is not required to reach the conclusion stated in Final Report Section 6.0, and therefore is not discussed in detail.

A-1
Contains pages
A-1 thru A-4
D. Christensen
11/15/95

HWI# 6329-152 A

Logbook was found with raw data. 11/28/95

Samples NOT correlated with peaks due to logbook MISSING. However, this is of NO consequence because ALL samples ARE LESS than DETECTION limit.
DLC 11/15/95

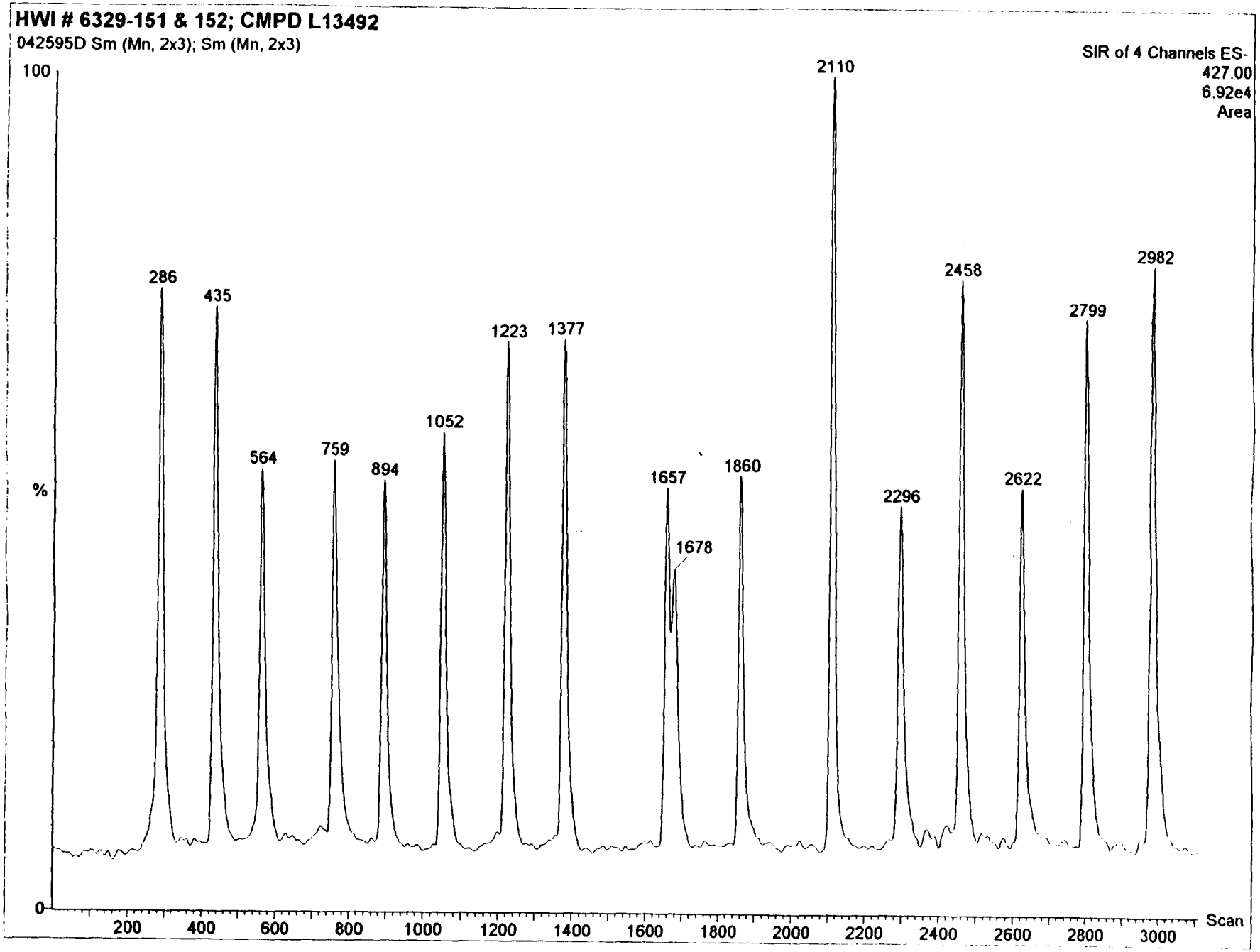
Study:	Single Dose Dermal Absorption
Protocol Number:	TP3016.AB
Test Material:	T-6067, T-6068, and T-6069 in Rabbits (L13492)
Matrix:	Liver
R Squared Value:	Screening
Response Factor Amount:	N/A
Analyst:	DLC
Date Analyzed:	4/25/95
Method:	
Instrument:	Fisons VG 2000 Electrospray MS
LABBASE FILE	042595D

Group Dose	Sample #	Ion Count Area *	Extracted wt g	Dilution factor	Concentration µg/g **	Total mass of liver g	Total amount of FC-95 per liver mg
Group 1: Distilled Water 2.0 mL/kg	F52725	N/D			N/D		N/D
	F52711	N/D			N/D		N/D
Group 2: 4 mg/kg T-6067	F52715	N/D			N/D		N/D
	F52718	N/D			N/D		N/D
	F52723	N/D			N/D		N/D
Group 3: 16 mg/kg T-6067	F52724	N/D			N/D		N/D
	F52637	N/D			N/D		N/D
	F52788	N/D			N/D		N/D
	F52733	N/D			N/D		N/D
Group 4: 40 mg/kg T-6067	F52774	N/D			N/D		N/D
	F52731	N/D			N/D		N/D
	F52734	N/D			N/D		N/D
	F52714	N/D			N/D		N/D
	F52802	N/D			N/D		N/D
Group 5: Fabric T-6068	F52728	N/D			N/D		N/D
	F52729	N/D			N/D		N/D
	F52716	N/D			N/D		N/D
	F52712	N/D			N/D		N/D
Group 6: Fabric T-6068	F52739	N/D			N/D		N/D
	F52736	N/D			N/D		N/D
	F52726	N/D			N/D		N/D

DLC 11/15/95

* SIR of M 412 + 413

** The concentration was calculated by using the standard curve and multiplying the result by 4/5. The 4/5 factor is the result of a miscalculation in applying formula 8.4 in Method AMDT-M-4-0. 137 mg of liver was used in this calculation rather than 171 mg. The concentrations in the standard curve are therefore 5/4 larger than they should be. By multiplying the calculated concentration in the standard curve by 4/5, the correct result is obtained.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042595D Sm (Mn, 2x3); Sm (Mn, 2x3)
SIR of 4 Channels ES-
427.00
6.92e4
Area
100
%
0
286
435
564
759
894
1052
1223
1377
1657
1678
1860
2110
2296
2458
2622
2799
2982
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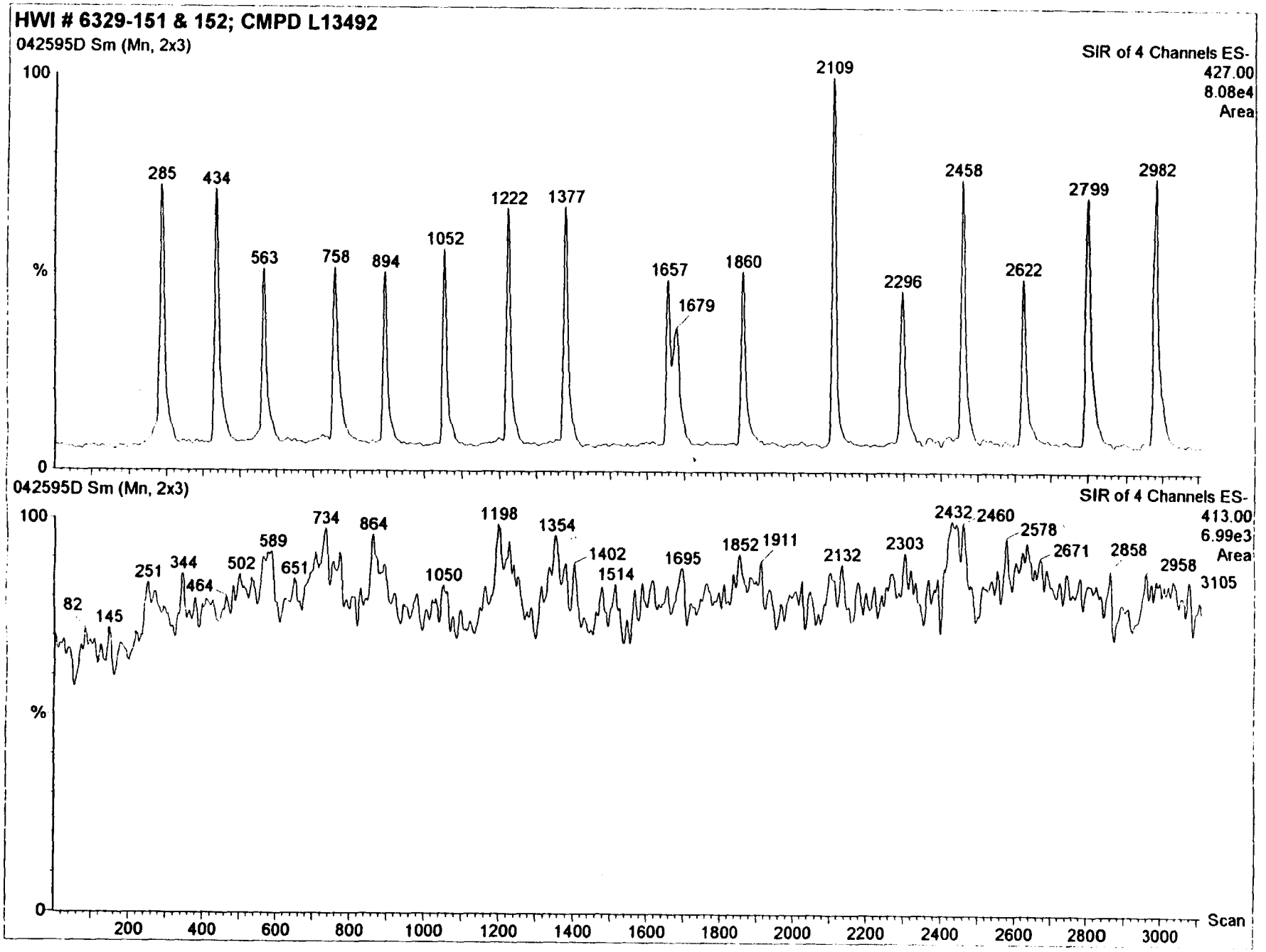
A-3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042595D Sm (Mn, 2x3)
SIR of 4 Channels ES-
427.00
8.08e4
Area
100
%
0
285
434
563
758
894
1052
1222
1377
1657
1679
1860
2109
2296
2458
2622
2799
2982
042595D Sm (Mn, 2x3)
SIR of 4 Channels ES-
413.00
6.99e3
Area
3105
100
%
0
82
145
251
344
464
502
589
651
734
864
1050
1198
1354
1402
1514
1695
1852
1911
2132
2303
2432
2460
2578
2671
2858
2958
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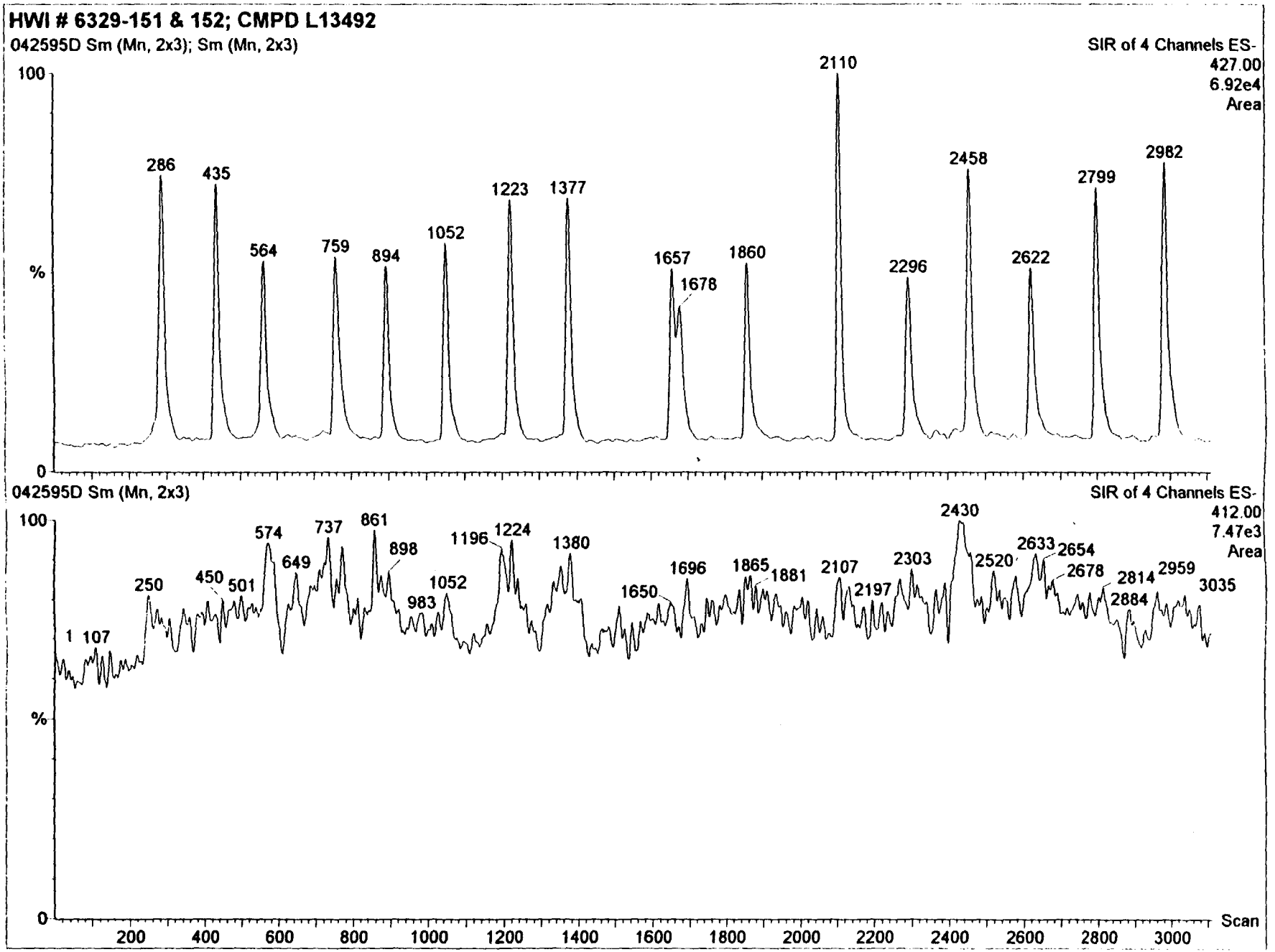
800
1000
1200
1400
1600
1800
2000
2200
2400
2600
2800
3000
Scan

000100


HWI # 6329-151 & 152; CMPD L13492
042595D Sm (Mn, 2x3); Sm (Mn, 2x3)
SIR of 4 Channels ES-
427.00
6.92e4
Area
100
%
0
286
435
564
759
894
1052
1223
1377
1657
1678
1860
2110
2296
2458
2622
2799
2982
042595D Sm (Mn, 2x3)
SIR of 4 Channels ES-
412.00
7.47e3
Area
100
%
0
1
107
250
450
501
574
649
737
861
898
983
1052
1196
1224
1380
1650
1696
1865
1881
2107
2197
2303
2430
2520
2633
2654
2678
2814
2884
2959
3035
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
2400
2600
2800
3000
Scan

9.11.3 Summary and raw data; ppm F^- in serum as determined by thermal extraction followed by analysis using Skalar segmented flow analyzer with ion selective electrode.

DDW 12819
Skalar DATA

RE: 6329-152 SERUM SAMPLES
AMDT 11095.1
Date of Analysis: 8/22, 8/23, and 8/28/95
Analyst: DDW

The samples are burned in the Dohrman at 950 C using 0.10 mL of the serum. The gas is collected in 2.0 mL of 1:1 TISAB/Milli-Q water. The samples are then analyzed on a Skalar Segmented Flow Analyzer using the Ion Specific Electrode (ISE) Method.

TISAB buffer is added to each sample as it proceeds through the system. The sample then goes through a heated mixing coil before the potential between the ion selective electrode and the reference electrode is measured. The signal is amplified and related to the fluoride concentration.

The instrument was calibrated in the ranges of 0.015 - 0.15 ppm and 0.15 - 1.50 ppm fluoride. The standard curve for the high range was plotted using the inverse logarithm option. The standard curve for the low range is linear. All standards and samples were then calculated by the Skalar software using these curves. All results below 0.0001 ppm appear on the raw data as #.####.

A quality control standard was analyzed every 10 samples to check for accuracy and drift.

Raw data is taken from the appropriate calibrated range of the Skalar printout and summarized on an Excel spreadsheet. The final results are adjusted for the collection volume and any subsequent dilutions.

Deborah D Wright

USW 1/28/95
Skalar Data

SUMMARY OF 6329-152
SERUM SAMPLES
AMDT 11095.1

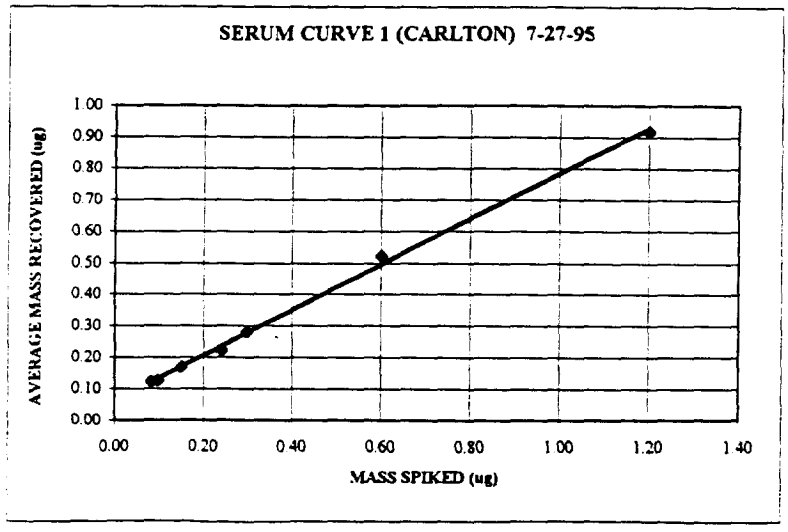
	Sample ID	Fluoride in Sample (ppm) Day 1	Fluoride in Sample (ppm) Day 2	Fluoride in Sample (ppm) Day 4	Fluoride in Sample (ppm) Day 8
	F52711-1	0.84	0.42	0.48*	0.45
GROUP 1	F52719-1	0.68	0.34	0.73	ND
Dose Level : 0 mg/kg	F52725-1	0.66	0.31	0.96*	0.78
	F52721-1	0.51	ND	0.84*	0.63
	F52727-1	0.48	0.60	0.82*	0.53
	F52806-1	0.53	0.62	0.92	0.49
	F52713-1	1.36	0.49	0.64	0.31
	F52718	1.16	0.42	0.99	0.33
GROUP 2	F52723-1	0.93	0.52	1.25	0.32
Dose Level : 4 mg/kg	F52715-1	1.00	0.54	0.69	0.49
	F52738-1	1.17	0.43	1.40	0.38
	F52809-1	1.49	0.61	0.72	0.33
	F52724-1	1.21	0.52	0.64	0.74
	F52737-1	1.17	0.48	0.64	0.72
GROUP 3	F52801-1	1.05	0.92	1.20	1.29
Dose Level : 16 mg/kg	F52720-2		0.73	0.92	0.92
	F52733-1	1.11	0.64	0.70	0.54
	F52788-1	0.32	0.74	0.50	ND
	F52730-1	1.04	0.59	0.39	
	F52731-1	0.95	0.03	0.39	
GROUP 4	F52802-1	1.11	0.68	0.59	
Dose Level : 40 mg/kg	F52714-1	0.87	0.85	0.93	
	F52734-1	1.07	0.69	1.35	
	F52774-1	1.23	0.72	0.92	
	F52712-1	1.28	0.95	0.69	
	F52729-1	1.08*	0.62	0.67	
GROUP 5	F52803-1	1.31	0.63	0.92	
Dose Level : 10 x 10 fabric	F52716-1	1.38	0.65	0.77	
	F52728-1	1.44	0.77	0.68	
	F52787-1	1.18	0.68	0.76	
	F52717-1	1.71	0.62	1.03	
	F52735-1	1.48	0.74	0.67	
GROUP 6	F52736-1	1.16	0.57	0.60*	
Dose Level : 10 x 10 fabric	F52726-1	0.94	0.79*	0.54	
	F52732-1	1.33	0.69	0.42	
	F52739-1	0.90	0.58	0.33	

* Average of duplicate analysis taken for final result. DDW

WSW 12845
Skalar Data
HWI 6329-152
AMDT 11095.1

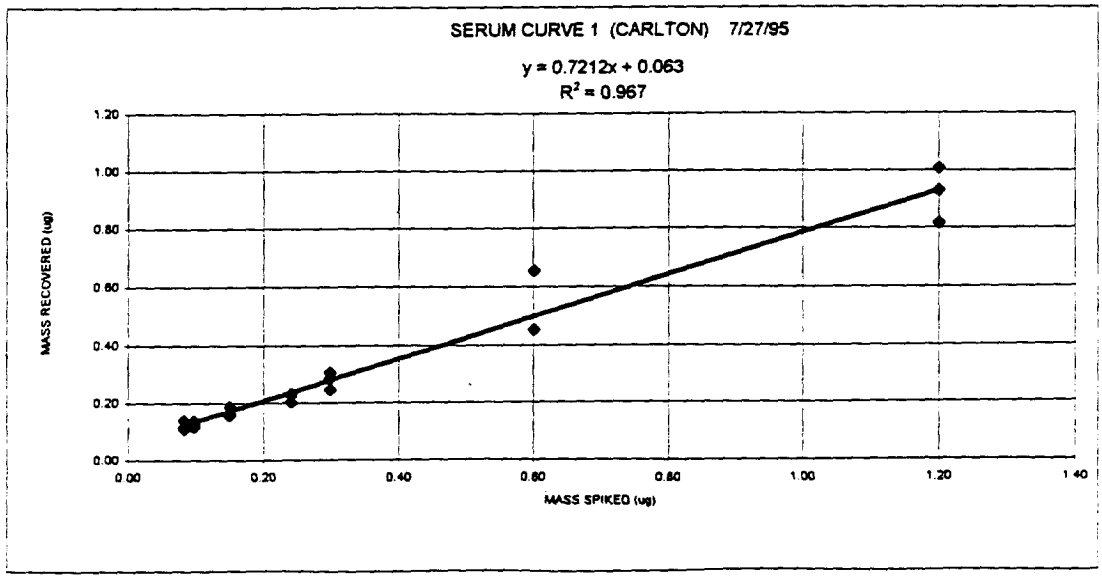
SERUM CURVE 2
7-27-95
CARLTON

Sample ID	Skalar Result (ppm)	DI:TISAB final vol (mL)	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Average Mass Recovered (ug F-)	% Recovery
Spk 34-1	0.07	2.0	0.004	34.00			
Spk 34-2	0.06	2.0	0.004	34.00	0.08	0.12	151%
Spk 34-3	0.06	2.0	0.004	34.00			
Spk 40-1	0.06	2.0	0.004	40.00			
Spk 40-2	0.06	2.0	0.004	40.00	0.10	0.13	132%
Spk 40-3	0.07	2.0	0.004	40.00			
Spk 62-1	0.08	2.0	0.004	62.00			
Spk 62-2	0.08	2.0	0.004	62.00	0.15	0.17	114%
Spk 62-3	0.09	2.0	0.004	62.00			
Spk 100-1	0.10	2.0	0.004	100.0			
Spk 100-2	0.11	2.0	0.004	100.0	0.24	0.22	93%
Spk 100-3	0.12	2.0	0.004	100.0			
Spk 124-1	0.12	2.0	0.004	124.0			
Spk 124-2	0.14	2.0	0.004	124.0	0.30	0.28	94%
Spk 124-3	0.15	2.0	0.004	124.0			
Spk 250-1	0.33	2.0	0.004	250.0			
Spk 250-2	0.23	2.0	0.004	250.0	0.60	0.52	87%
Spk 250-3	0.23	2.0	0.004	250.0			
Spk 500-1	0.47	2.0	0.004	500.0			
Spk 500-2	0.41	2.0	0.004	500.0	1.20	0.92	77%
Spk 500-3	0.50	2.0	0.004	500.0			



SERUM CURVE 2
7-27-95
CARLTON

Sample ID	Skalar Result (ppm)	DI:TISAB final vol (mL)	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery		
Spk 34-1	0.07	2.0	0.004	34.00	0.08	0.14	171%	STANDARD DEVIATION :	0.1787
Spk 34-2	0.06	2.0	0.004	34.00	0.08	0.11	138%	% RSD :	11.8165
Spk 34-3	0.06	2.0	0.004	34.00	0.08	0.12	145%		
Spk 40-1	0.06	2.0	0.004	40.00	0.10	0.12	123%	STANDARD DEVIATION :	0.1072
Spk 40-2	0.06	2.0	0.004	40.00	0.10	0.12	128%	% RSD :	8.1286
Spk 40-3	0.07	2.0	0.004	40.00	0.10	0.14	144%		
Spk 62-1	0.08	2.0	0.004	62.00	0.15	0.16	110%	STANDARD DEVIATION :	0.1072
Spk 62-2	0.08	2.0	0.004	62.00	0.15	0.16	106%	% RSD :	9.4074
Spk 62-3	0.09	2.0	0.004	62.00	0.15	0.19	126%		
Spk 100-1	0.10	2.0	0.004	100.0	0.24	0.20	85%	STANDARD DEVIATION :	0.0666
Spk 100-2	0.11	2.0	0.004	100.0	0.24	0.23	96%	% RSD :	7.1794
Spk 100-3	0.12	2.0	0.004	100.0	0.24	0.23	98%		
Spk 124-1	0.12	2.0	0.004	124.0	0.30	0.25	84%	STANDARD DEVIATION :	0.0975
Spk 124-2	0.14	2.0	0.004	124.0	0.30	0.29	97%	% RSD :	10.3277
Spk 124-3	0.15	2.0	0.004	124.0	0.30	0.31	103%		
Spk 250-1	0.33	2.0	0.004	250.0	0.60	0.66	109%	STANDARD DEVIATION :	0.1942
Spk 250-2	0.23	2.0	0.004	250.0	0.60	0.45	76%	% RSD :	22.3705
Spk 250-3	0.23	2.0	0.004	250.0	0.60	0.45	76%		
Spk 500-1	0.47	2.0	0.004	500.0	1.20	0.93	77%	STANDARD DEVIATION :	0.0795
Spk 500-2	0.41	2.0	0.004	500.0	1.20	0.82	68%	% RSD :	10.3961
Spk 500-3	0.50	2.0	0.004	500.0	1.20	1.01	84%		



1995-08-22 13:41 **OutPut of : 950822B1**
Operator : DDW
Date of the Analysis : 1995-08-22 09:04
Analysis File Name : C:\SKALAR\DATA\HWIDATA\SERUM\950822B1

Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	DI:TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery
1	Tracer	1.50	1.46	98%								
2	Drift	1.50	1.47	98%								
3	Wash		ND									
4	Standard 1	0.015	0.015	103%								
5	Standard 2	0.03	0.03	94%								
6	Standard 3	0.06	0.06	105%								
7	Standard 4	0.09	0.09	100%								
8	Standard 5	0.12	0.12	98%								
9	Standard 6	0.15	0.15	101%								
10	Standard 7	0.30	0.28	93%								
11	Standard 8	0.60	0.62	103%								
12	Standard 9	1.20	1.23	103%								
13	Standard 10	1.50	1.47	98%								
14	Drift	1.50	1.48	99%								
15	Wash		ND									
16	SERUM BLK 1		0.07		2.0	0.10	1.48					
17	SERUM BLK 2		0.04		2.0	0.10	0.82					
18	SPK 62-1		0.07		2.0	0.10	1.46	0.004	62.00	0.15	0.15	98%
19	SPK 62-2		0.11		2.0	0.10	2.18	0.004	62.00	0.15	0.22	146%
20	SPK 62-3		0.12		2.0	0.10	2.49	0.004	62.00	0.15	0.25	167%
21	SPK 250-1		0.12		2.0	0.10	2.49	0.004	250.0	0.60	0.25	42%
22	SPK 250-2		0.27		2.0	0.10	5.44	0.004	250.0	0.60	0.54	91%
23	SPK 250-3		0.27		2.0	0.10	5.34	0.004	250.0	0.60	0.53	89%
24	BLK		0.09		2.0	0.10	1.71					
25	BLK		0.07		2.0	0.10	1.35					
26	Drift	1.50	1.46	97%								
27	Wash		ND									
28	F52713-1		0.07		2.0	0.10	1.36					
29	F52718-29		0.06		2.0	0.10	1.16					
30	F52723-1		0.05		2.0	0.10	0.93					

HWI 6329 152 Serum
Skalar Data
Curve 2 Calibr

000107

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Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	DI:TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery
31	F52715-1		0.05		2.0	0.10	1.00					
32	F52738-1		0.06		2.0	0.10	1.17					
33	F52809-1		0.07		2.0	0.10	1.49					
34	F52724-1		0.06		2.0	0.10	1.21					
35	F52737-1		0.06		2.0	0.10	1.17					
36	F52801-1		0.05		2.0	0.10	1.05					
37	F52729-1		0.04		2.0	0.10	0.81					
38	Drift	1.50	1.46	98%								
39	Wash		ND									
40	SPK 62-1		0.07		2.0	0.10	1.43	0.004	62.00	0.15	0.14	96%
41	SPK 62-2		0.12		2.0	0.10	2.42	0.004	62.00	0.15	0.24	163%
42	SPK 250-1		0.17		2.0	0.10	3.32	0.004	250.0	0.60	0.33	55%
43	SPK 250-2		0.26		2.0	0.10	5.14	0.004	250.0	0.60	0.51	86%
44	BLK		0.06		2.0	0.10	1.16					
45	BLK C		0.04		2.0	0.10	0.88					
46	SPK 62-1		0.08		2.0	0.10	1.59	0.004	62.00	0.15	0.16	107%
47	SPK 62-2		0.08		2.0	0.10	1.66	0.004	62.00	0.15	0.17	111%
48	SPK 250-1		0.20		2.0	0.10	4.06	0.004	250.0	0.60	0.41	68%
49	SPK 250-2		0.22		2.0	0.10	4.38	0.004	250.0	0.60	0.44	73%
50	Drift	1.50	1.48	99%								
51	Wash		ND									
52	SPK 250-3		0.24		2.0	0.10	4.86	0.004	250.0	0.60	0.49	81%
53	SPK 250-4		0.24		2.0	0.10	4.76	0.004	250.0	0.60	0.48	79%
54	BLK		0.05		2.0	0.10	1.03					
55	BLK		0.04		2.0	0.10	0.72					
56	F52733-1		0.06		2.0	0.10	1.11					
57	F52788-1		0.02		2.0	0.10	0.32					
58	F52730-1		0.05		2.0	0.10	1.04					
59	F52731-1		0.05		2.0	0.10	0.95					
60	F52802-1		0.06		2.0	0.10	1.11					
61	F52714-1		0.04		2.0	0.10	0.87					
62	Drift	1.50	1.46	97%								
63	Wash		ND									
64	F52734-1		0.05		2.0	0.10	1.07					
65	F52774-1		0.06		2.0	0.10	1.23					
66	F52712-1		0.06		2.0	0.10	1.28					

Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	DI:TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery
67	F52729-1		0.07		2.0	0.10	1.35					
68	SPK 62-1		0.08		2.0	0.10	1.57	0.004	62.00	0.15	0.16	105%
69	SPK 62-2		0.10		2.0	0.10	1.96	0.004	62.00	0.15	0.20	132%
70	SPK 62-3		0.14		2.0	0.10	2.71	0.004	62.00	0.15	0.27	182%
71	SPK 250-1		0.26		2.0	0.10	5.10	0.004	250.0	0.60	0.51	85%
72	BLK		0.17		2.0	0.10	3.40					
73	F52803-1		0.07		2.0	0.10	1.31					
74	Drift	1.50	1.48	98%								
75	Wash		ND									
76	F52716-1		0.07		2.0	0.10	1.38					
77	F52728-1		0.07		2.0	0.10	1.44					
78	F52787-1		0.06		2.0	0.10	1.18					
79	F52717-1		0.09		2.0	0.10	1.71					
80	F52735-1		0.07		2.0	0.10	1.48					
81	F52736-1		0.06		2.0	0.10	1.16					
82	F52726-1		0.05		2.0	0.10	0.94					
83	F52732-1		0.07		2.0	0.10	1.33					
84	F52739-1		0.05		2.0	0.10	0.90					
85	SPK 62-1		0.08		2.0	0.10	1.68	0.004	62.00	0.15	0.17	113%
86	Drift	1.50	1.50	100%								
87	Wash		ND									
88	SPK 62-2		0.13		2.0	0.10	2.66	0.004	62.00	0.15	0.27	178%
89	SPK 250-1		0.34		2.0	0.10	6.72	0.004	250.0	0.60	0.67	112%
90	SPK 250-2		0.36		2.0	0.10	7.28	0.004	250.0	0.60	0.73	121%
91	Drift	1.50	1.51	101%								
92	Wash		ND									

1995-08-23 11:10 OutPut of : 950822C1
Operator : DDW
Date of the Analysis : 1995-08-22 13:41
Analysis File Name : C:\SKALAR\DATA\HWIDATA\SERUM\950822C1

Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	DI:TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery
1	Tracer	1.50	1.48	98%								
2	Drift	1.50	1.49	99%								
3	Wash		ND									
4	Standard 1	0.015	0.015	103%								
5	Standard 2	0.03	0.03	94%								
6	Standard 3	0.06	0.06	106%								
7	Standard 4	0.09	0.09	98%								
8	Standard 5	0.12	0.12	99%								
9	Standard 6	0.15	0.15	101%								
10	Standard 7	0.30	0.28	94%								
11	Standard 8	0.60	0.61	102%								
12	Standard 9	1.20	1.24	103%								
13	Standard 10	1.50	1.46	97%								
14	Drift	1.50	1.49	99%								
15	Wash		ND									
16	SERUM BLK 1		0.05		2.0	0.10	1.06					
17	SERUM BLK 2		0.05		2.0	0.10	0.99					
18	SPK 62-1		0.12		2.0	0.10	2.48	0.004	62.00	0.15	0.25	167%
19	SPK 62-2		0.09		2.0	0.10	1.77	0.004	62.00	0.15	0.18	119%
20	SPK 62-3		0.11		2.0	0.10	2.24	0.004	62.00	0.15	0.22	150%
21	SPK 250-1		0.19		2.0	0.10	3.74	0.004	250.0	0.60	0.37	62%
22	SPK 250-2		0.30		2.0	0.10	5.98	0.004	250.0	0.60	0.60	100%
23	SPK 250-3		0.28		2.0	0.10	5.50	0.004	250.0	0.60	0.55	92%
24	BLK		0.08		2.0	0.10	1.65					
25	BLK		0.06		2.0	0.10	1.11					
26	Drift	1.50	1.45	97%								
27	Wash		ND									
28	F52711-1		0.04		2.0	0.10	0.84					
29	F52719-1		0.03		2.0	0.10	0.68					
30	F52725-1		0.03		2.0	0.10	0.66					

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Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	DI:TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery
31	F52721-1		0.03		2.0	0.10	0.51					
32	F52727-1		0.02		2.0	0.10	0.48					
33	F52806-1		0.03		2.0	0.10	0.53					
34	F52711-2		0.02		2.0	0.10	0.42					
35	F52719-2		0.02		2.0	0.10	0.34					
36	F52725-2		0.02		2.0	0.10	0.31					
37	F52721-2		ND		2.0	0.10	ND					
38	Drift	1.50	1.49	99%								
39	Wash		ND									
40	SPK 62-1		0.11		2.0	0.10	2.22	0.004	62.00	0.15	0.22	149%
41	SPK 250-1		0.31		2.0	0.10	6.26	0.004	250.0	0.60	0.63	104%
42	BLK		0.06		2.0	0.10	1.16					
43	BLK		0.05		2.0	0.10	0.92					
44	SPK 62-1		0.07		2.0	0.10	1.34	0.004	62.00	0.15	0.13	90%
45	SPK 62-2		0.09		2.0	0.10	1.88	0.004	62.00	0.15	0.19	126%
46	SPK 62-3		0.09		2.0	0.10	1.76	0.004	62.00	0.15	0.18	118%
47	SPK 62-4		0.10		2.0	0.10	2.09	0.004	62.00	0.15	0.21	141%
48	SPK 250-1		0.18		2.0	0.10	3.58	0.004	250.0	0.60	0.36	60%
49	SPK 250-2		0.29		2.0	0.10	5.88	0.004	250.0	0.60	0.59	98%
50	Drift	1.50	1.45	97%								
51	Wash		ND									
52	SPK 250-3		0.27		2.0	0.10	5.46	0.004	250.0	0.60	0.55	91%
53	BLK		0.06		2.0	0.10	1.11					
54	BLK		0.04		2.0	0.10	0.73					
55	F52727-2		0.03		2.0	0.10	0.60					
56	F52806-2		0.03		2.0	0.10	0.62					
57	F52713-2		0.02		2.0	0.10	0.49					
58	F52718-2		0.02		2.0	0.10	0.42					
59	F52723-2		0.03		2.0	0.10	0.52					
60	F52715-2		0.03		2.0	0.10	0.54					
61	F52738-2		0.02		2.0	0.10	0.43					
62	Drift	1.50	1.53	102%								
63	Wash		ND									
64	F52809-2		0.03		2.0	0.10	0.61					
65	F52724-2		0.03		2.0	0.10	0.52					
66	F52737-2		0.02		2.0	0.10	0.48					

Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	DI:TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery
67	SPK 62-1		0.05		2.0	0.10	1.03	0.004	62.00	0.15	0.10	69%
68	SPK 62-2		0.07		2.0	0.10	1.39	0.004	62.00	0.15	0.14	94%
69	SPK 250-1		0.14		2.0	0.10	2.88	0.004	250.0	0.60	0.29	48%
70	SPK 250-2		0.24		2.0	0.10	4.70	0.004	250.0	0.60	0.47	78%
71	Drift	1.50	1.51	101%								
72	Wash		ND									

1995-08-23 11:00 OutPut of : 950823A1
Operator : DDW
Date of the Analysis : 1995-08-23 06:55
Analysis File Name : C:\SKALAR\DATA\HWIDATA\SERUM\950823A1

Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	DL TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery
1	Tracer	1.50	1.45	97%								
2	Drift	1.50	1.47	98%								
3	Wash		ND									
4	Standard 1	0.015	0.018	117%								
5	Standard 2	0.03	0.03	89%								
6	Standard 3	0.06	0.06	101%								
7	Standard 4	0.09	0.09	102%								
8	Standard 5	0.12	0.12	99%								
9	Standard 6	0.15	0.15	100%								
10	Standard 7	0.30	0.28	93%								
11	Standard 8	0.60	0.62	103%								
12	Standard 9	1.20	1.23	103%								
13	Standard 10	1.50	1.47	98%								
14	Drift	1.50	1.51	101%								
15	Wash		ND									
16	SERUM BLK		0.06		2.0	0.10	1.15					
17	SERUM BLK		0.05		2.0	0.10	0.94					
18	SPK 62-1		0.08		2.0	0.10	1.51	0.004	62.00	0.15	0.15	101%
19	SPK 62-2		0.10		2.0	0.10	2.01	0.004	62.00	0.15	0.20	135%
20	SPK 62-3		0.10		2.0	0.10	2.02	0.004	62.00	0.15	0.20	136%
21	SPK 250-1		0.14		2.0	0.10	2.74	0.004	250.0	0.60	0.27	46%
22	SPK 250-2		0.21		2.0	0.10	4.16	0.004	250.0	0.60	0.42	69%
23	SPK 250-3		0.23		2.0	0.10	4.50	0.004	250.0	0.60	0.45	75%
24	SPK 250-4		0.23		2.0	0.10	4.54	0.004	250.0	0.60	0.45	76%
25	SPK 250-5		0.26		2.0	0.10	5.18	0.004	250.0	0.60	0.52	86%
26	Drift	1.50	1.51	100%								
27	Wash		ND									
28	SPK 250-6		0.39		2.0	0.10	7.74	0.004	250.0	0.60	0.77	129%
29	BLK		0.07		2.0	0.10	1.30					
30	BLK		0.03		2.0	0.10	0.64					

Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	DI TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery
31	F52801-2		0.05		2.0	0.10	0.92					
32	F52720-2		0.04		2.0	0.10	0.73					
33	F52733-2		0.03		2.0	0.10	0.64					
34	F52788-2		0.04		2.0	0.10	0.74					
35	F52730-2		0.03		2.0	0.10	0.59					
36	F52731-2		0.00		2.0	0.10	0.03					
37	F52802-2		0.03		2.0	0.10	0.68					
38	Drift	1.50	1.51	100%								
39	Wash		ND									
40	F52714-2		0.04		2.0	0.10	0.85					
41	F52734-2		0.03		2.0	0.10	0.69					
42	F52774-2		0.04		2.0	0.10	0.72					
43	SPK 62-1		0.10		2.0	0.10	1.92	0.004	62.00	0.15	0.19	129%
44	SPK 250-1		0.15		2.0	0.10	2.92	0.004	250.0	0.60	0.29	49%
45	SPK 250-2		0.17		2.0	0.10	3.46	0.004	250.0	0.60	0.35	58%
46	BLK		0.05		2.0	0.10	1.03					
47	BLK		0.05		2.0	0.10	1.03					
48	SPK 62-1		0.05		2.0	0.10	1.03	0.004	62.00	0.15	0.10	69%
49	SPK 62-2		0.08		2.0	0.10	1.64	0.004	62.00	0.15	0.16	110%
50	Drift	1.50	1.53	102%								
51	Wash		ND									
52	SPK 62-3		0.12		2.0	0.10	2.46	0.004	62.00	0.15	0.25	165%
53	SPK 250-1		0.13		2.0	0.10	2.55	0.004	250.0	0.60	0.26	43%
54	SPK 250-2		0.22		2.0	0.10	4.36	0.004	250.0	0.60	0.44	73%
55	SPK 250-3		0.29		2.0	0.10	5.82	0.004	250.0	0.60	0.58	97%
56	SPK 250-4		0.25		2.0	0.10	5.06	0.004	250.0	0.60	0.51	84%
57	BLK		0.07		2.0	0.10	1.30					
58	BLK		0.06		2.0	0.10	1.13					
59	F52712-2		0.05		2.0	0.10	0.95					
60	F52729-2		0.03		2.0	0.10	0.62					
61	F52803-2		0.03		2.0	0.10	0.63					
62	Drift	1.50	1.52	101%								
63	Wash		ND									
64	F52716-2		0.03		2.0	0.10	0.65					
65	F52728-2		0.04		2.0	0.10	0.77					
66	F52787-2		0.03		2.0	0.10	0.68					

Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	DI TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery
67	F52717-2		0.03		2.0	0.10	0.62					
68	SPK 62-1		0.05		2.0	0.10	0.97	0.004	62.00	0.15	0.10	65%
69	SPK 62-2		0.09		2.0	0.10	1.83	0.004	62.00	0.15	0.18	123%
70	SPK 250-1		0.10		2.0	0.10	2.06	0.004	250.0	0.60	0.21	34%
71	SPK 250-2		0.24		2.0	0.10	4.76	0.004	250.0	0.60	0.48	79%
72	SPK 250-3		0.18		2.0	0.10	3.64	0.004	250.0	0.60	0.36	61%
73	Drift	1.50	1.54	102%								
74	Wash		ND									

1995-08-23 15:22 OutPut of : 950823B1
Operator : DDW
Date of the Analysis : 1995-08-23 10:50
Analysis File Name : C:\SKALAR\DATA\HWIDATA\SERUM\950823B1

Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	DI:TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery
1	Tracer	1.50	1.48	98%								
2	Drift	1.50	1.48	99%								
3	Wash		ND									
4	Standard 1	0.015	0.016	106%								
5	Standard 2	0.03	0.03	95%								
6	Standard 3	0.06	0.06	101%								
7	Standard 4	0.09	0.09	102%								
8	Standard 5	0.12	0.12	98%								
9	Standard 6	0.15	0.15	100%								
10	Standard 7	0.30	0.28	92%								
11	Standard 8	0.60	0.62	104%								
12	Standard 9	1.20	1.23	102%								
13	Standard 10	1.50	1.47	98%								
14	Drift	1.50	1.49	99%								
15	Wash		ND									
16	SERUM BLK		0.05		2.0	0.10	1.04					
17	SERUM BLK		0.05		2.0	0.10	0.93					
18	SERUM BLK		0.03		2.0	0.10	0.67					
19	SPK 62-1		0.05		2.0	0.10	1.03	0.004	62.00	0.15	0.10	69%
20	SPK 62-2		0.10		2.0	0.10	2.08	0.004	62.00	0.15	0.21	140%
21	SPK 62-3		0.11		2.0	0.10	2.13	0.004	62.00	0.15	0.21	143%
22	SPK 250-1		0.10		2.0	0.10	1.94	0.004	250.0	0.60	0.19	32%
23	SPK 250-2		0.23		2.0	0.10	4.66	0.004	250.0	0.60	0.47	78%
24	SPK 250-3		0.21		2.0	0.10	4.16	0.004	250.0	0.60	0.42	69%
25	SPK 124-1		0.20		2.0	0.10	4.08	0.004	124.0	0.30	0.41	137%
26	Drift	1.50	1.47	98%								
27	Wash		ND									
28	SPK 124-2		0.21		2.0	0.10	4.12	0.004	124.0	0.30	0.41	138%
29	BLK		0.13		2.0	0.10	2.53					
30	BLK		0.06		2.0	0.10	1.17					

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Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	DI:TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery
31	F52726-2		0.04		2.0	0.10	0.83					
32	F52735-2		0.04		2.0	0.10	0.74					
33	F52732-2		0.03		2.0	0.10	0.69					
34	F52736-2		0.03		2.0	0.10	0.57					
35	F52739-2		0.03		2.0	0.10	0.58					
36	F52711-4		0.03		2.0	0.10	0.53					
37	F52721-4		0.02		2.0	0.10	0.47					
38	Drift	1.50	1.50	100%								
39	Wash		ND									
40	F52719-4		0.26		2.0	0.10	5.10					
41	F52727-4		0.04		2.0	0.10	0.70					
42	F52725-4		0.03		2.0	0.10	0.60					
43	F52723-4		0.06		2.0	0.10	1.25					
44	SPK 124-1		0.17		2.0	0.10	3.30	0.004	124.0	0.30	0.33	111%
45	BLK		0.04		2.0	0.10	0.89					
46	BLK		0.04		2.0	0.10	0.84					
47	SPK 62-1		0.06		2.0	0.10	1.14	0.004	62.00	0.15	0.11	76%
48	SPK 62-2		0.08		2.0	0.10	1.67	0.004	62.00	0.15	0.17	112%
49	SPK 62-3		0.10		2.0	0.10	2.06	0.004	62.00	0.15	0.21	139%
50	Drift	1.50	1.48	99%								
51	Wash		ND									
52	SPK 250-1		0.17		2.0	0.10	3.42	0.004	250.0	0.60	0.34	57%
53	SPK 250-2		0.25		2.0	0.10	4.96	0.004	250.0	0.60	0.50	83%
54	SPK 250-3		0.29		2.0	0.10	5.88	0.004	250.0	0.60	0.59	98%
55	BLK		0.11		2.0	0.10	2.23					
56	BLK		0.08		2.0	0.10	1.57					
57	BLK		0.02		2.0	0.10	0.48					
58	F52726-2		0.04		2.0	0.10	0.75					
59	F52732-2		ND		2.0	0.10	ND					
60	F52739-2		ND		2.0	0.10	ND					
61	F52735-2		0.02		2.0	0.10	0.43					
62	Drift	1.50	1.45	96%								
63	Wash		ND									
64	F52736-2		0.03		2.0	0.10	0.57					
65	F52711-4		ND		2.0	0.10	ND					
66	BLK		0.11		2.0	0.10	2.25					

Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	Df TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery
67	BLK		0.18		2.0	0.10	3.64					
68	BLK		0.04		2.0	0.10	0.79					
69	BLK		0.02		2.0	0.10	0.42					
70	SPK 62-1		0.08		2.0	0.10	1.58	0.004	62.00	0.15	0.16	106%
71	SPK 62-2		0.08		2.0	0.10	1.65	0.004	62.00	0.15	0.17	111%
72	SPK 250-1		0.17		2.0	0.10	3.48	0.004	250.0	0.60	0.35	58%
73	SPK 250-2		0.18		2.0	0.10	3.56	0.004	250.0	0.60	0.36	59%
74	Drift	1.50	1.49	100%								
75	Wash		ND									
76	SPK 250-3		0.21		2.0	0.10	4.10	0.004	250.0	0.60	0.41	68%
77	SPK 124-1		0.17		2.0	0.10	3.32	0.004	124.0	0.30	0.33	111%
78	SPK 124-2		0.16		2.0	0.10	3.20	0.004	124.0	0.30	0.32	107%
79	BLK		0.08		2.0	0.10	1.68					
80	BLK		0.07		2.0	0.10	1.38					
81	F52711-4		0.04		2.0	0.10	0.90					
82	F52719-4		0.04		2.0	0.10	0.73					
83	SPK 124-1		0.13		2.0	0.10	2.62	0.004	124.0	0.30	0.26	88%
84	Drift	1.50	1.47	98%								
85	Wash		ND									

1995-08-28 15:03 **OutPut of : 950828A1**
Operator : DDW
Date of the Analysis : 1995-08-28 07:32
Analysis File Name : C:\SKALAR\DATA\HWIDATA\SERUM\950828A1

Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	DI:TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ng F-)	Mass Recovered (ng F-)	% Recovery
1	Tracer	1.50	1.45	97%								
2	Drift	1.50	1.47	98%								
3	Wash		ND									
4	Standard 1	0.015	0.014	91%								
5	Standard 2	0.03	0.03	105%								
6	Standard 3	0.06	0.06	102%								
7	Standard 4	0.09	0.09	99%								
8	Standard 5	0.12	0.12	99%								
9	Standard 6	0.15	0.15	100%								
10	Standard 7	0.30	0.28	93%								
11	Standard 8	0.60	0.62	103%								
12	Standard 9	1.20	1.23	103%								
13	Standard 10	1.50	1.47	98%								
14	Drift	1.50	1.49	99%								
15	Wash		ND									
16	SERUM BLANK		0.06		2.0	0.10	1.15					
17	SERUM BLANK		0.06		2.0	0.10	1.15					
18	SPK 62-1		0.10		2.0	0.10	1.92	0.004	62.00	0.15	0.19	129%
19	SPK 62-2		0.09		2.0	0.10	1.73	0.004	62.00	0.15	0.17	116%
20	SPK 250-1		0.20		2.0	0.10	3.90	0.004	250.0	0.60	0.39	65%
21	SPK 250-2		0.21		2.0	0.10	4.28	0.004	250.0	0.60	0.43	71%
22	SPK 124-1		0.19		2.0	0.10	3.84	0.004	124.0	0.30	0.38	129%
23	SPK 124-2		0.18		2.0	0.10	3.52	0.004	124.0	0.30	0.35	118%
24	BLANK		0.06		2.0	0.10	1.20					
25	F52725-4		0.07		2.0	0.10	1.32					
26	Drift	1.50	1.46	97%								
27	Wash		ND									
28	F52721-4		0.06		2.0	0.10	1.21					
29	F52727-4		0.05		2.0	0.10	0.93					
30	F52806-4		0.05		2.0	0.10	0.92					

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DDW 9/28/95
Skalar Data

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Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	DI:TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery
31	F52713-4		0.03		2.0	0.10	0.64					
32	F52718-4		0.05		2.0	0.10	0.99					
33	F52723-4		0.04		2.0	0.10	0.73					
34	F52715-4		0.03		2.0	0.10	0.69					
35	F52738-4		0.07		2.0	0.10	1.40					
36	SPK 62-1		0.05		2.0	0.10	0.93	0.004	62.00	0.15	0.09	63%
37	SPK 62-2		0.12		2.0	0.10	2.30	0.004	62.00	0.15	0.23	155%
38	Drift	1.50	1.48	99%								
39	Wash		ND									
40	SPK 124-1		0.15		2.0	0.10	2.93	0.004	124.0	0.30	0.29	98%
41	BLANK-1		0.05		2.0	0.10	1.04					
42	BLANK-2		0.04		2.0	0.10	0.75					
43	SPK 62-1		0.08		2.0	0.10	1.58	0.004	62.00	0.15	0.16	106%
44	SPK 62-2		0.08		2.0	0.10	1.69	0.004	62.00	0.15	0.17	114%
45	SPK 250-1		0.30		2.0	0.10	6.04	0.004	250.0	0.60	0.60	101%
46	SPK 250-2		0.26		2.0	0.10	5.14	0.004	250.0	0.60	0.51	86%
47	BLK		0.06		2.0	0.10	1.21					
48	F52809-4		0.04		2.0	0.10	0.72					
49	F52724-4		0.03		2.0	0.10	0.64					
50	Drift	1.50	1.47	98%								
51	Wash		ND									
52	F52737-4		0.03		2.0	0.10	0.64					
53	F52801-4		0.06		2.0	0.10	1.20					
54	F52720-4		0.05		2.0	0.10	0.92					
55	F52733-4		0.04		2.0	0.10	0.70					
56	F52788-4		0.02		2.0	0.10	0.50					
57	F52730-4		0.02		2.0	0.10	0.39					
58	F52731-4		0.02		2.0	0.10	0.39					
59	F52802-4		0.03		2.0	0.10	0.59					
60	SPK 62-1		0.05		2.0	0.10	0.95	0.004	62.00	0.15	0.10	64%
61	SPK 62-2		0.10		2.0	0.10	2.05	0.004	62.00	0.15	0.20	138%
62	Drift	1.50	1.49	99%								
63	Wash		ND									
64	SPK 250-1		0.18		2.0	0.10	3.52	0.004	250.0	0.60	0.35	59%
65	SPK 250-2		0.24		2.0	0.10	4.70	0.004	250.0	0.60	0.47	78%
66	SPK 124-1		0.16		2.0	0.10	3.20	0.004	124.0	0.30	0.32	107%

Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	DI:TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery
67	BLK		0.08		2.0	0.10	1.62					
68	BLK		0.06		2.0	0.10	1.25					
69	F52714-4		0.05		2.0	0.10	0.93					
70	F52734-4		0.07		2.0	0.10	1.35					
71	F52774-4		0.05		2.0	0.10	0.92					
72	F52712-4		0.03		2.0	0.10	0.69					
73	F52729-4		0.03		2.0	0.10	0.67					
74	Drift	1.50	1.49	99%								
75	Wash		ND									
76	F52803-4		0.05		2.0	0.10	0.92					
77	F52716-4		0.04		2.0	0.10	0.77					
78	F52728-4		0.03		2.0	0.10	0.68					
79	F52787-4		0.04		2.0	0.10	0.76					
80	SPK 62-1		0.07		2.0	0.10	1.31	0.004	62.00	0.15	0.13	88%
81	SPK 62-2		0.11		2.0	0.10	2.20	0.004	62.00	0.15	0.22	148%
82	SPK 124-1		0.09		2.0	0.10	1.81	0.004	124.0	0.30	0.18	61%
83	SPK 124-2		0.15		2.0	0.10	2.97	0.004	124.0	0.30	0.30	100%
84	Drift	1.50	1.51	100%								
85	Wash		ND									

1995-08-29 06:38 OutPut of : 950828B1
Operator : DDW
Date of the Analysis : 1995-08-28 11:47
Analysis File Name : C:\SKALAR\DATA\HWIDATA\SERUM\950828B1

Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	DI:TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery
1	Tracer	1.50	1.46	98%								
2	Drift	1.50	1.47	98%								
3	Wash		ND									
4	Standard 1	0.015	0.011	74%								
5	Standard 2	0.03	0.04	131%								
6	Standard 3	0.06	0.06	97%								
7	Standard 4	0.09	0.09	96%								
8	Standard 5	0.12	0.12	99%								
9	Standard 6	0.15	0.15	101%								
10	Standard 7	0.30	0.28	93%								
11	Standard 8	0.60	0.62	103%								
12	Standard 9	1.20	1.23	102%								
13	Standard 10	1.50	1.47	98%								
14	Drift	1.50	1.50	100%								
15	Wash		ND									
16	SERUM BLK 1		0.04		2.0	0.10	0.84					
17	SERUM BLK 2		0.03		2.0	0.10	0.69					
18	SPK 62-1		0.11		2.0	0.10	2.16	0.004	62.00	0.15	0.22	145%
19	SPK 62-2		0.09		2.0	0.10	1.89	0.004	62.00	0.15	0.19	127%
20	SPK 124-1		0.29		2.0	0.10	5.86	0.004	124.0	0.30	0.59	197%
21	SPK 124-2		0.13		2.0	0.10	2.55	0.004	124.0	0.30	0.26	86%
22	BLK		0.06		2.0	0.10	1.20					
23	BLK		0.04		2.0	0.10	0.71					
24	F52717-4		0.05		2.0	0.10	1.03					
25	F52735-4		0.03		2.0	0.10	0.67					
26	Drift	1.50	1.46	97%								
27	Wash		ND									
28	F52736-4		0.03		2.0	0.10	0.64					
29	F52726-4		0.03		2.0	0.10	0.54					
30	F52732-4		0.02		2.0	0.10	0.42					

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Skalar Data
Cum 2 Carlton

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Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	DI TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery
31	F52739-4		0.02		2.0	0.10	0.33					
32	F52711-8		0.02		2.0	0.10	0.45					
33	F52719-8		ND		2.0	0.10	ND					
34	BLK		0.03		2.0	0.10	0.60					
35	SPK 62-1		0.05		2.0	0.10	1.09	0.004	62.00	0.15	0.11	73%
36	SPK 62-2		0.06		2.0	0.10	1.17	0.004	62.00	0.15	0.12	79%
37	SPK 124-1		0.08		2.0	0.10	1.58	0.004	124.0	0.30	0.16	53%
38	Drift	1.50	1.46	97%								
39	Wash		ND									
40	SPK 124-2		0.17		2.0	0.10	3.48	0.004	124.0	0.30	0.35	117%
41	BLK		0.11		2.0	0.10	2.15					
42	BLK		0.05		2.0	0.10	0.93					
43	F52725-8		0.04		2.0	0.10	0.78					
44	F52721-8		0.03		2.0	0.10	0.63					
45	F52727-		0.03		2.0	0.10	0.53					
46	F52806-8		0.02		2.0	0.10	0.49					
47	F52713-8		0.02		2.0	0.10	0.31					
48	F52718-8		0.02		2.0	0.10	0.33					
49	F52723-8		0.02		2.0	0.10	0.32					
50	Drift	1.50	1.46	98%								
51	Wash		ND									
52	F52715-8		0.02		2.0	0.10	0.49					
53	F52738-8		0.02		2.0	0.10	0.38					
54	F52809-8		0.02		2.0	0.10	0.33					
55	SPK 62-1		0.05		2.0	0.10	0.90	0.004	62.00	0.15	0.09	61%
56	SPK 62-2		0.06		2.0	0.10	1.30	0.004	62.00	0.15	0.13	87%
57	SPK 124-1		0.10		2.0	0.10	1.98	0.004	124.0	0.30	0.20	67%
58	SPK 124-2		0.12		2.0	0.10	2.44	0.004	124.0	0.30	0.24	82%
59	BLK		0.04		2.0	0.10	0.72					
60	BLK		0.02		2.0	0.10	0.39					
61	SPK 62-1		0.08		2.0	0.10	1.50	0.004	62.00	0.15	0.15	101%
62	Drift	1.50	1.46	97%								
63	Wash		ND									
64	SPK 62-2		0.08		2.0	0.10	1.60	0.004	62.00	0.15	0.16	107%
65	SPK 124-1		0.14		2.0	0.10	2.79	0.004	124.0	0.30	0.28	94%
66	SPK 124-2		0.14		2.0	0.10	2.85	0.004	124.0	0.30	0.29	96%

Sample #	Sample ID	Skalar Standard (ppm)	Skalar Result (ppm)	% Recovery	DI:TISAB final vol (mL)	Qty Sampl (mL)	Actual ppm F- in Sample	mL FC 95 Solution Spiked	Conc FC 95 Soln (ppm)	Mass Spiked (ug F-)	Mass Recovered (ug F-)	% Recovery
67	BLK		0.05		2.0	0.10	0.95					
68	BLK		0.04		2.0	0.10	0.76					
69	F52724-8		0.04		2.0	0.10	0.74					
70	F52737-8		0.04		2.0	0.10	0.72					
71	F52801-8		0.06		2.0	0.10	1.29					
72	F52720-8		0.05		2.0	0.10	0.92					
73	F52733-8		0.03		2.0	0.10	0.54					
74	Drift	1.50	1.47	98%								
75	Wash		ND									
76	F52788-8		ND		2.0	0.10	ND					
77	SPK 62-1		0.06		2.0	0.10	1.12	0.004	62.00	0.15	0.11	75%
78	SPK 62-2		0.07		2.0	0.10	1.31	0.004	62.00	0.15	0.13	88%
79	SPK 124-1		0.11		2.0	0.10	2.15	0.004	124.0	0.30	0.21	72%
80	Drift	1.50	1.47	98%								
81	Wash		ND									

DDW 8/31/95
AMT 11095.1
HWI 6329-152
Serum Curve 2
Carlton

1995-08-22 13:41 OutPut of : 950822B1

Software : version 6.1 c1990,93

Operator : DDW

Date of the Analysis : 1995-08-22 09:04

Analysis File Name : C:\SKALAR\DATA\HWIDATA\SERUM\950822B1

Fluoride 1.5
Calibration order = Inverse Logarithm

Slope *: s = #.#####*

$$\text{Result} = 10^{\left[\frac{x - c1}{s}\right]}$$

x = corrected value of the sample
c1 = corrected value of the concentration 1
s = Slope of the electrode

a2 = -0.00000
a1 = 0.00072
a0 = -1.20078

Fluoride L
Calibration order = 2

Correlation : r = 0.99917

Result = a2 * x^2 + a1 * x + a0

a2 = -0.00000
a1 = 0.00025
a0 = 0.00551

Sampler
Type : SA1000
Number : 1
Sample Time : 50 sec.
Wash Time : 120 sec.
Air Time : 1 sec.
Take up : Single
sPecial : None
needle Height : 70 mm.

Diluter
needle Height : 80 mm
dilution Factor : 10
dilution Volume : 2.5 ml.
Resample : 1
Dilution runs : 1

User file : . TXT
Reproces : No

1995-08-22 13:41 OutPut of : 950822B1

Fluoride 1.5

Path number : 3
Signal type : Debubbled
Decolor : Yes
system Number : 0
diLute : No
Resample : No
dil Threshold : 4095
diG output : 0
Window event : Off

s1 sTandard : Ignore
s2 sTandard : Ignore
s3 sTandard : Ignore
s4 sTandard : Ignore
s5 sTandard : Ignore
s6 sTandard : 0.150
s7 sTandard : 0.300
s8 sTandard : 0.600
s9 sTandard : 1.200
s10 sTandard : 1.500
Order : Inverse Logarithm
Dimension : PPM
start Value : 500 DU
trigger Limit : 1800 Sec
Peak shape : Pointed
stArt ignore : 60 Sec
eNd ignore : 120 Sec
Measure window : 75 %
Filter : No
Regeneration : No
formUla :
output : ##.###

Fluoride L

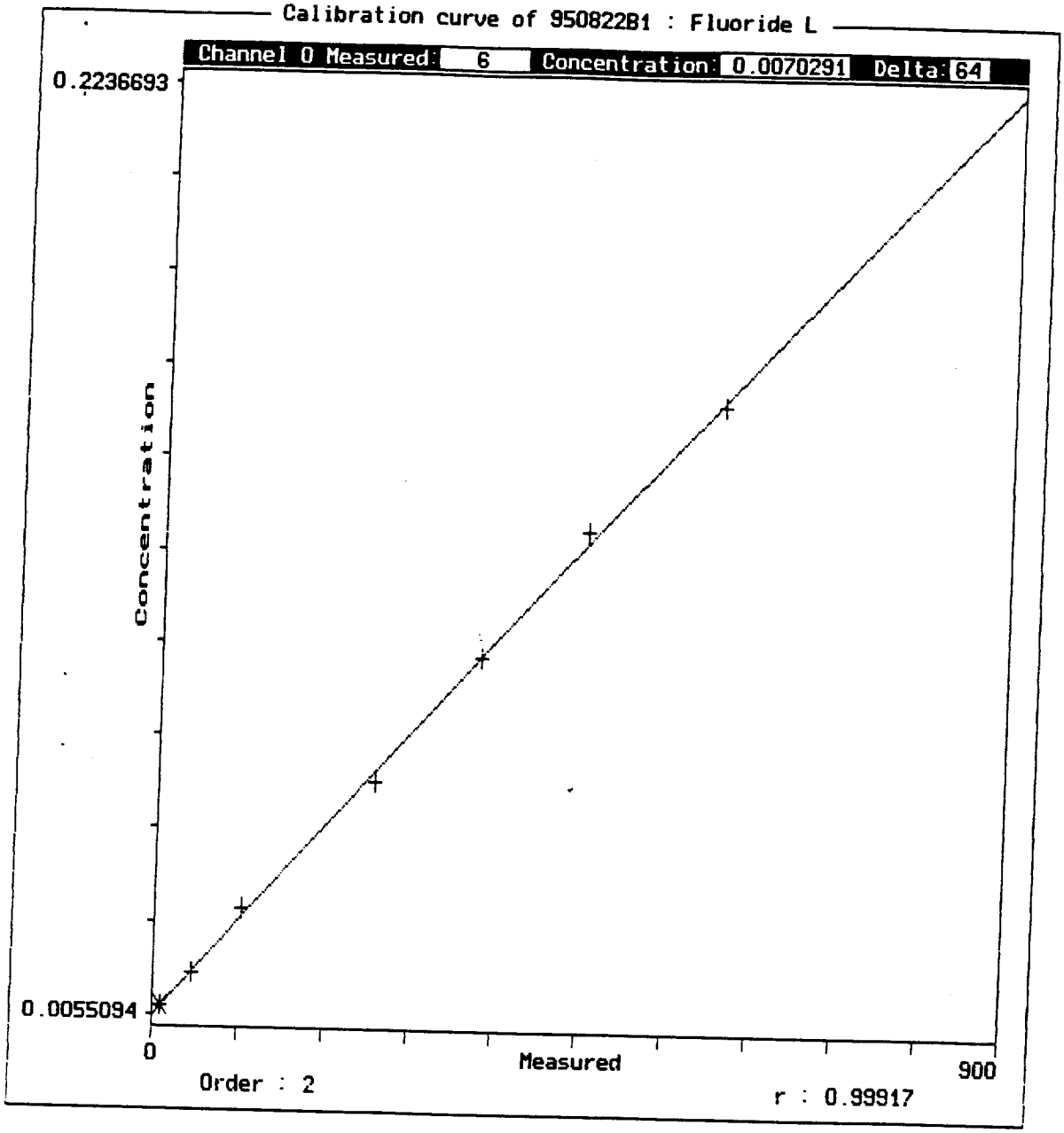
Path number : 0
Signal type : Debubbled
Decolor : No
system Number : 0
diLute : No
Resample : No
dil Threshold : 4095
diG output : 0
Window event : Off

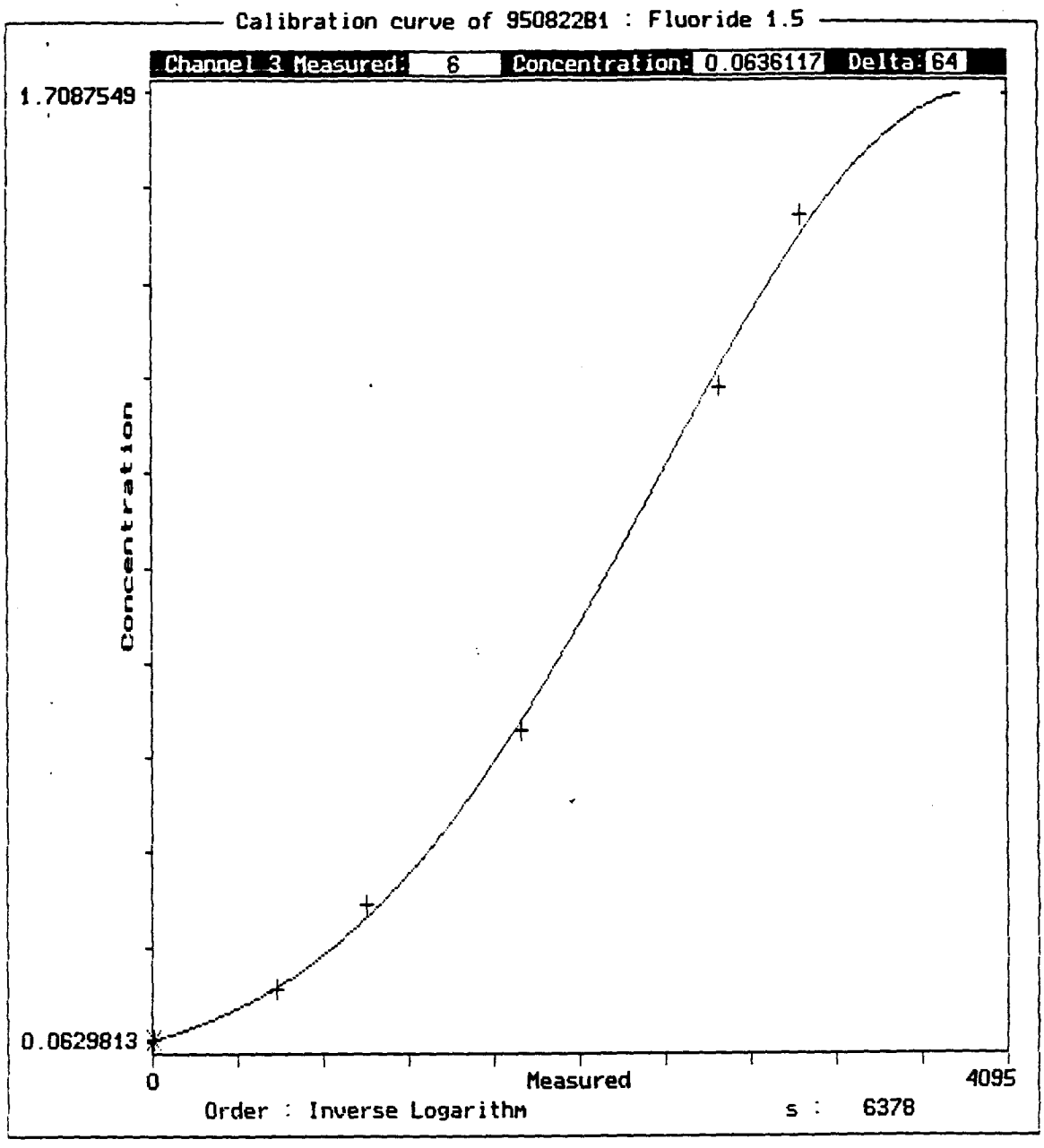
1995-08-22 13:41 OutPut of : 950822B1

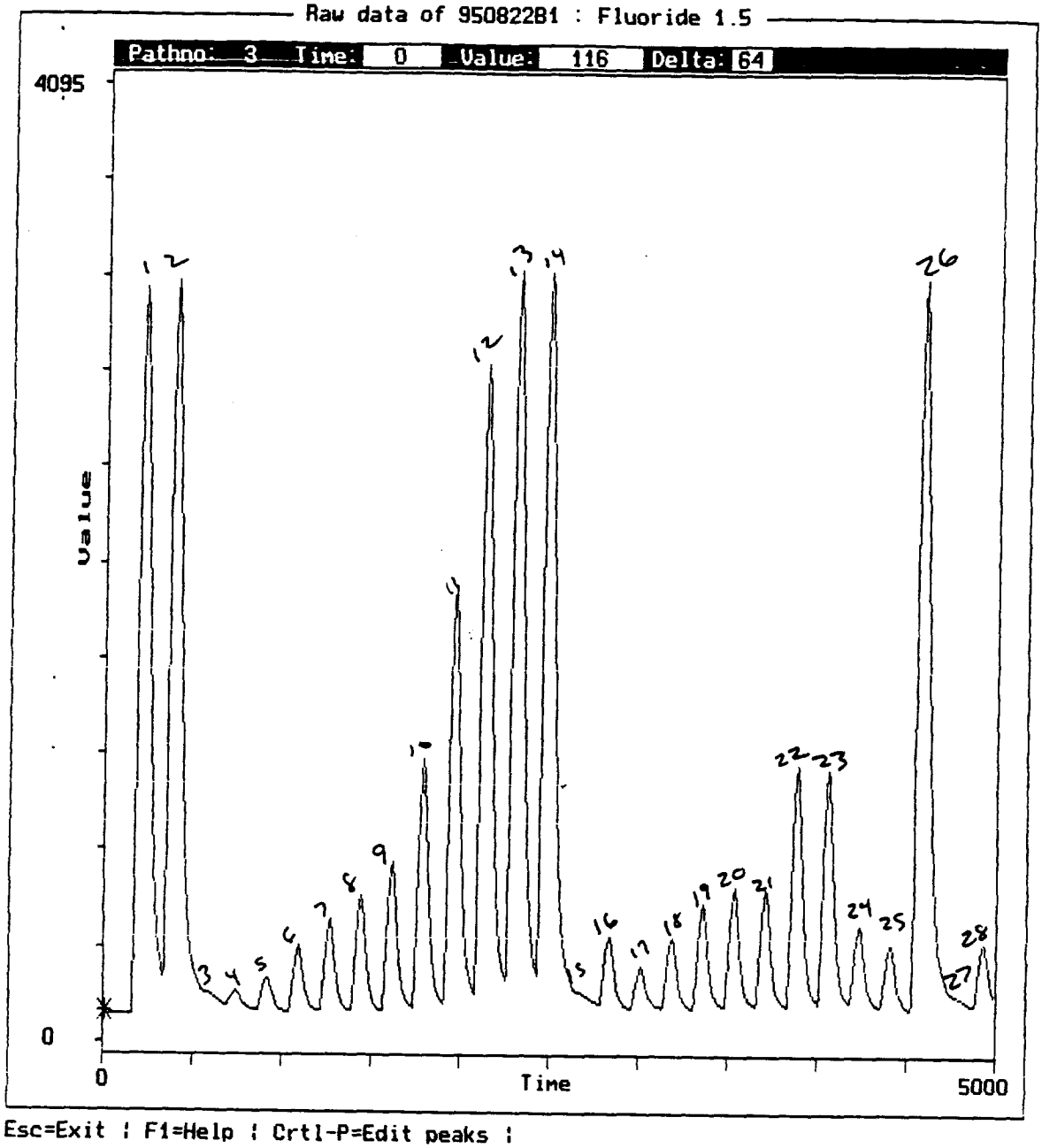
```
s1   sTandard :    0.015
s2   sTandard :    0.030
s3   sTandard :    0.060
s4   sTandard :    0.090
s5   sTandard :    0.120
s6   sTandard :    0.150
s7   sTandard : Ignore
s8   sTandard : Ignore
s9   sTandard : Ignore
s10  sTandard : Ignore
Order : 2
Dimension : PPM
start Value     : 500  DU
trigger Limit   : 1800 Sec
Peak shape      : Pointed
stArt ignore    : 60   Sec
eNd   ignore    : 120  Sec
Measure window : 75   %
Filter          : No
Regeneration    : No
formUla    : c4:=c3
output     : #.####
```

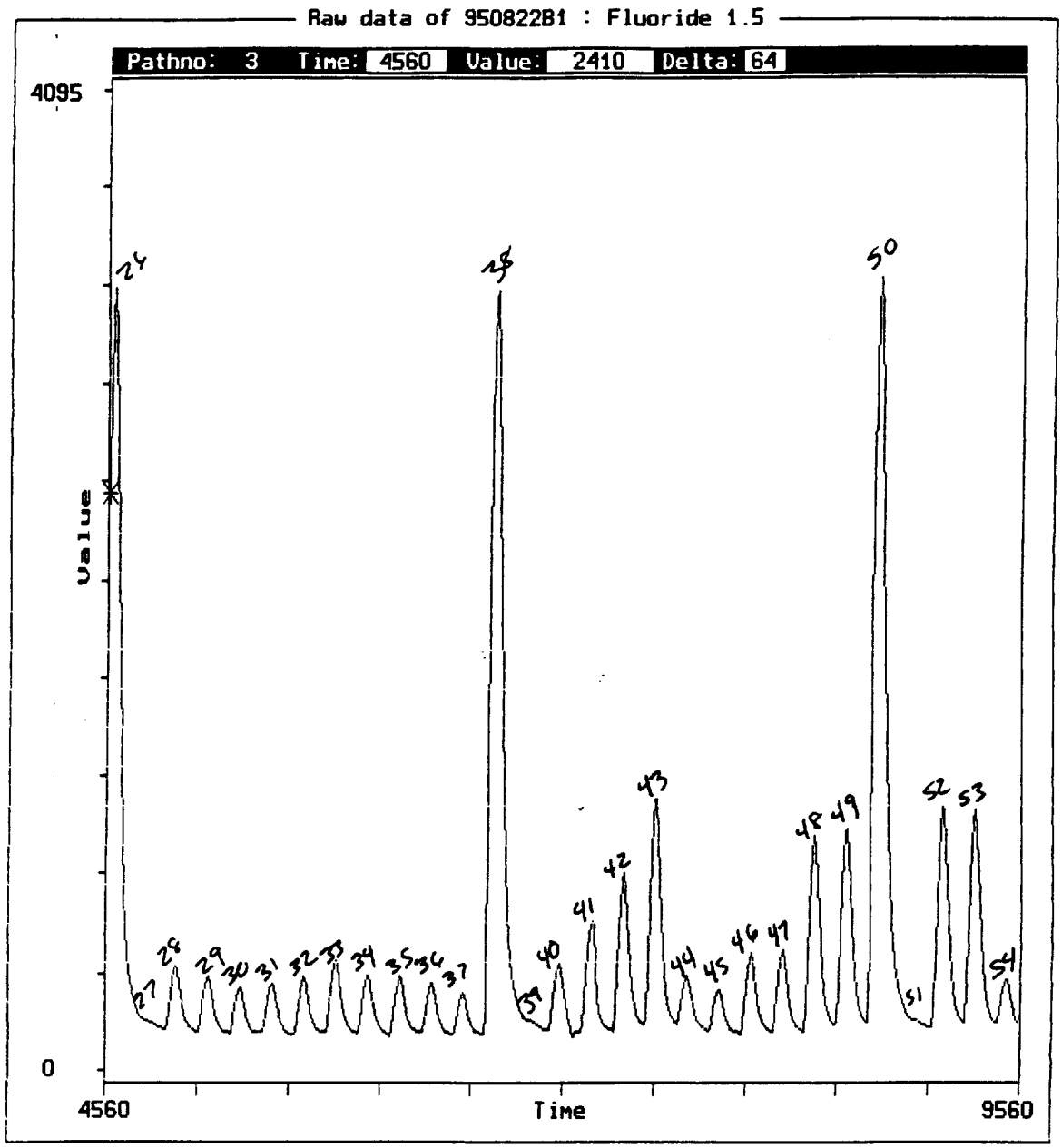
Pos	Typ	Ident	Fluoride 1.5 PPM Ch	Result	F	Time	Fluoride L PPM Ch	Result	F	Time
wt	iw	Initial Wash	3	0.063		65	4	0.0055		0
1	t	Tracer	3	1.464		210	4	0.6754		0
2	d	Drift	3	1.468		384	4	0.6770		0
3	w	Wash	3	0.063		626	4	0.0055		0
4	s1	Standard 1	3	0.067		731	4	0.0154		0
5	s2	Standard 2	3	0.073		909	4	0.0282		0
6	s3	Standard 3	3	0.091		1085	4	0.0629		0
7	s4	Standard 4	3	0.108		1257	4	0.0902		0
8	s5	Standard 5	3	0.127		1435	4	0.1171		0
9	s6	Standard 6	3	0.157		1611	4	0.1512		0
10	s7	Standard 7	3	0.278		1786	4	0.2529		0
11	s8	Standard 8	3	0.618		1960	4	0.4152		0
12	s9	Standard 9	3	1.232		2136	4	0.6046		0
13	s10	Standard 10	3	1.466		2310	4	0.6763		0
14	d	Drift	3	1.480		2486	4	0.6810		0
15	w	Wash	3	0.063		2727	4	0.0055		0
16	u	SERUM BLK 1	3	0.098		2837	4	0.0738		0
17	u	SERUM BLK 2	3	0.079		3007	4	0.0410		0
18	u	SPK 62-1	3	0.097		3185	4	0.0730		0
19	u	SPK 62-2	3	0.121		3363	4	0.1088		0
20	u	SPK 62-3	3	0.133		3539	4	0.1246		0
21	u	SPK 250-1	3	0.133		3713	4	0.1246		0
22	u	SPK 250-2	3	0.272		3888	4	0.2488		0
23	u	SPK 250-3	3	0.267		4063	4	0.2453		0
24	u	BLK	3	0.105		4237	4	0.0856		0
25	u	BLK	3	0.094		4416	4	0.0673		0
26	d	Drift	3	1.457		4588	4	0.6732		0
27	w	Wash	3	0.063		4823	4	0.0055		0
28	u	F52713-1	3	0.094		4938	4	0.0681		0
29	u	F52718-29	3	0.088		5113	4	0.0582		0
30	u	F52723-1	3	0.082		5289	4	0.0467		0
31	u	F52715-1	3	0.084		5461	4	0.0502		0
32	u	F52738-1	3	0.089		5635	4	0.0584		0
33	u	F52809-1	3	0.098		5813	4	0.0743		0
34	u	F52724-1	3	0.090		5991	4	0.0604		0
35	u	F52737-1	3	0.089		6163	4	0.0587		0
36	u	F52801-1	3	0.085		6340	4	0.0527		0
37	u	F52729-1	3	0.079		6515	4	0.0405		0
38	d	Drift	3	1.464		6690	4	0.6754		0
39	w	Wash	3	0.063		6929	4	0.0055		0
40	u	SPK 62-1	3	0.096		7042	4	0.0715		0
41	u	SPK 62-2	3	0.131		7229	4	0.1212		0
42	u	SPK 250-1	3	0.166		7392	4	0.1612		0
43	u	SPK 250-2	3	0.257		7566	4	0.2380		0
44	u	BLK	3	0.088		7741	4	0.0579		0
45	u	BLK C	3	0.081		7917	4	0.0440		0
46	u	SPK 62-1	3	0.101		8092	4	0.0794		0
47	u	SPK 62-2	3	0.103		8267	4	0.0829		0
48	u	SPK 250-1	3	0.203		8441	4	0.1957		0
49	u	SPK 250-2	3	0.219		8617	4	0.2097		0
50	d	Drift	3	1.479		8792	4	0.6807		0
51	w	Wash	3	0.063		9031	4	0.0055		0
52	u	SPK 250-3	3	0.243		9142	4	0.2278		0
53	u	SPK 250-4	3	0.238		9318	4	0.2241		0

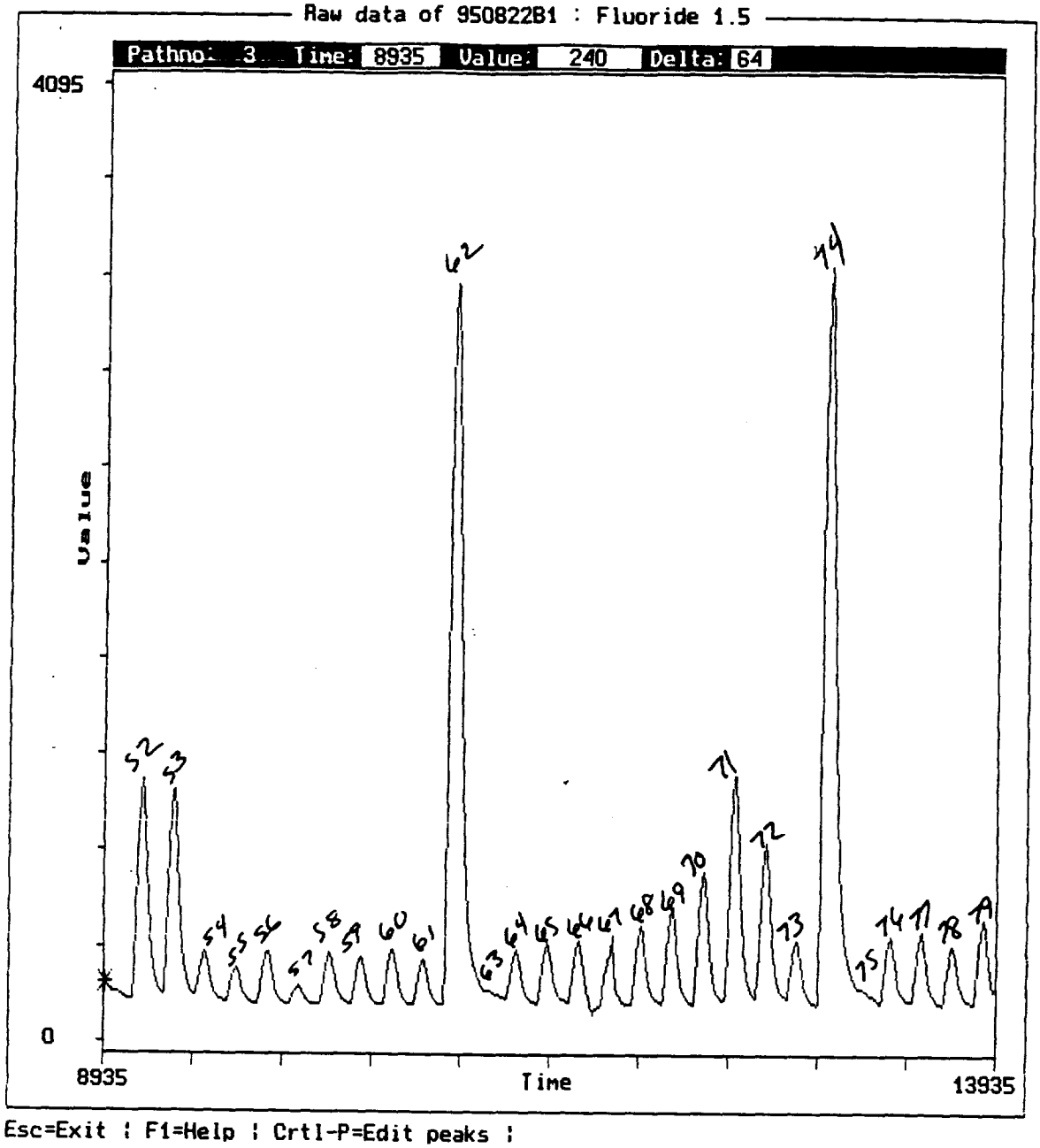
			Fluoride 1.5			Fluoride L		
			PPM			*PPM*		
Pos	Typ	Ident	Ch	Result	F Time	Ch	Result	F Time
54	u	BLK	3	0.085	9490	4	0.0517	0
55	u	BLK	3	0.077	9668	4	0.0362	0
56	u	F52733-1	3	0.087	9842	4	0.0554	0
57	u	F52788-1	3	0.068	10017	4	0.0161	0
58	u	F52730-1	3	0.085	10193	4	0.0520	0
59	u	F52731-1	3	0.083	10367	4	0.0477	0
60	u	F52802-1	3	0.087	10545	4	0.0557	0
61	u	F52714-1	3	0.081	10719	4	0.0435	0
62	d	Drift	3	1.462	10893	4	0.6748	0
63	w	Wash	3	0.063	11126	4	0.0055	0
64	u	F52734-1	3	0.086	11240	4	0.0537	0
65	u	F52774-1	3	0.090	11417	4	0.0617	0
66	u	F52712-1	3	0.092	11596	4	0.0641	0
67	u	F52729-1	3	0.094	11785	4	0.0676	0
68	u	SPK 62-1	3	0.100	11945	4	0.0785	0
69	u	SPK 62-2	3	0.114	12120	4	0.0981	0
70	u	SPK 62-3	3	0.143	12294	4	0.1356	0
71	u	SPK 250-1	3	0.255	12469	4	0.2371	0
72	u	BLK	3	0.170	12645	4	0.1650	0
73	u	F52803-1	3	0.093	12820	4	0.0654	0
74	d	Drift	3	1.475	12995	4	0.6793	0
75	w	Wash	3	0.063	13230	4	0.0055	0
76	u	F52716-1	3	0.095	13345	4	0.0691	0
77	u	F52728-1	3	0.097	13521	4	0.0720	0
78	u	F52787-1	3	0.089	13695	4	0.0592	0
79	u	F52717-1	3	0.105	13871	4	0.0853	0
80	u	F52735-1	3	0.098	14045	4	0.0738	0
81	u	F52736-1	3	0.088	14221	4	0.0579	0
82	u	F52726-1	3	0.083	14396	4	0.0472	0
83	u	F52732-1	3	0.093	14571	4	0.0666	0
84	u	F52739-1	3	0.081	14745	4	0.0450	0
85	u	SPK 62-1	3	0.104	14920	4	0.0839	0
86	d	Drift	3	1.503	15097	4	0.6888	0
87	w	Wash	3	0.063	15338	4	0.0055	0
88	u	SPK 62-2	3	0.140	15450	4	0.1328	0
89	u	SPK 250-1	3	0.336	15621	4	0.2886	0
90	u	SPK 250-2	3	0.364	15800	4	0.3039	0
91	d	Drift	3	1.508	15972	4	0.6906	0
92	w	Wash	3	0.063	16213	4	0.0055	0
wt	rw	RunOut Wash	3	0.063	16447	4	0.0055	0


Calibration curve of 950822B1 : Fluoride L
Channel 0 Measured: 6
Concentration: 0.0070291
Delta: 64
0.2236693
0.0055094
Concentration
0
Measured
900
Order : 2
r : 0.99917

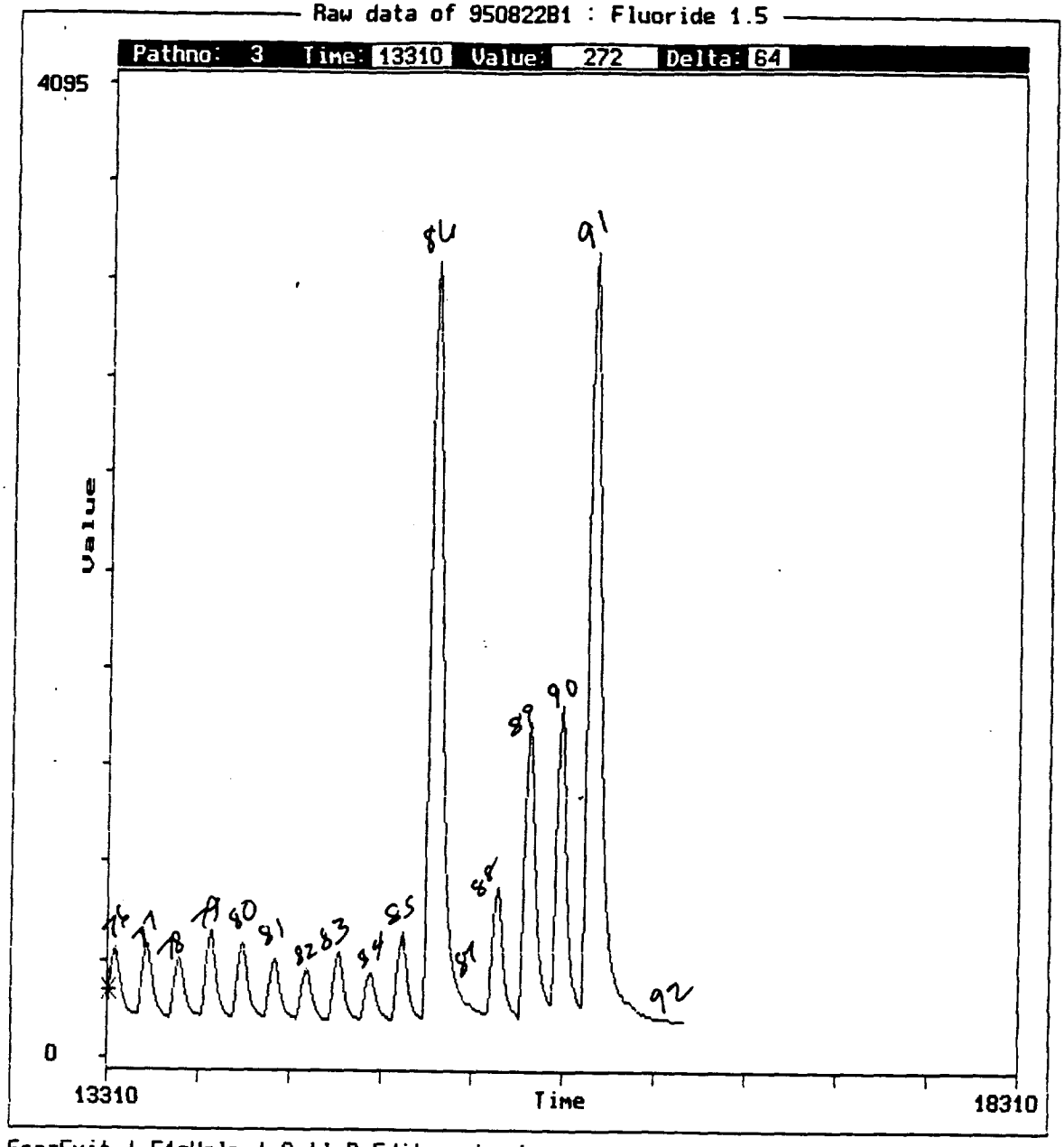

Calibration curve of 950822B1 : Fluoride 1.5
Channel 3 Measured: 6
Concentration: 0.0636117
Delta: 64
1.7087549
0.0629813
Concentration
0
Measured
4095
Order : Inverse Logarithm
s : 6378


Raw data of 95082281 : Fluoride 1.5
Pathno: 3 Time: 0 Value: 116 Delta: 64
4095
0
Value
0
Time
5000
Esc=Exit ¦ F1=Help ¦ Crtl-P=Edit peaks ¦


Raw data of 950822B1 : Fluoride 1.5
Pathno: 3 Time: 4560 Value: 2410 Delta: 64
4095
Value
0
4560
Time
9560
Esc=Exit ¦ F1=Help ¦ Crtl-P=Edit peaks ¦



Esc=Exit ¦ F1=Help ¦ Crtl-P=Edit peaks ¦


Raw data of 950822B1 : Fluoride 1.5
Pathno: 3 Time: 13310 Value: 272 Delta: 64
4095
Value
0
13310
Time
18310
84
91
89
90
88
92
Esc=Exit ¦ F1=Help ¦ Crtl-P=Edit peaks ¦

DDW 8/31/95
AMDT 11095.1
HWI 6329-152
Serum Curve 2
Carlton

1995-08-22 17:22 OutPut of : 950822C1

Software : version 6.1 c1990,93

Operator : DDW

Date of the Analysis : 1995-08-22 13:41

Analysis File Name : C:\SKALAR\DATA\HWIDATA\SERUM\950822C1

Fluoride 1.5
Calibration order = Inverse Logarithm

Slope : s = #.#####

$$\text{Result} = 10^{\left[\frac{x - c1}{s}\right]}$$

x = corrected value of the sample
c1 = corrected value of the concentration 1
s = Slope of the electrode

a2 = -0.00000
a1 = 0.00071
a0 = -1.19998

Fluoride L
Calibration order = 2

Correlation : r = 0.99910

Result = a2 * x^2 + a1 * x + a0

a2 = 0.00000
a1 = 0.00023
a0 = 0.00817

Sampler
Type : SA1000
Number : 1
Sample Time : 50 sec.
Wash Time : 120 sec.
Air Time : 1 sec.
Take up : Single
sPecial : None
needle Height : 70 mm.

Diluter
needle Height : 80 mm
dilution Factor : 10
dilution Volume : 2.5 ml.
Resample : 1
Dilution runs : 1

User file : . TXT
Reproces : No

1995-08-22 17:22 OutPut of : 950822C1

Fluoride 1.5

Path number : 3
Signal type : Debubbled
Decolor : Yes
system Number : 0
diLute : No
Resample : No
dil Threshold : 4095
diG output : 0
Window event : Off

s1 sTandard : Ignore
s2 sTandard : Ignore
s3 sTandard : Ignore
s4 sTandard : Ignore
s5 sTandard : Ignore
s6 sTandard : 0.150
s7 sTandard : 0.300
s8 sTandard : 0.600
s9 sTandard : 1.200
s10 sTandard : 1.500
Order : Inverse Logarithm
Dimension : PPM
start Value : 500 DU
trigger Limit : 1800 Sec
Peak shape : Pointed
stArt ignore : 60 Sec
eNd ignore : 120 Sec
Measure window : 75 %
Filter : No
Regeneration : No
formUla :
output : ##.###

Fluoride L

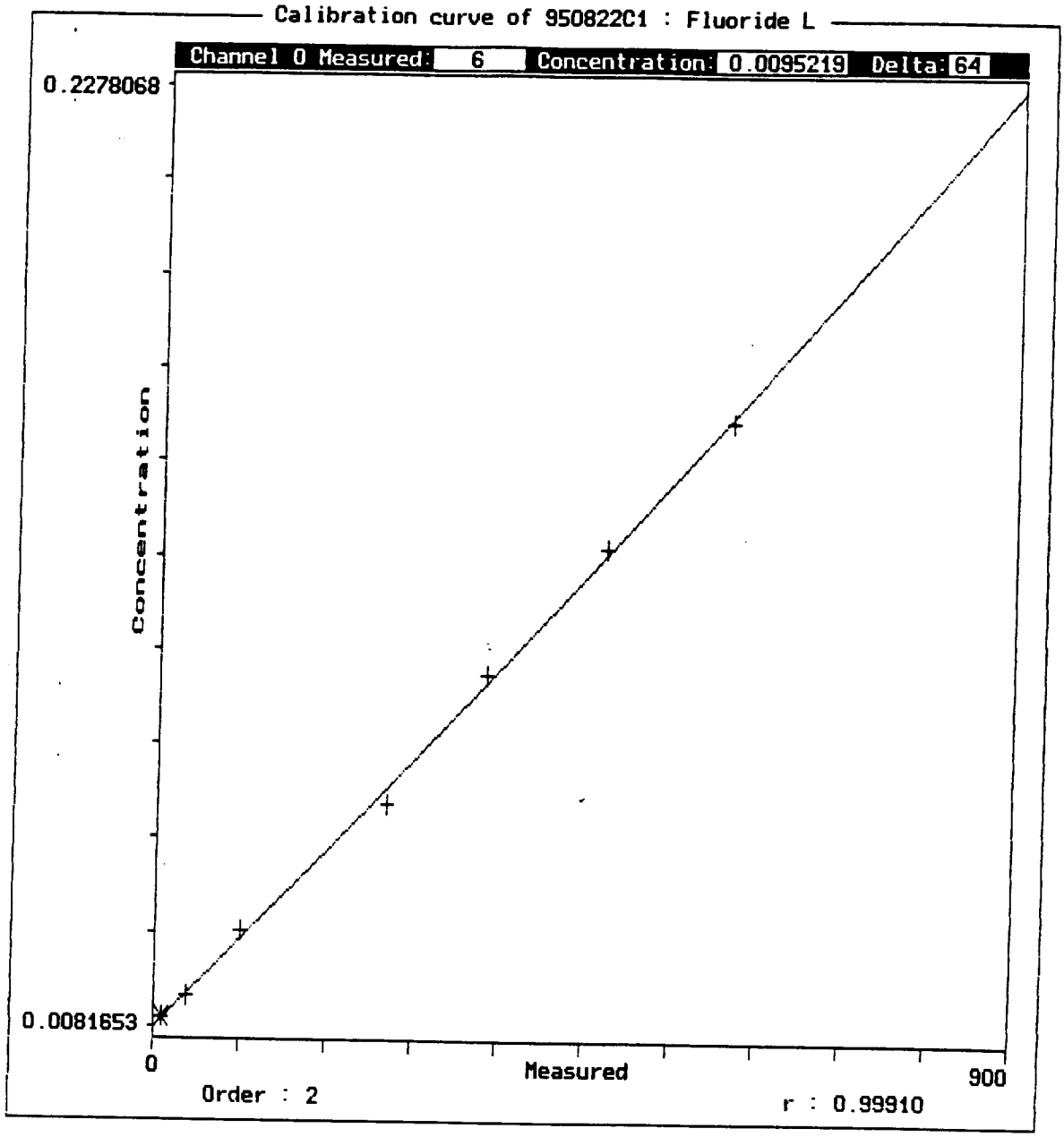
Path number : 0
Signal type : Debubbled
Decolor : No
system Number : 0
diLute : No
Resample : No
dil Threshold : 4095
diG output : 0
Window event : Off

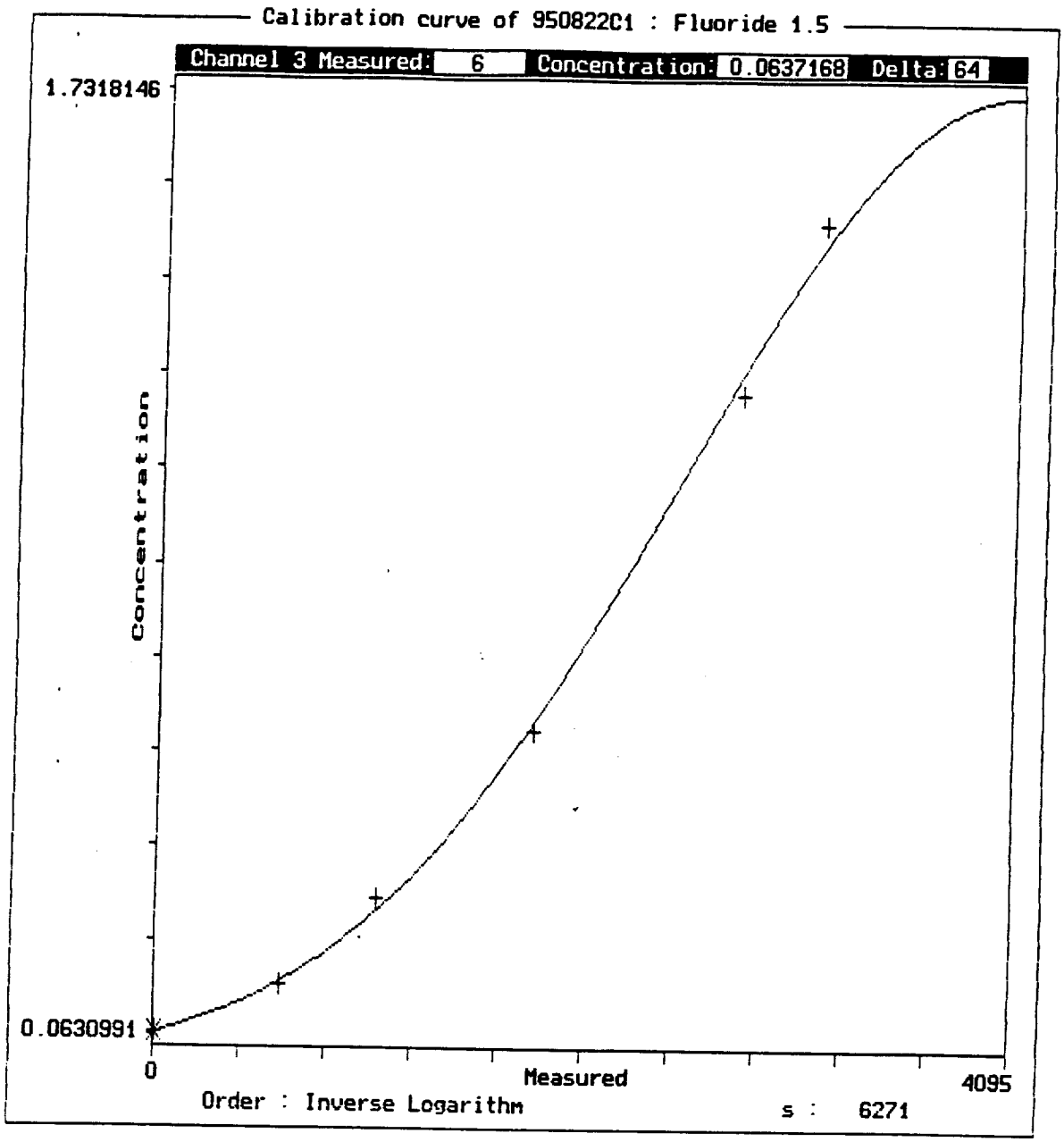
1995-08-22 17:22 OutPut of : 950822C1

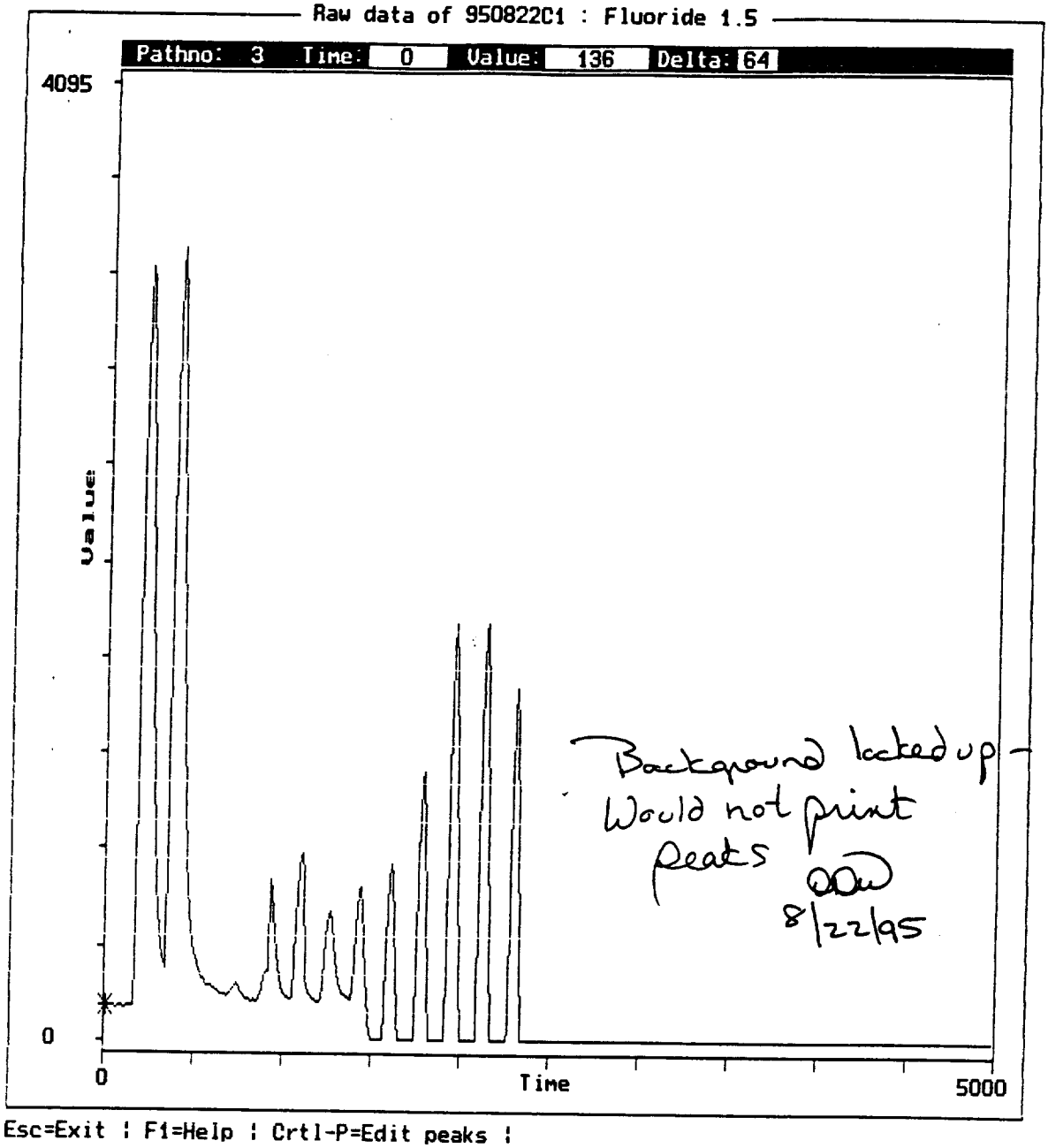
```
s1   sTandard :    0.015
s2   sTandard :    0.030
s3   sTandard :    0.060
s4   sTandard :    0.090
s5   sTandard :    0.120
s6   sTandard :    0.150
s7   sTandard : Ignore
s8   sTandard : Ignore
s9   sTandard : Ignore
s10  sTandard : Ignore
Order : 2
Dimension : PPM
start Value     : 500  DU
trigger Limit   : 1800 Sec
Peak shape      : Pointed
stArt ignore    : 60   Sec
eNd   ignore    : 120  Sec
Measure window  : 75   %
Filter          : No
Regeneration    : No
formUla    : c4:=c3
output     : #.####
```

			Fluoride 1.5			Fluoride L		
			PPM			*PPM*		
Pos	Typ	Ident	Ch	Result	F Time	Ch	Result	F Time
wt	iw	Initial Wash	3	0.063	65	4	0.0082	0
1	t	Tracer	3	1.476	210	4	0.9301	0
2	d	Drift	3	1.488	386	4	0.9386	0
3	w	Wash	3	0.063	619	4	0.0082	0
4	s1	Standard 1	3	0.066	734	4	0.0154	0
5	s2	Standard 2	3	0.073	911	4	0.0282	0
6	s3	Standard 3	3	0.092	1086	4	0.0636	0
7	s4	Standard 4	3	0.108	1261	4	0.0885	0
8	s5	Standard 5	3	0.129	1435	4	0.1183	0
9	s6	Standard 6	3	0.156	1611	4	0.1510	0
10	s7	Standard 7	3	0.281	1785	4	0.2692	0
11	s8	Standard 8	3	0.614	1959	4	0.4788	0
12	s9	Standard 9	3	1.238	2134	4	0.7912	0
13	s10	Standard 10	3	1.462	2308	4	0.9213	0
14	d	Drift	3	1.488	2483	4	0.9386	0
15	w	Wash	3	0.063	2723	4	0.0082	0
16	u	SERUM BLK 1	3	0.086	2831	4	0.0528	0
17	u	SERUM BLK 2	3	0.084	3006	4	0.0493	0
18	u	SPK 62-1	3	0.134	3184	4	0.1240	0
19	u	SPK 62-2	3	0.108	3360	4	0.0885	0
20	u	SPK 62-3	3	0.124	3533	4	0.1120	0
21	u	SPK 250-1	3	0.187	3710	4	0.1854	0
22	u	SPK 250-2	3	0.299	3884	4	0.2835	0
23	u	SPK 250-3	3	0.275	4060	4	0.2644	0
24	u	BLK	3	0.104	4231	4	0.0823	0
25	u	BLK	3	0.087	4408	4	0.0556	0
26	d	Drift	3	1.449	4584	4	0.9125	0
27	w	Wash	3	0.063	4789	4	0.0082	0
28	u	F52711-1	3	0.080	4935	4	0.0418	0
29	u	F52719-1	3	0.076	5111	4	0.0340	0
30	u	F52725-1	3	0.075	5283	4	0.0328	0
31	u	F52721-1	3	0.071	5459	4	0.0257	0
32	u	F52727-1	3	0.071	5633	4	0.0239	0
33	u	F52806-1	3	0.072	5811	4	0.0264	0
34	u	F52711-2	3	0.069	5977	4	0.0209	0
35	u	F52719-2	3	0.067	6155	4	0.0172	0
36	u	F52725-2	3	0.066	6335	4	0.0154	0
37	u	F52721-2	3	0.063	6509	4	0.0088	0
38	d	Drift	3	1.489	6685	4	0.9390	0
39	w	Wash	3	0.063	6926	4	0.0082	0
40	u	SPK 62-1	3	0.124	7033	4	0.1112	0
41	u	SPK 250-1	3	0.313	7213	4	0.2938	0
42	u	BLK	3	0.089	7384	4	0.0581	0
43	u	BLK	3	0.082	7558	4	0.0460	0
44	u	SPK 62-1	3	0.094	7736	4	0.0671	0
45	u	SPK 62-2	3	0.112	7911	4	0.0940	0
46	u	SPK 62-3	3	0.108	8084	4	0.0880	0
47	u	SPK 62-4	3	0.119	8261	4	0.1047	0
48	u	SPK 250-1	3	0.179	8434	4	0.1767	0
49	u	SPK 250-2	3	0.294	8611	4	0.2791	0
50	d	Drift	3	1.454	8785	4	0.9157	0
51	w	Wash	3	0.063	9025	4	0.0082	0
52	u	SPK 250-3	3	0.273	9136	4	0.2630	0
53	u	BLK	3	0.087	9309	4	0.0556	0

			Fluoride 1.5			Fluoride L		
			PPM			*PPM*		
Pos	Typ	Ident	Ch	Result	F Time	Ch	Result	F Time
54	u	BLK	3	0.077	9482	4	0.0367	0
55	u	F52727-2	3	0.074	9658	4	0.0300	0
56	u	F52806-2	3	0.074	9838	4	0.0312	0
57	u	F52713-2	3	0.071	10004	4	0.0245	0
58	u	F52718-2	3	0.069	10184	4	0.0209	0
59	u	F52723-2	3	0.072	10351	4	0.0261	0
60	u	F52715-2	3	0.072	10536	4	0.0271	0
61	u	F52738-2	3	0.069	10711	4	0.0216	0
62	d	Drift	3	1.526	10886	4	0.9653	0
63	w	Wash	3	0.063	11127	4	0.0082	0
64	u	F52809-2	3	0.074	11234	4	0.0305	0
65	u	F52724-2	3	0.072	11413	4	0.0261	0
66	u	F52737-2	3	0.071	11587	4	0.0241	0
67	u	SPK 62-1	3	0.085	11761	4	0.0514	0
68	u	SPK 62-2	3	0.096	11936	4	0.0697	0
69	u	SPK 250-1	3	0.150	12112	4	0.1442	0
70	u	SPK 250-2	3	0.235	12286	4	0.2310	0
71	d	Drift	3	1.512	12462	4	0.9553	0
72	w	Wash	3	0.063	12703	4	0.0082	0
wt	rw	RunOut Wash	3	0.063	12937	4	0.0082	0


Calibration curve of 950822C1 : Fluoride L
Channel 0 Measured: 6
Concentration: 0.0095219
Delta: 64
0.2278068
0.0081653
Concentration
0
Measured
900
Order : 2
r : 0.99910


Calibration curve of 950822C1 : Fluoride 1.5
Channel 3 Measured: 6
Concentration: 0.0637168
Delta: 64
1.7318146
Concentration
0.0630991
0
Measured
4095
Order : Inverse Logarithm
s : 6271



Esc=Exit ¦ F1=Help ¦ Crtl-P=Edit peaks ¦

DDW 8/31/95
AMDT 11095.1
HWI 6329-■
Serum Curve 2
Carlton

1995-08-23 11:00 OutPut of : 950823A1

Software : version 6.1 c1990,93

Operator : DDW

Date of the Analysis : 1995-08-23 06:55

Analysis File Name : C:\SKALAR\DATA\HWIDATA\SERUM\950823A1

Fluoride 1.5
Calibration order = Inverse Logarithm

Slope : s = #.#####

$$\text{Result} = 10^{\left[\frac{x - c1}{s}\right]}$$

x = corrected value of the sample
c1 = corrected value of the concentration 1
s = Slope of the electrode

a2 = -0.00000
a1 = 0.00076
a0 = -1.19531

Fluoride L
Calibration order = 2

Correlation : r = 0.99897

$\text{Result} = a2 * x^2 + a1 * x + a0$

a2 = -0.00000
a1 = 0.00028
a0 = 0.00600

Sampler	Type	: SA1000
	Number	: 1
	Sample Time	: 50 sec.
	Wash Time	: 120 sec.
	Air Time	: 1 sec.
	Take up	: Single
	sPecial	: None
	needle Height	: 70 mm.
Diluter	needle Height	: 80 mm
	dilution Factor	: 10
	dilution Volume	: 2.5 ml.
	Resample	: 1
	Dilution runs	: 1
	User file	: . TXT
	Reproces	: No

1995-08-23 11:00 OutPut of : 950823A1

Fluoride 1.5

Path number	: 3
Signal type	: Debubbled
Decolor	: Yes
system Number	: 0
diLute	: No
Resample	: No
dil Threshold	: 4095
diG output	: 0
Window event	: Off
s1 sTandard	: Ignore
s2 sTandard	: Ignore
s3 sTandard	: Ignore
s4 sTandard	: Ignore
s5 sTandard	: Ignore
s6 sTandard	: 0.150
s7 sTandard	: 0.300
s8 sTandard	: 0.600
s9 sTandard	: 1.200
s10 sTandard	: 1.500
Order	: Inverse Logarithm
Dimension	: PPM
start Value	: 500 DU
trigger Limit	: 1800 Sec
Peak shape	: Pointed
stArt ignore	: 60 Sec
eNd ignore	: 120 Sec
Measure window	: 75 %
Filter	: No
Regeneration	: No
formUla	:
output	: ##.###

Fluoride L

Path number	: 0
Signal type	: Debubbled
Decolor	: No
system Number	: 0
diLute	: No
Resample	: No
dil Threshold	: 4095
diG output	: 0
Window event	: Off

1995-08-23 11:00 OutPut of : 950823A1

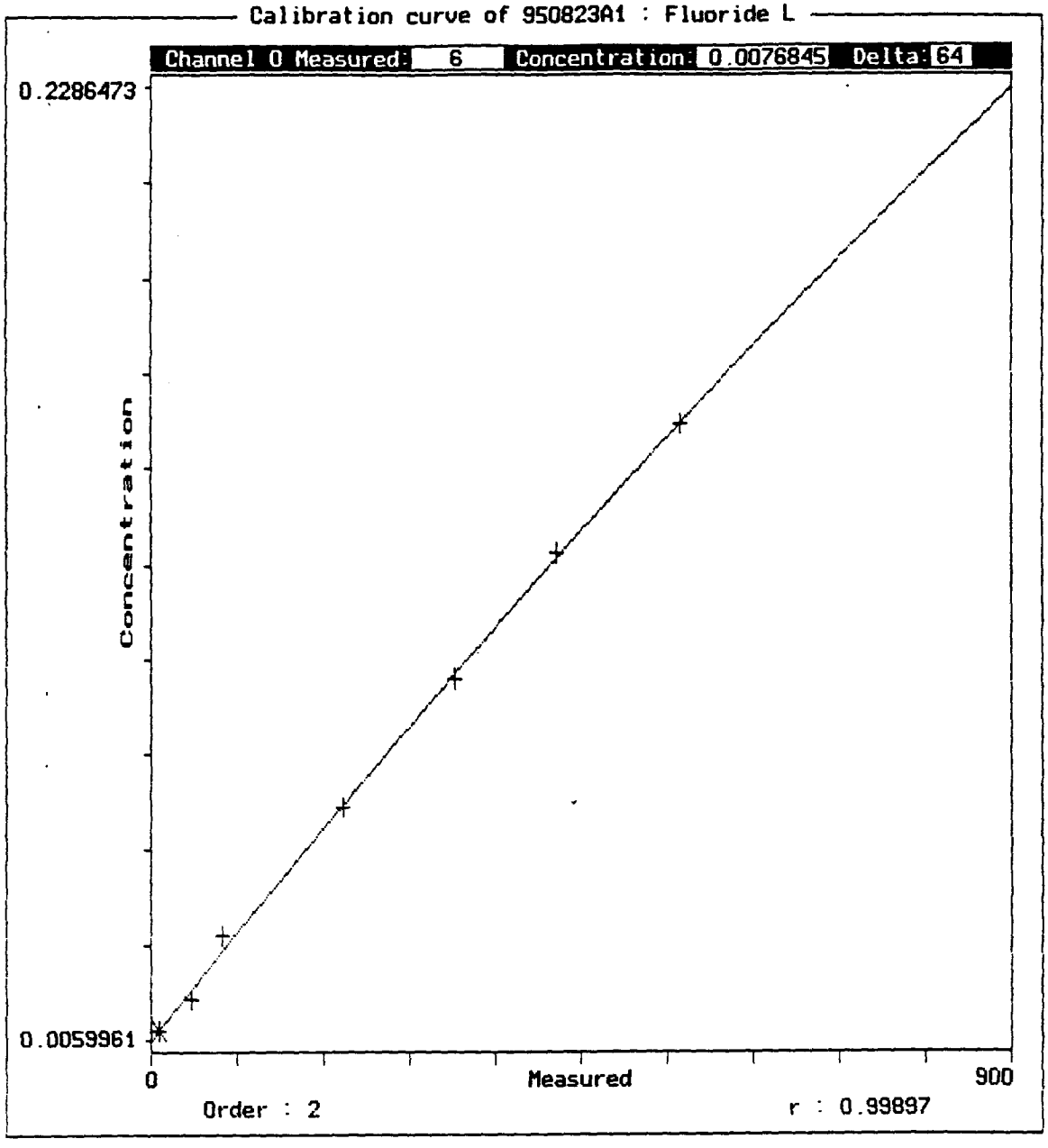
```
 s1   sTandard :    0.015
 s2   sTandard :    0.030
 s3   sTandard :    0.060
 s4   sTandard :    0.090
 s5   sTandard :    0.120
 s6   sTandard :    0.150
 s7   sTandard : Ignore
 s8   sTandard : Ignore
 s9   sTandard : Ignore
 s10  sTandard : Ignore
Order : 2
Dimension : PPM
start Value     : 500  DU
trigger Limit   : 1800 Sec
Peak shape      : Pointed
stArt ignore    : 60   Sec
eNd   ignore    : 120  Sec
Measure window  : 75   %
Filter          : No
Regeneration    : No
formUla    : c4:=c3
output     : #.####
```

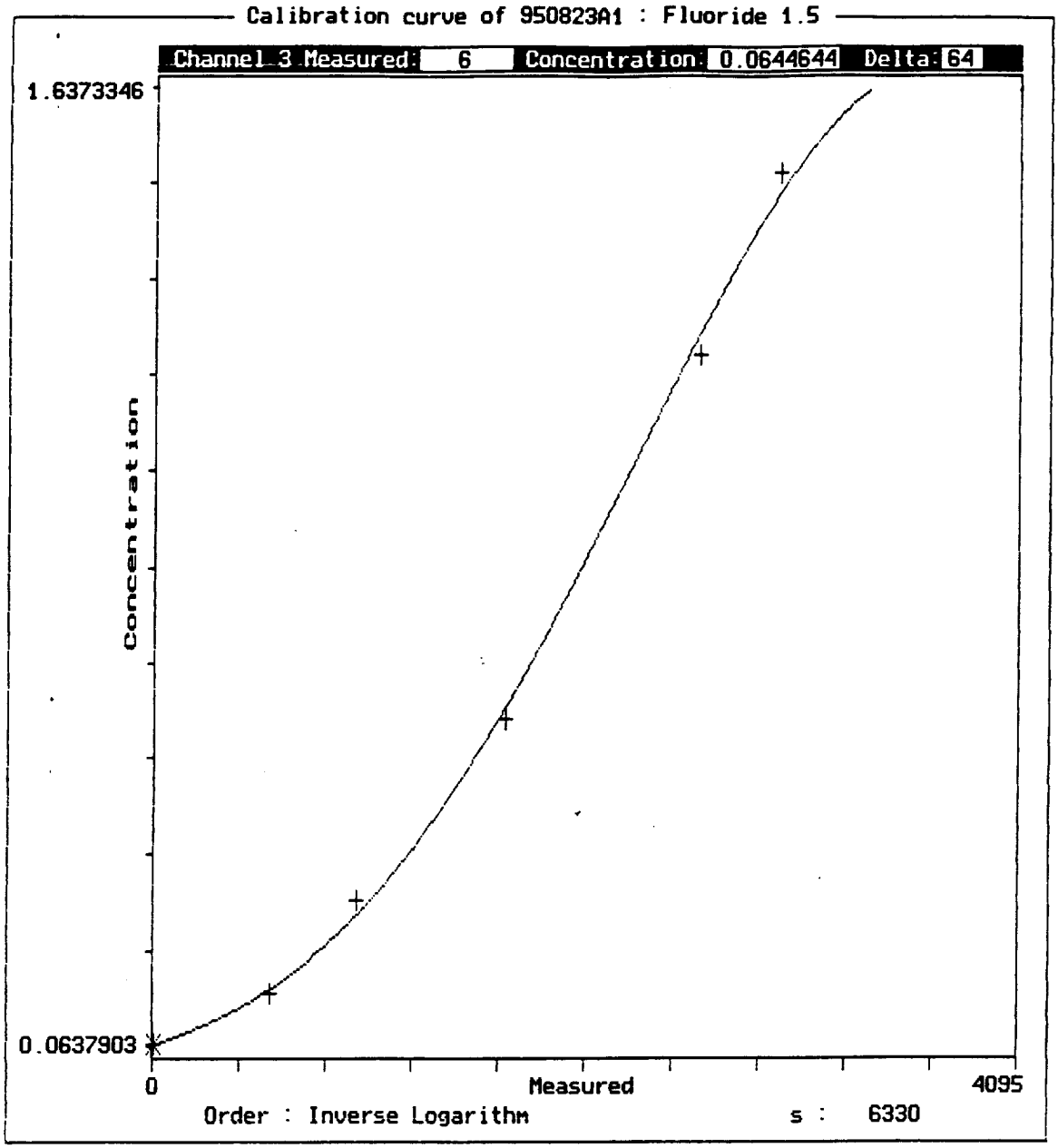
			Fluoride 1.5			Fluoride L			
			PPM			*PPM*			
Pos	Typ	Ident	Ch	Result	F Time	Ch	Result	F Time	
wt	iw	Initial Wash	3	0.064	65	4	0.0060	0	
1	t	Tracer	3	1.452	209	4	0.5049	0	
2	d	Drift	3	1.469	384	4	0.5067	0	
3	w	Wash	3	0.064	615	4	0.0060	0	
4	s1	Standard 1	3	0.069	732	4	0.0175	0	
5	s2	Standard 2	3	0.073	910	4	0.0266	0	
6	s3	Standard 3	3	0.090	1086	4	0.0605	0	
7	s4	Standard 4	3	0.109	1260	4	0.0914	0	
8	s5	Standard 5	3	0.129	1436	4	0.1186	0	
9	s6	Standard 6	3	0.157	1610	4	0.1503	0	
10	s7	Standard 7	3	0.278	1786	4	0.2419	0	
11	s8	Standard 8	3	0.618	1960	4	0.3698	0	
12	s9	Standard 9	3	1.233	2134	4	0.4794	0	
13	s10	Standard 10	3	1.466	2310	4	0.5064	0	
14	d	Drift	3	1.510	2485	4	0.5109	0	
15	w	Wash	3	0.064	2724	4	0.0060	0	
16	u	SERUM BLK	3	0.088	2833	4	0.0576	0	
17	u	SERUM BLK	3	0.082	3010	4	0.0468	0	
18	u	SPK 62-1	3	0.098	3186	4	0.0753	0	
19	u	SPK 62-2	3	0.115	3361	4	0.1004	0	
20	u	SPK 62-3	3	0.115	3533	4	0.1012	0	
21	u	SPK 250-1	3	0.144	3711	4	0.1371	0	
22	u	SPK 250-2	3	0.208	3887	4	0.1954	0	
23	u	SPK 250-3	3	0.225	4063	4	0.2081	0	
24	u	SPK 250-4	3	0.227	4237	4	0.2094	0	
25	u	SPK 250-5	3	0.259	4412	4	0.2308	0	
26	d	Drift	3	1.505	4587	4	0.5104	0	
27	w	Wash	3	0.064	4819	4	0.0060	0	
28	u	SPK 250-6	3	0.387	4937	4	0.2952	0	
29	u	BLK	3	0.092	5112	4	0.0651	0	
30	u	BLK	3	0.075	5284	4	0.0321	0	
31	u	F52801-2	3	0.082	5462	4	0.0458	0	
32	u	F52720-2	3	0.077	5636	4	0.0365	0	
33	u	F52733-2	3	0.075	5812	4	0.0321	0	
34	u	F52788-2	3	0.077	5986	4	0.0371	0	
35	u	F52730-2	3	0.074	6162	4	0.0294	0	
36	u	F52731-2	3	0.062	6338	4	0.0015	0	
37	u	F52802-2	3	0.076	6513	4	0.0341	0	
38	d	Drift	3	1.506	6688	4	0.5106	0	
39	w	Wash	3	0.064	6930	4	0.0060	0	
40	u	F52714-2	3	0.080	7037	4	0.0425	0	
41	u	F52734-2	3	0.076	7215	4	0.0346	0	
42	u	F52774-2	3	0.077	7391	4	0.0362	0	
43	u	SPK 62-1	3	0.112	7565	4	0.0961	0	
44	u	SPK 250-1	3	0.153	7740	4	0.1460	0	
45	u	SPK 250-2	3	0.173	7915	4	0.1657	0	low vol
46	u	BLK	3	0.085	8089	4	0.0517	0	
47	u	BLK	3	0.085	8264	4	0.0517	0	
48	u	SPK 62-1	3	0.085	8438	4	0.0517	0	
49	u	SPK 62-2	3	0.102	8616	4	0.0821	0	
50	d	Drift	3	1.529	8790	4	0.5129	0	
51	w	Wash	3	0.064	9025	4	0.0060	0	
52	u	SPK 62-3	3	0.132	9140	4	0.1230	0	
53	u	SPK 250-1	3	0.136	9314	4	0.1277	0	

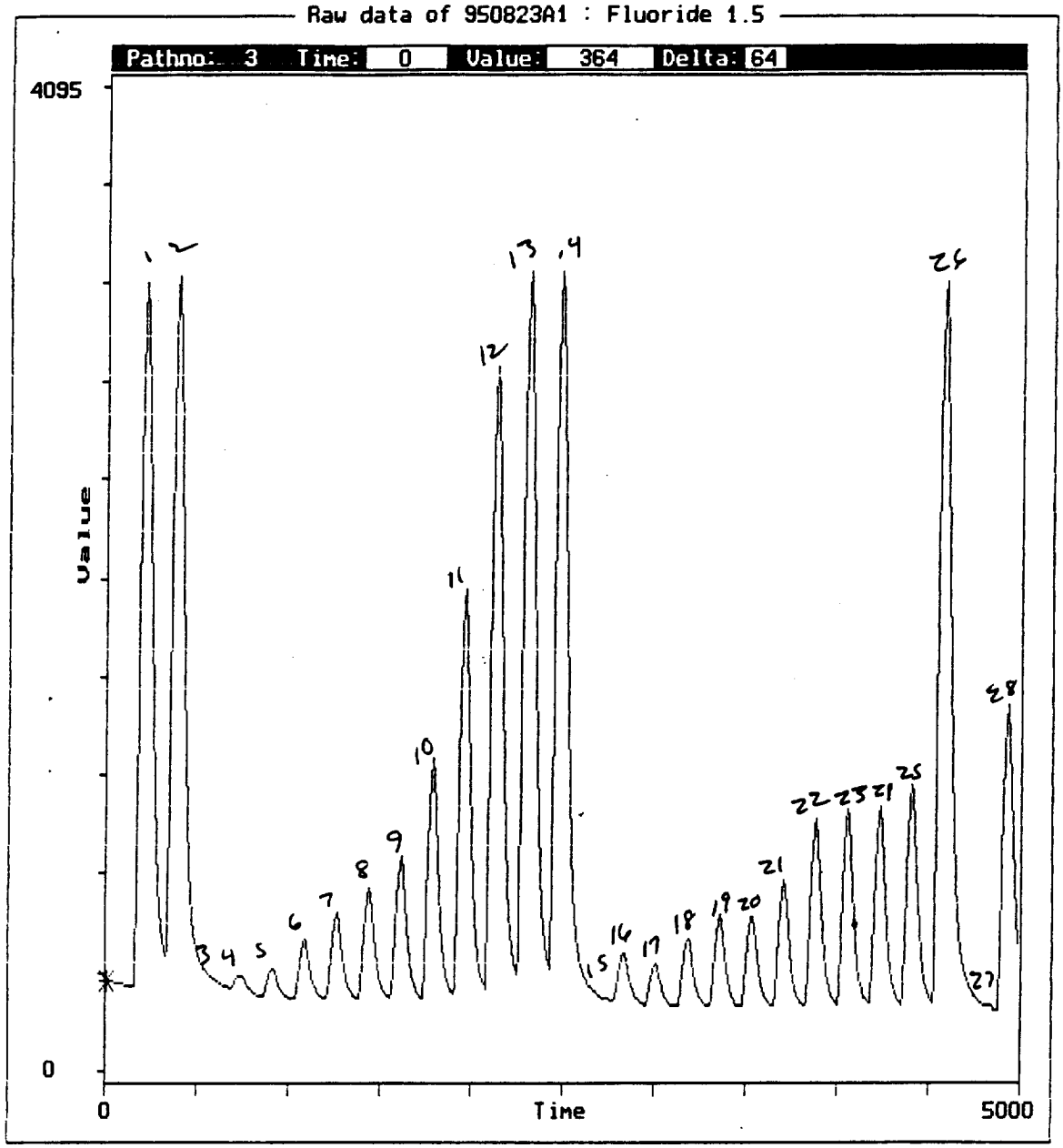
000147

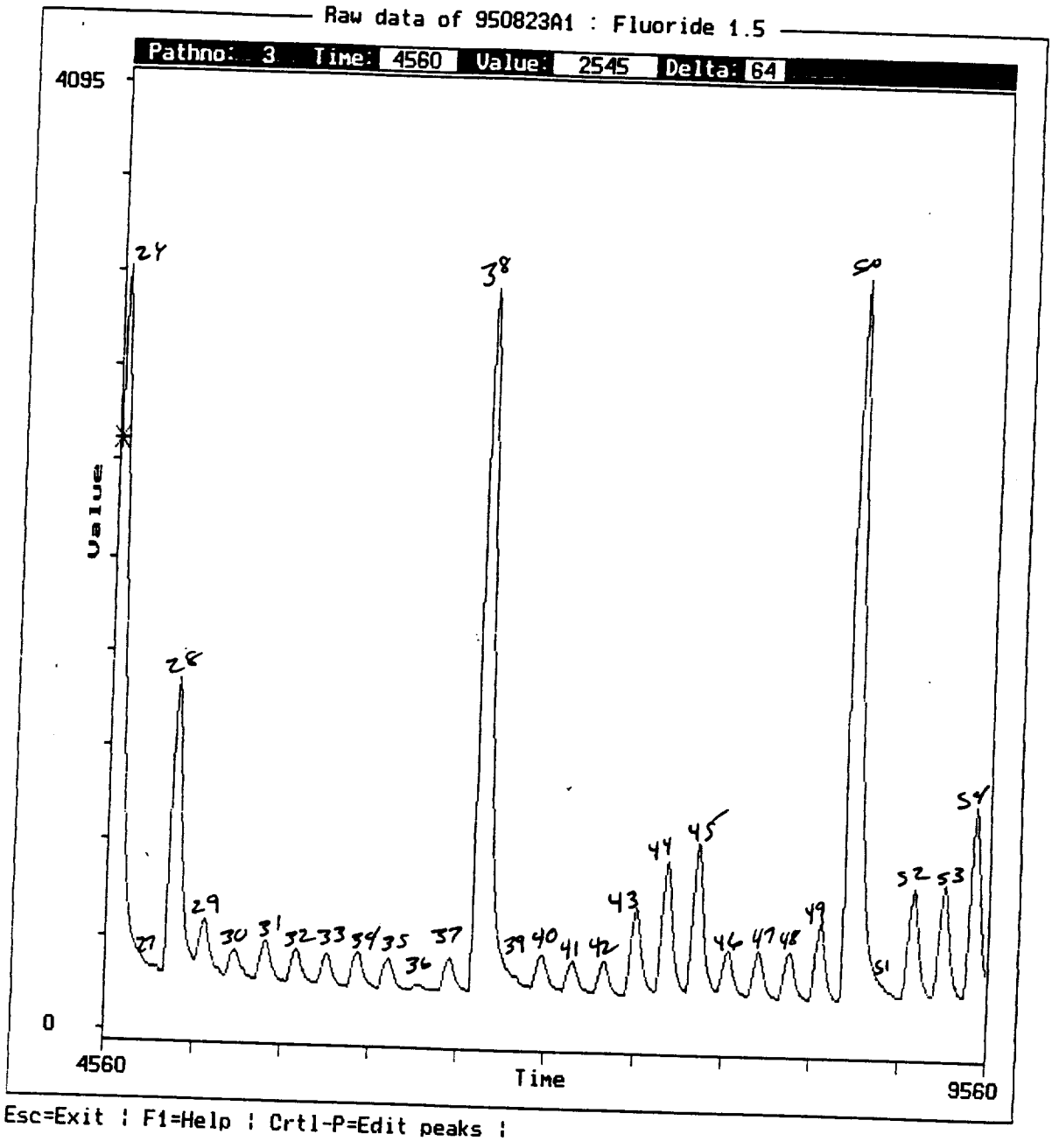
45

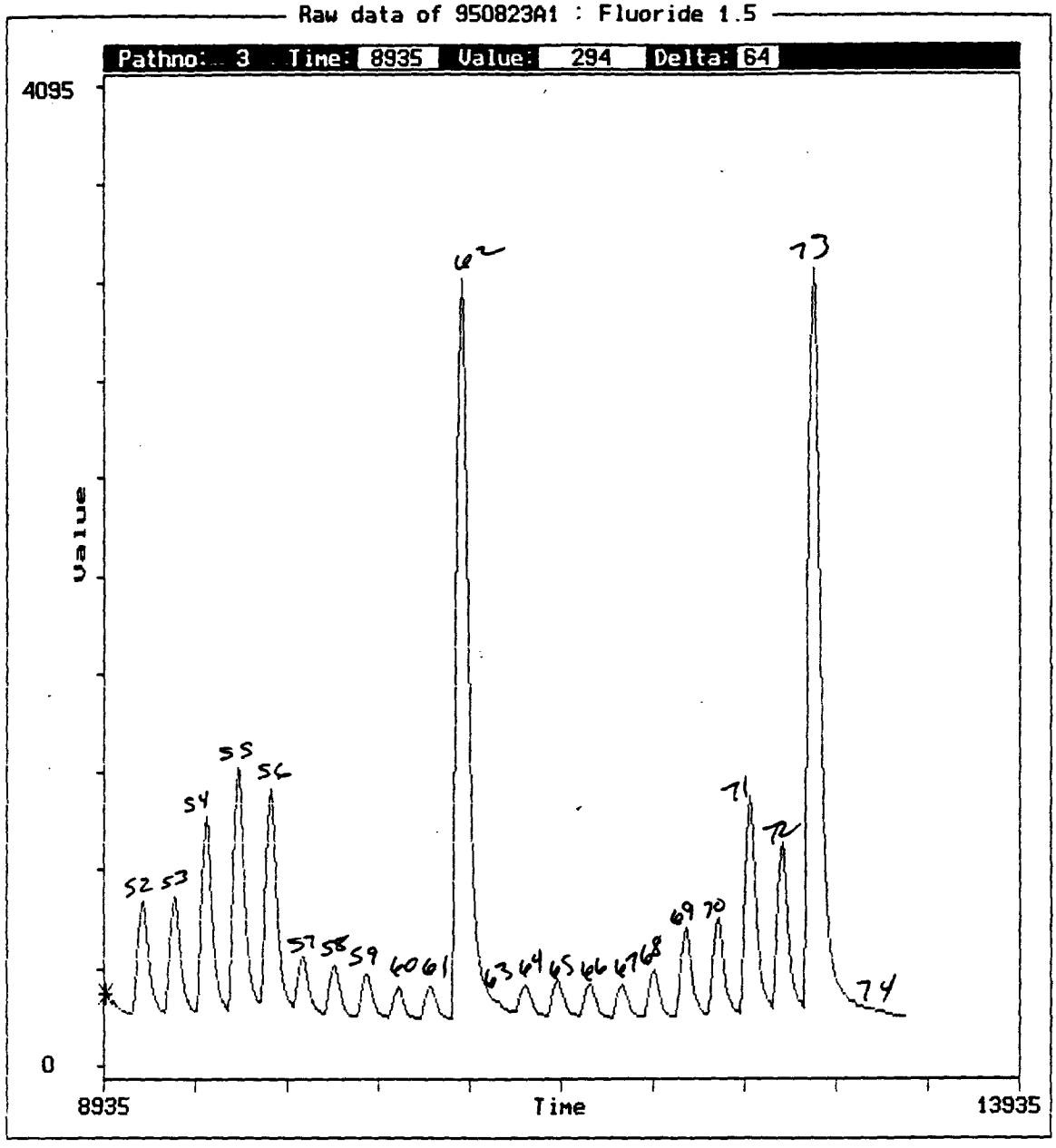
			Fluoride 1.5			Fluoride L		
			PPM			*PPM*		
Pos	Typ	Ident	Ch	Result	F Time	Ch	Result	F Time
54	u	SPK 250-2	3	0.218	9490	4	0.2034	0
55	u	SPK 250-3	3	0.291	9666	4	0.2496	0
56	u	SPK 250-4	3	0.253	9839	4	0.2269	0
57	u	BLK	3	0.092	10014	4	0.0651	0
58	u	BLK	3	0.087	10189	4	0.0563	0
59	u	F52712-2	3	0.083	10365	4	0.0477	0
60	u	F52729-2	3	0.075	10535	4	0.0310	0
61	u	F52803-2	3	0.075	10715	4	0.0316	0
62	d	Drift	3	1.522	10891	4	0.5121	0
63	w	Wash	3	0.064	11131	4	0.0060	0
64	u	F52716-2	3	0.075	11238	4	0.0324	0
65	u	F52728-2	3	0.078	11413	4	0.0384	0
66	u	F52787-2	3	0.076	11592	4	0.0341	0
67	u	F52717-2	3	0.075	11764	4	0.0310	0
68	u	SPK 62-1	3	0.083	11941	4	0.0487	0
69	u	SPK 62-2	3	0.109	12117	4	0.0914	0
70	u	SPK 250-1	3	0.117	12292	4	0.1030	0
71	u	SPK 250-2	3	0.238	12468	4	0.2170	0
72	u	SPK 250-3	3	0.182	12645	4	0.1746	0
73	d	Drift	3	1.537	12817	4	0.5137	0
74	w	Wash	3	0.064	13048	4	0.0060	0
wt	rw	RunOut Wash	3	0.064	13292	4	0.0060	0


Calibration curve of 950823A1 : Fluoride L
Channel 0 Measured: 6
Concentration: 0.0076845
Delta: 64
0.2286473
0.0059961
Concentration
0
Measured
900
Order : 2
r : 0.99897


Calibration curve of 950823A1 : Fluoride 1.5
Channel 3 Measured: 6 Concentration: 0.0644644 Delta: 64
1.6373346
0.0637903
Concentration
0
Measured
4095
Order : Inverse Logarithm
s : 6330


Raw data of 950823A1 : Fluoride 1.5
Pathno: 3 Time: 0 Value: 364 Delta: 64
4095
0
Value
0
Time
5000
Esc=Exit ; F1=Help ; Crtl-P=Edit peaks ;


Raw data of 950823A1 : Fluoride 1.5
Pathno: 3 Time: 4560 Value: 2545 Delta: 64
4095
0
Value
4560
Time
9560
24
25
27
28
29
30
31
32
33
34
35
36
37
38
39
40
41
42
43
44
45
46
47
48
49
50
51
52
53
54
Esc=Exit ¦ F1=Help ¦ Crtl-P=Edit peaks ¦


Raw data of 950823A1 : Fluoride 1.5
Pathno: 3 Time: 8935 Value: 294 Delta: 64
4095
0
Value
8935
Time
13935
52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74
Esc=Exit ¦ F1=Help ¦ Crtl-P=Edit peaks ¦

DDW 8/31/95
AMDT 11095.1
HWI 6329-152
Serum Curve2
Carlton

1995-08-23 15:23 OutPut of : 950823B1

Software : version 6.1 c1990,93

Operator : DDW

Date of the Analysis : 1995-08-23 10:50

Analysis File Name : C:\SKALAR\DATA\HWIDATA\SERUM\950823B1

Fluoride 1.5
Calibration order = Inverse Logarithm

Slope : s = #.#####

$$\text{Result} = 10^{\left[\frac{x - c1}{s}\right]}$$

x = corrected value of the sample
c1 = corrected value of the concentration 1
s = Slope of the electrode

a2 = -0.00000
a1 = 0.00070
a0 = -1.18413

Fluoride L
Calibration order = 2

Correlation : r = 0.99924

$\text{Result} = a2 * x^2 + a1 * x + a0$

a2 = -0.00000
a1 = 0.00027
a0 = 0.00378

Sampler	Type	: SA1000
	Number	: 1
	Sample Time	: 50 sec.
	Wash Time	: 120 sec.
	Air Time	: 1 sec.
	Take up	: Single
	sPecial	: None
	needle Height	: 70 mm.
Diluter	needle Height	: 80 mm
	dilution Factor	: 10
	dilution Volume	: 2.5 ml.
	Resample	: 1
	Dilution runs	: 1
	User file	: . TXT
	Reproces	: No

1995-08-23 15:23 OutPut of : 950823B1

Fluoride 1.5

Path number	: 3
Signal type	: Debubbled
Decolor	: Yes
system Number	: 0
diLute	: No
Resample	: No
dil Threshold	: 4095
diG output	: 0
Window event	: Off
s1 sTandard	: Ignore
s2 sTandard	: Ignore
s3 sTandard	: Ignore
s4 sTandard	: Ignore
s5 sTandard	: Ignore
s6 sTandard	: 0.150
s7 sTandard	: 0.300
s8 sTandard	: 0.600
s9 sTandard	: 1.200
s10 sTandard	: 1.500
Order	: Inverse Logarithm
Dimension	: PPM
start Value	: 500 DU
trigger Limit	: 1800 Sec
Peak shape	: Pointed
stArt ignore	: 60 Sec
eNd ignore	: 120 Sec
Measure window	: 75 %
Filter	: No
Regeneration	: No
formUla	:
output	: ##.###

Fluoride L

Path number	: 0
Signal type	: Debubbled
Decolor	: No
system Number	: 0
diLute	: No
Resample	: No
dil Threshold	: 4095
diG output	: 0
Window event	: Off

1995-08-23 15:23 OutPut of : 950823B1

```
s1   sTandard :    0.015
s2   sTandard :    0.030
s3   sTandard :    0.060
s4   sTandard :    0.090
s5   sTandard :    0.120
s6   sTandard :    0.150
s7   sTandard : Ignore
s8   sTandard : Ignore
s9   sTandard : Ignore
s10  sTandard : Ignore
Order : 2
Dimension : PPM
start Value     : 500  DU
trigger Limit   : 1800 Sec
Peak shape      : Pointed
stArt ignore    : 60   Sec
eNd   ignore    : 120  Sec
Measure window : 75   %
Filter          : No
Regeneration    : No
formUla    : c4:=c3
output     : #.####
```

Pos	Typ	Ident	Fluoride 1.5 PPM Ch	Result	F	Time	Fluoride L PPM Ch	Result	F	Time	
wt	iw	Initial Wash	3	0.065		65	4	0.0038		0	
1	t	Tracer	3	1.476		212	4	0.5182		0	
2	d	Drift	3	1.484		386	4	0.5190		0	
3	w	Wash	3	0.065		627	4	0.0038		0	
4	s1	Standard 1	3	0.070		730	4	0.0159		0	
5	s2	Standard 2	3	0.076		911	4	0.0284		0	
6	s3	Standard 3	3	0.092		1087	4	0.0604		0	
7	s4	Standard 4	3	0.111		1263	4	0.0921		0	
8	s5	Standard 5	3	0.129		1439	4	0.1175		0	
9	s6	Standard 6	3	0.157		1614	4	0.1507		0	
10	s7	Standard 7	3	0.277		1786	4	0.2461		0	
11	s8	Standard 8	3	0.621		1960	4	0.3798		0	
12	s9	Standard 9	3	1.225		2136	4	0.4895		0	
13	s10	Standard 10	3	1.470		2312	4	0.5177		0	
14	d	Drift	3	1.489		2487	4	0.5196		0	
15	w	Wash	3	0.065		2727	4	0.0038		0	
16	u	SERUM BLK	3	0.087		2840	4	0.0519		0	
17	u	SERUM BLK	3	0.084		3012	4	0.0467		0	
18	u	SERUM BLK	3	0.078		3189	4	0.0334		0	
19	u	SPK 62-1	3	0.087		3363	4	0.0514		0	
20	u	SPK 62-2	3	0.119		3538	4	0.1042		0	
21	u	SPK 62-3	3	0.121		3714	4	0.1066		0	
22	u	SPK 250-1	3	0.114		3888	4	0.0971		0	
23	u	SPK 250-2	3	0.233		4065	4	0.2171		0	
24	u	SPK 250-3	3	0.208		4239	4	0.1981		0	
25	u	SPK 124-1	3	0.204		4415	4	0.1951		0	
26	d	Drift	3	1.468		4589	4	0.5174		0	
27	w	Wash	3	0.065		4825	4	0.0038		0	
28	u	SPK 124-2	3	0.206		4940	4	0.1966		0	
29	u	BLK	3	0.136		5116	4	0.1266		0	
30	u	BLK	3	0.090		5288	4	0.0583		0	
31	u	F52726-2	3	0.082		5464	4	0.0413		0	
32	u	F52735-2	3	0.080		5638	4	0.0368		0	
33	u	F52732-2	3	0.079		5817	4	0.0347		0	
34	u	F52736-2	3	0.076		5994	4	0.0287		0	
35	u	F52739-2	3	0.076		6163	4	0.0289		0	
36	u	F52711-4	3	0.075		6336	4	0.0263		0	
37	u	F52721-4	3	0.074		6503	4	0.0236		0	
38	d	Drift	3	1.504		6691	4	0.5211		0	
39	w	Wash	3	0.065		6933	4	0.0038		0	
40	u	F52719-4	3	0.255		7055	4	0.2321		0	low val. – air spike ~0.06 peak ht low
41	u	F52727-4	3	0.079		7217	4	0.0350		0	
42	u	F52725-4	3	0.077		7393	4	0.0302		0	
43	u	F52723-4	3	0.093		7566	4	0.0624		0	
44	u	SPK 124-1	3	0.165		7742	4	0.1594		0	
45	u	BLK	3	0.083		7917	4	0.0444		0	
46	u	BLK	3	0.082		8092	4	0.0418		0	
47	u	SPK 62-1	3	0.090		8267	4	0.0568		0	
48	u	SPK 62-2	3	0.105		8438	4	0.0837		0	
49	u	SPK 62-3	3	0.118		8618	4	0.1032		0	
50	d	Drift	3	1.483		8792	4	0.5190		0	
51	w	Wash	3	0.065		9029	4	0.0038		0	
52	u	SPK 250-1	3	0.171		9142	4	0.1649		0	
53	u	SPK 250-2	3	0.248		9317	4	0.2273		0	

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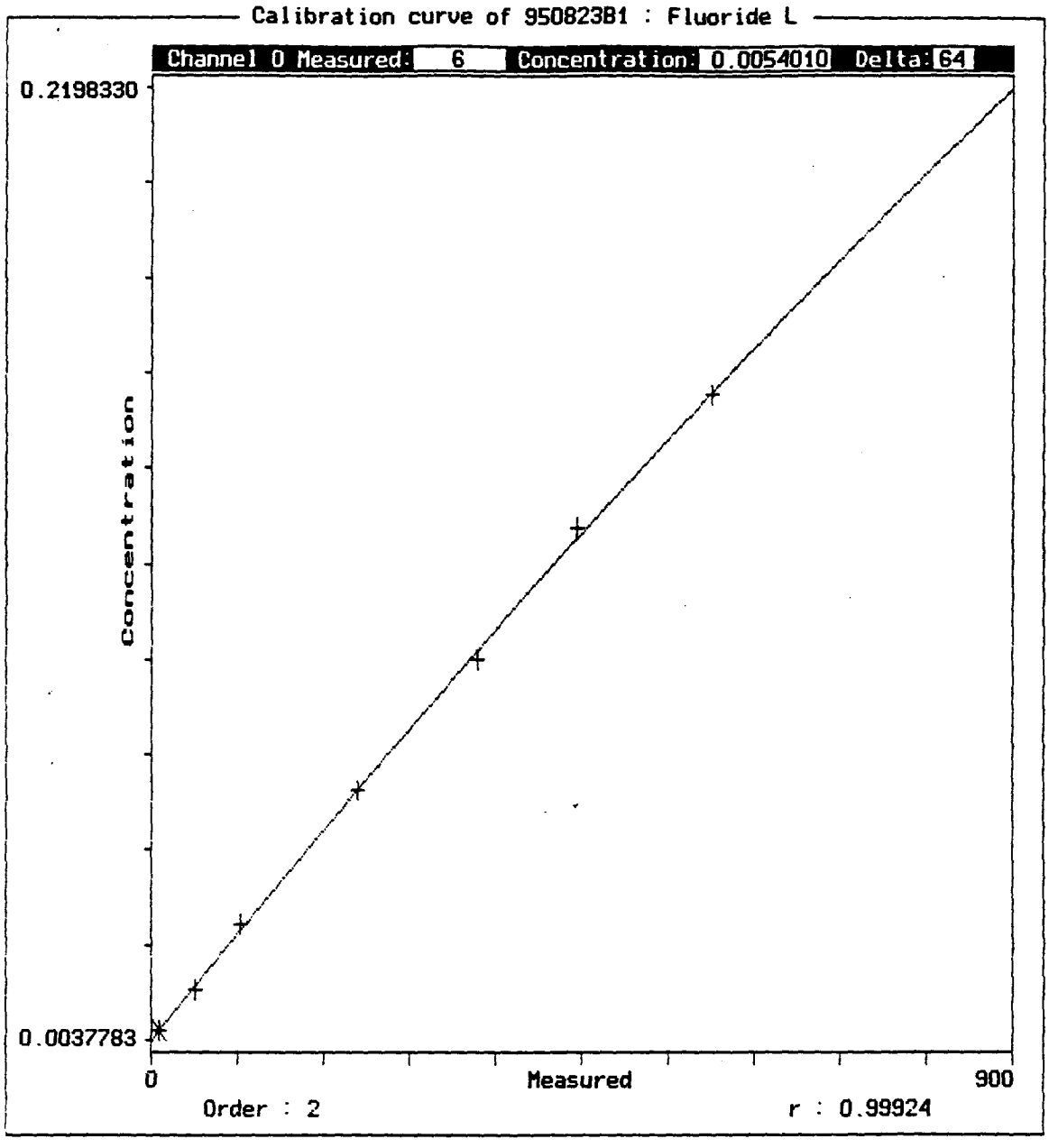
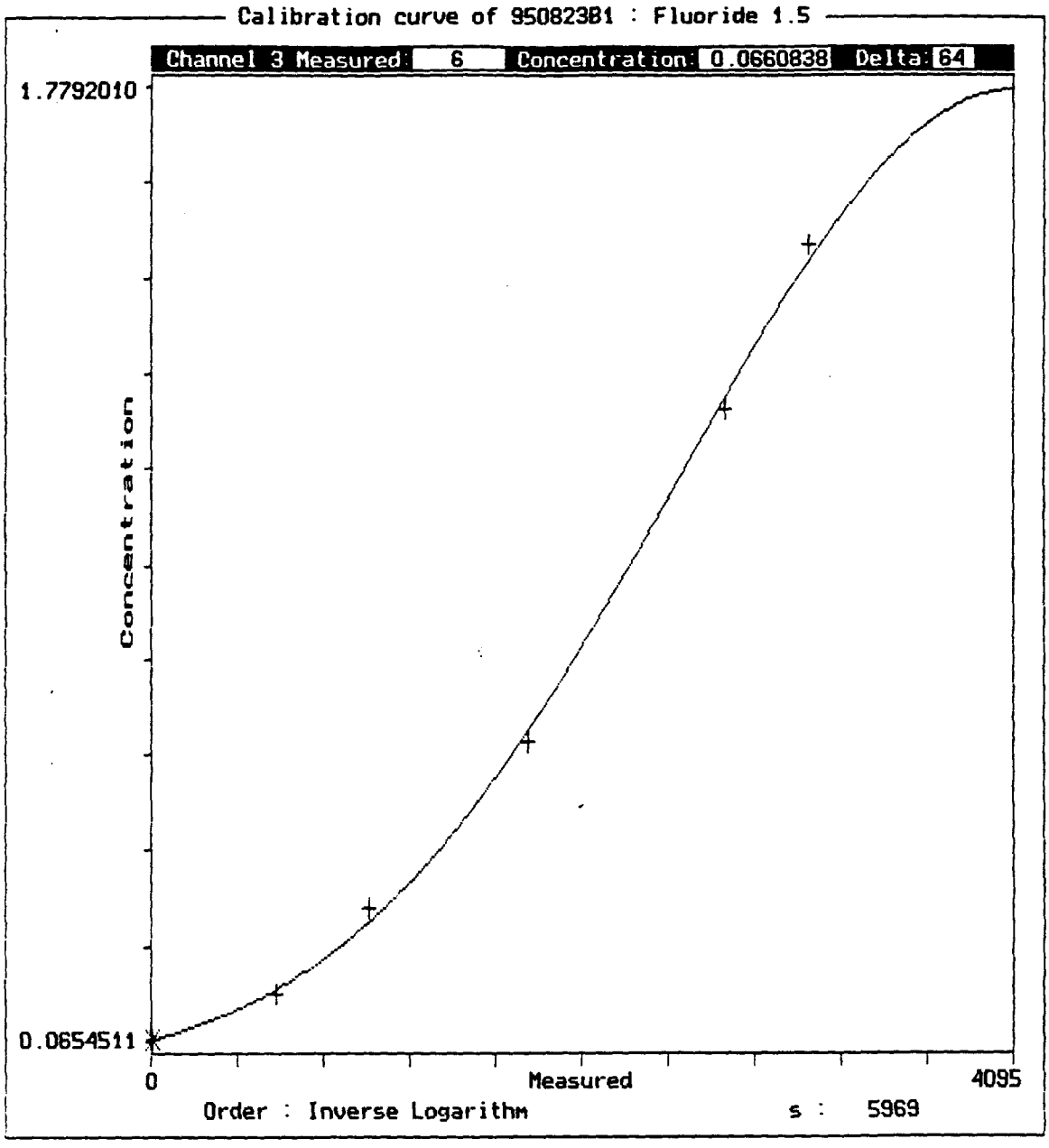
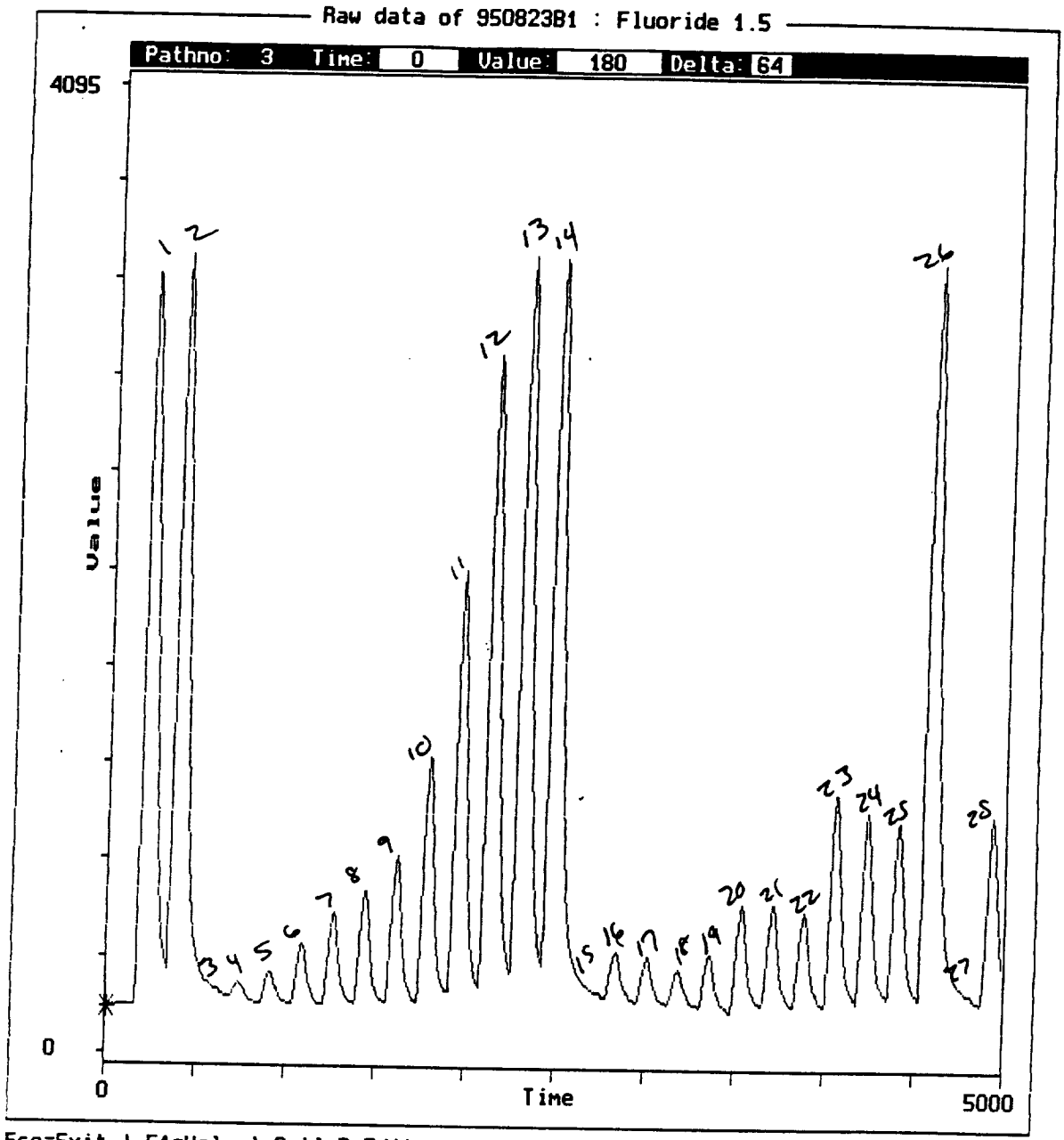
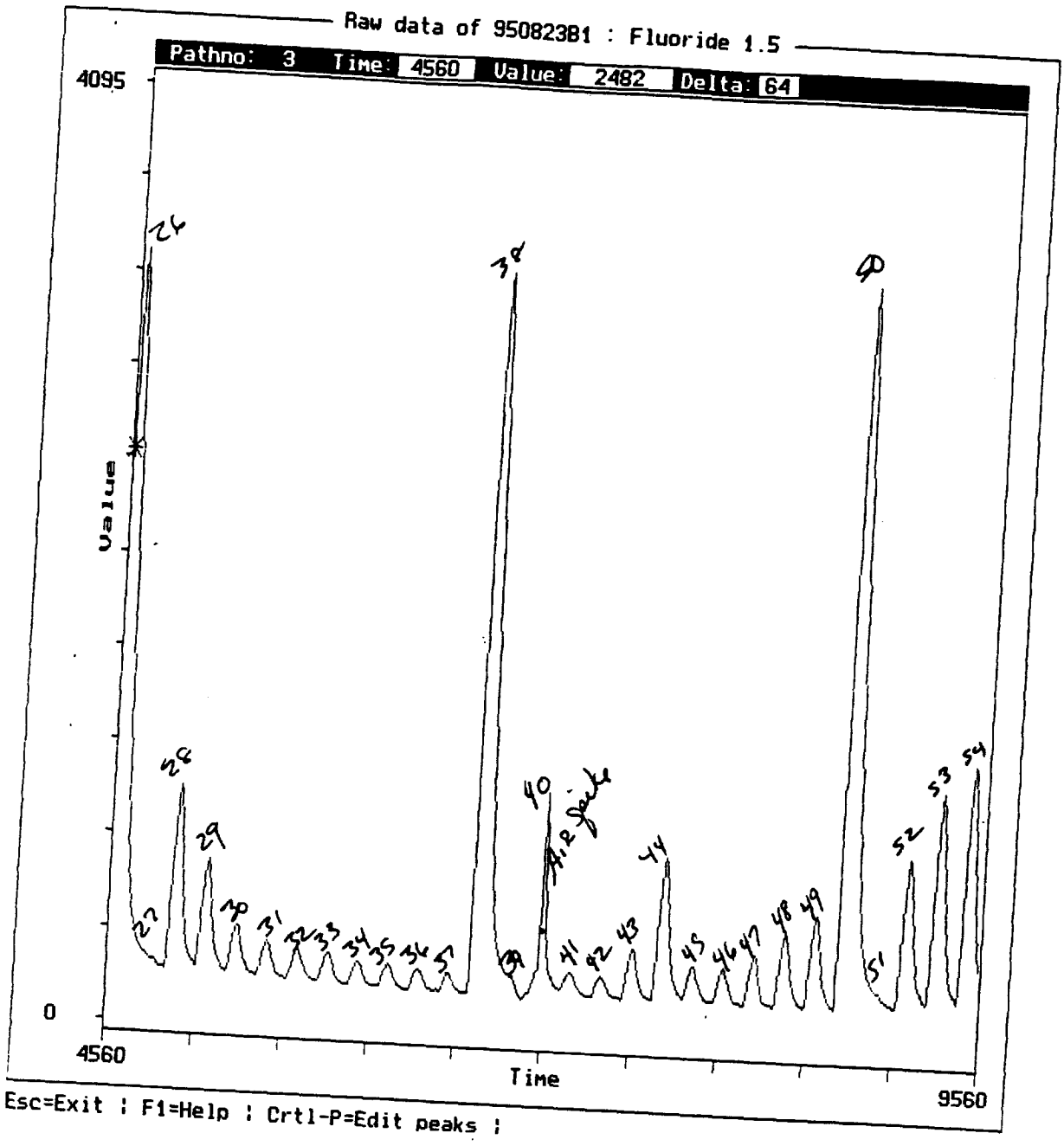
			Fluoride 1.5			Fluoride L		
			PPM			*PPM*		
Pos	Typ	Ident	Ch	Result	F Time	Ch	Result	F Time
54	u	SPK 250-3	3	0.294	9494	4	0.2558	0
55	u	BLK	3	0.124	9669	4	0.1115	0
56	u	BLK	3	0.102	9841	4	0.0787	0
57	u	BLK	3	0.074	10017	4	0.0239	0
58	u	F52726-2	3	0.080	10193	4	0.0373	0
59	u	F52732-2	3	0.070	10367	4	0.0140	0
60	u	F52739-2	3	0.068	10543	4	0.0100	0
61	u	F52735-2	3	0.073	10719	4	0.0215	0
62	d	Drift	3	1.447	10891	4	0.5152	0
63	w	Wash	3	0.065	11082	4	0.0038	0
64	u	F52736-2	3	0.076	11245	4	0.0287	0
65	u	F52711-4	3	0.063	11433	4	#.####	0
66	u	BLK	3	0.125	11594	4	0.1127	0
67	u	BLK	3	0.182	11769	4	0.1761	0
68	u	BLK	3	0.081	11942	4	0.0397	0
69	u	BLK	3	0.073	12123	4	0.0212	0
70	u	SPK 62-1	3	0.102	12289	4	0.0792	0
71	u	SPK 62-2	3	0.105	12469	4	0.0827	0
72	u	SPK 250-1	3	0.174	12645	4	0.1685	0
73	u	SPK 250-2	3	0.178	12819	4	0.1717	0
74	d	Drift	3	1.494	12993	4	0.5200	0
75	w	Wash	3	0.065	13222	4	0.0038	0
76	u	SPK 250-3	3	0.205	13343	4	0.1953	0
77	u	SPK 124-1	3	0.166	13519	4	0.1599	0
78	u	SPK 124-2	3	0.160	13693	4	0.1539	0
79	u	BLK	3	0.105	13869	4	0.0839	0
80	u	BLK	3	0.096	14044	4	0.0688	0
81	u	F52711-4	3	0.084	14214	4	0.0449	0
82	u	F52719-4	3	0.079	14396	4	0.0366	0
83	u	SPK 124-1	3	0.139	14567	4	0.1311	0
84	d	Drift	3	1.471	14743	4	0.5177	0
85	w	Wash	3	0.065	14966	4	0.0038	0
wt	rw	RunOut Wash	3	0.065	15218	4	0.0038	0

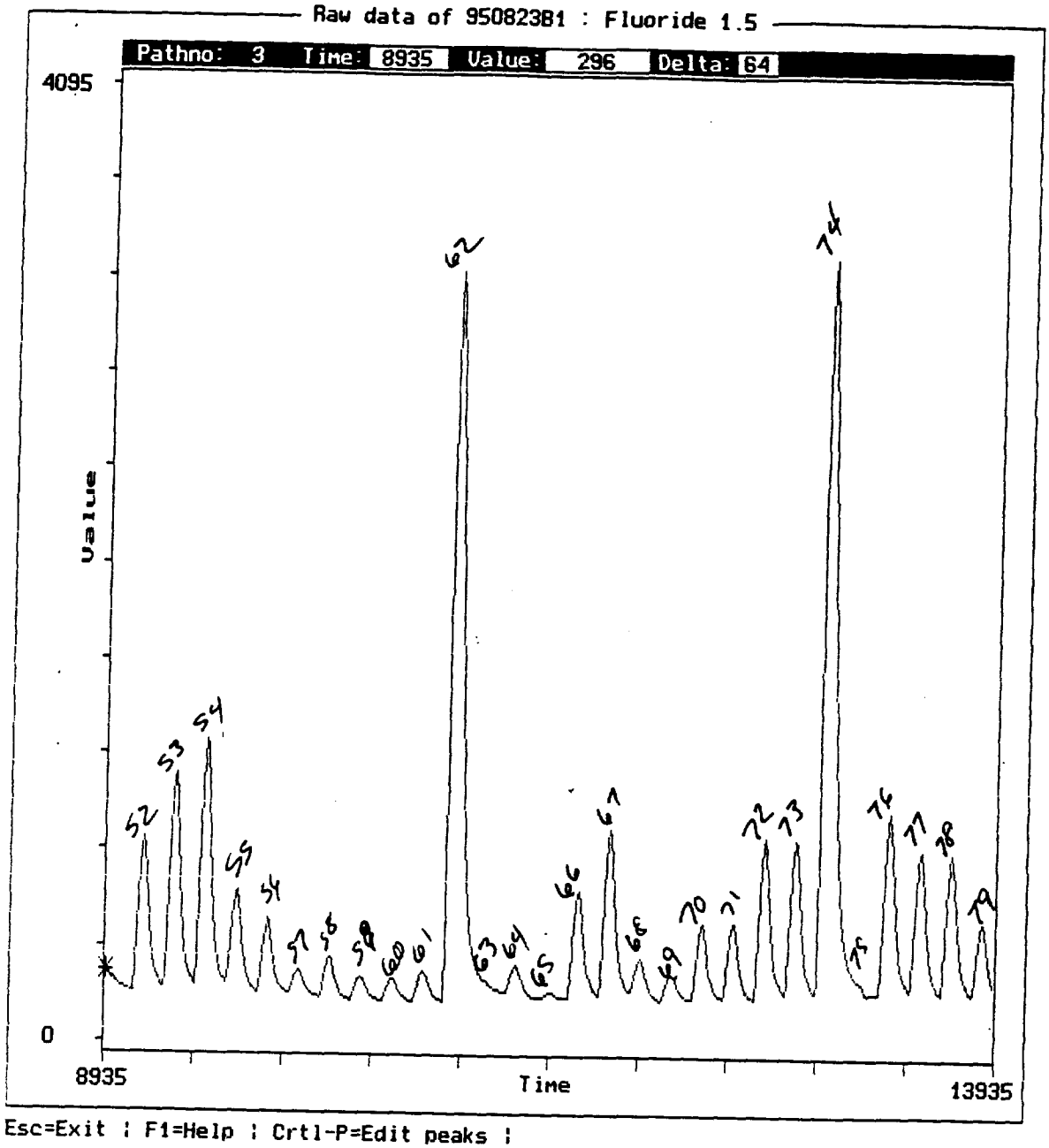
SC

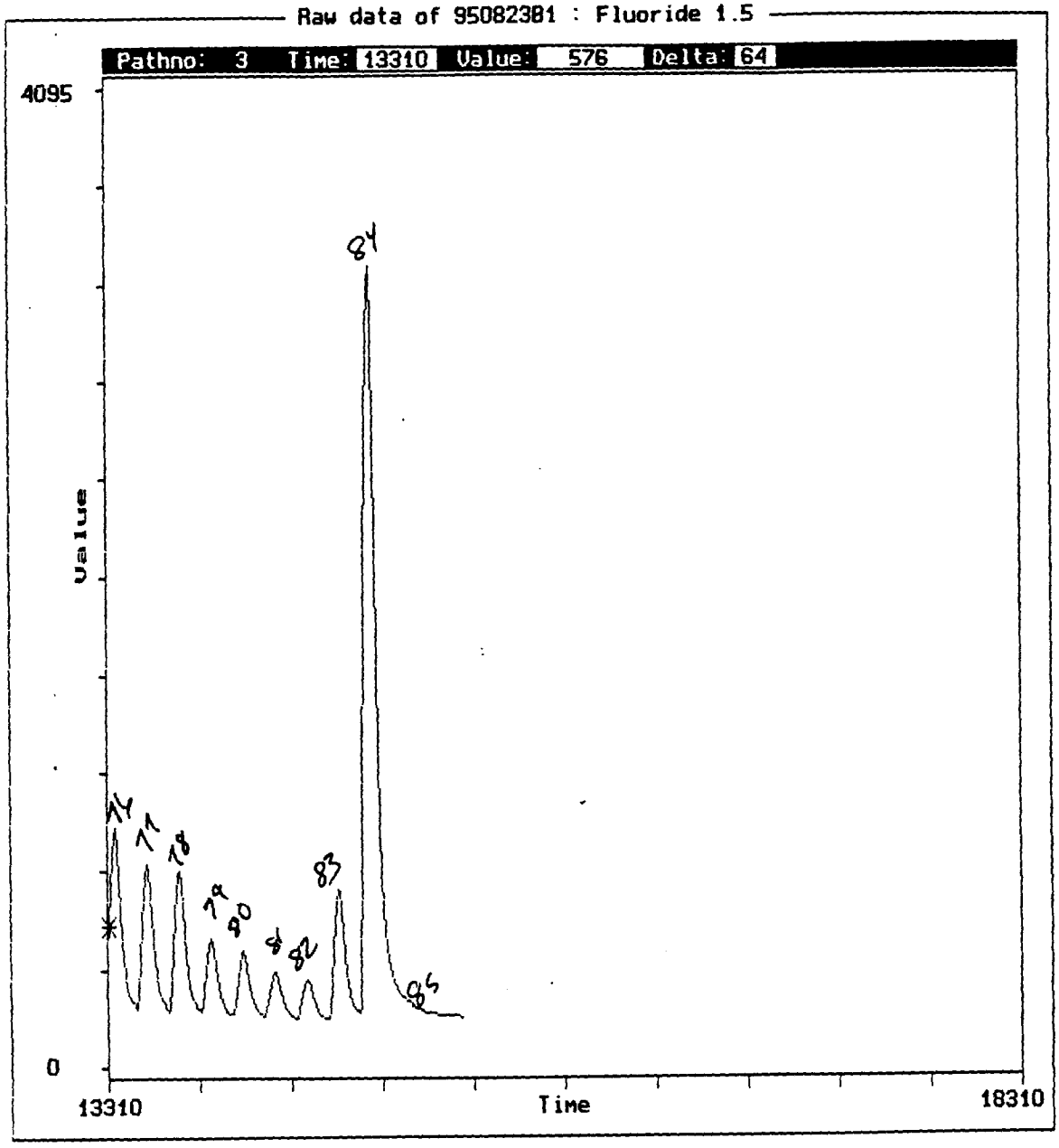

Calibration curve of 950823B1 : Fluoride L
Channel 0 Measured: 6 Concentration: 0.0054010 Delta: 64
0.2198330
0.0037783
Concentration
0
Measured
900
Order : 2
r : 0.99924


Calibration curve of 95082381 : Fluoride 1.5
Channel 3 Measured: 6
Concentration: 0.0660838
Delta: 64
1.7792010
0.0654511
Concentration
0
Measured
4095
Order : Inverse Logarithm
s : 5969


Raw data of 950823B1 : Fluoride 1.5
Pathno: 3 Time: 0 Value: 180 Delta: 64
4095
Value
0
0
Time
5000
Esc=Exit ¦ F1=Help ¦ Crtl-P=Edit peaks ¦


Raw data of 950823B1 : Fluoride 1.5
Pathno: 3 Time: 4560 Value: 2482 Delta: 64
4095
Value
0
4560
Time
9560
Esc=Exit ; F1=Help ; Crtl-P=Edit peaks ;


Raw data of 95082381 : Fluoride 1.5
Pathno: 3 Time: 8935 Value: 296 Delta: 64
4095
0
Value
8935
Time
13935
Esc=Exit ¦ F1=Help ¦ Crtl-P=Edit peaks ¦


Raw data of 95082381 : Fluoride 1.5
Pathno: 3 Time: 13310 Value: 576 Delta: 64
4095
Value
0
13310
Time
18310
84
77
78
79
80
81
82
83
85
Esc=Exit ¦ F1=Help ¦ Crtl-P=Edit peaks ¦

DDW 8/31/95
AMDT 11095.1
HWI 6329-152
Serum Curve 2
Carlton

Software : version 6.1 c1990,93

Operator : DDW

Date of the Analysis : 1995-08-28 07:32

Analysis File Name : C:\SKALAR\DATA\HWIDATA\SERUM\950828A1

Fluoride 1.5
Calibration order = Inverse Logarithm

Slope : s = #.#####

$$\text{Result} = 10^{\left[\frac{x - c1}{s}\right]}$$

x = corrected value of the sample
c1 = corrected value of the concentration 1
s = Slope of the electrode

a2 = -0.00000
a1 = 0.00075
a0 = -1.16846

Fluoride L
Calibration order = 2

Correlation : r = 0.99897

$\text{Result} = a2 * x^2 + a1 * x + a0$

a2 = -0.00000
a1 = 0.00032
a0 = 0.00531

Sampler	Type	: SA1000
	Number	: 1
	Sample Time	: 50 sec.
	Wash Time	: 120 sec.
	Air Time	: 1 sec.
	Take up	: Single
	sPecial	: None
	needle Height	: 70 mm.
Diluter	needle Height	: 80 mm
	dilution Factor	: 10
	dilution Volume	: 2.5 ml.
	Resample	: 1
	Dilution runs	: 1
	User file	: . TXT
	Reproces	: No

1995-08-28 15:03 OutPut of : 950828A1

Fluoride 1.5

```
Path number    :  3
Signal type    : Debubbled
Decolor        : Yes
system Number  : 0
diLute         : No
Resample       : No
dil Threshold  : 4095
diG output     : 0
Window event   : Off

s1   sTandard  : Ignore
s2   sTandard  : Ignore
s3   sTandard  : Ignore
s4   sTandard  : Ignore
s5   sTandard  : Ignore
s6   sTandard  :    0.150
s7   sTandard  :    0.300
s8   sTandard  :    0.600
s9   sTandard  :    1.200
s10  sTandard  :    1.500
Order : Inverse Logarithm
Dimension : PPM
start Value    : 500  DU
trigger Limit  : 1800 Sec
Peak shape     : Pointed
stArt ignore   : 60   Sec
eNd   ignore   : 120  Sec
Measure window : 75   %
Filter         : No
Regeneration   : No
formUla    :
output     : ##.###
```

Fluoride L

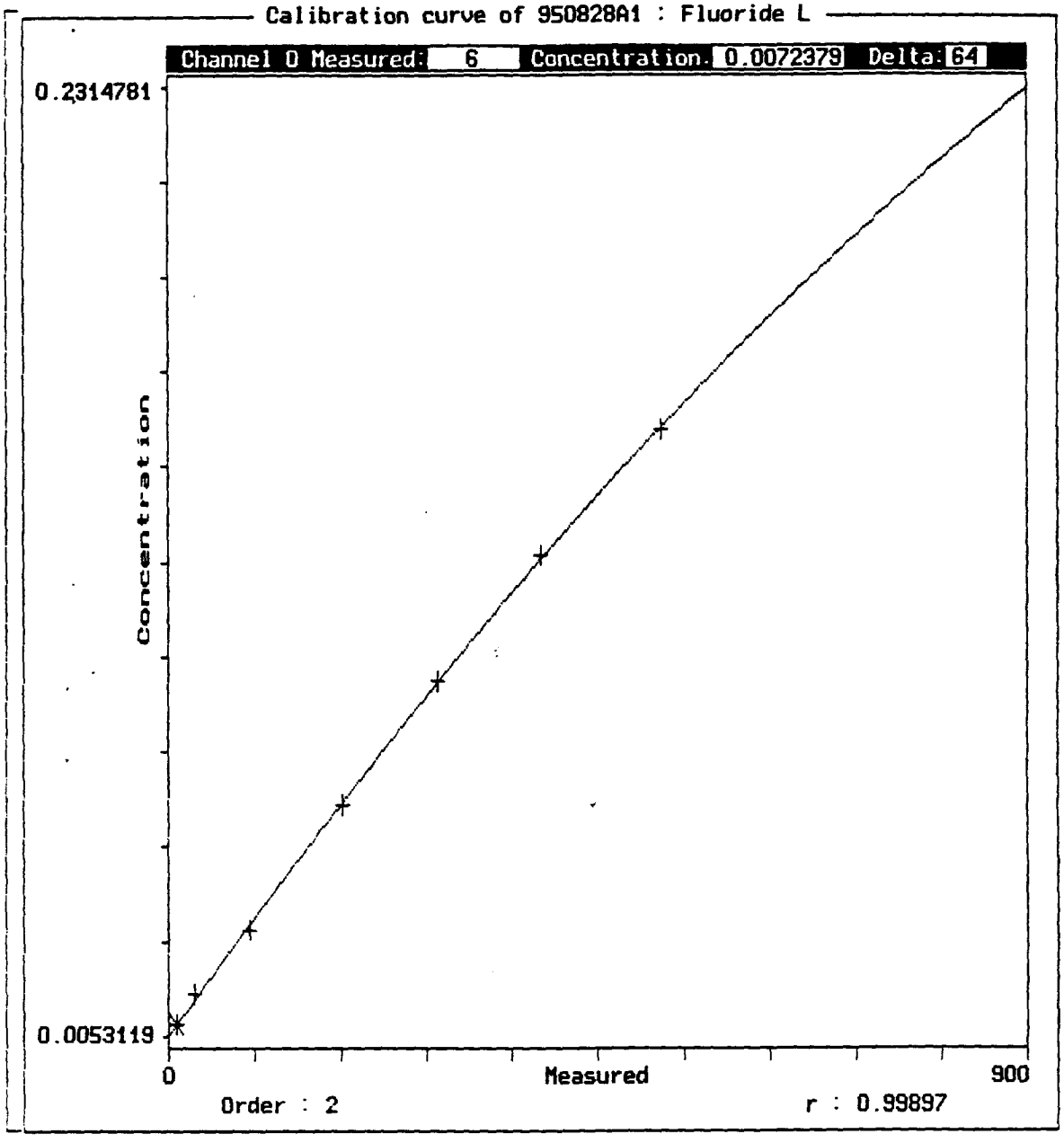
```
Path number    :  0
Signal type    : Debubbled
Decolor        : No
system Number  : 0
diLute         : No
Resample       : No
dil Threshold  : 4095
diG output     : 0
Window event   : Off
```

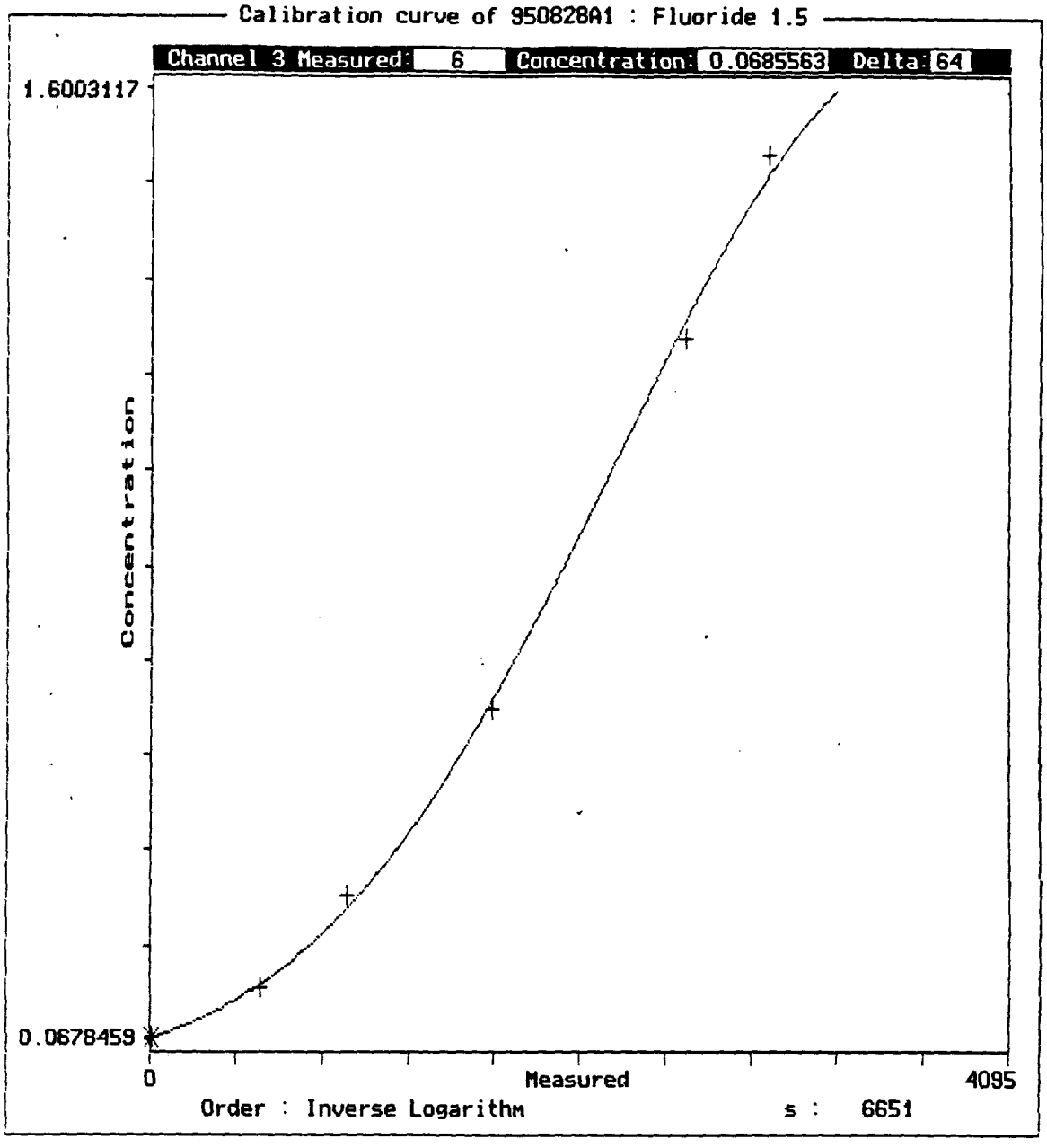
1995-08-28 15:03 OutPut of : 950828A1

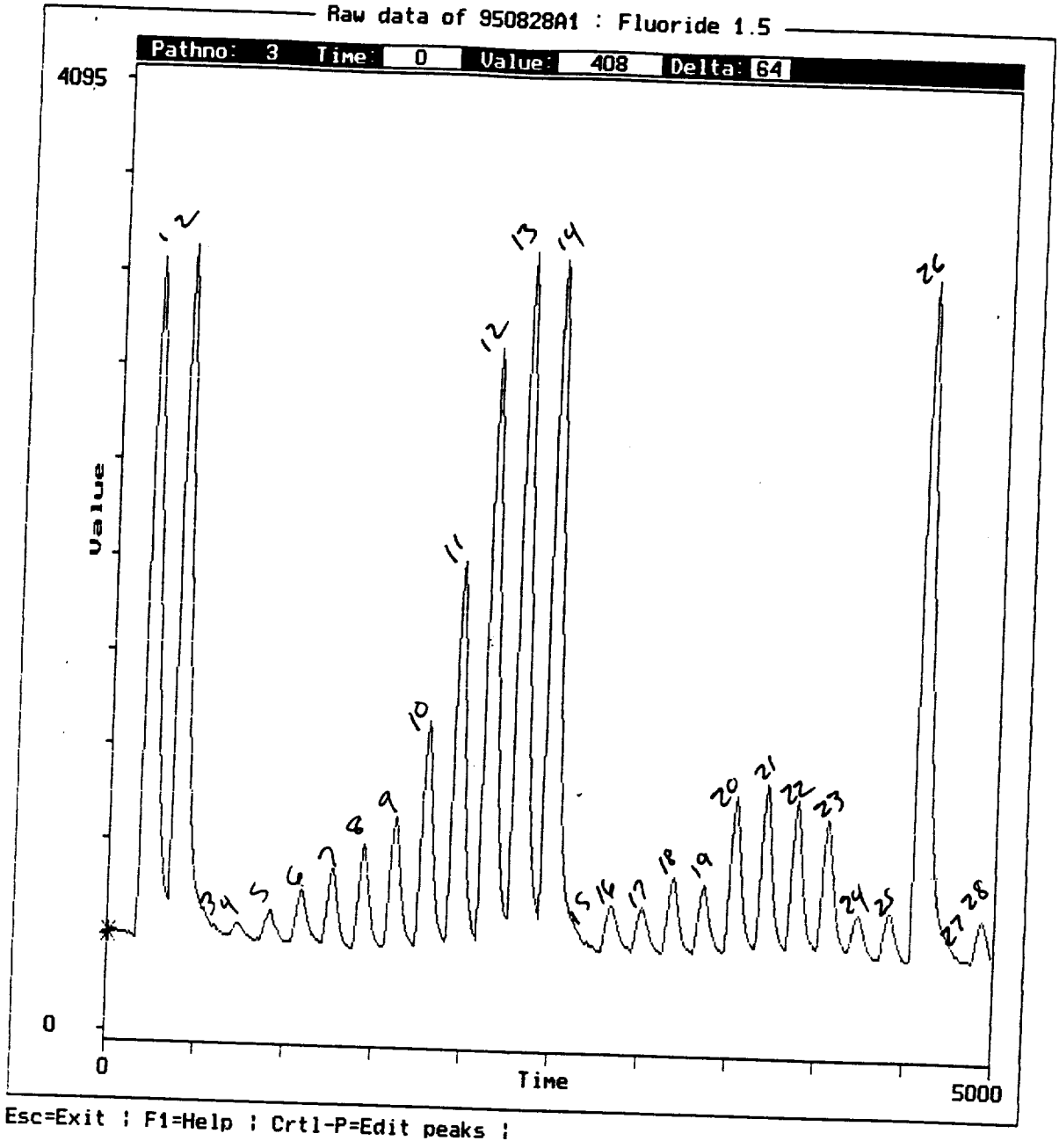
```
s1   sTandard :    0.015
s2   sTandard :    0.030
s3   sTandard :    0.060
s4   sTandard :    0.090
s5   sTandard :    0.120
s6   sTandard :    0.150
s7   sTandard : Ignore
s8   sTandard : Ignore
s9   sTandard : Ignore
s10  sTandard : Ignore
Order : 2
Dimension : PPM
start Value     : 500  DU
trigger Limit   : 1800 Sec
Peak shape      : Pointed
stArt ignore    : 60   Sec
eNd   ignore    : 120  Sec
Measure window  : 75   %
Filter          : No
Regeneration    : No
formUla    : c4:=c3
output     : #.####
```

			Fluoride 1.5			Fluoride L		
			PPM			*PPM*		
Pos	Typ	Ident	Ch	Result	F Time	Ch	Result	F Time
wt	iw	Initial Wash	3	0.068	65	4	0.0053	0
1	t	Tracer	3	1.453	209	4	0.2787	0
2	d	Drift	3	1.471	385	4	0.2740	0
3	w	Wash	3	0.068	587	4	0.0053	0
4	s1	Standard 1	3	0.071	733	4	0.0136	0
5	s2	Standard 2	3	0.078	901	4	0.0315	0
6	s3	Standard 3	3	0.092	1085	4	0.0609	0
7	s4	Standard 4	3	0.109	1260	4	0.0895	0
8	s5	Standard 5	3	0.129	1436	4	0.1188	0
9	s6	Standard 6	3	0.156	1611	4	0.1507	0
10	s7	Standard 7	3	0.279	1786	4	0.2370	0
11	s8	Standard 8	3	0.616	1960	4	0.3222	0
12	s9	Standard 9	3	1.233	2136	4	0.3177	0
13	s10	Standard 10	3	1.467	2312	4	0.2751	0
14	d	Drift	3	1.485	2488	4	0.2699	0
15	w	Wash	3	0.068	2687	4	0.0053	0
16	u	SERUM BLANK	3	0.091	2836	4	0.0577	0
17	u	SERUM BLANK	3	0.091	3012	4	0.0577	0
18	u	SPK 62-1	3	0.113	3188	4	0.0961	0
19	u	SPK 62-2	3	0.107	3362	4	0.0867	0
20	u	SPK 250-1	3	0.195	3538	4	0.1857	0
21	u	SPK 250-2	3	0.214	3713	4	0.1995	0
22	u	SPK 124-1	3	0.192	3887	4	0.1833	0
23	u	SPK 124-2	3	0.176	4063	4	0.1697	0
24	u	BLANK	3	0.092	4237	4	0.0598	0
25	u	F52725-4	3	0.095	4413	4	0.0659	0
26	d	Drift	3	1.462	4589	4	0.2764	0
27	w	Wash	3	0.068	4830	4	0.0053	0
28	u	F52721-4	3	0.092	4940	4	0.0606	0
29	u	F52727-4	3	0.085	5113	4	0.0467	0
30	u	F52806-4	3	0.085	5285	4	0.0458	0
31	u	F52713-4	3	0.079	5464	4	0.0321	0
32	u	F52718-4	3	0.087	5640	4	0.0497	0
33	u	F52723-4	3	0.081	5812	4	0.0367	0
34	u	F52715-4	3	0.080	5986	4	0.0345	0
35	u	F52738-4	3	0.097	6164	4	0.0700	0
36	u	SPK 62-1	3	0.085	6334	4	0.0467	0
37	u	SPK 62-2	3	0.126	6514	4	0.1152	0
38	d	Drift	3	1.482	6690	4	0.2708	0
39	w	Wash	3	0.068	6932	4	0.0053	0
40	u	SPK 124-1	3	0.152	7039	4	0.1466	0
41	u	BLANK-1	3	0.088	7213	4	0.0518	0
42	u	BLANK-2	3	0.081	7391	4	0.0376	0
43	u	SPK 62-1	3	0.102	7565	4	0.0791	0
44	u	SPK 62-2	3	0.106	7740	4	0.0847	0
45	u	SPK 250-1	3	0.302	7915	4	0.2478	0
46	u	SPK 250-2	3	0.257	8091	4	0.2256	0
47	u	BLK	3	0.092	8263	4	0.0604	0
48	u	F52809-4	3	0.080	8435	4	0.0361	0
49	u	F52724-4	3	0.079	8614	4	0.0321	0
50	d	Drift	3	1.469	8791	4	0.2746	0
51	w	Wash	3	0.068	9031	4	0.0053	0
52	u	F52737-4	3	0.079	9138	4	0.0321	0
53	u	F52801-4	3	0.092	9315	4	0.0601	0

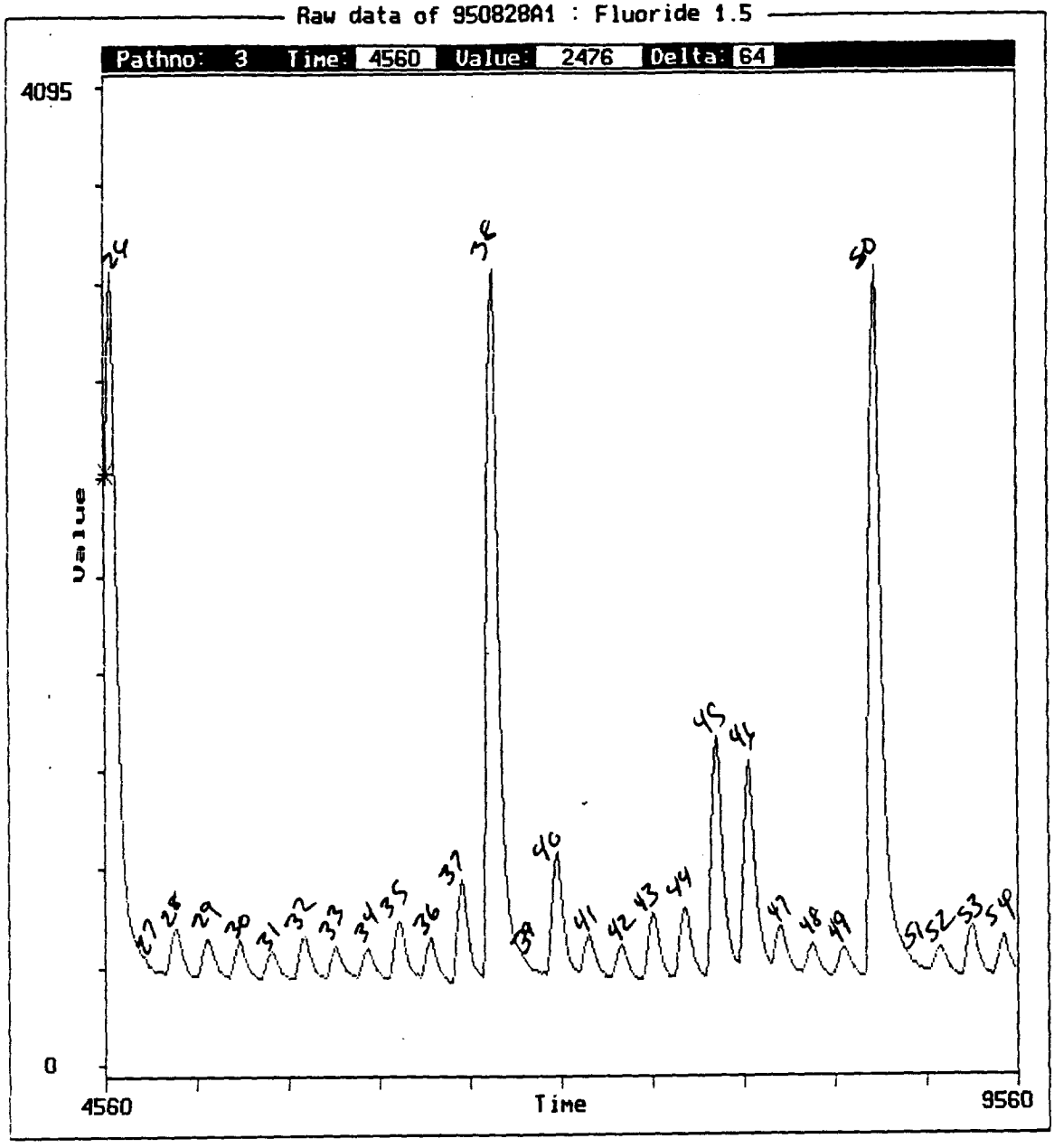
			Fluoride 1.5			Fluoride L		
			PPM			*PPM*		
Pos	Typ	Ident	Ch	Result	F Time	Ch	Result	F Time
54	u	F52720-4	3	0.085	9484	4	0.0458	0
55	u	F52733-4	3	0.080	9666	4	0.0351	0
56	u	F52788-4	3	0.075	9840	4	0.0249	0
57	u	F52730-4	3	0.073	10016	4	0.0196	0
58	u	F52731-4	3	0.073	10192	4	0.0193	0
59	u	F52802-4	3	0.077	10366	4	0.0293	0
60	u	SPK 62-1	3	0.086	10536	4	0.0476	0
61	u	SPK 62-2	3	0.117	10715	4	0.1024	0
62	d	Drift	3	1.492	10892	4	0.2679	0
63	w	Wash	3	0.068	11096	4	0.0053	0
64	u	SPK 250-1	3	0.176	11241	4	0.1694	0
65	u	SPK 250-2	3	0.235	11415	4	0.2128	0
66	u	SPK 124-1	3	0.160	11591	4	0.1543	0
67	u	BLK	3	0.103	11769	4	0.0811	0
68	u	BLK	3	0.093	11941	4	0.0624	0
69	u	F52714-4	3	0.085	12113	4	0.0467	0
70	u	F52734-4	3	0.096	12291	4	0.0674	0
71	u	F52774-4	3	0.085	12461	4	0.0461	0
72	u	F52712-4	3	0.080	12641	4	0.0345	0
73	u	F52729-4	3	0.079	12817	4	0.0336	0
74	d	Drift	3	1.488	12993	4	0.2690	0
75	w	Wash	3	0.068	13224	4	0.0053	0
76	u	F52803-4	3	0.085	13336	4	0.0461	0
77	u	F52716-4	3	0.081	13516	4	0.0385	0
78	u	F52728-4	3	0.079	13688	4	0.0342	0
79	u	F52787-4	3	0.081	13866	4	0.0382	0
80	u	SPK 62-1	3	0.095	14042	4	0.0653	0
81	u	SPK 62-2	3	0.122	14218	4	0.1101	0
82	u	SPK 124-1	3	0.109	14391	4	0.0903	0
83	u	SPK 124-2	3	0.154	14570	4	0.1483	0
84	d	Drift	3	1.506	14744	4	0.2632	0
85	w	Wash	3	0.068	14981	4	0.0053	0
wt	rw	RunOut Wash	3	0.068	15219	4	0.0053	0

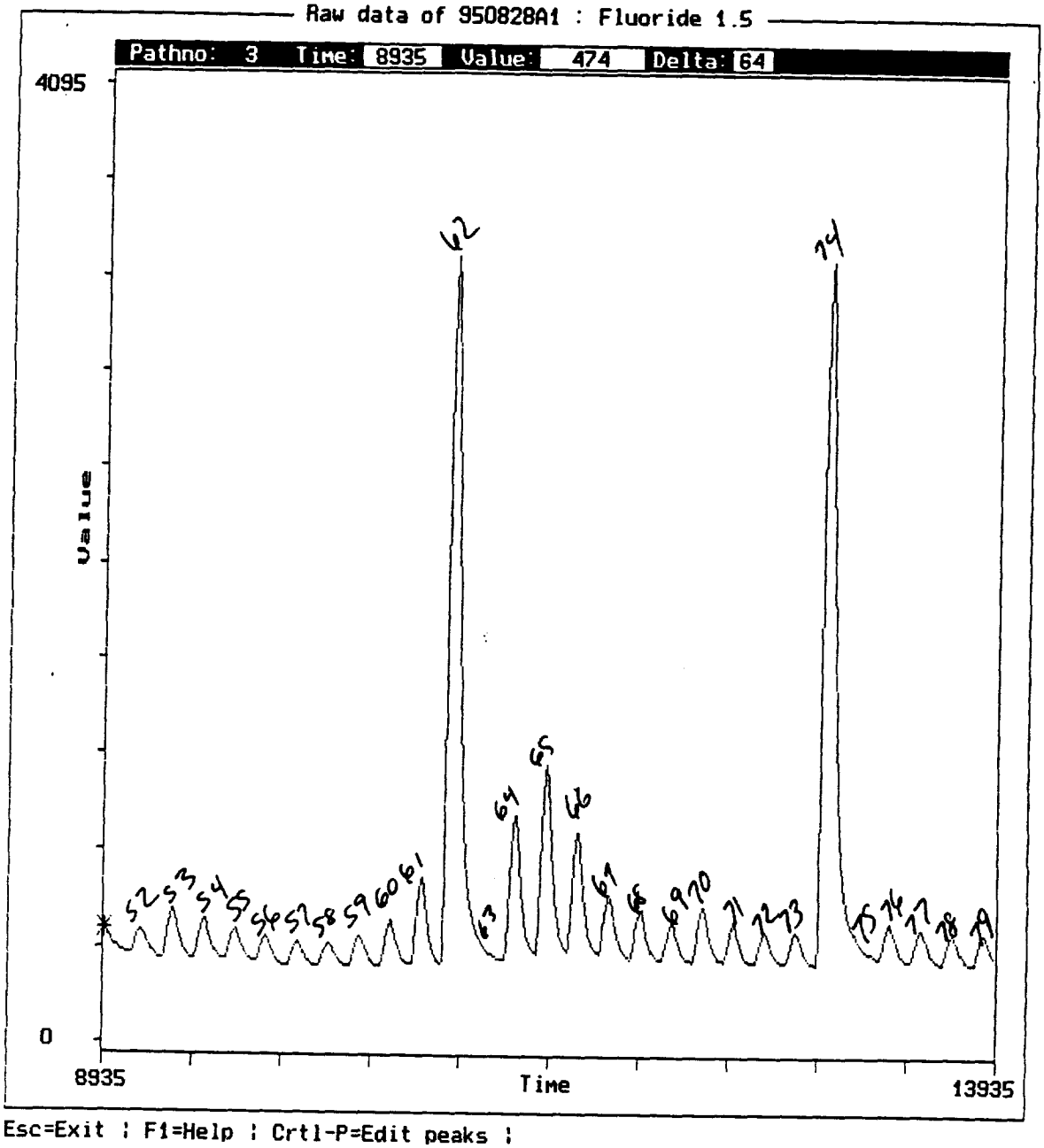

Calibration curve of 950828A1 : Fluoride L
Channel 0 Measured: 6
Concentration: 0.0072379
Delta: 64
0.2314781
0.0053119
Concentration
0
Measured
900
Order : 2
r : 0.99897

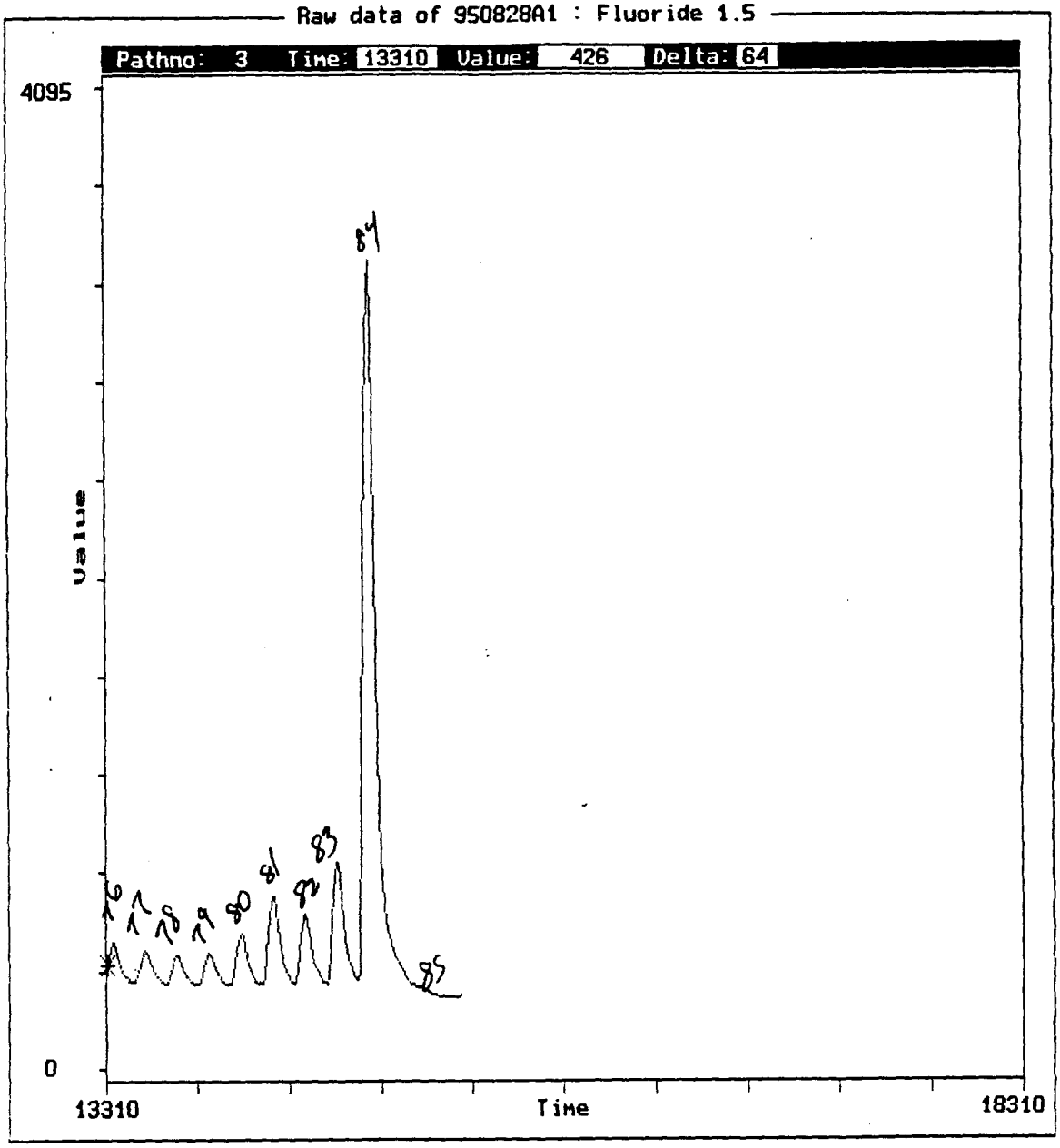

Calibration curve of 950828A1 : Fluoride 1.5
Channel 3 Measured: 6
Concentration: 0.0685563
Delta: 64
1.6003117
0.0678459
Concentration
0
Measured
4095
Order : Inverse Logarithm
s : 6651


Raw data of 950828A1 : Fluoride 1.5
Pathno: 3 Time: 0 Value: 408 Delta: 64
4095
0
Value
0
Time
5000
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28
Esc=Exit ; F1=Help ; Crtl-P=Edit peaks ;

000172


Raw data of 950828A1 : Fluoride 1.5
Pathno: 3 Time: 4560 Value: 2476 Delta: 64
4095
Value
0
4560
Time
9560
Esc=Exit ¦ F1=Help ¦ Crtl-P=Edit peaks ¦


Raw data of 950828A1 : Fluoride 1.5
Pathno: 3 Time: 8935 Value: 474 Delta: 64
4095
0
Value
8935
Time
13935
Esc=Exit ¦ F1=Help ¦ Crtl-P=Edit peaks ¦



Esc=Exit ¦ F1=Help ¦ Crtl-P=Edit peaks ¦

Printout of Sample Table : N2946- Inserted in System Table of : group 1

0	iw	Initial Wash	1	1.0000
1	t	Tracer	1	1.000
2	d	Drift	1	1.000
3	w	Wash	1	1.000
4	s1	Standard 1	1	1.000
5	s2	Standard 2	1	1.000
6	s3	Standard 3	1	1.000
7	s4	Standard 4	1	1.000
8	s5	Standard 5	1	1.000
9	s6	Standard 6	1	1.000
10	s7	Standard 7	1	1.000
11	s8	Standard 8	1	1.000

000175

DDW 8/31/95
AMDT 11095.1
HWI 6329-152
SerumCurve2
Carlton

1995-08-29 06:38 OutPut of : 950828B1

Software : version 6.1 c1990,93

Operator : DDW

Date of the Analysis : 1995-08-28 11:47

Analysis File Name : C:\SKALAR\DATA\HWIDATA\SERUM\950828B1

Fluoride 1.5
Calibration order = Inverse Logarithm

Slope : s = #.#####

$$\text{Result} = 10^{\left[\frac{x - c1}{s}\right]}$$

x = corrected value of the sample
c1 = corrected value of the concentration 1
s = Slope of the electrode

a2 = -0.00000
a1 = 0.00071
a0 = -1.18575

Fluoride L
Calibration order = 2

Correlation : r = 0.99313

Result = a2 * x^2 + a1 * x + a0

a2 = 0.00000
a1 = 0.00020
a0 = 0.00438

Sampler
Type : SA1000
Number : 1
Sample Time : 50 sec.
Wash Time : 120 sec.
Air Time : 1 sec.
Take up : Single
sPecial : None
needle Height : 70 mm.

Diluter
needle Height : 80 mm
dilution Factor : 10
dilution Volume : 2.5 ml.
Resample : 1
Dilution runs : 1

User file : . TXT
Reproces : No

000176

1995-08-29 06:38 OutPut of : 950828B1

Fluoride 1.5

Path number : 3
Signal type : Debubbled
Decolor : Yes
system Number : 0
diLute : No
Resample : No
dil Threshold : 4095
diG output : 0
Window event : Off

s1 sTandard : Ignore
s2 sTandard : Ignore
s3 sTandard : Ignore
s4 sTandard : Ignore
s5 sTandard : Ignore
s6 sTandard : 0.150
s7 sTandard : 0.300
s8 sTandard : 0.600
s9 sTandard : 1.200
s10 sTandard : 1.500
Order : Inverse Logarithm
Dimension : PPM
start Value : 500 DU
trigger Limit : 1800 Sec
Peak shape : Pointed
stArt ignore : 60 Sec
eNd ignore : 120 Sec
Measure window : 75 %
Filter : No
Regeneration : No
formUla :
output : ##.###

Fluoride L

Path number : 0
Signal type : Debubbled
Decolor : No
system Number : 0
diLute : No
Resample : No
dil Threshold : 4095
diG output : 0
Window event : Off

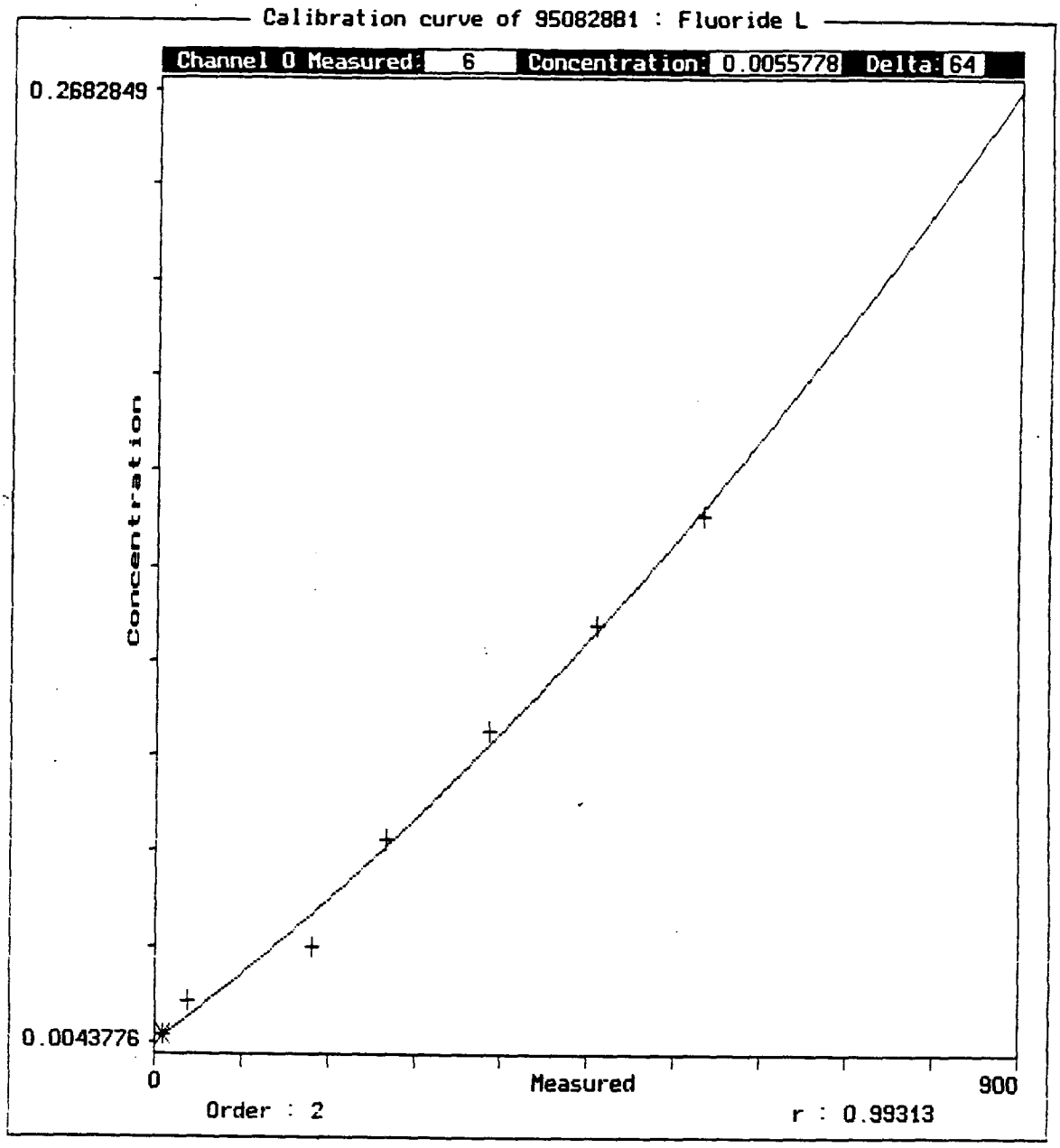
1995-08-29 06:38 OutPut of : 950828B1

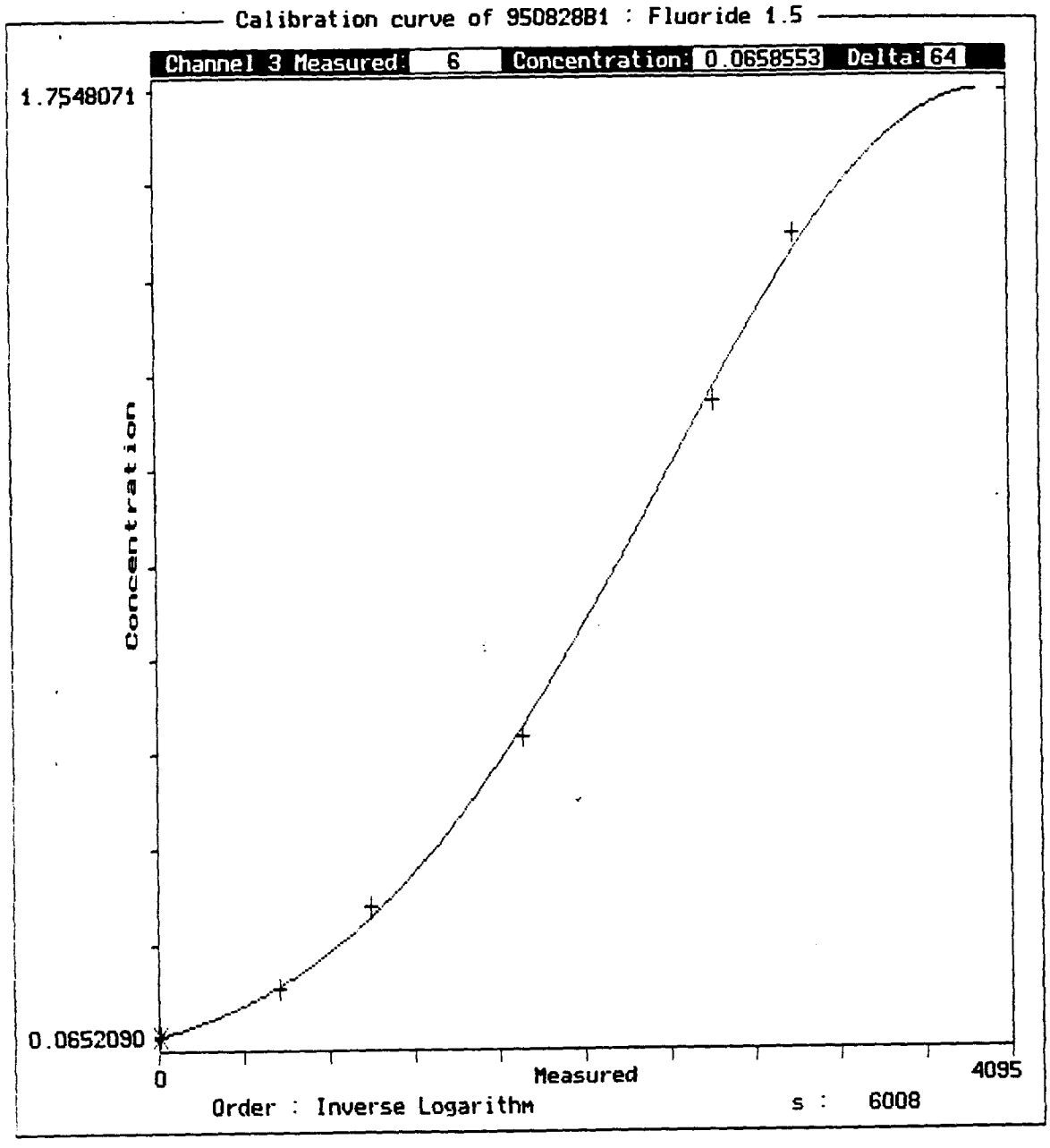
```
s1   sTandard :    0.015
s2   sTandard :    0.030
s3   sTandard :    0.060
s4   sTandard :    0.090
s5   sTandard :    0.120
s6   sTandard :    0.150
s7   sTandard : Ignore
s8   sTandard : Ignore
s9   sTandard : Ignore
s10  sTandard : Ignore
Order : 2
Dimension : PPM
start Value     : 500  DU
trigger Limit   : 1800 Sec
Peak shape      : Pointed
stArt ignore    : 60   Sec
eNd   ignore    : 120  Sec
Measure window  : 75   %
Filter          : No
Regeneration    : No
formUla    : c4:=c3
output     : #.####
```

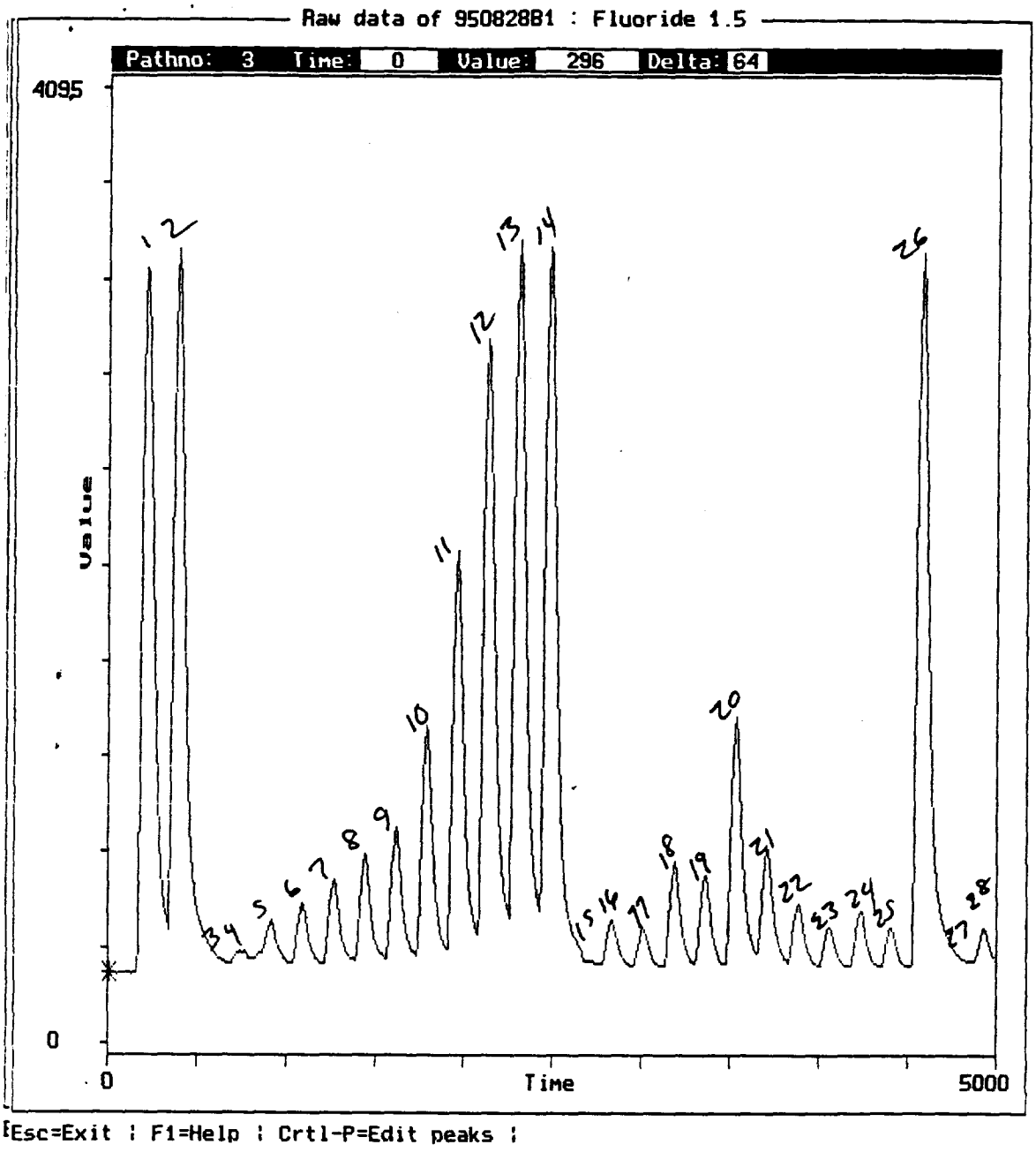
000178

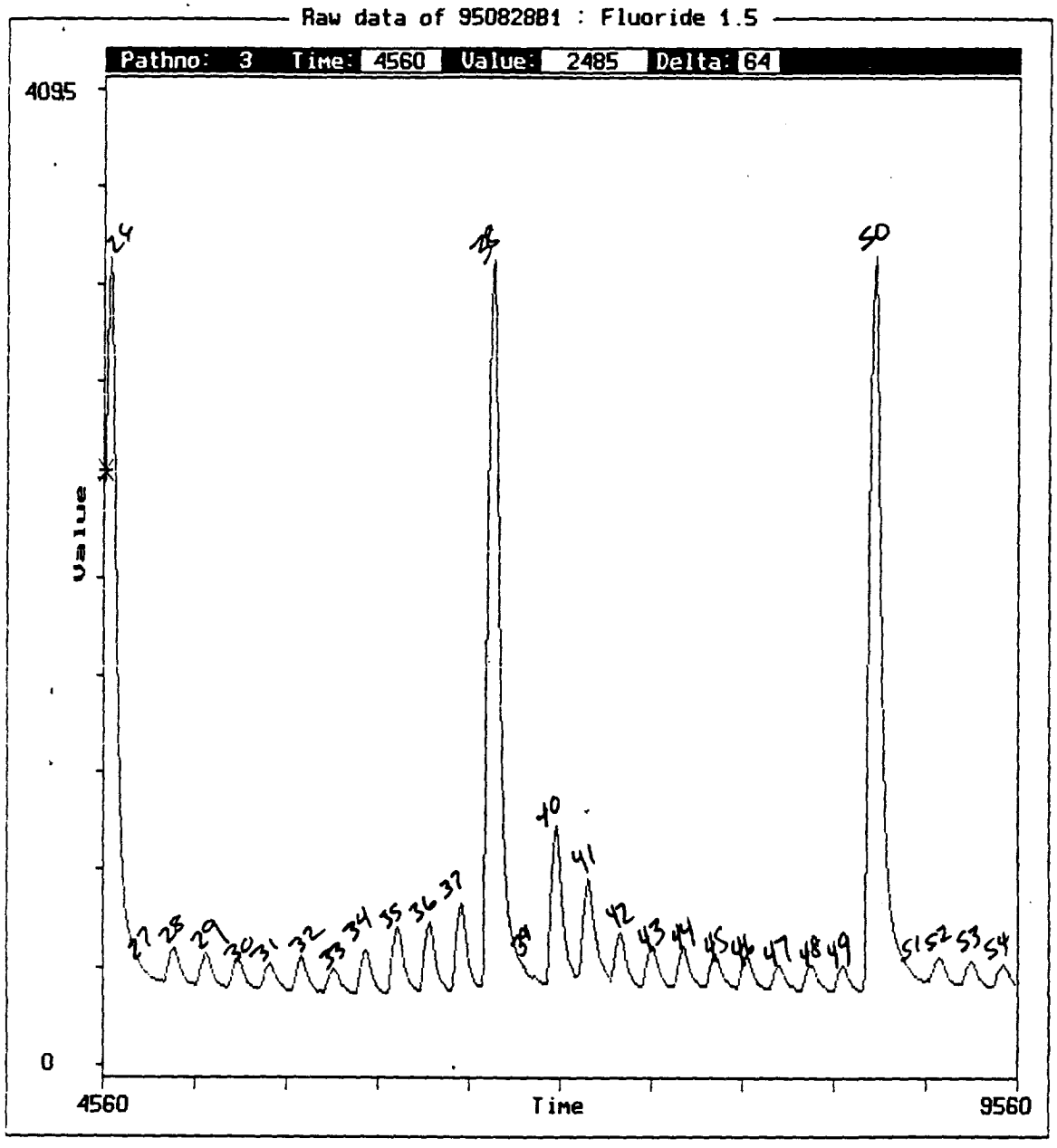
			Fluoride 1.5			Fluoride L		
			PPM			*PPM*		
Pos	Typ	Ident	Ch	Result F	Time	Ch	Result F	Time
wt	iw	Initial Wash	3	0.065	65	4	0.0044	0
1	t	Tracer	3	1.464	212	4	1.5923	0
2	d	Drift	3	1.474	387	4	1.6075	0
3	w	Wash	3	0.065	616	4	0.0044	0
4	s1	Standard 1	3	0.069	727	4	0.0111	0
5	s2	Standard 2	3	0.085	909	4	0.0394	0
6	s3	Standard 3	3	0.095	1087	4	0.0580	0
7	s4	Standard 4	3	0.113	1263	4	0.0861	0
8	s5	Standard 5	3	0.133	1439	4	0.1182	0
9	s6	Standard 6	3	0.156	1615	4	0.1522	0
10	s7	Standard 7	3	0.280	1787	4	0.3134	0
11	s8	Standard 8	3	0.618	1963	4	0.6720	0
12	s9	Standard 9	3	1.228	2137	4	1.2898	0
13	s10	Standard 10	3	1.470	2313	4	1.6007	0
14	d	Drift	3	1.498	2487	4	1.6438	0
15	w	Wash	3	0.065	2686	4	0.0044	0
16	u	SERUM BLK 1	3	0.086	2832	4	0.0420	0
17	u	SERUM BLK 2	3	0.082	3011	4	0.0343	0
18	u	SPK 62-1	3	0.127	3189	4	0.1082	0
19	u	SPK 62-2	3	0.118	3363	4	0.0946	0
20	u	SPK 124-1	3	0.293	3540	4	0.3291	0
21	u	SPK 124-2	3	0.139	3714	4	0.1277	0
22	u	BLK	3	0.097	3892	4	0.0602	0
23	u	BLK	3	0.082	4064	4	0.0353	0
24	u	F52717-4	3	0.092	4242	4	0.0516	0
25	u	F52735-4	3	0.081	4414	4	0.0334	0
26	d	Drift	3	1.455	4588	4	1.5790	0
27	w	Wash	3	0.065	4815	4	0.0044	0
28	u	F52736-4	3	0.080	4936	4	0.0318	0
29	u	F52726-4	3	0.078	5116	4	0.0269	0
30	u	F52732-4	3	0.074	5288	4	0.0210	0
31	u	F52739-4	3	0.072	5463	4	0.0163	0
32	u	F52711-8	3	0.075	5635	4	0.0227	0
33	u	F52719-8	3	0.070	5812	4	0.0123	0
34	u	BLK	3	0.079	5992	4	0.0298	0
35	u	SPK 62-1	3	0.093	6165	4	0.0545	0
36	u	SPK 62-2	3	0.096	6341	4	0.0585	0
37	u	SPK 124-1	3	0.108	6515	4	0.0791	0
38	d	Drift	3	1.460	6689	4	1.5857	0
39	w	Wash	3	0.065	6904	4	0.0044	0
40	u	SPK 124-2	3	0.174	7041	4	0.1784	0
41	u	BLK	3	0.126	7215	4	0.1074	0
42	u	BLK	3	0.089	7389	4	0.0467	0
43	u	F52725-8	3	0.084	7566	4	0.0390	0
44	u	F52721-8	3	0.080	7739	4	0.0316	0
45	u	F52727-	3	0.077	7914	4	0.0267	0
46	u	F52806-8	3	0.076	8092	4	0.0247	0
47	u	F52713-8	3	0.071	8266	4	0.0157	0
48	u	F52718-8	3	0.072	8439	4	0.0163	0
49	u	F52723-8	3	0.072	8613	4	0.0161	0
50	d	Drift	3	1.463	8789	4	1.5898	0
51	w	Wash	3	0.065	9028	4	0.0044	0
52	u	F52715-8	3	0.076	9133	4	0.0243	0
53	u	F52738-8	3	0.073	9313	4	0.0191	0

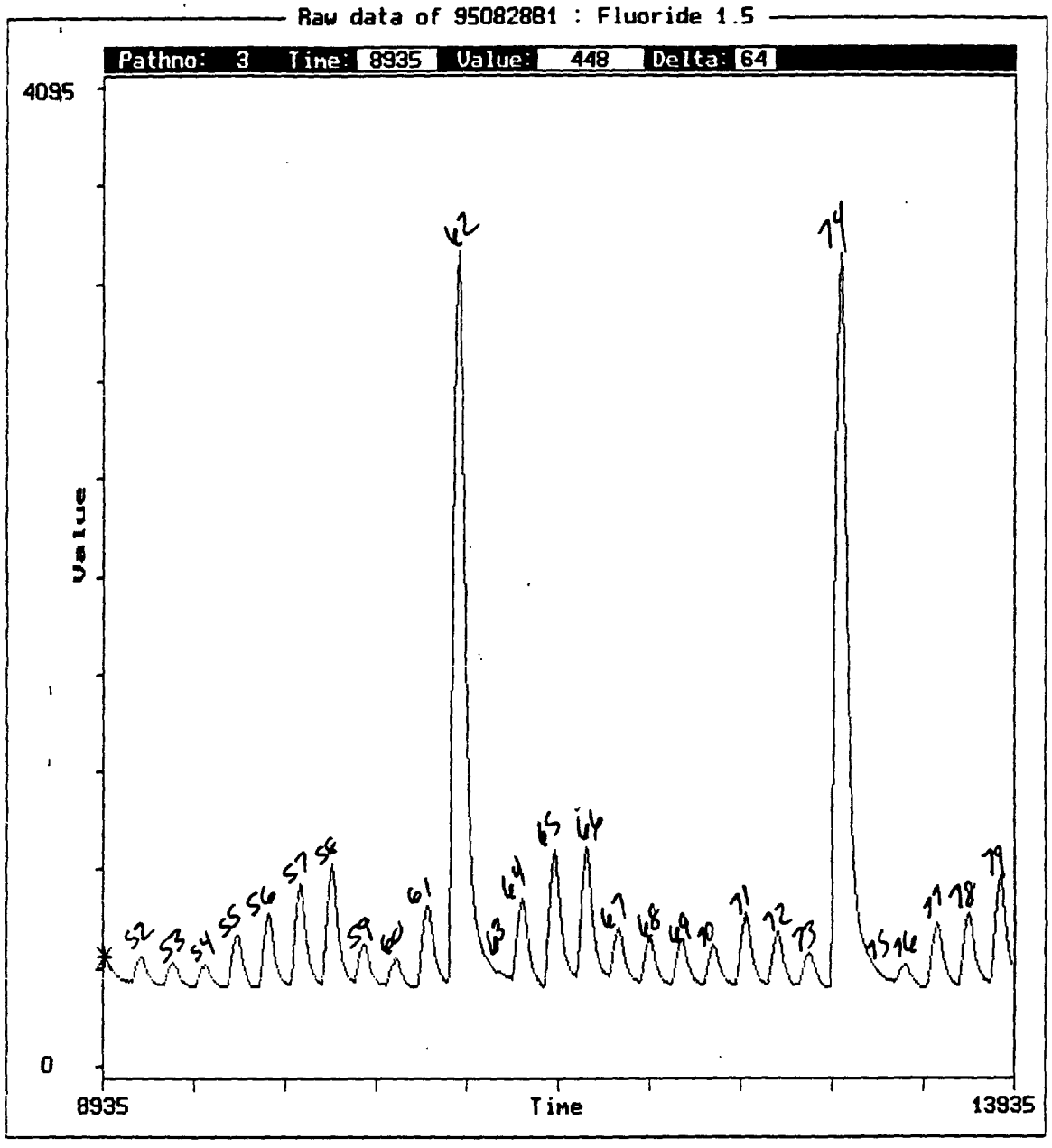
			Fluoride 1.5			Fluoride L		
			PPM			*PPM*		
Pos	Typ	Ident	Ch	Result	F Time	Ch	Result	F Time
54	u	F52809-8	3	0.072	9488	4	0.0165	0
55	u	SPK 62-1	3	0.088	9662	4	0.0451	0
56	u	SPK 62-2	3	0.100	9838	4	0.0648	0
57	u	SPK 124-1	3	0.121	10014	4	0.0991	0
58	u	SPK 124-2	3	0.136	10190	4	0.1220	0
59	u	BLK	3	0.083	10364	4	0.0362	0
60	u	BLK	3	0.074	10538	4	0.0197	0
61	u	SPK 62-1	3	0.106	10714	4	0.0752	0
62	d	Drift	3	1.460	10888	4	1.5857	0
63	w	Wash	3	0.065	11129	4	0.0044	0
64	u	SPK 62-2	3	0.109	11240	4	0.0799	0
65	u	SPK 124-1	3	0.147	11414	4	0.1393	0
66	u	SPK 124-2	3	0.149	11592	4	0.1425	0
67	u	BLK	3	0.089	11765	4	0.0477	0
68	u	BLK	3	0.084	11941	4	0.0378	0
69	u	F52724-8	3	0.083	12114	4	0.0369	0
70	u	F52737-8	3	0.083	12290	4	0.0362	0
71	u	F52801-8	3	0.099	12466	4	0.0643	0
72	u	F52720-8	3	0.088	12640	4	0.0460	0
73	u	F52733-8	3	0.078	12814	4	0.0269	0
74	d	Drift	3	1.465	12988	4	1.5940	0
75	w	Wash	3	0.065	13219	4	0.0044	0
76	u	F52788-8	3	0.071	13336	4	0.0154	0
77	u	SPK 62-1	3	0.094	13514	4	0.0558	0
78	u	SPK 62-2	3	0.100	13692	4	0.0653	0
79	u	SPK 124-1	3	0.126	13866	4	0.1074	0
80	d	Drift	3	1.468	14039	4	1.5982	0
81	w	Wash	3	0.065	14280	4	0.0044	0
wt	rw	RunOut Wash	3	0.065	14514	4	0.0044	0

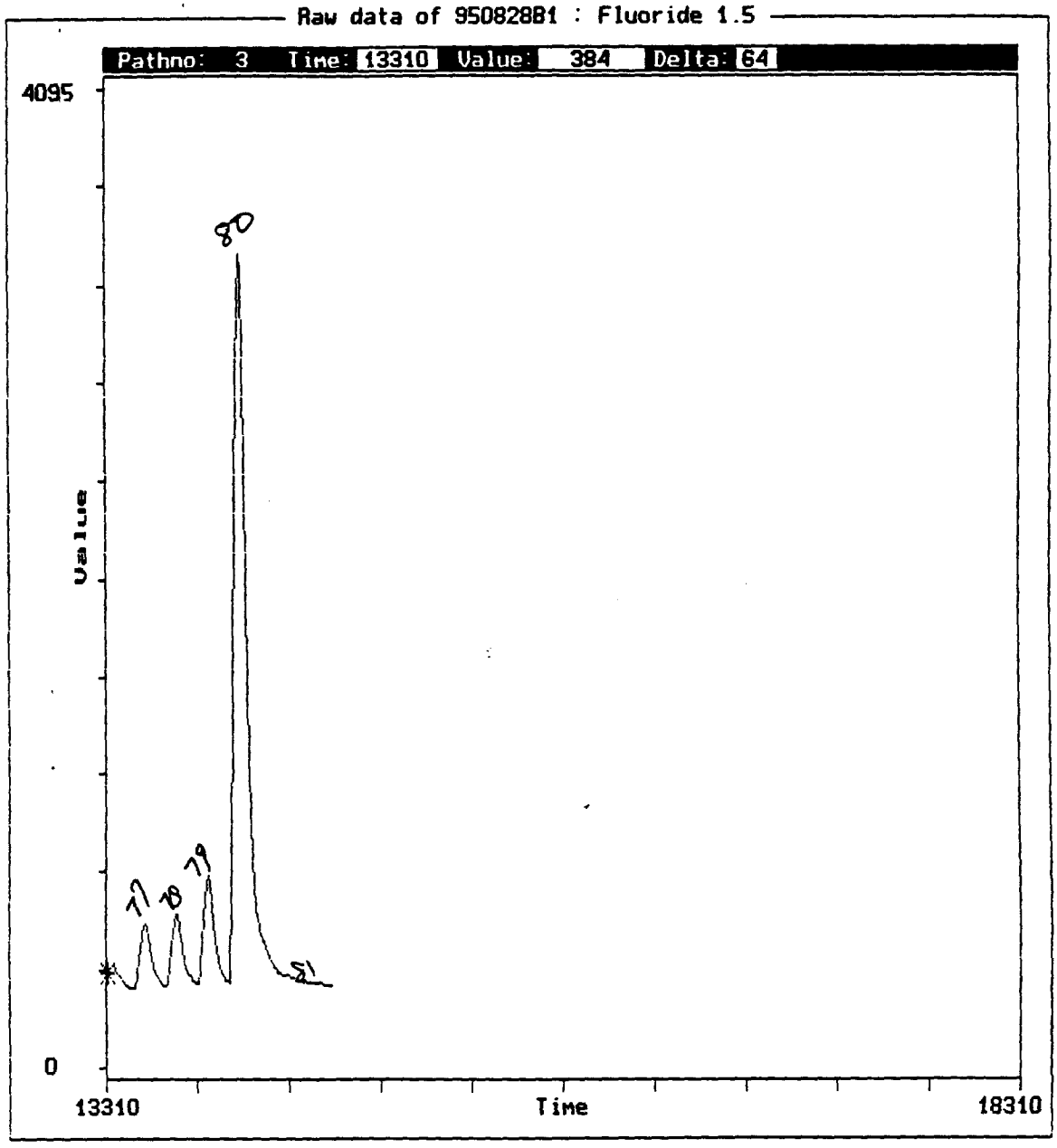

Calibration curve of 95082881 : Fluoride L
Channel 0 Measured: 6
Concentration: 0.0055778
Delta: 64
0.2682849
0.0043776
Concentration
0
Measured
900
Order : 2
r : 0.99313


Calibration curve of 950828B1 : Fluoride 1.5
Channel 3 Measured: 6
Concentration: 0.0658553
Delta: 64
1.7548071
0.0652090
Concentration
0
Measured
4095
Order : Inverse Logarithm
s : 6008


Raw data of 950828B1 : Fluoride 1.5
Pathno: 3 Time: 0 Value: 296 Delta: 64
4095
0
Value
0
Time
5000
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28
Esc=Exit ¦ F1=Help ¦ Crtl-P=Edit peaks ¦


Raw data of 95082881 : Fluoride 1.5
Pathno: 3 Time: 4560 Value: 2485 Delta: 64
4095
Value
0
4560
Time
9560
Esc=Exit ¦ F1=Help ¦ Crtl-P=Edit peaks ¦


Raw data of 95082881 : Fluoride 1.5
Pathno: 3 Time: 8935 Value: 448 Delta: 64
4095
Value
0
8935
Time
13935
Esc=Exit ¦ F1=Help ¦ Crtl-P=Edit peaks ¦



Esc=Exit ¦ F1=Help ¦ Crtl-P=Edit peaks ¦

9.11.4 Summary and raw data; ppm F^- in serum as determined by thermal extraction followed by analysis using Orion ion analyzer.

HWI 6329-152
AMDT 011095.1
Dohrmann Serum Analysis
Analysis Dates: 08/02/95 - 08/18/95

All serum samples were thermally extracted by a modified Dohrmann DX2000 Organic Halide Analyzer and collected in a 1:1 milli Q water and TISAB solution. The samples were measured on an Orion EA940 expandable ion analyzer. The Dohrmann was calibrated using 34ppm, 40ppm, 62ppm, 100ppm, 124ppm, 250ppm, and 500ppm FC-95 standards. The Orion was calibrated by direct measurement with no blank correction using 0.05ppm, 0.1ppm, 0.5ppm, 1.0ppm and 1.5ppm F^- standards. The slope, intercept, and correlation were recorded in the appropriate logbook.

A summary table is included, showing the ppm F^- in each sample (see page 2). An initial calibration curve with standard deviation, %RSD, R^2 value and equation of the line is on pages 3 - 5.

Pages 6 - 19 show the excel spreadsheet that was generated when the samples were being analyzed. The Dohrmann FC-95 initial calibration curve was not used to generate the data in the spreadsheet. Any Orion reading below 0.05 is below the Orion calibration and should be considered an estimate.

The FC-95 initial calibration curve was spiked into bovine serum. However, due to problems with blanks having higher readings than the samples, the blank samples and QC check samples after 08/11/95 were taken from study # HWI 6329-123 rabbit # F52423, F52346, and F52330. This would affect all the day 4, day 8 samples, and F52726, F52732, F52739, F52736 of the day 2 samples.

This study was discontinued after day 8 because nothing was found in the first 3 days of sampling.

Dean K. Plummer

L3492

HWI 6329-152

Fluoride concentration in rabbit serum (ppm F-)

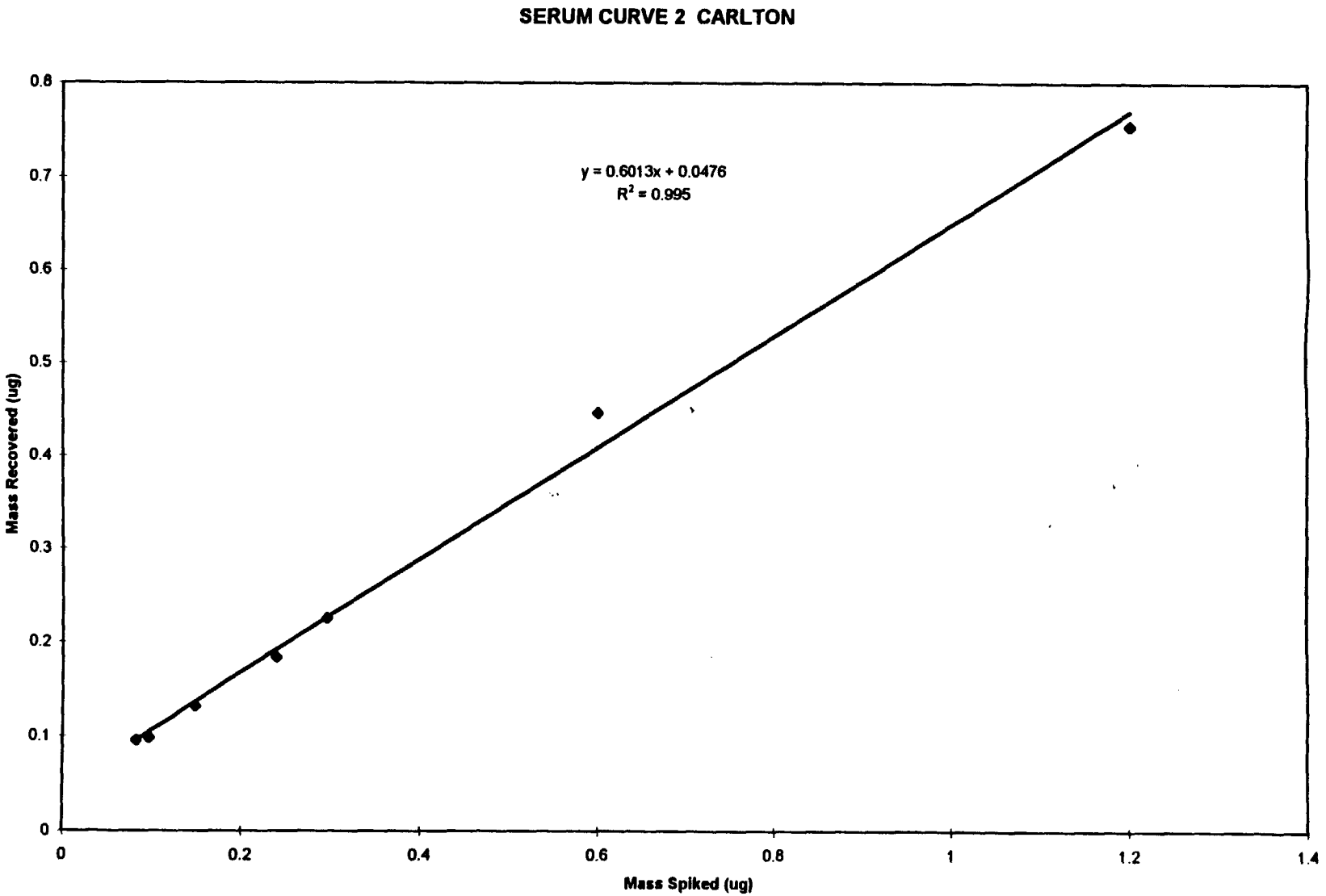
	Sample	Day 1	Day 2	Day 4	Day 8
Group 1					
Dosage: 0 mg/kg	F52711	0.593	0.381	0.594	0.484
	F52719	0.505	0.337	0.518	0.328
	F52725	0.531	0.296	0.691	0.600
	F52721	0.443	0.257	0.606	0.556
	F52727	0.408	0.526	0.496	0.461
	F52806	0.472	0.594	0.459	0.462
Group 2					
Dosage: 4 mg/kg	F52713	0.994	0.400	0.371	0.341
	F52718	0.810	0.402	0.590	0.349
	F52723	0.730	0.460	0.414	0.346
	F52715	0.749	0.410	0.377	0.344
	F52738	0.942	0.382	0.827	0.331
	F52809	1.12	0.384	0.391	0.303
Group 3					
Dosage: 16 mg/kg	F52724	0.866	0.369	0.373	0.578
	F52737	0.870	0.371	0.827	0.563
	F52801	0.761	0.609	0.718	0.856
	F52720	0.644	0.464	0.531	0.788
	F52733	0.775	0.451	0.436	0.491
	F52788	0.337	0.511	0.383	0.259
Group 4					
Dosage: 40 mg/kg	F52730	0.785	0.472	0.348	
	F52731	0.772	0.372	0.298	
	F52802	0.854	0.482	0.464	
	F52714	0.71	0.515	0.580	
	F52734	0.772	0.434	0.892	
	F52774	0.924	0.434	0.570	
Group 5					
Dosage: 10 X 10 fabric	F52712	0.934	0.649	0.436	
	F52729	0.686	0.457	0.452	
	F52803	0.948	0.448	0.474	
	F52716	0.964	0.419	0.411	
	F52728	1.06	0.549	0.452	
	F52787	0.890	0.536	0.443	
Group 6					
Dosage: 10 X 10 fabric	F52717	1.27	0.510	0.987	
	F52735	1.09	0.472	0.638	
	F52736	0.894	0.493	0.503	
	F52726	0.725	0.653	0.511	
	F52732	1.08	0.337	0.425	
	F52739	0.675	0.352	0.328	

STUDY # 6329-152 SERUM

Sample ID	Sample Date	Actual reading (ppm F-)	Sample Qty (mL or g)	TISAB final vol (mL)	mL FC95 spiked	onc. FC9 solution (ppm)	% recovery (ug/ug)	Actual ppm F- in sample	Mass spiked (ug F-)	Mass recovered (ug F-)		
blank-1	7/26/95	0.04392	0.1	2.0				0.8784		0.0878		
blank-2	7/26/95	0.03695	0.1	2.0				0.7390		0.0739		
blank-3	7/26/95	0.03742	0.1	2.0				0.7484		0.0748		
blank-4	7/26/95	0.04193	0.1	2.0				0.8386		0.0839		
blank-5	7/26/95	0.03201	0.1	2.0				0.6402		0.0640		
blank-6	7/26/95	0.03174	0.1	2.0				0.6348		0.0635		
blank-7	7/26/95	0.02625	0.1	2.0				0.5250		0.0525		
blank-8	7/26/95	0.03425	0.1	2.0				0.6850		0.0685		
blank-9	7/26/95	0.04265	0.1	2.0				0.8530		0.0853		
blank-10	7/26/95	0.03228	0.1	2.0				0.6456		0.0646		
blank-11	7/26/95	0.03017	0.1	2.0				0.6034		0.0603		
blank-12	7/26/95	0.03311	0.1	2.0				0.6622		0.0662		
blank-13	7/26/95	0.02693	0.1	2.0				0.5386		0.0539		
34ppm-1	7/26/95	0.05378	0.1	2.0	0.004	34	132%	1.0756	0.0817	0.1076	STDEV:	0.010105
34ppm-2	7/26/95	0.04430	0.1	2.0	0.004	34	109%	0.8860	0.0817	0.0886	%RSD:	11
34ppm-3	7/26/95	0.04601	0.1	2.0	0.004	34	113%	0.9202	0.0817	0.0920	AVERAGE:	0.0961
40ppm-1	7/26/95	0.04739	0.1	2.0	0.004	40	99%	0.9478	0.0961	0.0948	STDEV:	0.00448
40ppm-2	7/26/95	0.04881	0.1	2.0	0.004	40	102%	0.9762	0.0961	0.0976	%RSD:	4.5
40ppm-3	7/26/95	0.05178	0.1	2.0	0.004	40	108%	1.0356	0.0961	0.1036	AVERAGE:	0.0987
62ppm-1	7/26/95	0.06585	0.1	2.0	0.004	62	88%	1.3170	0.1489	0.1317	STDEV:	0.006838
62ppm-2	7/26/95	0.06287	0.1	2.0	0.004	62	84%	1.2574	0.1489	0.1257	%RSD:	5.2
62ppm-3	7/26/95	0.06969	0.1	2.0	0.004	62	94%	1.3938	0.1489	0.1394	AVERAGE:	0.1323
100ppm-1	7/26/95	0.08098	0.1	2.0	0.004	100	67%	1.6196	0.2402	0.1620	STDEV:	0.023949
100ppm-2	7/26/95	0.08998	0.1	2.0	0.004	100	75%	1.7996	0.2402	0.1800	%RSD:	13
100ppm-3	7/26/95	0.1047	0.1	2.0	0.004	100	87%	2.0940	0.2402	0.2094	AVERAGE:	0.1838
124ppm-1	7/26/95	0.09999	0.1	2.0	0.004	124	67%	1.9998	0.2978	0.2000	STDEV:	0.022645
124ppm-2	7/26/95	0.1159	0.1	2.0	0.004	124	78%	2.3180	0.2978	0.2318	%RSD:	10
124ppm-3	7/26/95	0.1219	0.1	2.0	0.004	124	82%	2.4380	0.2978	0.2438	AVERAGE:	0.2252

STUDY # 6329-152 SERUM

Sample ID	Sample Date	Actual reading (ppm F-)	Sample Qty (mL or g)	TISAB final vol (mL)	mL FC95 spiked	onc. FC9 solution (ppm)	% recovery (ug/ug)	Actual ppm F- in sample	Mass spiked (ug F-)	Mass recovered (ug F-)		
250ppm-1	7/26/95	0.1947	0.1	2.0	0.004	250	65%	3.8940	0.6004	0.3894	STDEV:	0.105373
250ppm-2	7/26/95	0.302	0.1	2.0	0.004	250	101%	6.0400	0.6004	0.6040	%RSD:	24
250ppm-3	7/26/95	0.1981	0.1	2.0	0.004	250	66%	3.9620	0.6004	0.3962	AVERAGE:	0.4460
250ppm-4	7/26/95	0.1972	0.1	2.0	0.004	250	66%	3.9440	0.6004	0.3944		
500ppm-1	7/26/95	0.3486	0.1	2.0	0.004	500	58%	6.9720	1.2008	0.6972	STDEV:	0.072032
500ppm-2	7/26/95	0.4286	0.1	2.0	0.004	500	71%	8.5720	1.2008	0.8572	%RSD:	9.6
500ppm-3	7/26/95	0.3745	0.1	2.0	0.004	500	62%	7.4900	1.2008	0.7490	AVERAGE:	0.7542
500ppm-4	7/26/95	0.3566	0.1	2.0	0.004	500	59%	7.1320	1.2008	0.7132		


SERUM CURVE 2 CARLTON
y = 0.6013x + 0.0476
R² = 0.995
Mass Recovered (ug)
Mass Spiked (ug)
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0
0.2
0.4
0.6
0.8
1
1.2
1.4

STUDY #6329-152 SERUM

Sample ID	Actual reading (ppm F-)	Sample Qty (mL or g)	TISAB final vol (mL)	mL FC95 spiked	Conc. FC95 solution (ppm)	% recovery (ug/ug)	Actual ppm F- in sample	Mass spiked (ug F-)	Mass recovered (ug F-)
BLANK	0.0512	0.1	2.0				1.024		0.102
BLANK	0.0314	0.1	2.0				0.628		0.0628
62PPM-1	0.0557	0.1	2.0	0.004	62	75%	1.11	0.149	0.111
62PPM-2	0.0867	0.1	2.0	0.004	62	116%	1.73	0.149	0.173
62PPM-3	0.0995	0.1	2.0	0.004	62	134%	1.99	0.149	0.199
250PPM-1	0.0982	0.1	2.0	0.004	250	33%	1.96	0.600	0.196
250PPM-2	0.240	0.1	2.0	0.004	250	80%	4.80	0.600	0.480
250PPM-3	0.235	0.1	2.0	0.004	250	78%	4.70	0.600	0.470
BLANK	0.164	0.1	2.0				3.27		0.327
BLANK	0.0878	0.1	2.0				1.76		0.176
BLANK	0.0631	0.1	2.0				1.26		0.126
BLANK	0.0404	0.1	2.0				0.807		0.0807
F52713DAY1	0.0497	0.1	2.0				0.994		0.0994
F52718DAY1	0.0405	0.1	2.0				0.810		0.0810
F52723DAY1	0.0365	0.1	2.0				0.730		0.0730
F52715DAY1	0.0374	0.1	2.0				0.749		0.0749
F52738DAY1	0.0471	0.1	2.0				0.942		0.0942
F52809DAY1	0.0561	0.1	2.0				1.12		0.112
F52724DAY1	0.0433	0.1	2.0				0.866		0.0866
F52737DAY1	0.0435	0.1	2.0				0.870		0.0870
F52801DAY1	0.0381	0.1	2.0				0.761		0.0761
F52720DAY1	0.0322	0.1	2.0				0.644		0.0644
62ppm-1	0.0501	0.1	2.0	0.004	62	67%	1.00	0.149	0.100
62ppm-2	0.0838	0.1	2.0	0.004	62	113%	1.68	0.149	0.168
250ppm-1	0.134	0.1	2.0	0.004	250	45%	2.68	0.600	0.268
250ppm-2	0.233	0.1	2.0	0.004	250	78%	4.67	0.600	0.467
BLANK	0.123	0.1	2.0				2.46		0.246
BLANK	0.0323	0.1	2.0				0.646		0.0646
SERUM BLANK	0.0478	0.1	2.0				0.956		0.0956
SERUM BLANK	0.0524	0.1	2.0				1.05		0.105
SERUM BLANK	0.0709	0.1	2.0				1.42		0.142
SERUM BLANK	0.0612	0.1	2.0				1.22		0.122
SERUM BLANK	0.0542	0.1	2.0				1.08		0.108
SERUM BLANK	0.0542	0.1	2.0				1.08		0.108

STUDY # 6329-152 SE.UM

Sample ID	Actual reading (ppm F-)	Sample Qty (mL or g)	TISAB final vol (mL)	mL FC95 spiked	Conc. FC95 solution (ppm)	% recovery (ug/ug)	Actual ppm F- in sample	Mass spiked (ug F-)	Mass recovered (ug F-)
SERUM BLANK	0.0636	0.1	2.0				1.27		0.127
SERUM BLANK	0.0540	0.1	2.0				1.08		0.108
SERUM BLANK	0.0526	0.1	2.0				1.05		0.105
SERUM BLANK	0.0585	0.1	2.0				1.17		0.117
SERUM BLANK	0.0464	0.1	2.0				0.928		0.0928
SERUM BLANK	0.0369	0.1	2.0				0.737		0.0737
SERUM BLANK	0.0318	0.1	2.0				0.636		0.0636
SPIKE 62-1	0.0604	0.1	2.0	0.004	62	81%	1.21	0.149	0.121
SPIKE 62-2	0.0610	0.1	2.0	0.004	62	82%	1.22	0.149	0.122
SPIKE 250-1	0.177	0.1	2.0	0.004	250	59%	3.54	0.600	0.354
SPIKE 250-2	0.184	0.1	2.0	0.004	250	61%	3.67	0.600	0.367
SPIKE 250-3	0.211	0.1	2.0	0.004	250	70%	4.22	0.600	0.422
SPIKE 250-4	0.204	0.1	2.0	0.004	250	68%	4.08	0.600	0.408
BLANK	0.170	0.1	2.0				3.41		0.341
BLANK	0.0644	0.1	2.0				1.29		0.129
BLANK	0.0476	0.1	2.0				0.952		0.0952
BLANK	0.0413	0.1	2.0				0.825		0.0825
BLANK	0.0346	0.1	2.0				0.692		0.0692
BLANK	0.0274	0.1	2.0				0.547		0.0547
F52733-DAY1	0.0388	0.1	2.0				0.775		0.0775
F52788-DAY1	0.0168	0.1	2.0				0.337		0.0337
F52730-DAY1	0.0392	0.1	2.0				0.785		0.0785
F52731-DAY1	0.0386	0.1	2.0				0.772		0.0772
F52802-DAY1	0.0427	0.1	2.0				0.854		0.0854
F52714-DAY1	0.0355	0.1	2.0				0.710		0.0710
F52734-DAY1	0.0386	0.1	2.0				0.772		0.0772
F52774-DAY1	0.0462	0.1	2.0				0.924		0.0924
F52712-DAY1	0.0467	0.1	2.0				0.934		0.0934
F52729-DAY1	0.0343	0.1	2.0				0.686		0.0686
62-PPM-1	0.0604	0.1	2.0	0.004	62	81%	1.21	0.149	0.121
62-PPM-2	0.0768	0.1	2.0	0.004	62	103%	1.54	0.149	0.154
62-PPM-3	0.104	0.1	2.0	0.004	62	139%	2.07	0.149	0.207
250-PPM-1	0.226	0.1	2.0	0.004	250	75%	4.53	0.600	0.453
BLANK	0.0983	0.1	2.0				1.97		0.197

STUDY # 6329-152 SERUM

Sample ID	Actual reading (ppm F-)	Sample Qty (mL or g)	TISAB final vol (mL)	mL FC95 spiked	Conc. FC95 solution (ppm)	% recovery (ug/ug)	Actual ppm F- in sample	Mass spiked (ug F-)	Mass recovered (ug F-)
F52803-DAY1	0.0474	0.1	2.0				0.948		0.0948
F52716-DAY1	0.0482	0.1	2.0				0.964		0.0964
F52728-DAY1	0.0529	0.1	2.0				1.06		0.106
F52787-DAY1	0.0445	0.1	2.0				0.890		0.0890
F52717-DAY1	0.0636	0.1	2.0				1.27		0.127
F52735-DAY1	0.0547	0.1	2.0				1.09		0.109
F52736-DAY1	0.0447	0.1	2.0				0.894		0.0894
F52726-DAY1	0.0362	0.1	2.0				0.725		0.0725
F52732-DAY1	0.0538	0.1	2.0				1.08		0.108
F52739-DAY1	0.0338	0.1	2.0				0.675		0.0675
62-PPM-1	0.0636	0.1	2.0	0.004	62	85%	1.27	0.149	0.127
62-PPM-2	0.105	0.1	2.0	0.004	62	141%	2.09	0.149	0.209
250-PPM-1	0.286	0.1	2.0	0.004	250	95%	5.73	0.600	0.573
250-PPM-2	0.272	0.1	2.0	0.004	250	91%	5.44	0.600	0.544
SERUM BLANK	0.0766	0.1	2.0				1.53		0.153
SERUM BLANK	0.0844	0.1	2.0				1.69		0.169
SERUM BLANK	0.0673	0.1	2.0				1.35		0.135
SERUM BLANK	0.0606	0.1	2.0				1.21		0.121
SERUM BLANK	0.0588	0.1	2.0				1.18		0.118
SERUM BLANK	0.0621	0.1	2.0				1.24		0.124
SERUM BLANK	0.0616	0.1	2.0				1.23		0.123
SERUM BLANK	0.0571	0.1	2.0				1.14		0.114
SERUM BLANK	0.0469	0.1	2.0				0.937		0.0937
SERUM BLANK	0.0586	0.1	2.0				1.17		0.117
SERUM BLANK	0.0422	0.1	2.0				0.844		0.0844
SERUM BLANK	0.0358	0.1	2.0				0.716		0.0716
SERUM BLANK	0.0363	0.1	2.0				0.725		0.0725
SPIKE 62-1	0.104	0.1	2.0	0.004	62	139%	2.07	0.149	0.207
SPIKE 62-2	0.0708	0.1	2.0	0.004	62	95%	1.42	0.149	0.142
SPIKE 62-3	0.0881	0.1	2.0	0.004	62	118%	1.76	0.149	0.176
SPIKE 250-1	0.162	0.1	2.0	0.004	250	54%	3.24	0.600	0.324
SPIKE 250-2	0.267	0.1	2.0	0.004	250	89%	5.34	0.600	0.534
SPIKE 250-3	0.245	0.1	2.0	0.004	250	81%	4.89	0.600	0.489
BLANK	0.140	0.1	2.0				2.81		0.281

STUDY # 6329-152 SERUM

Sample ID	Actual reading (ppm F-)	Sample Qty (mL or g)	TISAB final vol (mL)	mL FC95 spiked	Conc. FC95 solution (ppm)	% recovery (ug/ug)	Actual ppm F- in sample	Mass spiked (ug F-)	Mass recovered (ug F-)
BLANK	0.0564	0.1	2.0				1.13		0.113
BLANK	0.0406	0.1	2.0				0.812		0.0812
F52711-DAY1	0.0296	0.1	2.0				0.593		0.0593
F52719-DAY1	0.0253	0.1	2.0				0.505		0.0505
F52725-DAY1	0.0266	0.1	2.0				0.531		0.0531
F52721-DAY1	0.0222	0.1	2.0				0.443		0.0443
F52727-DAY1	0.0204	0.1	2.0				0.408		0.0408
F52806-DAY1	0.0236	0.1	2.0				0.472		0.0472
F52711-DAY2	0.0191	0.1	2.0				0.381		0.0381
F52719-DAY2	0.0169	0.1	2.0				0.337		0.0337
F52725-DAY2	0.0148	0.1	2.0				0.296		0.0296
F52721-DAY2	0.0128	0.1	2.0				0.257		0.0257
SPIKE 62-1	0.0922	0.1	2.0	0.004	62	124%	1.84	0.149	0.184
SPIKE 250-1	0.281	0.1	2.0	0.004	250	94%	5.62	0.600	0.562
SERUM BLANK	0.0668	0.1	2.0				1.34		0.134
SERUM BLANK	0.0515	0.1	2.0				1.03		0.103
SERUM BLANK	0.0787	0.1	2.0				1.57		0.157
SERUM BLANK	0.0452	0.1	2.0				0.904		0.0904
SERUM BLANK	0.0484	0.1	2.0				0.967		0.0967
SERUM BLANK	0.0490	0.1	2.0				0.979		0.0979
SERUM BLANK	0.0526	0.1	2.0				1.05		0.105
SERUM BLANK	0.0381	0.1	2.0				0.761		0.0761
SERUM BLANK	0.0407	0.1	2.0				0.814		0.0814
SPIKE 62-1	0.0528	0.1	2.0	0.004	62	71%	1.06	0.149	0.106
SPIKE 62-2	0.0742	0.1	2.0	0.004	62	100%	1.48	0.149	0.148
SPIKE 62-3	0.0688	0.1	2.0	0.004	62	92%	1.38	0.149	0.138
SPIKE 62-4	0.0842	0.1	2.0	0.004	62	113%	1.68	0.149	0.168
SPIKE 250-1	0.149	0.1	2.0	0.004	250	49%	2.97	0.600	0.297
SPIKE 250-2	0.258	0.1	2.0	0.004	250	86%	5.15	0.600	0.515
SPIKE 250-3	0.241	0.1	2.0	0.004	250	80%	4.82	0.600	0.482
BLANK	0.137	0.1	2.0				2.73		0.273
BLANK	0.0685	0.1	2.0				1.37		0.137
BLANK	0.0492	0.1	2.0				0.984		0.0984
BLANK	0.0394	0.1	2.0				0.787		0.0787

STUDY #6329-152 SERUM

Sample ID	Actual reading (ppm F-)	Sample Qty (mL or g)	TISAB final vol (mL)	mL FC95 spiked	Conc. FC95 solution (ppm)	% recovery (ug/ug)	Actual ppm F- in sample	Mass spiked (ug F-)	Mass recovered (ug F-)
BLANK	0.0407	0.1	2.0				0.814		0.0814
BLANK	0.0310	0.1	2.0				0.619		0.0619
F52727 DAY2	0.0263	0.1	2.0				0.526		0.0526
F52806 DAY2	0.0297	0.1	2.0				0.594		0.0594
F52713 DAY2	0.0200	0.1	2.0				0.400		0.0400
F52718 DAY2	0.0201	0.1	2.0				0.402		0.0402
F52723 DAY2	0.0230	0.1	2.0				0.460		0.0460
F52715 DAY2	0.0205	0.1	2.0				0.410		0.0410
F52738 DAY2	0.0191	0.1	2.0				0.382		0.0382
F52809 DAY2	0.0192	0.1	2.0				0.384		0.0384
F52724 DAY2	0.0185	0.1	2.0				0.369		0.0369
F52737 DAY2	0.0186	0.1	2.0				0.371		0.0371
SPIKE 62-1	0.0389	0.1	2.0	0.004	62	52%	0.777	0.149	0.0777
SPIKE 62-2	0.0546	0.1	2.0	0.004	62	73%	1.09	0.149	0.109
SPIKE 250-1	0.118	0.1	2.0	0.004	250	39%	2.37	0.600	0.237
SPIKE 250-2	0.223	0.1	2.0	0.004	250	74%	4.45	0.600	0.445
BLANK	0.103	0.1	2.0				2.06		0.206
SERUM BLANK	0.0522	0.1	2.0				1.04		0.104
SERUM BLANK	0.0474	0.1	2.0				0.948		0.0948
SERUM BLANK	0.0420	0.1	2.0				0.840		0.0840
SERUM BLANK	0.0330	0.1	2.0				0.659		0.0659
SERUM BLANK	0.0299	0.1	2.0				0.599		0.0599
SPIKE 62-1	0.0517	0.1	2.0	0.004	62	69%	1.03	0.149	0.103
SPIKE 62-2	0.0747	0.1	2.0	0.004	62	100%	1.49	0.149	0.149
SPIKE 62-3	0.0763	0.1	2.0	0.004	62	102%	1.53	0.149	0.153
SPIKE 250-1	0.112	0.1	2.0	0.004	250	37%	2.23	0.600	0.223
SPIKE 250-2	0.179	0.1	2.0	0.004	250	60%	3.57	0.600	0.357
SPIKE 250-3	0.197	0.1	2.0	0.004	250	66%	3.94	0.600	0.394
SPIKE 250-4	0.194	0.1	2.0	0.004	250	65%	3.89	0.600	0.389
SPIKE 250-5	0.223	0.1	2.0	0.004	250	74%	4.46	0.600	0.446
SPIKE 250-6	0.330	0.1	2.0	0.004	250	110%	6.60	0.600	0.660
BLANK	0.102	0.1	2.0				2.04		0.204
BLANK	0.0411	0.1	2.0				0.822		0.0822
BLANK	0.0185	0.1	2.0				0.370		0.0370

STUDY # 6329-152 SERUM

Sample ID	Actual reading (ppm F-)	Sample Qty (mL or g)	TISAB final vol (mL)	mL FC95 spiked	Conc. FC95 solution (ppm)	% recovery (ug/ug)	Actual ppm F- in sample	Mass spiked (ug F-)	Mass recovered (ug F-)
F52801-DAY2	0.0304	0.1	2.0				0.609		0.0609
F52720-DAY2	0.0232	0.1	2.0				0.464		0.0464
F52733-DAY2	0.0225	0.1	2.0				0.451		0.0451
F52788-DAY2	0.0255	0.1	2.0				0.511		0.0511
F52730-DAY2	0.0236	0.1	2.0				0.472		0.0472
F52731-DAY2	0.0186	0.1	2.0				0.372		0.0372
F52802-DAY2	0.0241	0.1	2.0				0.482		0.0482
F52714-DAY2	0.0258	0.1	2.0				0.515		0.0515
F52734-DAY2	0.0217	0.1	2.0				0.434		0.0434
F52774-DAY2	0.0217	0.1	2.0				0.434		0.0434
SPIKE 62-1	0.0673	0.1	2.0	0.004	62	90%	1.35	0.149	0.135
SPIKE 250-1	0.115	0.1	2.0	0.004	250	38%	2.30	0.600	0.230
SPIKE 250-2	0.215	0.1	2.0	0.004	250	72%	4.31	0.600	0.431
SERUM BLANK	0.0910	0.1	2.0				1.82		0.182
SERUM BLANK	0.0568	0.1	2.0				1.14		0.114
SERUM BLANK	0.0451	0.1	2.0				0.903		0.0903
SERUM BLANK	0.0438	0.1	2.0				0.877		0.0877
SERUM BLANK	0.0467	0.1	2.0				0.934		0.0934
SERUM BLANK	0.0435	0.1	2.0				0.870		0.0870
SERUM BLANK	0.0436	0.1	2.0				0.872		0.0872
SERUM BLANK	0.0335	0.1	2.0				0.670		0.0670
SERUM BLANK	0.0352	0.1	2.0				0.704		0.0704
SPIKE 62-1	0.0395	0.1	2.0	0.004	62	53%	0.790	0.149	0.0790
SPIKE 62-2	0.0631	0.1	2.0	0.004	62	85%	1.26	0.149	0.126
SPIKE 62-3	0.0926	0.1	2.0	0.004	62	124%	1.85	0.149	0.185
SPIKE 250-1	0.0998	0.1	2.0	0.004	250	33%	2.00	0.600	0.200
SPIKE 250-2	0.198	0.1	2.0	0.004	250	66%	3.96	0.600	0.396
SPIKE 250-3	0.256	0.1	2.0	0.004	250	85%	5.13	0.600	0.513
SPIKE 250-4	0.217	0.1	2.0	0.004	250	72%	4.34	0.600	0.434
BLANK	0.180	0.1	2.0				3.60		0.360
BLANK	0.0994	0.1	2.0				1.99		0.199
BLANK	0.0783	0.1	2.0				1.57		0.157
BLANK	0.0525	0.1	2.0				1.05		0.105
BLANK	0.0465	0.1	2.0				0.930		0.0930

STUDY #6329-152 SERUM

Sample ID	Actual reading (ppm F-)	Sample Qty (mL or g)	TISAB final vol (mL)	mL FC95 spiked	Conc. FC95 solution (ppm)	% recovery (ug/ug)	Actual ppm F- in sample	Mass spiked (ug F-)	Mass recovered (ug F-)
BLANK	0.0417	0.1	2.0				0.834		0.0834
BLANK	0.0385	0.1	2.0				0.770		0.0770
F52712 DAY2	0.0325	0.1	2.0				0.649		0.0649
F52729 DAY2	0.0229	0.1	2.0				0.457		0.0457
F52803 DAY2	0.0224	0.1	2.0				0.448		0.0448
F52716 DAY2	0.0209	0.1	2.0				0.419		0.0419
F52728 DAY2	0.0275	0.1	2.0				0.549		0.0549
F52787 DAY2	0.0268	0.1	2.0				0.536		0.0536
F52717 DAY2	0.0255	0.1	2.0				0.510		0.0510
SPIKE 62-1	0.0356	0.1	2.0	0.004	62	48%	0.712	0.149	0.0712
SPIKE 62-2	0.0697	0.1	2.0	0.004	62	94%	1.39	0.149	0.139
SPIKE250-1	0.0820	0.1	2.0	0.004	250	27%	1.64	0.600	0.164
SPIKE250-2	0.221	0.1	2.0	0.004	250	74%	4.41	0.600	0.441
SPIKE250-3	0.182	0.1	2.0	0.004	250	60%	3.63	0.600	0.363
SERUM BLANK	0.114	0.1	2.0				2.28		0.228
SERUM BLANK	0.0836	0.1	2.0				1.67		0.167
SERUM BLANK	0.0620	0.1	2.0				1.24		0.124
SERUM BLANK	0.0521	0.1	2.0				1.04		0.104
SERUM BLANK	0.0426	0.1	2.0				0.852		0.0852
SERUM BLANK	0.0375	0.1	2.0				0.751		0.0751
SERUM BLANK	0.0341	0.1	2.0				0.682		0.0682
SPIKE 62-1	0.0471	0.1	2.0	0.004	62	63%	0.943	0.149	0.0943
SPIKE 62-2	0.0592	0.1	2.0	0.004	62	79%	1.18	0.149	0.118
SPIKE 62-3	0.0481	0.1	2.0	0.004	62	65%	0.963	0.149	0.0963
SPIKE 62-4	0.0574	0.1	2.0	0.004	62	77%	1.15	0.149	0.115
SPIKE 62-5	0.0469	0.1	2.0	0.004	62	63%	0.939	0.149	0.0939
SPIKE 250-1	0.0871	0.1	2.0	0.004	250	29%	1.74	0.600	0.174
SPIKE 250-2	0.167	0.1	2.0	0.004	250	56%	3.33	0.600	0.333
SPIKE 250-3	0.184	0.1	2.0	0.004	250	61%	3.69	0.600	0.369
SPIKE 250-4	0.187	0.1	2.0	0.004	250	62%	3.73	0.600	0.373
SPIKE 250-5	0.162	0.1	2.0	0.004	250	54%	3.24	0.600	0.324
SPIKE 250-6	0.196	0.1	2.0	0.004	250	65%	3.91	0.600	0.391
SPIKE 124-1	0.171	0.1	2.0	0.004	124	115%	3.42	0.298	0.342
SPIKE 124-2	0.0473	0.1	2.0	0.004	124	32%	0.946	0.298	0.0946

STUDY # 6329-152 SER.M

Sample ID	Actual reading (ppm F-)	Sample Qty (mL or g)	TISAB final vol (mL)	mL FC95 spiked	Conc. FC95 solution (ppm)	% recovery (ug/ug)	Actual ppm F- in sample	Mass spiked (ug F-)	Mass recovered (ug F-)
SPIKE 124-3	0.0133	0.1	2.0	0.004	124	9%	0.265	0.298	0.0265
SPIKE 124-5	0.161	0.1	2.0	0.004	124	108%	3.21	0.298	0.321
SPIKE 124-6	0.155	0.1	2.0	0.004	124	104%	3.09	0.298	0.309
SERUM BLANK	0.143	0.1	2.0				2.85		0.285
SERUM BLANK	0.0822	0.1	2.0				1.64		0.164
SERUM BLANK	0.0677	0.1	2.0				1.35		0.135
SERUM BLANK	0.0383	0.1	2.0				0.767		0.0767
SERUM BLANK	0.0366	0.1	2.0				0.732		0.0732
SERUM BLANK	0.0272	0.1	2.0				0.545		0.0545
SPIKE 62-1	0.0431	0.1	2.0	0.004	62	58%	0.863	0.149	0.0863
SPIKE 62-2	0.0855	0.1	2.0	0.004	62	115%	1.71	0.149	0.171
SPIKE 62-3	0.0877	0.1	2.0	0.004	62	118%	1.75	0.149	0.175
SPIKE 250-1	0.0769	0.1	2.0	0.004	250	26%	1.54	0.600	0.154
SPIKE 250-2	0.217	0.1	2.0	0.004	250	72%	4.35	0.600	0.435
SPIKE 250-3	0.186	0.1	2.0	0.004	250	62%	3.72	0.600	0.372
SPIKE 124-1	0.179	0.1	2.0	0.004	124	120%	3.58	0.298	0.358
SPIKE 124-2	0.182	0.1	2.0	0.004	124	122%	3.64	0.298	0.364
BLANK	0.0966	0.1	2.0				1.93		0.193
BLANK	0.0395	0.1	2.0				0.790		0.0790
F52726 DAY 2***	0.0264	0.1	2.0				0.529		0.0529
F52735 DAY 2***	0.0225	0.1	2.0				0.450		0.0450
F52732 DAY 2***	0.0208	0.1	2.0				0.416		0.0416
F52736 DAY 2***	0.0179	0.1	2.0				0.357		0.0357
F52739 DAY 2***	0.0177	0.1	2.0				0.354		0.0354
F52711 DAY 4***	0.0164	0.1	2.0				0.328		0.0328
F52721 DAY 4***	0.0146	0.1	2.0				0.293		0.0293
F52719 DAY 4***	0.0171	0.1	2.0				0.342		0.0342
F52727 DAY 4***	0.0168	0.1	2.0				0.335		0.0335
F52725 DAY 4***	0.0170	0.1	2.0				0.341		0.0341
SPIKE 62-1	0.0407	0.1	2.0	0.004	62	55%	0.813	0.149	0.0813
SPIKE 124-1	0.130	0.1	2.0	0.004	124	88%	2.61	0.298	0.261
BLANK	0.0311	0.1	2.0				0.623		0.0623
BLANK	0.0291	0.1	2.0				0.582		0.0582
62PPM-1	0.0407	0.1	2.0	0.004	62	55%	0.814	0.149	0.0814

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STUDY #6329-152 SERUM

Sample ID	Actual reading (ppm F-)	Sample Qty (mL or g)	TISAB final vol (mL)	mL FC95 spiked	Conc. FC95 solution (ppm)	% recovery (ug/ug)	Actual ppm F- in sample	Mass spiked (ug F-)	Mass recovered (ug F-)
62PPM-2	0.0613	0.1	2.0	0.004	62	82%	1.23	0.149	0.123
62PPM-3	0.0798	0.1	2.0	0.004	62	107%	1.60	0.149	0.160
250PPM -1	0.141	0.1	2.0	0.004	250	47%	2.81	0.600	0.281
250PPM -2	0.223	0.1	2.0	0.004	250	74%	4.45	0.600	0.445
250PPM -3	0.267	0.1	2.0	0.004	250	89%	5.35	0.600	0.535
BLANK	0.0865	0.1	2.0				1.73		0.173
BLANK	0.0582	0.1	2.0				1.16		0.116
BLANK	0.0213	0.1	2.0				0.426		0.0426
F52726 DAY2	0.0326	0.1	2.0				0.653		0.0653
F52732 DAY2	0.0169	0.1	2.0				0.337		0.0337
F52739 DAY2	0.0176	0.1	2.0				0.352		0.0352
F52735 DAY2	0.0236	0.1	2.0				0.472		0.0472
F52736 DAY2	0.0246	0.1	2.0				0.493		0.0493
F52711 DAY2	0.0064	0.1	2.0				0.128		0.0128
BLANK	0.0888	0.1	2.0				1.78		0.178
BLANK	0.150	0.1	2.0				3.00		0.300
BLANK	0.0251	0.1	2.0				0.501		0.0501
BLANK	0.0175	0.1	2.0				0.350		0.0350
SPIKE 62-1	0.0556	0.1	2.0	0.004	62	75%	1.11	0.149	0.111
SPIKE 62-2	0.0570	0.1	2.0	0.004	62	77%	1.14	0.149	0.114
SPIKE 250-1	0.133	0.1	2.0	0.004	250	44%	2.66	0.600	0.266
SPIKE 250-2	0.138	0.1	2.0	0.004	250	46%	2.76	0.600	0.276
SPIKE 250-3	0.159	0.1	2.0	0.004	250	53%	3.18	0.600	0.318
SPIKE 124-1	0.122	0.1	2.0	0.004	124	82%	2.43	0.298	0.243
SPIKE 124-2	0.121	0.1	2.0	0.004	124	81%	2.42	0.298	0.242
BLANK	0.0568	0.1	2.0				1.14		0.114
BLANK	0.0457	0.1	2.0				0.913		0.0913
F52711 DAY4	0.0297	0.1	2.0				0.594		0.0594
F52719 DAY4	0.0259	0.1	2.0				0.518		0.0518
SPIKE 62-1	0.0563	0.1	2.0	0.004	62	76%	1.13	0.149	0.113
SPIKE 124-1	0.125	0.1	2.0	0.004	124	84%	2.51	0.298	0.251
BLANK	0.0673	0.1	2.0				1.35		0.135
BLANK	0.0302	0.1	2.0				0.603		0.0603
BLANK	0.0334	0.1	2.0				0.668		0.0668

STUDY # 6329-152 SERUM 1

Sample ID	Actual reading (ppm F-)	Sample Qty (mL or g)	TISAB final vol (mL)	mL FC95 spiked	Conc. FC95 solution (ppm)	% recovery (ug/ug)	Actual ppm F- in sample	Mass spiked (ug F-)	Mass recovered (ug F-)
SPIKE 62-1	0.0317	0.1	2.0	0.004	62	43%	0.635	0.149	0.0635
SPIKE 62-2	0.0656	0.1	2.0	0.004	62	88%	1.31	0.149	0.131
SPIKE 62-3	0.0567	0.1	2.0	0.004	62	76%	1.13	0.149	0.113
SPIKE 250-1	0.0942	0.1	2.0	0.004	250	31%	1.88	0.600	0.188
SPIKE 250-2	0.171	0.1	2.0	0.004	250	57%	3.42	0.600	0.342
SPIKE 250-3	0.180	0.1	2.0	0.004	250	60%	3.60	0.600	0.360
SPIKE 250-4	0.158	0.1	2.0	0.004	250	53%	3.16	0.600	0.316
SPIKE 250-5	0.172	0.1	2.0	0.004	250	57%	3.44	0.600	0.344
SPIKE 124-1	0.153	0.1	2.0	0.004	124	103%	3.06	0.298	0.306
SPIKE 124-2	0.129	0.1	2.0	0.004	124	87%	2.58	0.298	0.258
BLANK	0.0708	0.1	2.0				1.42		0.142
BLANK	0.0293	0.1	2.0				0.585		0.0585
F52725 DAY 4	0.0346	0.1	2.0				0.691		0.0691
F52721 DAY 4	0.0303	0.1	2.0				0.606		0.0606
F52727 DAY 4	0.0248	0.1	2.0				0.496		0.0496
F52806 DAY 4	0.0230	0.1	2.0				0.459		0.0459
F52713 DAY 4	0.0185	0.1	2.0				0.371		0.0371
F52718 DAY 4	0.0295	0.1	2.0				0.590		0.0590
F52723 DAY 4	0.0207	0.1	2.0				0.414		0.0414
F52715 DAY 4	0.0189	0.1	2.0				0.377		0.0377
F52738 DAY 4	0.0414	0.1	2.0				0.827		0.0827
SPIKE 62-1	0.0244	0.1	2.0	0.004	62	33%	0.487	0.149	0.0487
SPIKE 62-2	0.0784	0.1	2.0	0.004	62	105%	1.57	0.149	0.157
SPIKE 124-1	0.126	0.1	2.0	0.004	124	85%	2.52	0.298	0.252
SERUM BLANK	0.0334	0.1	2.0				0.668		0.0668
SERUM BLANK	0.0290	0.1	2.0				0.580		0.0580
SERUM BLANK	0.0239	0.1	2.0				0.478		0.0478
SPIKE 62-1	0.0414	0.1	2.0	0.004	62	56%	0.827	0.149	0.0827
SPIKE 62-2	0.0534	0.1	2.0	0.004	62	72%	1.07	0.149	0.107
SPIKE 62-3	0.0564	0.1	2.0	0.004	62	76%	1.13	0.149	0.113
SPIKE 250-1	0.127	0.1	2.0	0.004	250	42%	2.54	0.600	0.254
SPIKE 250-2	0.223	0.1	2.0	0.004	250	74%	4.45	0.600	0.445
SPIKE 250-3	0.172	0.1	2.0	0.004	250	57%	3.45	0.600	0.345
SPIKE 250-4	0.264	0.1	2.0	0.004	250	88%	5.29	0.600	0.529

STUDY #6329-152 SERUM

Sample ID	Actual reading (ppm F-)	Sample Qty (mL or g)	TISAB final vol (mL)	mL FC95 spiked	Conc. FC95 solution (ppm)	% recovery (ug/ug)	Actual ppm F- in sample	Mass spiked (ug F-)	Mass recovered (ug F-)
SPIKE 250-5	0.230	0.1	2.0	0.004	250	77%	4.60	0.600	0.460
blank	0.177	0.1	2.0				3.54		0.354
blank	0.0331	0.1	2.0				0.663		0.0663
F52809 DAY 4	0.0196	0.1	2.0				0.391		0.0391
F52724 DAY 4	0.0187	0.1	2.0				0.373		0.0373
F52737 DAY 4	0.0164	0.1	2.0				0.328		0.0328
F52801 DAY 4	0.0359	0.1	2.0				0.718		0.0718
F52720 DAY 4	0.0265	0.1	2.0				0.531		0.0531
F52733 DAY 4	0.0218	0.1	2.0				0.436		0.0436
F52788 DAY 4	0.0192	0.1	2.0				0.383		0.0383
F52730 DAY 4	0.0174	0.1	2.0				0.348		0.0348
F52731 DAY 4	0.0149	0.1	2.0				0.298		0.0298
F52802 DAY4	0.0232	0.1	2.0				0.464		0.0464
SPIKE 62-1	0.0325	0.1	2.0	0.004	62	44%	0.649	0.149	0.0649
SPIKE 62-2	0.0722	0.1	2.0	0.004	62	97%	1.44	0.149	0.144
SPIKE 250-1	0.130	0.1	2.0	0.004	250	43%	2.59	0.600	0.259
SPIKE 250-2	0.123	0.1	2.0	0.004	250	41%	2.47	0.600	0.247
SPIKE 250-3	0.199	0.1	2.0	0.004	250	66%	3.98	0.600	0.398
SPIKE 250-4	0.0964	0.1	2.0	0.004	250	32%	1.93	0.600	0.193
SPIKE 250 -5	0.119	0.1	2.0	0.004	250	40%	2.38	0.600	0.238
SPIKE 250-6	0.183	0.1	2.0	0.004	250	61%	3.66	0.600	0.366
SPIKE 250-7	0.138	0.1	2.0	0.004	250	46%	2.76	0.600	0.276
SPIKE 250-8	0.192	0.1	2.0	0.004	250	64%	3.83	0.600	0.383
SPIKE 124-1	0.114	0.1	2.0	0.004	124	76%	2.27	0.298	0.227
BLANK	0.0825	0.1	2.0				1.65		0.165
BLANK	0.0818	0.1	2.0				1.64		0.164
BLANK	0.0578	0.1	2.0				1.16		0.116
BLANK	0.0562	0.1	2.0				1.12		0.112
BLANK	0.0495	0.1	2.0				0.990		0.0990
BLANK	0.0379	0.1	2.0				0.758		0.0758
F52714 DAY 4	0.0290	0.1	2.0				0.580		0.0580
F52734 DAY4	0.0446	0.1	2.0				0.892		0.0892
F52774 DAY 4	0.0285	0.1	2.0				0.570		0.0570
F52712 DAY 4	0.0218	0.1	2.0				0.436		0.0436

STUDY # 6329 - 152 SERUM

Sample ID	Actual reading (ppm F-)	Sample Qty (mL or g)	TISAB final vol (mL)	mL FC95 spiked	Conc. FC95 solution (ppm)	% recovery (ug/ug)	Actual ppm F- in sample	Mass spiked (ug F-)	Mass recovered (ug F-)
F52729 DAY 4	0.0226	0.1	2.0				0.452		0.0452
F52803 DAY 4	0.0237	0.1	2.0				0.474		0.0474
F52716 DAY4	0.0206	0.1	2.0				0.411		0.0411
F52728 DAY 4	0.0191	0.1	2.0				0.381		0.0381
F52787 DAY 4	0.0222	0.1	2.0				0.443		0.0443
SPIKE 62-1	0.0307	0.1	2.0	0.004	62	41%	0.615	0.149	0.0615
SPIKE 62-2	0.0409	0.1	2.0	0.004	62	55%	0.817	0.149	0.0817
SPIKE 62-3	0.0710	0.1	2.0	0.004	62	95%	1.42	0.149	0.142
SPIKE 124-1	0.0571	0.1	2.0	0.004	124	38%	1.14	0.298	0.114
SPIKE 124-2	0.109	0.1	2.0	0.004	124	73%	2.19	0.298	0.219
SERUM BLANK	0.0397	0.1	2.0				0.794		0.0794
SERUM BLANK	0.0379	0.1	2.0				0.758		0.0758
SERUM BLANK	0.0379	0.1	2.0				0.758		0.0758
SERUM BLANK	0.0306	0.1	2.0				0.612		0.0612
SERUM BLANK	0.0277	0.1	2.0				0.554		0.0554
SPIKE 62-1	0.0898	0.1	2.0	0.004	62	121%	1.80	0.149	0.180
SPIKE 62-2	0.0913	0.1	2.0	0.004	62	123%	1.83	0.149	0.183
SPIKE 62-3	0.0769	0.1	2.0	0.004	62	103%	1.54	0.149	0.154
SPIKE 124-1	0.113	0.1	2.0	0.004	124	76%	2.25	0.298	0.225
SPIKE 124-2*	0.0326	0.1	2.0				0.652		0.0652
SPIKE 124-2*	0.270	0.1	2.0				5.40		0.540
SPIKE 124-4	0.121	0.1	2.0	0.004	124	81%	2.41	0.298	0.241
blank	0.0485	0.1	2.0				0.970		0.0970
blank	0.0299	0.1	2.0				0.597		0.0597
F52717 DAY4	0.0493	0.1	2.0				0.987		0.0987
F52735 DAY4	0.0319	0.1	2.0				0.638		0.0638
F52736 DAY4	0.0251	0.1	2.0				0.503		0.0503
F52726 DAY4	0.0256	0.1	2.0				0.511		0.0511
F52732 DAY4	0.0213	0.1	2.0				0.425		0.0425
F52739 DAY4	0.0164	0.1	2.0				0.328		0.0328
F52711 DAY 8	0.0242	0.1	2.0				0.484		0.0484
F52719 DAY 8	0.0164	0.1	2.0				0.328		0.0328
SPIKE 62-1	0.0332	0.1	2.0	0.004	62	45%	0.663	0.149	0.0663
SPIKE 62-2	0.0500	0.1	2.0	0.004	62	67%	0.999	0.149	0.100

STUDY # 6329-152 SERUM

Sample ID	Actual reading (ppm F-)	Sample Qty (mL or g)	TISAB final vol (mL)	mL FC95 spiked	Conc. FC95 solution (ppm)	% recovery (ug/ug)	Actual ppm F- in sample	Mass spiked (ug F-)	Mass recovered (ug F-)
SPIKE 62-3	0.0498	0.1	2.0	0.004	62	67%	0.995	0.149	0.100
SPIKE 62-4	0.0392	0.1	2.0	0.004	62	53%	0.784	0.149	0.0784
SPIKE 62-5	0.126	0.1	2.0	0.004	62	169%	2.52	0.149	0.252
SPIKE 62-6	0.0354	0.1	2.0	0.004	62	48%	0.709	0.149	0.0709
SPIKE 62-7	0.0421	0.1	2.0	0.004	62	57%	0.842	0.149	0.0842
SPIKE 62-8	0.0508	0.1	2.0	0.004	62	68%	1.02	0.149	0.102
SPIKE 62-9	0.0537	0.1	2.0	0.004	62	72%	1.07	0.149	0.107
SPIKE 124-1	0.0710	0.1	2.0	0.004	124	48%	1.42	0.298	0.142
SPIKE 124-2	0.144	0.1	2.0	0.004	124	96%	2.87	0.298	0.287
BLANK	0.0847	0.1	2.0				1.69		0.169
BLANK	0.0392	0.1	2.0				0.784		0.0784
F52725 DAY 8	0.0300	0.1	2.0				0.600		0.0600
F52721 DAY 8	0.0278	0.1	2.0				0.556		0.0556
F52727 DAY 8	0.0230	0.1	2.0				0.461		0.0461
F52806 DAY 8	0.0231	0.1	2.0				0.462		0.0462
F52713 DAY 8	0.0170	0.1	2.0				0.341		0.0341
F52718 DAY 8	0.0175	0.1	2.0				0.349		0.0349
F52723 DAY 8	0.0173	0.1	2.0				0.346		0.0346
F52715 DAY 8	0.0172	0.1	2.0				0.344		0.0344
F52738 DAY 8	0.0165	0.1	2.0				0.331		0.0331
F52809 DAY 8	0.0152	0.1	2.0				0.303		0.0303
SPIKE 62-1	0.0245	0.1	2.0	0.004	62	33%	0.490	0.149	0.0490
SPIKE 62-2	0.0353	0.1	2.0	0.004	62	47%	0.706	0.149	0.0706
SPIKE 62-3	0.0414	0.1	2.0	0.004	62	56%	0.828	0.149	0.0828
SPIKE 62-4	0.0579	0.1	2.0	0.004	62	78%	1.16	0.149	0.116
SPIKE 124-1	0.0925	0.1	2.0	0.004	124	62%	1.85	0.298	0.185
SPIKE 124-2	0.0865	0.1	2.0	0.004	124	58%	1.73	0.298	0.173
SPIKE 124-3	0.1048	0.1	2.0	0.004	124	70%	2.10	0.298	0.210
BLANK	0.0868	0.1	2.0				1.74		0.174
BLANK	0.0414	0.1	2.0				0.828		0.0828
SERUM BLANK	0.0310	0.1	2.0				0.620		0.0620
SERUM BLANK	0.0284	0.1	2.0				0.568		0.0568
SERUM BLANK	0.0195	0.1	2.0				0.390		0.0390
SPIKE 62-1	0.0558	0.1	2.0	0.004	62	75%	1.12	0.149	0.112

507000

STUDY #6329-152 SERUM

Sample ID	Actual reading (ppm F-)	Sample Qty (mL or g)	TISAB final vol (mL)	mL FC95 spiked	Conc. FC95 solution (ppm)	% recovery (ug/ug)	Actual ppm F- in sample	Mass spiked (ug F-)	Mass recovered (ug F-)
SPIKE 62-2	0.0619	0.1	2.0	0.004	62	83%	1.24	0.149	0.124
SPIKE 62-3	0.0604	0.1	2.0	0.004	62	81%	1.21	0.149	0.121
SPIKE 124-1	0.0933	0.1	2.0	0.004	124	63%	1.87	0.298	0.187
SPIKE 124-2	0.0551	0.1	2.0	0.004	124	37%	1.10	0.298	0.110
SPIKE 124-3	0.109	0.1	2.0	0.004	124	73%	2.19	0.298	0.219
SPIKE 124-4	0.109	0.1	2.0	0.004	124	73%	2.17	0.298	0.217
BLANK	0.0837	0.1	2.0				1.67		0.167
BLANK	0.0367	0.1	2.0				0.734		0.0734
BLANK	0.0305	0.1	2.0				0.610		0.0610
F52724 DAY8	0.0289	0.1	2.0				0.578		0.0578
F52737 DAY8	0.0282	0.1	2.0				0.563		0.0563
F52801 DAY8	0.0428	0.1	2.0				0.856		0.0856
F52720 DAY8	0.0394	0.1	2.0				0.788		0.0788
F52733 DAY8	0.0245	0.1	2.0				0.491		0.0491
F52788 DAY8	0.0129	0.1	2.0				0.259		0.0259
spike 62-1	0.0430	0.1	2.0	0.004	62	58%	0.860	0.149	0.0860
spike 62-2	0.0518	0.1	2.0	0.004	62	70%	1.04	0.149	0.104
spike 124-1	0.104	0.1	2.0	0.004	124	70%	2.08	0.298	0.208

* Forgot to spike blank serum with FC-95 therefore, another replicate was analyzed. DP2 (08/17/95)

** Instrument malfunction, the boat did not go all the way into the oven, another replicate was analyzed. DP2 (08/21/95)

*** Samples reanalyzed on following day, due to QC not meeting requirements. DP2 (08/21/95)

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