

FOSA or PFOSA

Ongoing - /

3M MEDICAL DEPARTMENT, CORPORATE TOXICOLOGY
Protocol for Study No. T-7132.2; ST-39
PHARMACOKINETIC STUDY OF PERFLUOROOCTANE SULFONAMIDE
IN RATS

Study Objective:

The objective of this study is to assess the potential for oral absorption, urinary and fecal clearance and biological persistence of Perfluorooctane Sulfonamide (PFOSA) in male Sprague Dawley rats after a single oral dose. Analysis of the serum, liver, urine and feces for potential metabolites of PFOSA will be performed by LCMS and perhaps other methods. Previous studies of the N-ethyl derivative of PFOSA have concluded that PFOSA is the ultimate metabolite in rats (1,2); however, the analytical technique used in these studies was unable to detect all potential metabolites, including perfluorooctanesulfonate (PFOS). No pharmacokinetic studies have yet been done on PFOSA to resolve this issue and search for other possible metabolites in the urine and feces. Recently, validated methods have been developed for the quantitation of PFOSA and its potential metabolite, PFOS, in serum and liver down to the low part per billion level (3). One goal of this study is to determine the potential for, if occurring, the extent of conversion of PFOSA to PFOS. This information will help to explain the pharmacokinetics of NETFOSE and its metabolites and provide data for proper risk characterization.

Research Client: 3M Specialty Chemicals Division
3M Center, Building 236
Saint Paul, MN 55144

Sponsor: 3M Specialty Chemicals Division
3M Center, Building 236
Saint Paul, MN 55144

Study Location: 3M Strategic Toxicology Laboratory
3M Center, Building 270-3S-06 room SB314
Saint Paul, MN 55144

Study Director: Andrew M. Seacat, Ph.D.
Toxicology Specialist
3M Medical Dept. / Corporate Toxicology
3M Center, Building 220-2E-02
Saint Paul, MN 55144
Ph.: 651-575-3161 FAX: 651-733-1773

Study Toxicologist: Deanna Luebker, MS
Advanced Research Toxicologist
3M Medical Dept. / Corporate Toxicology
3M Center, Building 220-2E-02
Saint Paul, MN 55144

000503

Ph: 651-737-1374 FAX: 651-733-1773

Proposed Study Timeline

In-Life Start Date: October 4th, 1999

In-Life End Date: November 2nd, 1999

Analytical Completion Date: TBA

Final Report Completion Date: TBA

Regulatory Compliance:

This study will be performed in the 3M Strategic Toxicology Laboratory under a defined protocol and classified as a "Class B Study" as explained in TOX SOP 0950, Strategic Toxicology Lab GLP Program Procedure.

Test Material:

Dan Hakes, Product Responsibility Liaison 3M Chemicals Division, will furnish high-purity PFOSA.

Identification:

Name: Perfluorooctanesulfonamide

Molecular Formula: $C_8F_{17}SO_2NH_2$

Lot Number: L-10009 (Prepared by George Moore, 3M SMD Lab, Bldg 236, April 1996)

Purity: Analysis by GCMS determined that the starting material was over 99% pure (4). Qualitative and quantitative compositional results that were derived from the single trial $^1H/^{19}F$ -NMR cross-integration analysis revealed that the composition was 65.8% $CF_3(CF_2)_x-SO_2-NH_2$ (Normal chain), 18.7% $CF_3(CF_2)_x-CF(CF_3)-(CF_2)_y-SO_2-NH_2$ (Internal monomethyl branch), 11.2% $(CF_3)_2CF-(CF_2)_x-SO_2-NH_2$ (Isopropyl branch), 3.5% $C_xF_{2x+1}-CF(CF_3)-SO_2-NH_2$ (Alpha branch) and 0.28% $(CF_3)_3C-(CF_2)_x-SO_2-NH_2$ (t-Butyl branch)(5). HPLC/MS characterization of the PFOSA sample revealed 9,600 ppm of PFOS, 1,100 ppm of $C_7F_{15}SO_2NH_2$, 510 ppm of $C_9F_{19}SO_2NH_2$, 6,600 ppm of $C_8F_{16}HSO_2NH_2$, 24,000 ppm of $C_{18}F_{36}HSO_2NH_2$, 1,200 ppm of $C_8F_{15}H_2SO_2NH_2$ and lower concentrations of several other amides. Based on the sum of the impurities, the purity of the PFOSA sample would be approximately 96 % (6 & appendix I).

Stability:

Documentation will be kept on file with the Sponsor.

Storage Conditions:

Upon receipt, test material will be stored tightly sealed at room temperature.

Characteristics:

Information on synthesis methods, composition or other characteristics that define the test material will be kept on file with the Sponsor.

Animals:

Species: Rat
Strain: Sprague Dawley
Source: Harlan
Age at initiation of treatment: 6-8 weeks
Weight at initiation of treatment: approximately 150-250g
Number and sex: 30 males

Table 1 - Dose Groups

Group	Dose	N	Euthanasia
1	0 mg/kg	15	5 each on days 1, 4 and 29 post dose
2	5 mg/kg	15	5 each on days 1, 4 and 29 post dose

Identification: ear tag with animal number or unique tail mark.
AUA Number: 2246

Husbandry:

Housing:

All rats from groups 1 and 2, which are to be sacrificed on day 29 post dose, will be housed individually in metabolism cages for portions of the study (see Table 2). When not in metabolism cages, these rats will be group housed in standard cages. All other rats will be group housed in standard cages throughout the study.

Diet/Water:

Harlan Teklad LM-485 Mouse/Rat Sterilizable Diet, supplied by Harlan Teklad, Madison, WI, and tap water will be provided to all rats *ad libitum* throughout the study.

Environment:

Environmental controls for the animal room will be set to maintain a temperature of $72 \pm 3^{\circ}\text{F}$, humidity of 30-70%, a minimum of 10 exchanges of room air per hour and a 12 hour light/dark cycle.

Dose and Dosing Procedures:

Method of administration/Dose preparation:

A single 5mg/kg dose of PFOSA will be administered via oral gavage to rats in group 2 on day zero of the study. The PFOSA will be prepared from a stock solution of 100mg/ml PFOSA in acetone. A final 0.1% (1mg/ml) uniform suspension (or emulsion) of PFOSA in 2% Tween 80 and 1% acetone will be prepared using a 15 ml dounce tissue grinder. A volume of 5 ml suspension / kg body weight will be administered to each rat. Re-suspension of solids will be performed with 5 strokes of the tissue grinder pestel before each sample is drawn-up in the syringe for dosing. A single 5

ml / kg body weight dose of vehicle, 2% Tween 80 and 1% acetone, will be administered via oral gavage to rats in group 1 on day zero of the study.

Observation of Animals:

Clinical Observations:

Each animal will be observed daily (excluding weekends and holidays) for mortality and morbidity and notable findings will be recorded. Additional findings will be recorded as they are observed.

Body Weights:

Each animal will be weighed immediately prior to treatment, weekly thereafter and immediately prior to euthanasia.

Specimen Collection:

Frequency (See table 2):

Urine and feces collections will be made on days 1 - 4 post dose.

Necropsies will be performed on days 1, 4 and 29 post dose.

Table 2 - Schedule

Sun	Mon	Tues	Wed	Thurs	Fri	Sat
Oct 3	Oct 4 day 0 DOSING	Oct 5 day 1 PD Collection, Dy 1 PD sac	Oct 6 day 2 PD Collection	Oct 7 day 3 PD Collection	Oct 8 day 4 PD Collection Switch to reg cages. Dy 4 PD sac.	Oct 9 day 5 PD
Oct 10 day 6 PD	Oct 11 day 7 PD	Oct 12 day 8 PD	Oct 13 day 9 PD	Oct 14 day 10 PD	Oct 15 day 11 PD	Oct 16 day 12 PD
Oct 17 day 13 PD	Oct 18 day 14 PD	Oct 19 day 15 PD	Oct 20 day 16 PD	Oct 21 day 17 PD	Oct 22 day 18 PD	Oct 23 day 19 PD
Oct 24 day 20 PD	Oct 25 day 21 PD	Oct 26 day 22 PD	Oct 27 day 23 PD	Oct 28 day 24 PD	Oct 29 day 25 PD	Oct 30 day 26 PD
Oct 31 day 27 PD	Nov 1 day 28 PD	Nov 2 day 29 PD Dy 29 PD sac.				

Method of Specimen Collection:

Urine and feces will be collected from each metabolism cage at the designated times. The initial volume of urine will be recorded, the sides of the urine collection apparatus will be washed with approximately 5-10ml deionized water and the final volume of urine will be brought to 15 ml with additional deionized water. Daily feces weight will be recorded for each animal. At the designated times, animals will be euthanized by CO₂ and gross necropsy performed. During necropsy, blood (≈ 6 ml) will be collected via the abdominal aorta and transferred to blood collection tubes without anticoagulant. Blood samples will be allowed to clot for a period

of 15 to 30 minutes at room temperature and the clot will be spun down in a centrifuge at 1100 x g for 5 minutes. The serum will be transferred to labeled 1.5 ml microfuge tubes and centrifuged again at 2000 x g to remove any remaining red blood cells. Each sera sample will then be transferred to a separate labeled polypropylene microfuge tube and frozen in dry ice. Livers will be removed, weighed, placed individually into labeled sterile sample bags and flash frozen in liquid nitrogen then maintained on dry ice.

Specimen Handling:

Specimens will temporarily be stored in a freezer set to maintain -60 to -80°C. For metabolite analysis, these specimens will be packed in dry ice and shipped to:

Kris Hansen, Ph.D.
3M Environmental Technology and Safety Services
935 Bush Avenue
St. Paul, MN 55133-3331
Ph: 612-778-6081, FAX: 612-778-6176.

All results will be provided for inclusion in the final report.

The number, type and date of collection of specimens to be generated for analysis are as follows:

Table 3 - Specimens

<u>Specimen</u>	day 1 post dose	day 2 post dose	Day 3 post dose	day 4 post dose	day 29 post dose	<u>Total</u>
Serum (5/group/day)	10			10	10	30
Liver (5/group/day)	10			10	10	30
Urine (5/group/day)	10	10	10	10		40
Feces (5/group/day)	10	10	10	10		40

Data Analysis:

Data collected on parent compound and identifiable metabolites will be analyzed for toxicokinetic parameters and for statistically significant differences between groups using ANOVA and /or Students T-test.

Responsibilities:

- Deanna Luebker and Andrew Seacat will be responsible for dosing the animals, collecting in-life specimens, performing the necropsies and collecting and sending tissue specimens for analysis.
- Kris Hansen, 3M Environmental, will be responsible for analytical evaluation of the
- Andrew Seacat will draft a final report and ensure the report receives appropriate 3M review before a final report is issued.

000508

Signatures:

Andrew M. Seacat 10/27/99
Andrew M. Seacat Ph.D.
Toxicology Specialist
Study Director
Date

Deanna Luebker 10/27/99
Deanna Luebker, MS
Advanced Research Toxicologist
Study Toxicologist
Date

Daniel A. Hoot 11/1/99
Sponsor Representative
Date

000509

References:

1. Grossman M.R. and Bowen J.M. (1990) Tissue analysis of fluorinated sulfonamide pesticide: an evaluation of distribution, elimination, and potential for bioaccumulation in orally exposed rats. M.S. Thesis, Univ. of Georgia, Athens, GA. (also possibly published as: Grossman Mark R. and Bowen J.M. (1990) Tissue distribution and elimination of a fluorinated sulfonamide pesticide in rats. *Fundam. Appl. Toxicol.*, but not found).
2. Grossman M.R., Mispagel, M.E. and Bowen J.M. (1992) Distribution and tissue in rats during and after prolonged dietary exposure to a highly fluorinated sulfonamide pesticide. *J. Agric. Food Chem.* 40, 2505 – 2509.
3. K.J. Hansen, L.A. Clemen, M.E. Ellefson, H.O. Johnson. (1999). Compound Specific Characterization of Organic Fluorochemicals in General Population Human Sera Samples. 3M Environmental Lab, St. Paul, MN 55133.
4. Payfer R.M. GC/MS analyses of PFOSA (L-10009). SA&C Analytical Request No. 59426. Report 9/24/99. 3M SA&C Lab Building 236-2B-11.
5. Tom Kestner Chemical Characterization of PFOSA, L-10009, by 1H and 19F-NMR Spectroscopy Requests # 59426. 3M Specialty Adhesives & Chemicals Analytical Laboratory / SMMD-236-2B-11, September 25, 1999.
6. DeRoos F.L. Characterization of PFOSA Samples, T-7132-1 (L-10009) and TN-A-1584. Request # A-151254. Report 10/7/99. Corporate Analytical Technology Center, Building 201-1-29, CATC – Chromatography Group.

Appendix I:

Tel: 736-0665
201-1W-29

Corporate Analytical Technology Center

To: Larry A. Wendling/US-Corporate/3M/US
cc: Andrew Seacat/US-Corporate/3M/US
Subject: Characterization of PFOSA Sample for Toxicology

Larry,

I have completed the HPLC/MS characterization of the PFOSA sample (T-7132-, L-10009 prepared by G. Moore 4/96) that is proposed to be used for the animal feeding study. I found 9,600 ppm of PFOS, 1,100 ppm of $C_7F_{15}SO_2NH_2$, 510 ppm of $C_9F_{19}SO_2NH_2$, 6,600 ppm of $C_8F_{16}HSO_2NH_2$, 24,000 ppm of $C_{18}F_{36}HSO_2NH_2$, 1,200 ppm of $C_8F_{15}H_2SO_2NH_2$ and lower concentrations of several other amides. Based on the sum of the impurities, the purity of the PFOSA sample would be approximately 96 %.

I've thought a little more about apparent presence of $C_{18}F_{36}HSO_2NH_2$ in the sample. The identification of this compound was based primarily on the observed molecular weight and the relative elution order in the chromatogram. Rather than $C_{18}F_{36}HSO_2NH_2$, it seems more probable that this compound is actually $(C_8F_{17}SO_2)_2NH$. Confirmation of this identification will require additional analyses. I also need to think more about whether the other partially hydrogenated perfluoroamides may actually be of this

The PFOS was quantified using a standard curve prepared by analyzing PFOS standards so its concentration should be accurate. The amide impurities, however, including the partially hydrogenated amides, were all quantified using a PFOSA standard curve assuming that they had the same mass spectral response factor as did PFOSA. While this assumption will introduce some error to the quantitative data, it is the best we can do since we do not have standards for each of these amides. Also, this calculated purity assumes that all of the impurities are amenable to HPLC and are detected by the analysis. While not always true, this sample was analyzed by Rick Payfer using GC/MS and by CATC using our GC method. Neither of the analyses found volatile or semivolatile impurities, e.g., N-methyl FOSE, N-ethyl FOSE, etc. at concentrations greater than 50 ppm.

I also carried out a semiquantitative assessment of the purity of the PFOSA by comparing the PFOSA response to a PFOSA standard curve. Using this technique, the purity of the PFOSA sample was found to be 118 %. These analyses were carried out in duplicate, with triplicate injections of each solution, so they should be relatively accurate as analytical variation would be averaged. In general, however, it is not highly accurate to use a chromatographic method to quantify a relatively pure material due to the extremely large dilution factor, in this case >100,000, that must be applied. It is not expected that instrumental variation would bias the purity high. It is possible that the purity of the sample is actually higher than the purity of the PFOSA that we are using as a standard! If this were true, the purity would be determined to be > 100%.

Andrew Seacat has reviewed the concentrations of the impurities that were determined and calculated that the concentration of PFOS will not adversely affect the study. At 9,600 ppm, he calculates that the rats receiving 3 mg/kg/day would ingest app. 0.20 mg PFOS total in

Appendix I Continued:

28 days assuming a 0.25 kg rat. If we further assume that 30 % of that is deposited in the liver (based on previous feeding studies) and liver is approximately 12 grams, then the predicted concentration in the liver from the residual PFOS would be $200 \text{ ug PFOS} \times 0.3 / 12 \text{ g liver weight} = 5 \text{ ug/gram}$, or 5 ppm, which in itself should not impose any toxicological consequences. Any PFOS measured significantly over that could be attributed to metabolism of PFOSA to PFOS.

He believes that the 1-H and 2-H perfluoro amides are interesting as they may be subject to metabolic attack, however these amides should be analytically distinct from the Perfluorinated species on down the line, and would not amount to a foreseeable toxic concentration and therefore would be acceptable if no other substitute can be prepared. Andrew is still thinking about whether the $(\text{C}_8\text{F}_{17}\text{SO}_2)_2\text{NH}$ will have a negative impact on the study.

Please give me a call at 6-0665 if you have any questions concerning our analyses or if i can provide additional information.

Fred L. DeRoos
CATC

Laboratory Report
Analytical Report of Data for
PFOSA Pharmacokinetic Study in Rats
(Sera and Liver)

Laboratory Report No. FACT-TOX-145 (W2814)

Requester Project No. T-7132.2

Testing Laboratory

3M Environmental Technology & Safety Services
3M Environmental Laboratory
Fluorine Analytical Chemistry Team (FACT)
2-3E-09
935 Bush Avenue,
St. Paul, MN 55106

Laboratory Contact

Kris Hansen, Ph.D.
Bldg. 2-3E-09
P.O. Box 3331
St. Paul, MN 55133-3331
Phone: (651) 778-6018
FAX: (651) 778-6176

Requester

Andrew Seacat and Deanna Luebker
3M Company
Toxicology Services
220-2E-02
St. Paul, MN

1 Introduction

This study was designed to preliminarily assess the adsorption and metabolism by the Sprague Dawley rat of the perfluorooctanesulfonyl amide (PFOSA, $C_8F_{17}SO_2NH_2$, CAS # 31506-32-8). Test animals were dosed by oral gavage with 5 mg/kg of PFOSA suspended in 2% TWEEN 80; control animals were given 2% TWEEN 80 with no PFOSA. Subsets of the dosed group and the control group were sacrificed on day 1, day 4, and day 29 post-dose. Sera and liver collected from the sacrificed animals were sent to the Environmental Lab and analyzed quantitatively for perfluorooctane sulfonate (PFOS, $C_8F_{17}SO_3^-$, CAS # 2795-39-3), PFOSA, and perfluorooctanoate (POAA, $C_7F_{15}CO_2^-$, CAS # 2991-51-7).

PFOS and PFOSA were detected in the livers of all dosed animals. PFOS levels were highest in the day 4 group, but even liver from animals sacrificed 29 days post dose were determined to contain high levels of PFOS. PFOSA levels in the livers of the dosed animals were highest in the group sacrificed on day 1; on day 29, the average PFOSA level was approximately 2.5 % of the day 1 levels. POAA was not detected above the limit of quantitation in the livers of dosed animals.

As in the liver extracts, sera extracts collected 4 days post-dose evidenced the highest PFOS levels. Serum PFOSA levels decreased from day 1 to day 4 to day 29; on day 29, sera from four out of five animals did not contain PFOSA above the limit of detection. POAA was detected in the sera of animals collected on day 1 and day 4. Screening studies of the dose material suggest that a small amount of POAA was present in the PFOSA dosing solution.

PFOSAB, $(C_8F_{17}SO_2)_2NH$, was detected in the livers of dosed animals. This analyte was also present in the solution used to dose the animals. No investigations of PFOSAB in the sera were conducted.

The solution used to dose the test animals was analyzed to confirm the theoretical dose levels. The PFOSA concentration in the TWEEN 80 solution was determined to be 1330 +/- 400 $\mu g/mL$. PFOS and POAA were also identified in the dose solution. The concentration of PFOS was estimated to be approximately 18.5 $\mu g/mL$; POAA levels are approximated at 2.5 $\mu g/mL$. PFOS and POAA data were collected from a single analysis and should be considered to be of screening quality only. A more thorough characterization of the material used to dose the animals was requested by 3M Toxicology and was conducted by Fred Deroos prior to initiation of the study. Results of this more comprehensive characterization are available from the requestors.

Although rigorous quality control measures and 3M Environmental Laboratory Standard Operating Procedures were followed, the acquisition of this data was not necessarily collected according to applicable Good Laboratory Practices. Neither the dose material nor the analytical standards were given a certificate of analysis; dose material was characterized by NMR and LCMS and the analytical standards were characterized by NMR. The data presented here is not suitable for distribution to any government agency in defense of product suitability or safety. This data is the highest quality data available at this time.

2 Sample Receipt

On 10/6/99, 10/8/99, and 11/1/99 Deanna Luebker from 3M Toxicology Services shipped rat urine, feces, liver, and sera, samples to the FACT group in the 3M Environmental Laboratory; samples were received frozen on dry ice and were stored at $-20^{\circ}C$.

3 Holding Times

There is no holding time criteria associated with these samples for LC/ESMSMS analysis.

Methods - Analytical and Preparatory

Analytical methods

ETS-8-4.1, Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical Compounds from Serum for Analysis Using HPLC-Electrospray/Mass Spectrometry
ETS-8-6.0, Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical Compounds from Liver for Analysis Using HPLC-Electrospray/Mass Spectrometry
ETS-8-5.1, Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemicals in Serum Extracts Using HPLC-Electrospray/Mass Spectrometry
ETS-8-7.0, Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemical on Liver Extracts Using HPLC-Electrospray/Mass Spectrometry

Sample preparation –HPLC/ESMSMS: ion-pairing extraction

Analyte is extracted from sample matrix with ion pairing reagent [tetrabutyl ammonium hydrogen sulfate (TBA)] in a pH-controlled environment. The cationic reagent selectively targets anionic fluorochemicals. Once the anion-TBA pair is formed, the analyte is transferred into a non-polar organic solvent (methyl-*tert*-butyl ether), dried, and reconstituted in methanol for MS analysis.

HPLC/ESMSMS

In HPLC, an aliquot of extract is injected and passed through a reverse phase liquid chromatographic column. Based on the affinity of the analyte for the stationary phase in the column relative to the liquid mobile phase, the analyte is retained for a characteristic amount of time. For example, in a standard solution PFOS may elute at 8.0 minutes. Retention times between a standard PFOS solution and the analyte extracted from tissue in this analysis were matched on the HPLC system to within 1%.

Following HPLC separation, ESMSMS provides a rapid and accurate means for analyzing a wide range of organic compounds, including fluorochemicals. Electrospray, an ionization technique used primarily for detection of molecular ions, is generally operated at relatively mild temperatures. Molecules are ionized, and a primary ion, characteristic of the analyte, is selected. This ion is bombarded with high-energy gas; subsequent collisions create smaller secondary ionic fragments unique to the primary ion, which are detected.

For example, for PFOS ($C_8F_{17}SO_3^-$) analysis, ion 499 is selected as the characteristic primary ion. This ion is fragmented into other ions such as 80 amu (corresponding to SO_3^-), and 230 amu ($C_3F_6SO_3^-$). Each of these secondary fragments is detected and can be used to differentiate PFOS from other compounds that might have the same characteristic 499 amu primary ion, but different chemical compositions and secondary ion fragmentation patterns.

4 Analysis

4.1 Calibration

Quantitation of the target analytes is based on linear regression analysis (weighted $1/x$) of two extracted matrix curves bracketing each group of samples. Quantitation of each analyte is based in the response of one or more specific daughter ions using the multiple response-monitoring mode of the instrument.

A mid-level, extracted matrix calibration check standard is analyzed every 5-10 samples to monitor instrumental drift. Calibration check standards are compliant if instrumental response is within +/- 30% of the expected value.

4.2 Blanks

Two extraction blanks, utilizing water as surrogate matrix, are extracted with each batch of samples. Additionally, two samples of blank matrix (both liver and sera) are extracted along with each batch of samples and curves.

Blanks are compliant if no target analyte is detected above the limit of detection for a specific analyte. In this study, all blanks were compliant.

4.3 Surrogates

Tetra-hydroperfluorooctanesulfonate is used as a surrogate in this analysis. Surrogate response is monitored to confirm gross instrumental failure, if necessary. In the non-diluted samples, the surrogate response was acceptable (+/-50%) for all analyses.

4.4 Matrix Spikes

Duplicate matrix spike analyses utilizing all target analytes were prepared and analyzed for one control animal. With one exception, PFOS recoveries were within the acceptable range of 70-130%. One liver matrix spike showed a 66 % recovery for PFOS. All PFOSA matrix spike recoveries were acceptable for PFOSA. As POAA was not a target analyte in this analysis, no matrix spikes were prepared. Although the recovery of POAA from sera and liver has been verified for other studies, POAA results should be considered screening quality only.

One matrix spike was prepared for each of the target analytes in the dose verification study. Spike recovery for PFOSA was 130%, PFOS was 115%, and POAA was 121%.

4.5 Laboratory Control Samples

Laboratory Control Samples are not a component of this study.

4.6 Sample Related Comments

Other matrices submitted with this study will be analyzed as needed upon written request from the sponsors. In the event of further analysis, an additional report will be generated.

Dose verification was conducted by evaluating diluted aliquots of the dose against an unextracted standard curve. Triplicate dilutions of the TWEEN 80 dose solution were analyzed for determination of PFOSA; a single dilution was analyzed for determination of PFOS and POAA.

5 Data Summary

The table of results detailing the sera analysis is available in Attachment A. Results of the liver analysis are presented in Attachment B.

PFOS and PFOSA were detected in the livers of all dosed animals. PFOS levels were highest in the day 4 group, but even day 29 group animals were determined to contain high levels of PFOS. PFOSA levels in the livers of the dosed animals were highest in the group sacrificed on day 1; on day 29, the average PFOSA level was approximately 2.5 % of the day 1 levels. POAA was not detected above the limit of quantitation in dosed animals.

As in the liver extracts, sera extracts collected 4 days post-dose evidenced the highest PFOS levels. Serum PFOSA levels decreased from day 1 to day 4 to day 29; on day 29, sera from four out of five animals did not contain PFOSA above the limit of detection. POAA was detected in the sera of animals collected on day 1 and day 4. Screening studies of the dose material suggest that a small amount of POAA was present in the PFOSA dosing solution.

PFOSAB was detected in the livers of dosed animals. This analyte was also present in the solution used to dose the animals. No investigations of PFOSAB in the sera were conducted.

The solution used to dose the test animals was analyzed to confirm dose levels. The PFOSA concentration in the TWEEN 80 solution was determined to be 1330 +/- 400 µg/mL. PFOS and POAA were also identified in the dose solution. The concentration of PFOS was estimated to be approximately 18.5 µg/mL; POAA levels are approximated at 2.5 µg/mL. PFOS and POAA data were collected from a single analysis and should be considered to be of screening quality only.

6 Data Quality Objectives and Data Integrity

No circumstances existed during the present study that would have adversely affected the quality or integrity of the data. The following data quality objectives were followed during the study:

- Linearity – The correlation coefficient (r^2) of the standard curve was equal to or greater than 0.98 using 1/x weighting.
- Limits of detection (defined as ½ the LOQ) – PFOS in sera is 4.88 ng / mL; PFOS in liver is 15.3 ng / g; PFOSA in sera is 2.47 ng / mL; PFOSA in liver is 6.2 ng / g
- Limits of quantitation (defined as the lowest extracted standard accurate to within 30% of theoretical value) - PFOS in sera is 9.76 ng / mL; PFOS in liver is 30.7 ng / g; PFOSA in sera is 4.93 ng / mL; PFOSA in liver is 12.4 ng / g
- Duplication frequency/acceptable precision - < 30%
- Spike frequency/acceptable recoveries – 70% to 130%; PFOS recovery was 66 % for a single matrix spike in liver (see Section 4.4)
- Use of confirmatory methods – no confirmatory methods were used
- Demonstration of specificity – specificity was demonstrated by chromatographic retention time and mass spectral characterization of a single product ion

Given the parameters listed above, and assuming spike recoveries form a suitable indication of endogenous analyte recovery, data are quantitative to +/- 30%. The validity of this assumption has not been verified by other techniques.

7 Attachments

- Attachment A: Table of results of sera analysis
- Attachment B: Table of results of liver analysis
- Attachment C: Sample receipt
- Attachment D: Screening results of dose verification
- Attachment E: Extraction and analytical data

8 Signatures

Lisa A. Clemen
Lisa Clemen, Advanced Chemist

12/06/99
Date

Kris Hansen
Kris Hansen, Ph.D., Principal Analytical Investigator

12/06/99
Date

Laboratory Report
Analytical Report of Data for
PFOSA Pharmacokinetic Study in Rats
(Sera and Liver)

Laboratory Report No. FACT-TOX-145 (W2814)

Requester Project No. T-7132.2

Attachment A

Table of Results - Sera Analysis

FACT-TOX-145

Study: TOX-145 Pharmacokinetic Study of PFOSA in Rats
 Product Number(Test Substance): T-7132.2
 Matrix: Rat Serum
 Method/Revision: ETS-4-1 and ETS-4-5.1
 Analytical Equipment System Number: Soup 020199, Davey 070799, Amelia 062498
 Instrument Software/Version: Masslynx 3.3
 Filename: See Attachments
 R-Squared Value: See Attachments
 Slope: See Attachments
 Y-Intercept: See Attachments
 Dates of Extraction/Analyst: 10/19/99, 11/03/99 SEE/MCH
 Dates of Analysis/Analyst: 10/19/99, 10/25/99, 10/27/99, 11/04/99, 11/05/99 MMH/IAS
 Date of Data Reduction/Analyst: 10/20/99, 10/26/99, 10/28/99, 11/05/99, 11/08/99 IAS/MMH

Sample Data

RAT SERUM

Group Dose	Sample #	PFOS Conc. ug/mL	PFOS Reported ug/mL	Average PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	POAA Conc. ng/mL	POAA Reported ng/mL	Average POAA ug/mL	RSD Std. Dev. MS/MSD RPD	PFOSA Conc. ng/mL	PFOSA Reported ug/mL	Average PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	H2O Blk-10199-11	0.00	<LOD	<LOD	<LOD	NA	NA	NA	NA	0.450	<LOD	<LOD	<LOD
	H2O Blk-10199-11	0.00	<LOD	<LOD	<LOD	NA	NA	NA	NA	0.320	<LOD	<LOD	<LOD
	H2O Blk-11039-9	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD	0.450	<LOD	<LOD	<LOD
	H2O Blk-11039-9	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD	0.320	<LOD	<LOD	<LOD
Matrix Blk	RBS10199-Sera Blk-11	0.00	<LOD	<LOD	<LOD	NA	NA	NA	NA	0.00	<LOD	<LOD	<LOD
	RBS10199-Sera Blk-11	0.00	<LOD	<LOD	<LOD	NA	NA	NA	NA	0.00	<LOD	<LOD	<LOD
	RTS10199-Sera Blk-11	0.00	<LOD	<LOD	<LOD	NA	NA	NA	NA	0.390	<LOD	<LOD	<LOD
	RTS10199-Sera Blk-12	0.00	<LOD	<LOD	<LOD	NA	NA	NA	NA	0.320	<LOD	<LOD	<LOD
	RBS11039-Sera Blk-9	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
QC 250 ppb	RBS11039-Sera Blk-9	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
	RTS10199-250 ppb-MS-11	231	93%	96%	5%	NA	NA	NA	NA	243	98%	102%	8%
	RTS10199-250 ppb-MSD-11	243	98%	96%	5%	NA	NA	NA	NA	264	100%	102%	8%
	RTS11039-250 ppb-MS-9	186	75%	72%	8%	NA	NA	NA	NA	246	99%	99%	0%
Group 1 0.0 mg/kg	RTS11039-250 ppb-MSD-10	172	69%	72%	8%	NA	NA	NA	NA	245	99%	99%	0%
	9R03230M, Day1	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD	1.41	<LOD	<LOD	<LOD
	9R03231M, Day1	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD	0.720	<LOD	<LOD	<LOD
	9R03232M, Day1	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD	0.330	<LOD	<LOD	<LOD
	9R03233M, Day1	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD	0.340	<LOD	<LOD	<LOD
	9R03234M, Day1	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD	0.300	<LOD	<LOD	<LOD
Group 2 0.0 mg/kg	9R03235M, Day4	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD	0.430	<LOD	<LOD	<LOD
	9R03236M, Day4	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD	0.290	<LOD	<LOD	<LOD
	9R03237M, Day4	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD	0.430	<LOD	<LOD	<LOD
	9R03238M, Day4	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD	0.320	<LOD	<LOD	<LOD
	9R03239M, Day4	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD	0.270	<LOD	<LOD	<LOD
	9R03240M, Day29	0.00	<LOD	<LOD	<LOD	NA	NA	NA	NA	0.00	<LOD	<LOD	<LOD
Group 3 0.0 mg/kg	9R03241M, Day29	0.00	<LOD	<LOD	<LOD	NA	NA	NA	NA	0.00	<LOD	<LOD	<LOD
	9R03242M, Day29	0.00	<LOD	<LOD	<LOD	NA	NA	NA	NA	0.00	<LOD	<LOD	<LOD
	9R03243M, Day29	0.00	<LOD	<LOD	<LOD	NA	NA	NA	NA	0.00	<LOD	<LOD	<LOD
	9R03244M, Day29	0.00	<LOD	<LOD	<LOD	NA	NA	NA	NA	0.00	<LOD	<LOD	<LOD
	9R03245M, Day1	139	6.94	10.8	21.9	108	0.108	0.121	0.0177	260	0.260	0.313	24.4
	9R03246M, Day1	216	10.8	128	1.80	147	0.147	0.120	0.0177	437	0.437	0.313	0.0761
Group 4 5.0 mg/kg	9R03247M, Day1	187	9.35	103	1.80	128	0.128	0.120	0.0177	255	0.255	0.313	0.0761
	9R03248M, Day1	133	6.65	103	1.80	103	0.103	0.120	0.0177	334	0.334	0.313	0.0761
	9R03249M, Day1	147	7.34	120	1.80	120	0.120	0.120	0.0177	277	0.277	0.313	0.0761
	9R03250M, Day4	156	7.78	68.0	1.56	68.0	0.0680	0.0845	0.0177	61.3	0.0613	0.0629	11.2
	9R03251M, Day4	150	7.48	67.4	1.56	67.4	0.0674	0.0845	0.0177	58.3	0.0583	0.0629	0.00702
	9R03252M, Day4	226	11.3	105	1.56	105	0.105	0.0845	0.0177	75.3	0.0753	0.0629	0.00702
Group 5 5.0 mg/kg	9R03254-1M, Day4	172	8.61	101	1.56	101	0.101	0.0845	0.0177	60.7	0.0607	0.0629	0.00702
	9R03254-2M, Day4	158	7.91	81.4	1.56	81.4	0.0814	0.0845	0.0177	59.0	0.0590	0.0629	0.00702
	9R03255M, Day29	359	3.59	NA	NA	NA	NA	NA	NA	0.140	<LOD	<LOD	<LOD
	9R03256M, Day29	398	3.98	NA	NA	NA	NA	NA	NA	1.20	<LOD	<LOD	<LOD
	9R03257M, Day29	404	4.04	NA	NA	NA	NA	NA	NA	1.09	<LOD	<LOD	<LOD
	9R03258M, Day29	413	4.13	NA	NA	NA	NA	NA	NA	1.16	<LOD	<LOD	<LOD
Group 6 5.0 mg/kg	9R03259M, Day29	736	7.36	4.62	1.54	NA	NA	NA	NA	5.01	0.00501	0.00501	N/A
	9R03259M, Day29	736	7.36	4.62	1.54	NA	NA	NA	NA	5.01	0.00501	0.00501	N/A

Limit of Quantitation (LOQ): PFOS = 5.55 ng/mL, POAA = 49.3 ng/mL, PFOSA = 4.79 ng/mL

Limit of Detection (LOD): PFOS = 1.74 ng/mL, POAA = 24.7 ng/mL, PFOSA = 1.51 ng/mL

Date Entered/By: 10/21/99, 10/29/99, 11/05/99 LAC, 11/08/99 GML

Date Verified/By: 11/16/99 GML

PFOS = Perfluorooctanesulfonate

POAA = Perfluorooctanoate

PFOSA = Perfluorooctanesulfonamide

NA = Not Applicable or Not Analyzed

NS = Not Spiked

Laboratory Report
Analytical Report of Data for
PFOSA Pharmacokinetic Study in Rats
(Sera and Liver)

Laboratory Report No. FACT-TOX-145 (W2814)

Requester Project No. T-7132.2

Attachment B

Table of Results - Liver Analysis

FACT-TOX-145

Study:
Product Number(Test Substance):
Matrix:
Method/Revision:
Analytical Equipment System Number:
Instrument Software/Version:
Date of Extraction/Analyst:
Date of Analysis/Analyst:
Date of Data Reduction/Analyst:
Sample Data

TOX-145 Pharmacokinetic Study of PFOSA in Rats
T-7132.2
Rat Liver
ETS-8-6.0 and ETS-8-7.0
Soup 020199, Amelia 062498
Masslynx 3.3
10/13/99, 11/04/99 SEE/RW/SAL
10/15/99, 10/19/99 MMH, 11/05/99 IAS, 11/10/99 IAS
10/18/99, 10/20/99, 11/08/99, 11/11/99 IAS/MMH

Filename:
R-Squared Value:
Slope:
Y-Intercept:

See Attachments
See Attachments
See Attachments
See Attachments

RAT LIVER

Group Dose	Sample #	PFOS Calc. Conc. ng/g	Total Amount of PFOS ug/g	Average PFOS ug/g	RSD Std. Dev. MS/MSD RPD	PFOSA Calc. Conc. ng/g	Total Amount of PFOSA ug/g	Average PFOSA ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	H2O Blk-11	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
	H2O Blk-11	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
	H2O Blk-2	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
	H2O Blk-2	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
Matrix Blk	RBL10199-Liver Blk-11	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
	RBL10199-Liver Blk-11	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
	RBL10199-Liver Blk-2	1.86	<LOD	<LOD	<LOD	0.760	<LOD	<LOD	<LOD
	RBL10199-Liver Blk-2	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
QC 250 ppb	9R03230M, Day1-MS-11	276	92%			313	104%		
	9R03230M, Day1-MSD-11	264	88%	90%	5%	283	94%	99%	10%
	9R03235M, Day4-MS-12	226	75%			229	76%		
	9R03235M, Day4-MSD-12	216	72%	73%	5%	215	71%	74%	7%
	9R03240M, Day29-MS-1	212	72%			352	119%		
	9R03240M, Day29-MSD-2	191	65%	68%	10%	330	112%	116%	7%
Group 1 0.0 mg/kg	9R03230M, Day1	0.00	<LOD			0.00	<LOD		
	9R03231M, Day1	0.00	<LOD			0.00	<LOD		
	9R03232M, Day1	0.00	<LOD			0.00	<LOD		
	9R03233M, Day1	0.00	<LOD			0.00	<LOD		
	9R03234M, Day1	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
Group 2 0.0 mg/kg	9R03235M, Day4	0.00	<LOD			2.35	<LOD		
	9R03236M, Day4	0.00	<LOD			0.00	<LOD		
	9R03250M, Day4	29.5	0.0295	*		22.0	0.0220	*	
	9R03238M, Day4	22.0	<LOQ			4.66	<LOD		
	9R03239M, Day4	0.00	<LOD	<LOQ	<LOQ	0.00	<LOD	<LOD	<LOD
Group 3 0.0 mg/kg	9R03240M, Day29	0.00	<LOD			2.57	<LOD		
	9R03241M, Day29	0.00	<LOD			1.72	<LOD		
	9R03242M, Day29	0.00	<LOD			0.724	<LOD		
	9R03243M, Day29	0.00	<LOD			0.162	<LOD		
	9R03244M, Day29	0.00	<LOD	<LOD	<LOD	0.00967	<LOD	<LOD	<LOD
Group 4 5.0 mg/kg	9R03245M, Day1	23660	23.7			6021	6.02		
	9R03246M, Day1	32260	32.3			7015	7.02		
	9R03247M, Day1	36179	36.2			6239	6.24		
	9R03248M, Day1	21200	21.2		21.7	6657	6.66		16.0
	9R03249M, Day1	27829	27.8	28.2	6.12	8811	8.81	6.95	1.11
Group 5 5.0 mg/kg	9R03237M, Day4	36791	36.8	*		3523	3.52	*	
	9R03251M, Day4	32619	32.6			3470	3.47		
	9R03252M, Day4	42606	42.6			3959	3.96		
	9R03253M, Day4	36945	36.9		9.56	3454	3.45		8.28
	9R03254M, Day4	38268	38.3	37.4	3.58	4105	4.11	3.70	0.307
Group 6 5.0 mg/kg	9R03255M, Day29	20305	20.3			85.5	0.0855		
	9R03256M, Day29	20638	20.6			175	0.175		
	9R03257M, Day29	22025	22.0			104	0.104		
	9R03258M, Day29	26172	26.2		17.3	142	0.142		53.0
	9R03259M, Day29	29867	29.9	23.8	4.12	303	0.303	0.162	0.0859

Limit of Quantitation Limit (LOQ): PFOS = 26.9 ng/g, PFOSA = 11.1 ng/g
Limit of Detection (LOD): PFOS = 8.45 ng/g, PFOSA = 3.50 ng/g

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
* Samples numbers may have been switched. 3237 should be 3250.
@ These samples may be falsely high, due to carryover from accidentally switching 3237 and 3250 GML 11/16/99

Date Entered/Analyst: 10/21/99, 10/22/99, 11/15/99 LAC, 11/08/99 GML
Date Verified/Analyst: 11/16/99 GML

Laboratory Report
Analytical Report of Data for
PFOSA Pharmacokinetic Study in Rats
(Sera and Liver)

Laboratory Report No. FACT-TOX-145 (W2814)

Requester Project No. T-7132.2

Attachment C

Sample Receipt

000523

10/06/1999

3M ENVIRONMENTAL LABORATORY
REQUEST FOR ANALYTICAL/FIELD WORK

1

LAB REQUEST NO. W2814
REQUESTOR NAME: DEANNA LUEBKER
DEPARTMENT: 0227
PROJECT NO: 0010535009
DATE RECEIVED: 10/06/1999
DESC: PFOSA PK STUDY IN RATS TOX-145

CONTRACT LAB(S):
EXP COMP DATE: 12/30/1999
DATE COMPLETED:
PROJECT LEAD: KJ HANSEN
PHONE NO: 651-778-6018
3M FAX NO: 651-778-6176

220-2E-02,7-1374

SAMPLE	DATE	CODE	DESCRIPTION	RESULT	MDL or (95% C.I.)
--------	------	------	-------------	--------	-------------------

1

KJH-SPEC		SPECIAL STUDY - KJH			
----------	--	---------------------	--	--	--

#

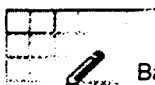
= UPDATED DATA

✓
KJH LAC ✓

000524

W2814

TOX-145 SAMPLES RECEIVED



Barbara A. Gramenz
10/07/99 10:08 AM

ET&SS Lab
2-3E-09

Fax: 778-6176

To: Kari J. Rambo/US-Corporate/3M/US@3M-Corporate
Grant A. Paul/US-Corporate/3M/US@3M-Corporate
Lisa A. Clemen/US-Corporate/3M/US@3M-Corporate
Kris J. Hansen/US-Corporate/3M/US@3M-Corporate

cc:

Subject: TOX-145 SAMPLES RECEIVED

On 10/6/99 we received 10 Liver, 10 Sera, 20 Feces and 20 Urine Samples for days 1 & 2 from 3M TOXICOLOGY. FACT- TOX -145. LAB REQUEST # W2814. PFOSA PK Study in Rats (T-7132.2). Samples received frozen on dry ice and have been stored in Freezer F-16 at -20+/-10C.

Barb

Ver. Sied sample receipt - 10/7/99 - RWW

PFOSA PK STUDY IN RATS

TOX-145 LR# W2814

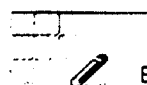
DAY 1 DATE 10/5/99 Gen. MALE POST DOSE

URINE ID#	FECES ID#	LIVER ID#	SERA ID#
9R03240	9R03240	9R03230	9R03230
9R03241	9R03241	9R03231	9R03231
9R03242	9R03242	9R03232	9R03232
9R03243	9R03243	9R03233	9R03233
9R03244	9R03244	9R03234	9R03234
9R03255	9R03255	9R03245	9R03245
9R03256	9R03256	9R03246	9R03246
9R03257	9R03257	9R03247	9R03247
9R03258	9R03258	9R03248	9R03248
9R03259	9R03259	9R03249	9R03249

DAY 2 DATE 10/6/99 Gen.MALE POST DOSE

URINE ID#	FECES ID#
9R03240	9R03240
9R03241	9R03241
9R03242	9R03242
9R03243	9R03243
9R03244	9R03244
9R03255	9R03255
9R03256	9R03256
9R03257	9R03257
9R03258	9R03258
9R03259	9R03259

TOX-145 SAMPLES RECEIVED



Barbara A. Gramenz
10/11/99 11:54 AM

ET&SS Lab
2-3E-09

Fax: 778-6176

To: Kari J. Rambo/US-Corporate/3M/US@3M-Corporate
Grant A. Paul/US-Corporate/3M/US@3M-Corporate
Lisa A. Clemen/US-Corporate/3M/US@3M-Corporate
Kris J. Hansen/US-Corporate/3M/US@3M-Corporate

cc:

Subject: TOX-145 days 3&4 SAMPLES RECEIVED

On 10/8/99 we received 10 Liver, 10 Sera, 20 Feces and 20 Urine Samples for day 3 10/7/99 & day 4 10/8/99 from 3M TOXICOLOGY. FACT- TOX -145. LAB REQUEST # W2814. PFOSA PK Study in Rats (T-7132.2). Samples received frozen on dry ice and have been stored in Freezer F-16 at -20+-10C.

Barb

sample verified 10-12-99/SC

PFOSA PK STUDY IN RATS

TOX-145

LR# W2814

DAY 3 DATE 10/7/99 Gen. MALE POST DOSE

URINE ID#	FECES ID#
9R03240	9R03240
9R03241	9R03241
9R03242	9R03242
9R03243	9R03243
9R03244	9R03244
9R03255	9R03255
9R03256	9R03256
9R03257	9R03257
9R03258	9R03258
9R03259	9R03259

DAY 4 DATE 10/8/99 Gen. MALE POST DOSE

URINE ID#	FECES ID#	LIVER ID#	SERA ID#
9R03240	9R03240	9R03235	9R03235
9R03241	9R03241	9R03236	9R03236
9R03242	9R03242	9R03237	9R03237
9R03243	9R03243	9R03238	9R03238
9R03244	9R03244	9R03239	9R03239
9R03255	9R03255	9R03250	9R03250
9R03256	9R03256	9R03251	9R03251
9R03257	9R03257	9R03252	9R03252
9R03258	9R03258	9R03253	
9R03259	9R03259	9R03254	② 9R03254

THERE ARE TWO 9R03254 SERA SAMPLES
ONE SHOULD BE 9R03253 BUT DONT KNOW WHICH ONE. BAG 10/11/99.

KTH, CAC

000529

PFOSA PK STUDY IN RATS
TOX-145 LR# W2814
DAY 3 DATE 10/7/99 Gen. MALE POST DOSE

URINE ID#	FECES ID#
9R03240	9R03240
9R03241	9R03241
9R03242	9R03242
9R03243	9R03243
9R03244	9R03244
9R03255	9R03255
9R03256	9R03256
9R03257	9R03257
9R03258	9R03258
9R03259	9R03259

DAY 4 DATE 10/8/99 Gen.MALE POST DOSE

URINE ID#	FECES ID#	LIVER ID#	SERA ID#
9R03240	9R03240	9R03235	9R03235
9R03241	9R03241	9R03236	9R03236
9R03242	9R03242	9R03237	9R03237
9R03243	9R03243	9R03238	9R03238
9R03244	9R03244	9R03239	9R03239
9R03255	9R03255	9R03250	9R03250
9R03256	9R03256	9R03251	9R03251
9R03257	9R03257	9R03252	9R03252
9R03258	9R03258	9R03253	
9R03259	9R03259	9R03254	② 9R03254

THERE ARE TWO 9R03254 SERA SAMPLES
ONE SHOULD BE 9R03253 BUT DONT KNOW WHICH ONE. BAG 10/11/99.
I have added a -1, -2 to the sera samples 9R03254 in order
to differentiate the two for testing. See 10/12/99

000530

BEST COPY AVAILABLE

Chain of Custody Record 3M Environmental Laboratory

09600

3M Requester <i>Deanna Luebker</i>
3M Project Lead <i>Andrew Seacat</i>
3M Project Number <i>T-7132.2</i> 0010535009
3M Department Number <i>62270</i> 4291
Lab Request/Template Number <i>W2814</i>

Form 35176 - PWO

3M Facility <i>Corporate Tox</i>	3M Project Title <i>PFOSA PK IN RATS T-7132.2 TDX14E</i>	3M Project Number <i>T-7132.2</i> 0010535009
Address <i>2002E-02</i>	3M Facility Contact <i>Deanna</i>	3M Department Number <i>62270</i> 4291
City, State, Zip Code	3M Telephone Number <i>7-13741</i>	Lab Request/Template Number <i>W2814</i>

Sampled By (Print) <i>Deanna Luebker</i>		Contractor		Number Containers of each		Total Containers of Each Item	☐ Samples Split See Attachment	Comments				
Sampler Signature <i>Deanna Luebker</i>		Date <i>10/8/99</i>		Preservatives								
Item #	Sample Description	Date	Time	Matrix	None	HNO ₃	H ₂ SO ₄	NaOH	VOC			
1	Dx3 Post Dose Urine	10/7	2pm	Urine								10
2	" " " " feces	10/7	2pm	Feces								10
3	Dx4 " " Urine	10/8	9am	Urine								10
4	" " " " feces	10/8	9am	Feces								10
5	Dx4 Post dose Liver	10/8	2pm	Liver								10
6	" " " " Serum	10/8	2pm	Serum								10
7												60
8												
9												
10												
11												
12												
13												

Item #	Relinquished By/Affiliation	Time	Date	Shipped Via	Received By/Affiliation	Time	Date
1-6	<i>Deanna Luebker / Tox</i>	<i>3:45 PM</i>	<i>10/8/99</i>	<i>Road Runner</i>	<i>Jim A. Clener / 3M</i>	<i>4:00pm</i>	<i>10/08/99</i>

Original: Accompanying Sample

Lab Page: 1 of 1

① (RE) UC 10/08/99

See Reverse Side For Instructions

000531

TOX-145 day 29 SAMPLES RECEIVED



Barbara A. Gramenz
11/02/99 02:13 PM

ET&SS Lab
2-3E-09

Fax: 778-6176

To: Kari J. Rambo/US-Corporate/3M/US@3M-Corporate
Grant A. Paul/US-Corporate/3M/US@3M-Corporate
Lisa A. Clemen/US-Corporate/3M/US@3M-Corporate
Kris J. Hansen/US-Corporate/3M/US@3M-Corporate

cc:

Subject: TOX-145 day 29 SAMPLES RECEIVED

On 11/2/99 we received 10 Liver, 10 Sera Samples for day 29 from 3M TOXICOLOGY. FACT- TOX -145. LAB REQUEST # W2814. PFOSA PK Study in Rats (T-7132.2). Samples received frozen on dry ice and have been stored in Freezer F-16 at -20+/-10C.

Barb

Samples verified - 11/2/99 - RWW

PFOSA PK STUDY IN RATS

TOX-145 LR# W2814

DAY 29 DATE 11/2/99 Gen.MALE POST DOSE

LIVER ID#	SERA ID#
9R03240	9R03240
9R03241	9R03241
9R03242	9R03242
9R03243	9R03243
9R03244	9R03244
9R03255	9R03255
9R03256	9R03256
9R03257	9R03257
9R03258	9R03258
9R03259	9R03259

Chain of Custody Record
3M Environmental Laboratory

09305

Form 35176 - PWO

3M Requester <i>Deanna Luebker</i>
3M Project Lead <i>Andrew Scacat</i>
3M Project Number <i>0010535009</i>
3M Department Number <i>227 4291</i>
Lab Request/Template Number <i>W 2814</i>

3M Facility <i>Corporate Tox</i>	3M Project Title <i>PDSA PK in Rats T-7132.2</i>	3M Project Number <i>0010535009</i>
Address <i>220 JE-02</i>	3M Facility Contact <i>Deanna Luebker</i>	3M Department Number <i>227 4291</i>
City, State, Zip Code	3M Telephone Number <i>7-1374</i>	Lab Request/Template Number <i>W 2814</i>

Sampled By (Print) <i>Deanna Luebker</i>		Contractor		Number Containers of each						Total Containers of Each Item	☐ Samples Split See Attachment Comments	
Sampler Signature <i>Deanna Luebker</i>		Date <i>11/2/99</i>		Preservatives								
Item #	Sample Description	Date	Time	Matrix	None	HNO ₃	H ₂ SO ₄	NaOH	VOC			
1	Rat Liver vehicle control	11/2/99	10a	Liver							5	
2	Rat Liver 5mg/kg PDSA	"	"	Liver							5	
3	Rat Sera vehicle control	"	"	Sera							5	
4	Rat Sera 5mg/kg PDSA	"	"	Sera							5	
5												
6												
7												
8												
9												
10												
11												
12												
13												

Item #	Relinquished By/Affiliation	Time	Date	Shipped Via	Received By/Affiliation	Time	Date
1-4	Deanna Luebker / Tox	12:15p	11/2/99	RoadRunner	Bobby Venz	1:30	11/2/99

000534

Laboratory Report
Analytical Report of Data for
PFOSA Pharmacokinetic Study in Rats
(Sera and Liver)

Laboratory Report No. FACT-TOX-145 (W2814)

Requester Project No. T-7132.2

Attachment D

Screening Results of Dose Verification

000535



Kris J. Hansen

11/04/99 02:29 PM



Tel: 778-6018
2-3E-09

Fax: 778-6176
Environmental Lab

To: Andrew Seacat/US-Corporate/3M/US@3M-Corporate
Deanna J. Luebker/US-Corporate/3M/US@3M-Corporate
cc: Lisa A. Clemen/US-Corporate/3M/US@3M-Corporate
Subject: TOX145: PFOSA PK, Dose Verification

Hi Andrew and Deanna:

We have completed our screen of the PFOSA dose in TWEEN. Error bars are about +/-30%. PFOSA was determined in triplicate and PFOS and POAA from a single analysis. I'd classify this data as screening quality and if this data poses any major questions about the interpretation of the tox data, we may need more data points for the PFOS and POAA. However, I did check for the presence of the POAA on two different instruments.

Also, in one of your voice mails, Andrew, you mentioned concern about photolysis of PFOSA to PFOS. All of the data that I've seen indicates that there may be photolysis of PFOSA to **POAA** (not verified yet), but there is no evidence of PFOSA photolysis to PFOS. Since the photolysis experiments were conducted under very strenuous conditions, I don't think you have much to worry about with your samples (at least not for a few dozen years!).

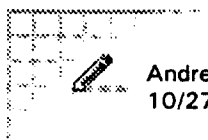
In TWEEN 80:

PFOSA	133.26% of expected dose of 1000 µg/mL	--> 1330 µg/mL	spike
recovery 130%			
PFOS	185% of expected dose of 10 µg/mL	--> 18.5 µg/mL	spike
recovery 115%			
POAA		--> 2.5 µg/mL	spike
recovery 121%			

Let me know if you have questions.

kjh

000526



Andrew Seacat
10/27/99 02:53 PM

3M Medical Dept. Toxicology Services
Andrew M. Seacat Ph.D.
3M Center 220-2E-02
Saint Paul, MN 55144
Tel: (651) 575-3161
Fax: (651) 733-1773

To: Kris J. Hansen/US-Corporate/3M/US@3M-Corporate
cc: Fred L. DeRoos/US-Corporate/3M/US@3M-Corporate
Deanna J. Luebker/US-Corporate/3M/US@3M-Corporate
Sue Chang/US-Corporate/3M/US@3M-Corporate
Subject: PFOSA dosing solution

Kris,

As we were discussing in our last Friday's meeting, we would like to have the PFOSA dosing solution checked for PFOSA concentration, and for PFOS and PFOA, which may under certain conditions arise from photolysis. We left the dosing solution with you. I understand there is a problem with the Tween 80 concentration being too high to allow sufficient dilution for analysis of some PFOS concentrations. Given that the starting material has approximately 1% PFOS in it could the dosing solution sample be analysed to see if there is significantly more PFOS in there? If not possible, then perhaps we could ask Fred Deroos to analyze the dosing solution?

The dosing solution was prepared as follows:

Stock PFOSA in acetone was made up at 52mg PFOSA/0.52 mL acetone = 100 mg/mL

This was diluted into 51.48 ml of 2% Tween 80 to achieve the dosing solution:

Final concentration of PFOSA = 1mg/ml

Final conc of acetone = 1%

Final conc Tween 80 = ~2%

Thanks,
Andrew

000537

TOX145
Tween 80- Dose Confirmation

Study TOX145
Date Analyzed 11/2/99
File # 110299a
Instrument Soup 020199

POAA

Sample ID	Dose conc. (ppb)	Expected Dose (ppb)	Amt of spike (ppb)	Dose + MS conc. (ppb)	% Recovery of Dose	% Recovery of MS	Date of Extraction
Tween dose-NA	4.82	NA	20.4	29.48	#VALUE!	121	11/2/99

PFOS

Sample ID	Dose conc. (ppb)	Expected Dose (ppb)	Amt of spike (ppb)	Dose + MS conc. (ppb)	% Recovery of Dose	% Recovery of MS	Date of Extraction
Tween dose-20ppb	36.9	20.0	20.7	60.8	185	115	11/2/99

↑
observed

↑
from email
expected

↑
1 aliquot

4.82 ppb POAA in TWEEN
36.9 ppb PFOS in TWEEN

if 20 ppb $\Rightarrow$ 10 ppm
Then 36.9 ppb $\Rightarrow$ 18. ppm

if 4.8 ppb POAA $\Rightarrow$ 2.4 ppm POAA

$$4.8 \text{ ng/mL} * 500 = \frac{2500 \text{ ng}}{\text{mL}} \Rightarrow 2.5 \mu\text{g/mL}$$

000538

Study	TOX145
Date Analyzed	10/29/99
File #	102999a
Instrument	Soup 020199

PFOSA in 2% Tween 80, Sample TN-A-3046

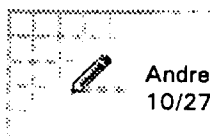
File #	% Recovery of Dose	Recovery of Dose amt (ppb)
10299013	132.64	265.27
10299014	134.31	268.62
10299017	132.83	265.65
Mean	133.26	266.51
Standard Dev.	0.917	1.83
%RSD	0.688	0.688

expected 200 ppb
recovered 265 ppb

PFOSA $\Rightarrow$ 3 analysis
+ 1 matrix spike

000539

PFOSA dosing solution



Andrew Seacat
10/27/99 02:53 PM

3M Medical Dept. Toxicology Services
Andrew M. Seacat Ph.D.
3M Center 220-2E-02
Saint Paul, MN 55144
Tel: (651) 575-3161
Fax: (651) 733-1773

To: Kris J. Hansen/US-Corporate/3M/US@3M-Corporate
cc: Fred L. DeRoos/US-Corporate/3M/US@3M-Corporate
Deanna J. Luebker/US-Corporate/3M/US@3M-Corporate
Sue Chang/US-Corporate/3M/US@3M-Corporate
Subject: PFOSA dosing solution

Kris,

As we were discussing in our last Friday's meeting, we would like to have the PFOSA dosing solution checked for PFOSA concentration, and for PFOS and PFOA, which may under certain conditions arise from photolysis. We left the dosing solution with you. I understand there is a problem with the Tween 80 concentraton being too high to allow sufficient dilution for analysis of some PFOS concentrations. Given that the starting material has approximately 1% PFOS in it could the dosing solution sample be analysed to see if there is significantly more PFOS in there? If not possible, then perhaps we could ask Fred Deroos to analyze the dosing solution?

The dosing solution was prepared as follows:

Stock PFOSA in acetone was made up at 52mg PFOSA/0.52 mL acetone = 100 mg/mL

This was diluted into 51.48 ml of 2% Tween 80 to acheive the dosing solution:

Final concentration of PFOSA = 1mg/ml

Final conc of acetone = 1%

Final conc Tween 80 = ~2%

Thanks,
Andrew

$$1\text{mg/mL} = 1000\text{ }\mu\text{g/mL} = 1000\text{ ppm PFOSA conc.}$$

$$1\% \text{ of } 1000\text{ ppm} = 10\text{ ppm} = \text{PFOS conc.}$$

PFOSA

Dose $1000\text{ ppm} \times \frac{5\text{ }\mu\text{L}}{25\text{ mL}} \times 1000 = 200\text{ ppb}$

MS $100\text{ ppm} \times \frac{0.025\text{ mL}}{25\text{ mL}} \times 1000 = 1000\text{ ppb}$

PFOS

$10\text{ ppm} \times \frac{50\text{ }\mu\text{L}}{25\text{ mL}} \times 1000 = 20\text{ ppb}$

$5\text{ ppm} \times \frac{0.100\text{ mL}}{25\text{ mL}} \times 1000 = 20\text{ ppb}$

Printed 11/04/99

[3M Restricted]

$$\frac{10\text{ }\mu\text{g}}{\text{mL}} \times \frac{50\text{ }\mu\text{L}}{25\text{ mL}} \times \frac{1000\text{ }\mu\text{L}}{1000\text{ }\mu\text{L}} = 20\text{ }\mu\text{g/mL}$$

$$500 / 25000 = 0.02\text{ }\mu\text{g/mL} = 20\text{ ng/mL}$$

Page 1

000540

TOX145
Tween 80- Dose Confirmation

Study TOX145
Date Analyzed 10/29/99
File # 102999a
Instrument Soup 020199

POAA

Sample ID	Dose conc. (ppb)	Expected Dose (ppb)	Amt of spike (ppb)	Dose + MS conc. (p	% Recovery of D _s	% Recovery of M _d	Date of Extraction
Tween dose-2ppb	1.67	2.00	2.04	3.51	24	90	10/29/99

PFOS

Sample ID	Dose conc. (ppb)	Expected Dose (ppb)	Amt of spike (ppb)	Dose + MS conc. (p	% Recovery of D _s	% Recovery of M _d	Date of Extraction
Tween dose-2ppb	4.13	2.00	2.07	6.40	207	110	10/29/99

PFOSA

Sample ID	Dose conc. (ppb)	Expected Dose (ppb)	Amt of spike (ppb)	Dose + MS conc. (p	% Recovery of D _s	% Recovery of M _d	Date of Extraction
Tween dose-200ppb	265.65	200	100	385.42	133	120	10/29/99

dilution level

1:5000

→ Andrew's claim

1:5000

① st. dev.
RSD

② prepare a 1:500 dilution
of dose; analyze
for PFOS / POAA
+ 20ppb spike
20ppb tendogenous
(20ppb)

3 dilutions of dose

1 MS of dose

000541

FACT: DATA REVIEW SUMMARY FORM

JAS 11/01/99 p. 1 of 2

Data Review Sign-Off

Date: 11/01/99
 Data Analyst: JAS
 FACT Study #: TOX145
 Matrix/Timepoints: Tween 80 dose confirmation sample
 Sample List: 102999a
 Set-up Analysis
 Date/Analyst: 10/29/99 JAS
 Instrument: Sing 220199

Analyst Review

Name / Date: JAS 11/01/99

Technical Review

Name / Date: kin 11/1/99

Quality Assurance Review

Name / Date: _____

Data Entry: _____

Comments / Justifications:

Analyte: POAA (CH1) ①
 Acceptable Linear Range: 1.00 ppb → 100 ppb
 LOQ: 1.00 ppb Extraction Blank: NA
 R²: 0.997121 Matrix Blank: NA
 Calibration Checks: OK - 25 ppb = 1% deviation
 MS/MSD: _____
 Data Status: _____

Analyte: PFOS (CH2) ②
 Acceptable Linear Range: 1.01 ppb → 101 ppb
 LOQ: 1.01 ppb Extracted Blank: NA
 R²: 0.996935 Matrix Blank: NA
 Calibration Checks: OK - Range is 2% for the 25 ppb calcheck
 MS/MSD: _____
 Data Status: _____

Analyte: PFOSA (CH3) ③
 Acceptable Linear Range: 10.10 ppb → 754 ppb
 LOQ: 10.10 ppb Extracted Blank: NA
 R²: 0.997261 Matrix Blank: NA
 Calibration Checks: OK - 25 ppb calcheck = 10%
 MS/MSD: _____
 Data Status: _____

① The 251 ppb, 502 ppb, and 752 ppb points were excluded from the curve in order to focus on the lower linear conc. points. Keeping the higher concentrations gave r² values of less than 0.98 and deviations greater than ± 30% for the lower conc. points. - JAS 11/01/99
 Some carryover was noticed in the MeOH BLKs. - JAS 11/01/99.
 ② The 251 ppb, 502 ppb, and 752 ppb points were excluded. Keeping the higher points gave r² values of less than 0.98 and deviations greater than ± 30% for the lower conc. curve points. Some carry over was noticed in the MeOH BLKs. - JAS 11/01/99.
 ③ The 1.01 ppb and 5.03 ppb points were dropped in order to focus on the higher conc. curve points. Deviations of the 1.01 ppb and 5.03 ppb

000542

FACT: DATA REVIEW SUMMARY FORM

Jas 11/01/99 p. 2 of 2

Data Review Sign-Off

Date: 11/01/99
 Data Analyst: JAS
 FACT Study #: Tox 145
 Matrix/Timepoints: Tween 80 dose confirmation sample
 Sample List: 102999a
 Set-up Analysis Date/Analyst: 11/29/99 JAS
 Instrument: Simp 920199

Analyst Review

Name / Date: JAS 11/01/99

Technical Review

Name / Date: kjh 11/1/99

Quality Assurance Review

Name / Date: _____

Data Entry: _____

Comments / Justifications:

③ (cont) curve points were greater than $\pm 30\%$ when keeping the upper linear range. — JAS 11/01/99
 Carryover was noticed in the Meth B/Ls. — JAS 11/01/99.
 ④ ISTD area counts varied throughout the sample run. Deviations were as high as 54% and as low as -65%. — JAS 11/01/99

Analyte: IS (CH4) ④
 Acceptable Linear Range: NA
 LOQ: NA Extraction Blank: NA
 R²: NA Matrix Blank: NA
 Calibration Checks: NA
 MS/MSD: _____
 Data Status: _____

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: JAS 11/01/99
 MS/MSD: _____
 Data Status: _____

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: _____

000543

Quantify Compound Summary Report
TOX145-tween 80 dose confirmation for POAA, PFOS, & POAA- ext. 10/29/99 ias

Sample List: C:\MASSLYNK\soup020199.pro\sampleDB\102999a
Last modified: Fri Oct 29 10:23:59 1999
Method: C:\MASSLYNK\soup020199.pro\MethDB\102999a
Last modified: Mon Nov 01 06:21:40 1999
Job Code:

Printed: Mon Nov 01 06:47:34 1999

Compound 1: POAA (Ch1)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1 102999001	MeOH Blk			7.154	42	bb	3.66	
2 102999002	MeOH Blk							
3 102999003	FCMIX-99086-43-1, 1ppb- 9/10/99 RWW	Standard	1.00	7.088	16	MM	1.02	2
4 102999004	FCMIX-99086-43-2, 5ppb- 9/10/99 RWW	Standard	5.02	7.129	53	MM	4.72	-6
5 102999005	FCMIX-99086-43-3, 10ppb- 9/10/99 RWW	Standard	10.00	7.101	108	bb	10.22	2
6 102999006	FCMIX-99086-43-4, 25ppb- 9/10/99 RWW	Standard	25.10	7.082	244	bb	23.74	-5
7 102999007	FCMIX-99086-43-5, 50ppb- 9/10/99 RWW	Standard	50.20	7.087	471	bb	46.43	-8
8 102999008	FCMIX-99086-43-6, 100ppb- 9/10/99 RWW	Standard	100.00	7.084	981	bb	97.28	-3
9 102999009	FCMIX-99086-43-7, 250ppb- 9/10/99 RWW	Standard	251.00	7.133	2481	bbX	246.70	-2
10 102999010	FCMIX-99086-43-8, 500ppb- 9/10/99 RWW	Standard	502.00	7.103	4254	bbX	423.45	-16
11 102999011	FCMIX-99086-43-9, 750ppb- 9/10/99 RWW	Standard	752.00	7.129	5542	bbX	551.77	-27
12 102999012	MeOH Blk			7.147	14	MM	0.92	
13 102999013	TOX145-PFOS/POAA in Tween-dose-2ppb- 10/29/99 IAS			7.152	22	MM	1.67	
14 102999014	TOX145-PFOS/POAA in Tween-ms-4ppb- 10/29/99 IAS			7.134	40	MM	3.51	
15 102999015	FCMIX-99086-43-4, 25ppb- 9/10/99 RWW	QC	25.10	7.147	259	bb	25.32	1
16 102999016	MeOH Blk							
17 102999017	TOX145-PFOSA in Tween-dose-200ppb- 10/29/99 IAS			7.131	12	MM	0.70	
18 102999018	TOX145-PFOSA in Tween-ms-300ppb- 10/29/99 IAS			7.160	11	MM	0.61	
19 102999019	FCMIX-99086-43-7, 250ppb- 9/10/99 RWW	QC	251.00	7.128	2363	bb	234.97	-6
20 102999020	MeOH Blk							
21 102999021	FCMIX-99086-43-1, 1ppb- 9/10/99 RWW	Standard	1.00	7.110	13	MM	0.73	-27
22 102999022	FCMIX-99086-43-2, 5ppb- 9/10/99 RWW	Standard	5.02	7.169	67	MM	6.15	23
23 102999023	FCMIX-99086-43-3, 10ppb- 9/10/99 RWW	Standard	10.00	7.174	113	MM	10.72	7
24 102999024	FCMIX-99086-43-4, 25ppb- 9/10/99 RWW	Standard	25.10	7.180	273	MM	26.64	6
25 102999025	FCMIX-99086-43-5, 50ppb- 9/10/99 RWW	Standard	50.20	7.129	542	bb	53.45	6
26 102999026	FCMIX-99086-43-6, 100ppb- 9/10/99 RWW	Standard	100.00	7.130	1024	MM	101.52	2
27 102999027	FCMIX-99086-43-7, 250ppb- 9/10/99 RWW	Standard	251.00	7.179	2338	bbX	232.52	-7
28 102999028	FCMIX-99086-43-8, 500ppb- 9/10/99 RWW	Standard	502.00	7.154	4210	bbX	418.97	-17
29 102999029	FCMIX-99086-43-9, 750ppb- 9/10/99 RWW	Standard	752.00	7.129	5411	bbX	538.72	-28

Sample List: C:\MASSLYNX\soup020199.pro\SampleDB\102999a
Last modified: Fri Oct 29 10:23:59 1999
Method: C:\MASSLYNX\soup020199.pro\MethDB\102999a
Last modified: Mon Nov 01 06:21:40 1999
Job Code:

Printed: Mon Nov 01 06:47:34 1999

Compound 2: PFOS (Ch2)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1 102999001	MeOH Blk			7.315	202	bb	1.56	
2 102999002	MeOH Blk			7.295	42	bb	0.00	
3 102999003	FCMIX-99086-43-1, 1ppb- 9/10/99 RWW	Standard	1.01	7.226	148	MM	0.93	-8
4 102999004	FCMIX-99086-43-2, 5ppb- 9/10/99 RWW	Standard	5.03	7.291	476	MM	4.74	-6
5 102999005	FCMIX-99086-43-3, 10ppb- 9/10/99 RWW	Standard	10.00	7.240	913	MM	9.81	-2
6 102999006	FCMIX-99086-43-4, 25ppb- 9/10/99 RWW	Standard	25.20	7.266	2108	bb	23.71	-6
7 102999007	FCMIX-99086-43-5, 50ppb- 9/10/99 RWW	Standard	50.30	7.248	4162	bd	47.57	-5
8 102999008	FCMIX-99086-43-6, 100ppb- 9/10/99 RWW	Standard	101.00	7.246	8473	bd	97.67	-3
9 102999009	FCMIX-99086-43-7, 250ppb- 9/10/99 RWW	Standard	252.00	7.272	22878	bbX	265.05	5
10 102999010	FCMIX-99086-43-8, 500ppb- 9/10/99 RWW	Standard	503.00	7.265	41708	bbX	483.87	-4
11 102999011	FCMIX-99086-43-9, 750ppb- 9/10/99 RWW	Standard	755.00	7.289	56477	bbX	655.48	-13
12 102999012	MeOH Blk			7.309	170	MM	1.18	
13 102999013	TOX145-PFOS/POAA in Tween-dose-2ppb- 10/29/99 IAS			7.313	424	bb	4.13	
14 102999014	TOX145-PFOS/POAA in Tween-ms-4ppb- 10/29/99 IAS			7.340	619	bb	6.40	
15 102999015	FCMIX-99086-43-4, 25ppb- 9/10/99 RWW	QC	25.20	7.331	2284	bb	25.75	2
16 102999016	MeOH Blk			7.338	63	MM	0.00	
17 102999017	TOX145-PFOSA in Tween-dose-200ppb- 10/29/99 IAS			7.314	373	MM	3.55	
18 102999018	TOX145-PFOSA in Tween-ms-300ppb- 10/29/99 IAS			7.321	398	MM	3.83	
19 102999019	FCMIX-99086-43-7, 250ppb- 9/10/99 RWW	QC	252.00	7.313	22863	bb	264.88	5
20 102999020	MeOH Blk			7.295	91	bb	0.27	
21 102999021	FCMIX-99086-43-1, 1ppb- 9/10/99 RWW	Standard	1.01	7.272	158	bb	1.04	3
22 102999022	FCMIX-99086-43-2, 5ppb- 9/10/99 RWW	Standard	5.03	7.330	530	bb	5.37	7
23 102999023	FCMIX-99086-43-3, 10ppb- 9/10/99 RWW	Standard	10.00	7.335	989	bb	10.70	7
24 102999024	FCMIX-99086-43-4, 25ppb- 9/10/99 RWW	Standard	25.20	7.342	2289	bb	25.80	2
25 102999025	FCMIX-99086-43-5, 50ppb- 9/10/99 RWW	Standard	50.30	7.291	4761	bb	54.53	8
26 102999026	FCMIX-99086-43-6, 100ppb- 9/10/99 RWW	Standard	101.00	7.313	8949	bb	103.19	2
27 102999027	FCMIX-99086-43-7, 250ppb- 9/10/99 RWW	Standard	252.00	7.363	22364	bbX	259.08	3
28 102999028	FCMIX-99086-43-8, 500ppb- 9/10/99 RWW	Standard	503.00	7.314	42068	bbX	488.05	-3
29 102999029	FCMIX-99086-43-9, 750ppb- 9/10/99 RWW	Standard	755.00	7.291	55588	bbX	645.14	-15

Sample List: C:\MASSLYNK\soup020199.pro\SampleDB\102999a
Last modified: Fri Oct 29 10:23:59 1999
Method: C:\MASSLYNK\soup020199.pro\MethDB\102999a
Last modified: Mon Nov 01 06:21:40 1999
Job Code:

Printed: Mon Nov 01 06:47:34 1999

Compound 3: PFOSA (Ch3)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1 102999001	MeOH Blk			7.833	18209	MM	66.15	
2 102999002	MeOH Blk			7.790	835	MM	2.32	
3 102999003	FCMix-99086-43-1, 1ppb- 9/10/99 RWW	Standard	1.01	7.741	1102	MMX	3.30	227
4 102999004	FCMix-99086-43-2, 5ppb- 9/10/99 RWW	Standard	5.03	7.809	1881	MMX	6.16	23
5 102999005	FCMix-99086-43-3, 10ppb- 9/10/99 RWW	Standard	10.10	7.803	2710	MM	9.21	-9
6 102999006	FCMix-99086-43-4, 25ppb- 9/10/99 RWW	Standard	25.10	7.805	6408	MM	22.80	-9
7 102999007	FCMix-99086-43-5, 50ppb- 9/10/99 RWW	Standard	50.30	7.766	10741	MM	38.71	-23
8 102999008	FCMix-99086-43-6, 100ppb- 9/10/99 RWW	Standard	101.00	7.809	21634	MM	78.74	-22
9 102999009	FCMix-99086-43-7, 250ppb- 9/10/99 RWW	Standard	251.00	7.857	68915	MM	252.44	1
10 102999010	FCMix-99086-43-8, 500ppb- 9/10/99 RWW	Standard	503.00	7.895	139095	MM	510.28	1
11 102999011	FCMix-99086-43-9, 750ppb- 9/10/99 RWW	Standard	754.00	7.941	199487	MM	732.15	-3
12 102999012	MeOH Blk			7.982	2140	MM	7.12	
13 102999013	TOX145-PFOS/POAA in Tween-dose-2ppb- 10/29/99 IAS			7.920	72407	bb	265.27	
14 102999014	TOX145-PFOS/POAA in Tween-ms-4ppb- 10/29/99 IAS			7.969	73318	bb	268.62	
15 102999015	FCMix-99086-43-4, 25ppb- 9/10/99 RWW	QC	25.10	7.984	8797	MM	31.57	26
16 102999016	MeOH Blk			7.989	596	MM	1.44	
17 102999017	TOX145-PFOSA in Tween-dose-200ppb- 10/29/99 IAS			7.943	72510	bd	265.65	
18 102999018	TOX145-PFOSA in Tween-ms-300ppb- 10/29/99 IAS			7.950	105109	db	385.42	
19 102999019	FCMix-99086-43-7, 250ppb- 9/10/99 RWW	QC	251.00	7.964	75327	MM	276.00	10
20 102999020	MeOH Blk			7.990	870	MM	2.45	
21 102999021	FCMix-99086-43-1, 1ppb- 9/10/99 RWW	Standard	1.01	7.947	783	MMX	2.13	111
22 102999022	FCMix-99086-43-2, 5ppb- 9/10/99 RWW	Standard	5.03	8.003	1994	MMX	6.58	31
23 102999023	FCMix-99086-43-3, 10ppb- 9/10/99 RWW	Standard	10.10	7.988	3433	MM	11.87	17
24 102999024	FCMix-99086-43-4, 25ppb- 9/10/99 RWW	Standard	25.10	7.948	8204	MM	29.39	17
25 102999025	FCMix-99086-43-5, 50ppb- 9/10/99 RWW	Standard	50.30	7.942	14786	MM	53.58	7
26 102999026	FCMix-99086-43-6, 100ppb- 9/10/99 RWW	Standard	101.00	7.965	30309	MM	110.61	10
27 102999027	FCMix-99086-43-7, 250ppb- 9/10/99 RWW	Standard	251.00	7.992	76998	MM	282.14	12
28 102999028	FCMix-99086-43-8, 500ppb- 9/10/99 RWW	Standard	503.00	7.989	140797	MM	516.53	3
29 102999029	FCMix-99086-43-9, 750ppb- 9/10/99 RWW	Standard	754.00	7.917	201774	MM	740.56	-2

Sample List: C:\MASSLYNX\soup020199.pro\SampleDB\102999a
 Last modified: Fri Oct 29 10:23:59 1999
 Method: C:\MASSLYNX\soup020199.pro\MethDB\102999a
 Last modified: Mon Nov 01 06:21:40 1999
 Job Code:

Printed: Mon Nov 01 06:47:34 1999

Compound 4: IS (Ch4)

# Name	Samplelist Text	Type	Std Conc	RT	Area Flags	Conc.	%Dev
1 102999001	MeOH Blk			7.170	15 bb	0.01	-99
2 102999002	MeOH Blk						
3 102999003	FCMIX-99086-43-1, 1ppb- 9/10/99 RWW	Standard	1.00	7.082	4292 bb	1.48	48
4 102999004	FCMIX-99086-43-2, 5ppb- 9/10/99 RWW	Standard	1.00	7.147	4289 bb	1.48	48
5 102999005	FCMIX-99086-43-3, 10ppb- 9/10/99 RWW	Standard	1.00	7.095	4424 bb	1.52	52
6 102999006	FCMIX-99086-43-4, 25ppb- 9/10/99 RWW	Standard	1.00	7.099	4486 bb	1.54	54
7 102999007	FCMIX-99086-43-5, 50ppb- 9/10/99 RWW	Standard	1.00	7.104	4446 bb	1.53	53
8 102999008	FCMIX-99086-43-6, 100ppb- 9/10/99 RWW	Standard	1.00	7.101	4093 bb	1.41	41
9 102999009	FCMIX-99086-43-7, 250ppb- 9/10/99 RWW	Standard	1.00	7.128	2630 bb	0.91	-9
10 102999010	FCMIX-99086-43-8, 500ppb- 9/10/99 RWW	Standard	1.00	7.120	1993 bb	0.69	-31
11 102999011	FCMIX-99086-43-9, 750ppb- 9/10/99 RWW	Standard	1.00	7.145	1842 bb	0.63	-37
12 102999012	MeOH Blk						
13 102999013	TOX145-PFOS/POAA in Tween-dose-2ppb- 10/29/99 IAS			7.169	2613 bb	0.90	-10
14 102999014	TOX145-PFOS/POAA in Tween-ms-4ppb- 10/29/99 IAS			7.150	1041 bb	0.36	-64
15 102999015	FCMIX-99086-43-4, 25ppb- 9/10/99 RWW	QC	1.00	7.163	2305 bb	0.79	-21
16 102999016	MeOH Blk						
17 102999017	TOX145-PFOA in Tween-dose-200ppb- 10/29/99 IAS			7.147	2529 bb	0.87	-13
18 102999018	TOX145-PFOA in Tween-ms-300ppb- 10/29/99 IAS			7.154	1016 bb	0.35	-65
19 102999019	FCMIX-99086-43-7, 250ppb- 9/10/99 RWW	QC	1.00	7.146	1991 bb	0.69	-31
20 102999020	MeOH Blk						
21 102999021	FCMIX-99086-43-1, 1ppb- 9/10/99 RWW	Standard	1.00	7.127	2320 bb	0.80	-20
22 102999022	FCMIX-99086-43-2, 5ppb- 9/10/99 RWW	Standard	1.00	7.163	2361 bb	0.81	-19
23 102999023	FCMIX-99086-43-3, 10ppb- 9/10/99 RWW	Standard	1.00	7.168	2396 bb	0.83	-17
24 102999024	FCMIX-99086-43-4, 25ppb- 9/10/99 RWW	Standard	1.00	7.174	2274 bb	0.78	-22
25 102999025	FCMIX-99086-43-5, 50ppb- 9/10/99 RWW	Standard	1.00	7.145	2435 bb	0.84	-16
26 102999026	FCMIX-99086-43-6, 100ppb- 9/10/99 RWW	Standard	1.00	7.147	2181 bb	0.75	-25
27 102999027	FCMIX-99086-43-7, 250ppb- 9/10/99 RWW	Standard	1.00	7.174	2059 bb	0.71	-29
28 102999028	FCMIX-99086-43-8, 500ppb- 9/10/99 RWW	Standard	1.00	7.146	1934 bb	0.67	-33
29 102999029	FCMIX-99086-43-9, 750ppb- 9/10/99 RWW	Standard	1.00	7.146	1818 bb	0.63	-37

Method Report

Page 1

Method file: C:\MASSLYNX\scup020199.pro\MethDB\102999a.mdb
 Last modified: Mon Nov 01 06:21:40 1999
 Compound: 1: POAA (Ch1)
 Printed: Mon Nov 01 07:36:09 1999

Compound Name: POAA (Ch1)
 Internal Ref.: [None]
 Quantify Trace: TIC
 Conc. of Standards: Conc. A
 User RF Value: 1.000000, Disabled
 User Peak Factor: 1.000000
 Integrate Window Factor: 2.000
 Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.173, Enabled
 Relative Retention Time: 0.000, Disabled
 RT Reference: [None]
 Time Window (mins): 0.312

Peak Selection: Nearest

Response Type: External
 Response: Areas

Polynomial Type: Linear
 Point of Origin: Exclude
 Fit Weighting: 1/X
 Axis Transformation: None

Units:

Peak-to-peak amplitude: 90

Smoothing: Enabled
 Window size (scans): 2
 Number of smooths: 1
 Smoothing method:

Join valley threshold: 30.00
 Reduce peak tailing: 200.00
 Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
 Detect shoulders: Disabled
 Shoulder threshold: 30.00

Relative height: 1.50, Disabled
 Absolute height: 0.00, Disabled
 Relative area: 2.00, Disabled
 Absolute area: 0.00, Disabled

000548

Method Report

Page 2

Method file: C:\MASSLYNX\scoup020199.pro\MethDB\102999a.mdb
Last modified: Mon Nov 01 06:21:40 1999
Compound: 2: PFOS (Ch2)
Printed: Mon Nov 01 07:36:09 1999

Compound Name: PFOS (Ch2)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. B
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.434, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.345

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 88

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000549

Las 11/01/99 7.10/4

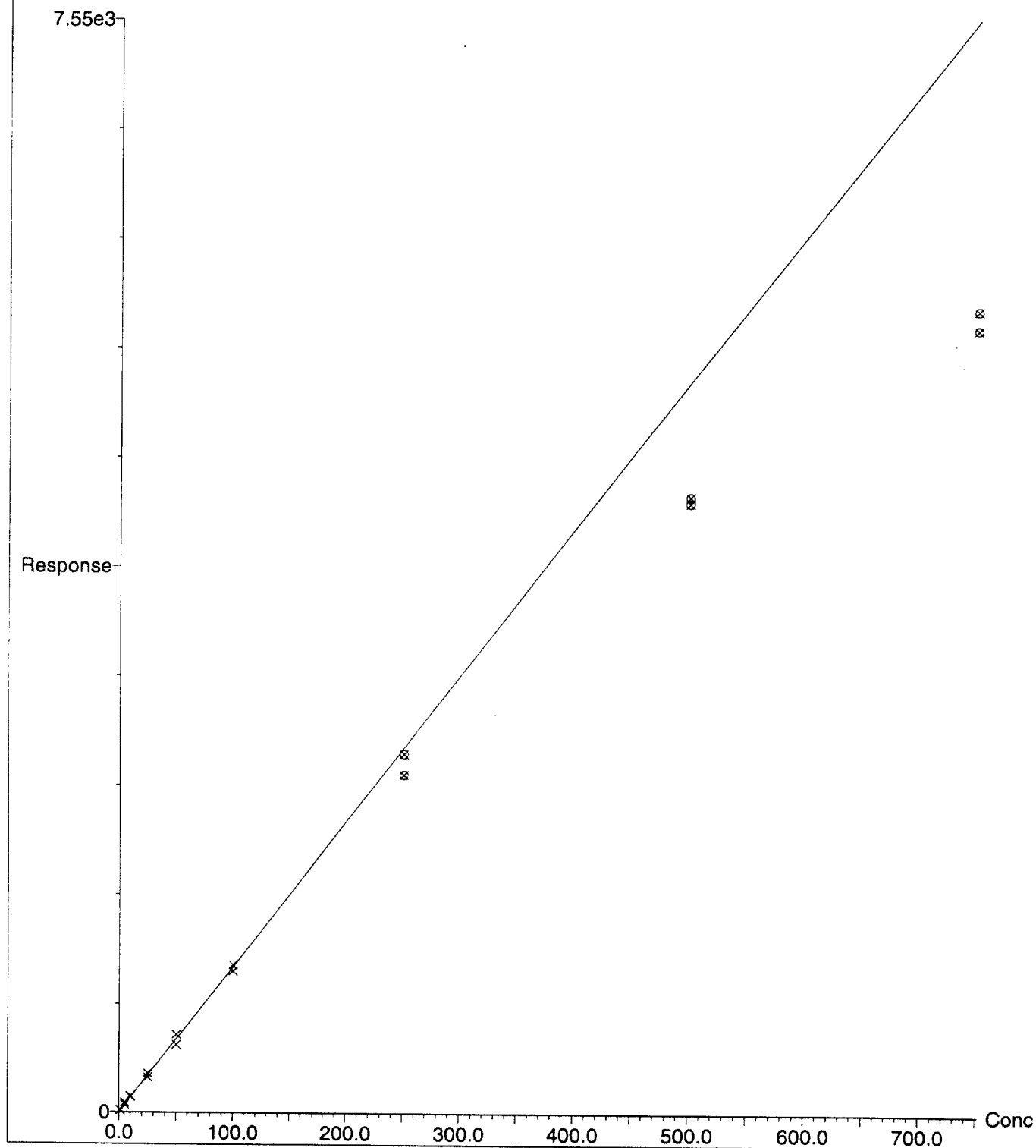
Compound 1 name: POAA (Ch1)

Coefficient of Determination: 0.997121

Calibration curve: $10.0347 * x + 5.23213$

Response type: External Std, Area

Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



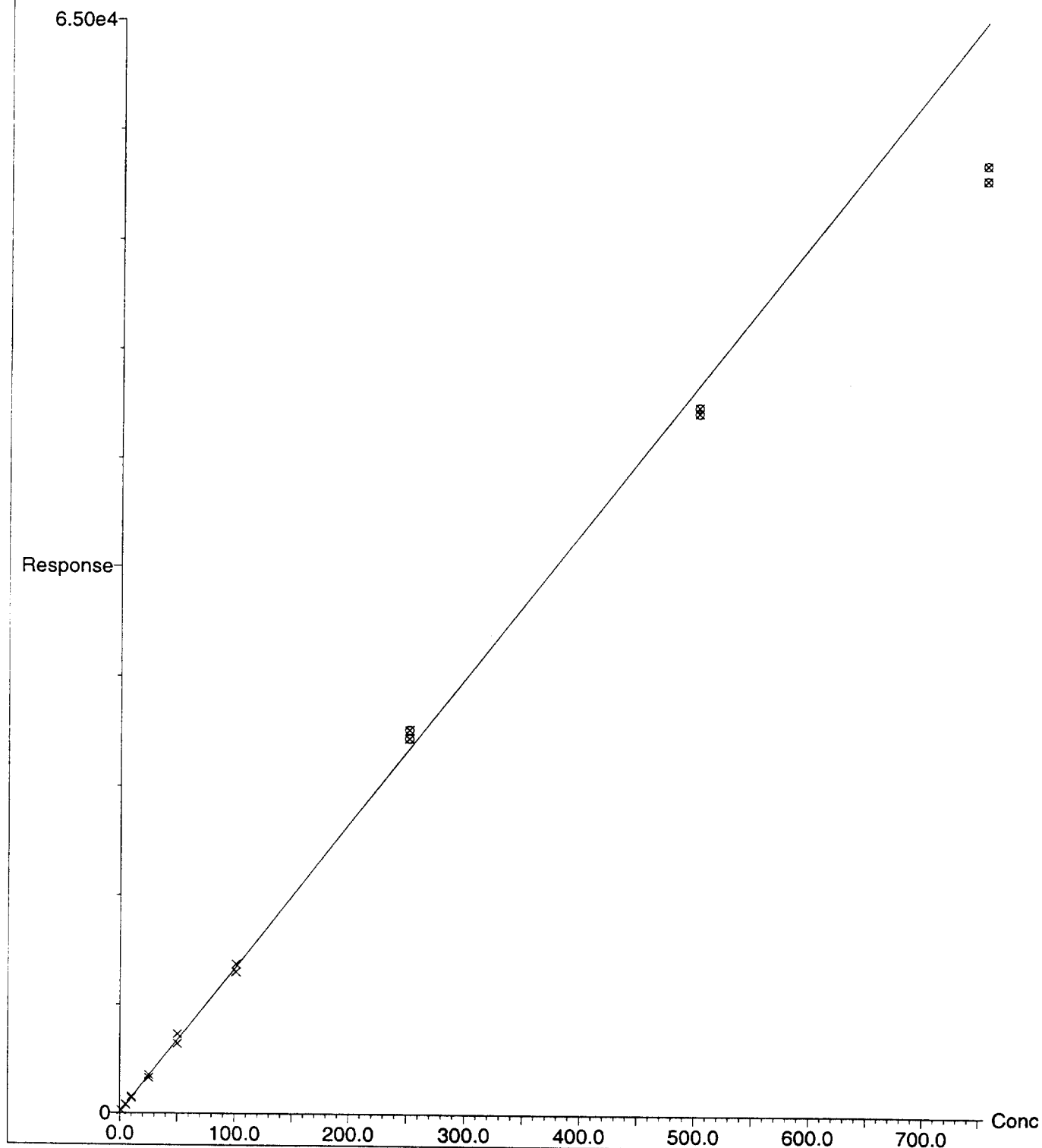
Compound 2 name: PFOS (Ch2)

Coefficient of Determination: 0.996935

Calibration curve: $86.0577 * x + 68.0228$

Response type: External Std, Area

Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



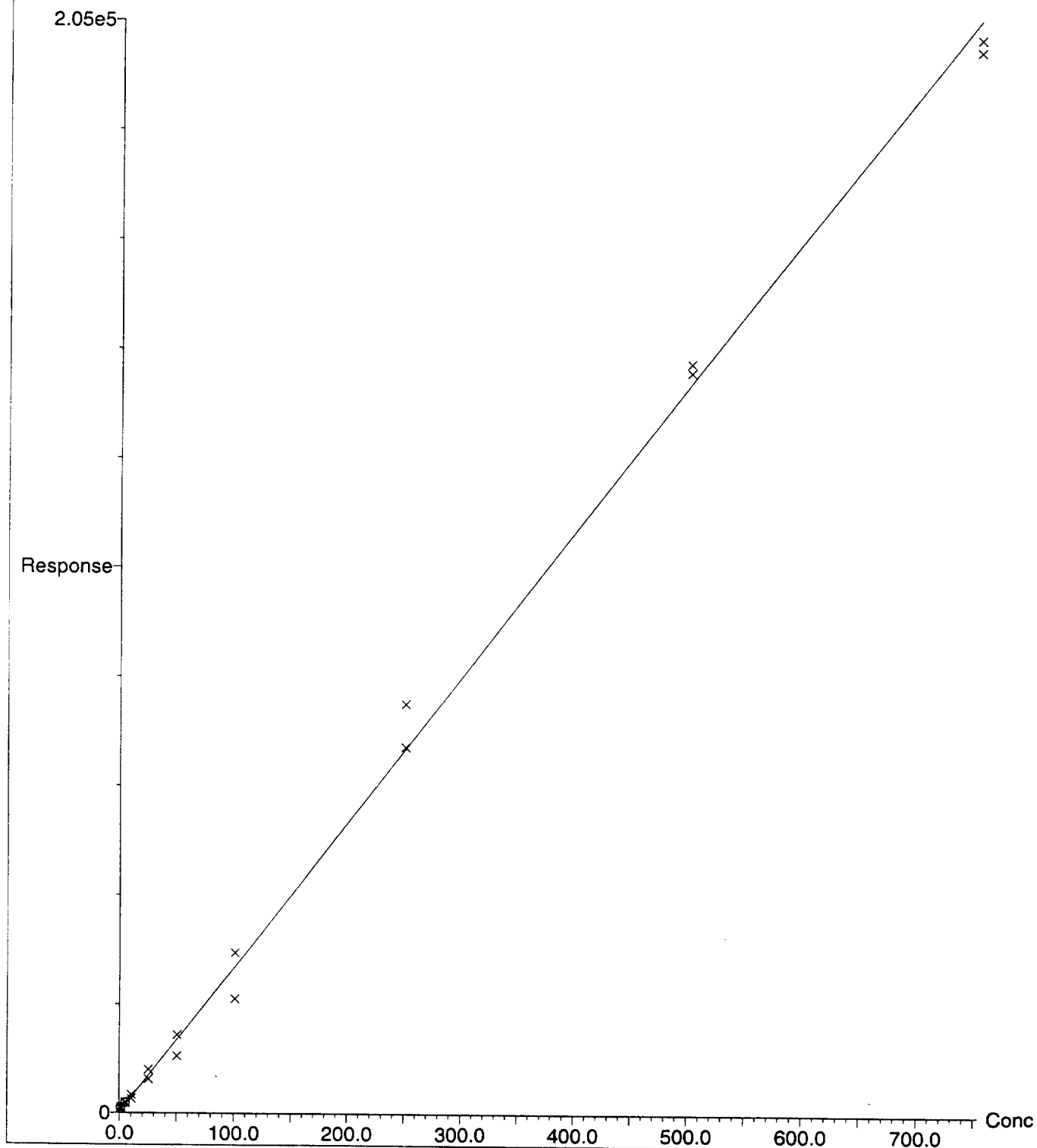
Compound 3 name: PFOSA (Ch3)

Coefficient of Determination: 0.997261

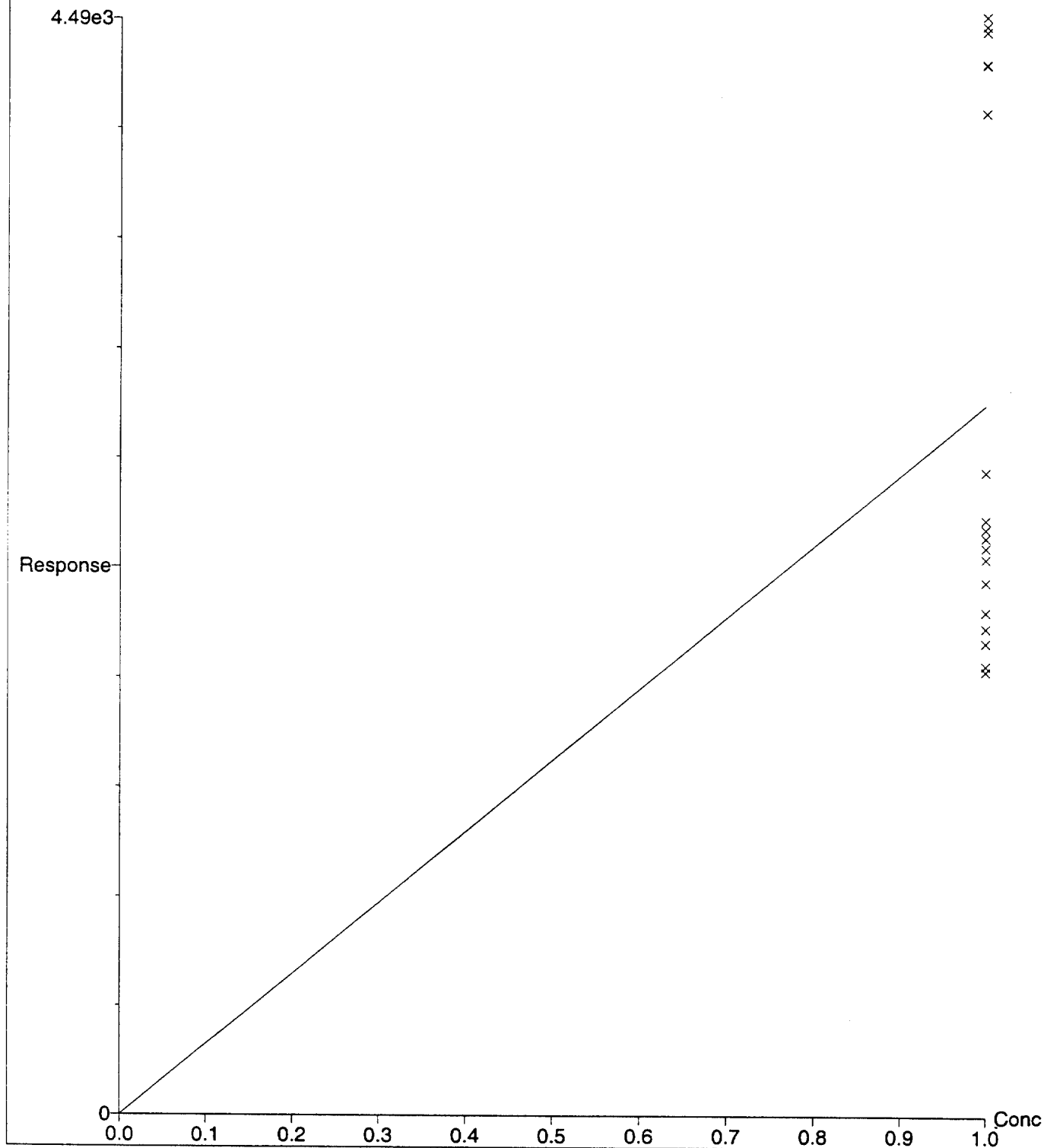
Calibration curve: $272.189 * x + 203.159$

Response type: External Std, Area

Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound 4 name: IS (Ch4)
Response Factor: 2904.16
Response type: External Std, Area
Curve type: RF



060553

Laboratory Report
Analytical Report of Data for
PFOSA Pharmacokinetic Study in Rats
(Sera and Liver)

Laboratory Report No. FACT-TOX-145 (W2814)

Requester Project No. T-7132.2

Attachment E
Extraction and Analytical Data

Extraction Worksheet ETS-8-6.0

Study # <u>Box-145</u>	Surrogate Std	FC Mix	FC Mix	FC Mix	Comments	Verified By
Matrix <u>Rat Liver</u>	Approx. ppm	approx.0.5 ppm	approx.5 ppm	approx.50 ppm		
Box # <u>Box 99-200</u>	Actual ppm	actual ppm	actual ppm	actual ppm		
WK/Day <u>4/1/99</u>	# <u>94086-13-11</u>	# <u>94086-47-2</u>	# <u>94086-47-1</u>	# <u>94095-33</u>		
Date/Spiked/Analyst	<u>10-13-99/SL</u>	<u>10-13-99/SL</u>	<u>10-13-99/SL</u>	<u>10-13-99/SL</u>		
RBL10139-250ppb-CCV-11-1 to 4	5 uL	-	10 uL	-	<u>10-13-99/SL</u>	<u>N/A</u>
9R03230M, Dy1, Control-250ppb-MS/D11	5 uL	-	10 uL	-		
9R03235M, Dy4, Control-250ppb-MS/D12	5 uL	-	10 uL	-		
RBL10139-H2O blk-11&12	5 uL	-	-	-		
RBL10139-Liver blk-11&12	5 uL	-	-	-		
RBL10139-5ppb-11&12	5 uL	2 uL	-	-		
RBL10139-10ppb-11&12	5 uL	4 uL	-	-		
RBL10139-25ppb-11&12	5 uL	10 uL	-	-		
RBL10139-50ppb-11&12	5 uL	20 uL	-	-		
RBL10139-100ppb-11&12	5 uL	40 uL	-	-		
RBL10139-250ppb-11&12	5 uL	-	10 uL	-		
RBL10139-500ppb-11&12	5 uL	-	20 uL	-		
RBL10139-750ppb-11&12	5 uL	-	30 uL	-		
RBL10139-1000ppb-11&12	5 uL	-	-	4 uL		
9R03230M, Dy1, Control	5 uL	-	-	-		
9R03231M, Dy1, Control	5 uL	-	-	-		<u>SL 10/13/99</u>
9R03232M, Dy1, Control	5 uL	-	-	-		<u>N/A</u>
9R03233M, Dy1, Control	5 uL	-	-	-		
9R03234M, Dy1, Control	5 uL	-	-	-		
9R03245M, Dy1, 5mg/kg	5 uL	-	-	-		
9R03246M, Dy1, 5mg/kg	5 uL	-	-	-		
9R03247M, Dy1, 5mg/kg	5 uL	-	-	-		
9R03248M, Dy1, 5mg/kg	5 uL	-	-	-		
9R03249M, Dy1, 5mg/kg	5 uL	-	-	-		
9R03235M, Dy4, Control	5 uL	-	-	-		
9R03236M, Dy4, Control	5 uL	-	-	-		
9R03237M, Dy4, Control	5 uL	-	-	-		
9R03238M, Dy4, Control	5 uL	-	-	-		
Blank - <u>Rabbit Liver</u>	Std# <u>94086-13-5</u>	amount = <u>1.0</u> ml	<u>0.163</u> g/ml			
Liver Extraction Method	ETS-8-6.0		Date & Initials			
Vortex 15 Sec.			<u>10-13-99/SL</u>			
Pipette Matrix, spike with appropriate surrogate or FC-Mix	Volume <u>1.0</u> ml					
Pipette 1 ml of 0.5 M TBA, pH 10. pH= <u>10</u>	<u>10-13-99/SL</u>	Std. # <u>94086-13-16</u>				<u>N/A 10/13/99</u>
Pipette 2 ml of 0.25 M Na ₂ CO ₃ /0.25 M NaHCO ₃ buffer		Std. # <u>94086-14-08</u>				
Dispense 5 ml of methyl-t-butyl ether		TN-A- <u>2946</u>				
Shake 20 min	Shaker speed: <u>300</u>					<u>N/A</u>
Centrifuge 20-25 min.	Centrifuge speed: <u>3500</u>					
Remove a 4 ml aliquot of organic layer						
Put on Nitrogen Evaporator to dryness	Temperature: <u>Room Temp.</u>					
Add methanol	Volume <u>1.0</u> ml	TN-A- <u>2945</u>			<u>10-14-99/SL</u>	
Vortex 30 sec.						
Filter using a 3cc B-D syringe with a 0.2 um filter into an autosampler vial						

* Watched and Verified Surrogate Spiking SL 10/13/99
 FN-1 3ml of 1000ppb #12 was added to 4ml of 750ppb during 4ml aliquot
 of organic layer. 1 ml of 1000ppb went into the correct tube. 10-13-99/SL

Extraction Worksheet ETS-8-6.0

[illegible]

Re: 10-13-98 Sc

10/12/99 Draft Attachment A

ETS-8-6.0

Extraction of PFOS from Liver

Form Completion
Verified By SAL 10/14/99

000556

Printed 10-12-99
See

Sample Weights/ Volumes

Prep Date(s): 10-12-99
Analyst(s): SEE
Sample Matrix: Rat Liver
Method/Revision: ETS-8-6.0
Target Analyte(s): PFOS, PFOSA

Study Number: TOX-145
Equipment Number: 0222-0900
Final Solvent & TN Number: MeOH / TN-A-2945
Matrix Blank/Identifier: Rabbit Liver
Box: 99-200

Sample ID	Initial Weight grams	Homogenized By:	Verified By:	Comments
9R03230M,Dy1,Control	0.9964	10/13/99-See	N/A	Used for MS/D 11
9R03231M,Dy1,Control	1.0159			N/A
9R03232M,Dy1,Control	1.0000			
9R03233M,Dy1,Control	0.9978			
9R03234M,Dy1,Control	1.0137			
9R03245M,Dy1,5mg/kg	1.0171		MMT 10/12/99	
9R03246M,Dy1,5mg/kg	1.0074		N/A	
9R03247M,Dy1,5mg/kg	1.0105			
9R03248M,Dy1,5mg/kg	1.0169			
9R03249M,Dy1,5mg/kg	0.9913			
9R03235M,Dy4,Control	1.0014			Used for MS/D 12
9R03236M,Dy4,Control	1.0067			N/A
9R03237M,Dy4,Control	1.0024			
9R03238M,Dy4,Control	0.9905			
9R03239M,Dy4,Control	1.0191			
9R03250M,Dy4,5mg/kg	1.0058			
9R03251M,Dy4,5mg/kg	1.0022			
9R03252M,Dy4,5mg/kg	1.0081			
9R03253M,Dy4,5mg/kg	0.9978			
9R03254M,Dy4,5mg/kg	1.0137			
		10-12-99	See	

Summary of method:

Notes:

Review Date _____

Rtl10139

Form Approved by/Date: _____

Form Completion

Verified By: SM 10/14/99

000557

Printed
10-14-98
Lan

Ion Pair Standard Curves--Tissues

Prep Date(s): 10/14/98
Analyst(s): SEE
Sample Matrix: Rat Liver
Method/Revision: ETS-8-6.0
Target Analyte(s): FC-Mix
Box Number: 99-200

Study Number: TOX-145
Equipment Number: 0222-0900
Final Solvent & TN Number: MeOH/TN-A-2945
Blank Tissue/Identifier: Rabbit Liver/99086-13-5

FC Mix Std Approx. 0.500 ppm: 99086-47-2
FC Mix Std Approx. 5.00 ppm: 99086-47-1
FC Mix Std Approx. 50.0 ppm: 99095-33
Surrogate Std Approx. 100 ppm: 99086-13-11

Blank liver initial density (g) for curves: 0.163
Final solvent volume (mL) for curves: 1.000

Actual Concentrations of Standards in the FC Mix

PFOS Std Conc ug/mL	PFOSA Std Conc ug/mL	PFOSEA Std Conc ug/mL	PFOSAA Std Conc ug/mL	EtFOSE-OH Std Conc ug/mL	M556 Std Conc ug/mL	All Am't Spiked mL	All Density g	All Vol. mL
0.5000	0.5005	0.4997	0.4999	0.5005	0.5010	0.002	0.163	1.000
0.5000	0.5050	0.4997	0.4999	0.5005	0.5010	0.004	0.163	1.000
0.5000	0.5050	0.4997	0.4999	0.5005	0.5010	0.010	0.163	1.000
0.5000	0.5050	0.4997	0.4999	0.5005	0.5010	0.020	0.163	1.000
0.5000	0.5050	0.4997	0.4999	0.5005	0.5010	0.040	0.163	1.000
5.000	5.005	4.997	4.999	5.005	5.010	0.010	0.163	1.000
5.000	5.005	4.997	4.999	5.005	5.010	0.020	0.163	1.000
5.000	5.005	4.997	4.999	5.005	5.010	0.030	0.163	1.000
50.00	50.05	49.97	49.99	50.05	50.10	0.004	0.163	1.000

Calculated Concentrations of Standards in the sample matrix

PFOS Std Conc. ng/g	PFOSA Std Conc. ng/g	PFOSEA Std Conc. ng/g	PFOSAA Std Conc. ng/g	EtFOSE-OH Std Conc. ng/g	M556 Std Conc. ng/g	Surrogate Std Conc. ug/mL	All Am't Spiked mL
6.13	6.14	6.13	6.13	6.14	6.15	100	0.005
12.3	12.4	12.3	12.3	12.3	12.3	Surrogate	
30.7	31.0	30.7	30.7	30.7	30.7	Final Conc.	
61.3	62.0	61.3	61.3	61.4	61.5	ng/g	
123	124	123	123	123	123	3077	
307	307	307	307	307	307		
613	614	613	613	614	615		
920	921	920	920	921	922		
1227	1228	1226	1227	1228	1229		

Calculated Concentrations of Standards in the 1.0 mL final solvent volume

PFOS Final Conc. ng/mL	PFOSA Final Conc. ng/mL	PFOSEA Final Conc. ng/mL	PFOSAA Final Conc. ng/mL	EtFOSE-OH Final Conc. ng/mL	M556 Std Conc. ng/mL	Surrogate Std Conc. ug/mL	All Am't Spiked mL
1.00	1.00	1.00	1.00	1.00	1.00	100	0.005
2.00	2.02	2.00	2.00	2.00	2.00	Surrogate	
5.00	5.05	5.00	5.00	5.01	5.01	Final Conc.	
10.0	10.1	10.0	10.0	10.0	10.0	ng/mL	
20.0	20.2	20.0	20.0	20.0	20.0	502	
50.0	50.1	50.0	50.0	50.1	50.1		
100	100	100	100	100	100		
150	150	150	150	150	150		
200	200	200	200	200	200		

Sample used for MS/MSD: 9R03230M, Dy1. Control

MS/MSD calc. In matrix Initial Wt (g): 0.996

Initial Volume (mL): 5.00

Approx. Std Conc.: 5.00 ug/mL

Am't Spiked (mL) Density
0.010 0.166

PFOS Final Conc. ng/g	PFOSA Final Conc. ng/g	PFOSEA Final Conc. ng/g	PFOSAA Final Conc. ng/g	EtFOSE-OH Final Conc. ng/g	M556 Std Conc. ng/g
301	301	301	301	301	302
301	301	301	301	301	302

Sample used for MS/MSD: 03230M, Dy1. Control

MS/MSD calc. In solvent Initial Wt (g): 0.996

Initial Volume (mL): 5.00

Approx. Std Conc.: 5.00 ug/mL

Am't Spiked (mL) Final Vol.
0.010 1.000

PFOS Final Conc. ng/g	PFOSA Final Conc. ng/g	PFOSEA Final Conc. ng/g	PFOSAA Final Conc. ng/g	EtFOSE-OH Final Conc. ng/g	M556 Std Conc. ng/g
50.0	50.1	50.0	50.0	50.1	50.1
50.0	50.1	50.0	50.0	50.1	50.1

+ ng/g should read ng/mL 11/4/98

Sample used for MS/MSD: 9R03233M, Dy4. control

MS/MSD calc. In matrix Initial Wt (g): 1.001

Initial Volume (mL): 5.00

Approx. Std Conc.: 5.00 ug/mL

Am't Spiked (mL) Density
0.010 0.167

PFOS Final Conc. ng/g	PFOSA Final Conc. ng/g	PFOSEA Final Conc. ng/g	PFOSAA Final Conc. ng/g	EtFOSE-OH Final Conc. ng/g	M556 Std Conc. ng/g
301	301	301	301	301	302
301	301	301	301	301	302

Sample used for MS/MSD: 03233M, Dy4. control

MS/MSD calc. In solvent Initial Wt (g): 1.001

Initial Volume (mL): 5.00

Approx. Std Conc.: 5.00 ug/mL

Am't Spiked (mL) Final Vol.
0.010 1.000

PFOS Final Conc. ng/g	PFOSA Final Conc. ng/g	PFOSEA Final Conc. ng/g	PFOSAA Final Conc. ng/g	EtFOSE-OH Final Conc. ng/g	M556 Std Conc. ng/g
50.0	50.1	50.0	50.0	50.1	50.1
50.0	50.1	50.0	50.0	50.1	50.1

+ ng/g should read ng/mL 11/4/98

Ion Pair Std Extraction Sheet

Verified
11/4/98

000558

DILUTIONS SUMMARY SHEET

Study: TOX-045

Dilution Date/Analyst: MCH 10/18/99 *MCH 10/18/99*

Box #: 99-200

Solvent/TN Number: MeOH TN-A-2945 MW 10/18/99

Extraction Date/Analyst: SEE, 10/13/99

Matrix/Timepoint: Rat Liver

[illegible]

Notes: D100=10 uL sample + 990 uL MeOH. 11/18/99

Form Completion

Verified By: 10-17-99 / *ser*

00000

Extraction Worksheet ETS-8-4.1

Study # <u>Box 145</u>	Surrogate Std	FC Mix	FC Mix	FC Mix	Comments	Verified By
Matrix <u>Rat Serum</u>	Approx. ppm	approx. 0.5 ppm	approx. 5 ppm	approx. 50 ppm		
Box # <u>99-211</u>	Actual ppm	actual ppm	actual ppm	actual ppm		
WK/Day <u>dy 1-4</u>	# <u>9906-13-11</u>	# <u>9906-47-2</u>	# <u>9906-47-1</u>	# <u>99095-33</u>		
Date Spiked/Analyst	<u>10-19-99/SC</u>	<u>10-19-99/SC</u>	<u>10-19-99/SC</u>	<u>10-19-99/SC</u>		
RBS10199-250ppb-CCV-11-1 to 4	5 uL	-	-	5 uL	n/A	n/A
RTS10199-250ppb-MS/D-11	5 uL	-	-	5 uL		
RTS10199-Serum Blank-11&12	5 uL	-	-	-		
RBS10199-H2O blk-11&12	5 uL	-	-	-		
RBS10199-Serum blk-11&12	5 uL	-	-	-		
RBS10199-5ppb-11&12	5 uL	10 uL	-	-		
RBS10199-10ppb-11&12	5 uL	20 uL	-	-		
RBS10199-25ppb-11&12	5 uL	-	5 uL	-		
RBS10199-50ppb-11&12	5 uL	-	10 uL	-		
RBS10199-100ppb-11&12	5 uL	-	20 uL	-		
RBS10199-250ppb-11&12	5 uL	-	-	5 uL		
RBS10199-500ppb-11&12	5 uL	-	-	10 uL		
RBS10199-750ppb-11&12	5 uL	-	-	15 uL		
RBS10199-1000ppb-11&12	5 uL	-	-	20 uL		
9R03230M, Dy1, Control	5 uL	-	-	-		
9R03231M, Dy1, Control	5 uL	-	-	-		
9R03232M, Dy1, Control	5 uL	-	-	-		
9R03233M, Dy1, Control	5 uL	-	-	-		10/19/99
9R03234M, Dy1, Control	5 uL	-	-	-		n/A
9R03245M, Dy1, 5mg/kg	5 uL	-	-	-		
9R03246M, Dy1, 5mg/kg	5 uL	-	-	-		
9R03247M, Dy1, 5mg/kg	5 uL	-	-	-		
9R03248M, Dy1, 5mg/kg	5 uL	-	-	-		
9R03249M, Dy1, 5mg/kg	5 uL	-	-	-		
9R03235M, Dy4, Control	5 uL	-	-	-		
9R03236M, Dy4, Control	5 uL	-	-	-		
9R03237M, Dy4, Control	5 uL	-	-	-		
9R03238M, Dy4, Control	5 uL	-	-	-		
Blank Rabbit to Serum Std# 99062-39 amount = 1.0 ml						
Serum Extraction Method ETS-8-4.1 : Date & Initials						
Vortex 15 Sec. 10-19-99/SC						
Pipette Matrix, spike with appropriate surrogate or FC-Mix Volume 1.0 ml						
Pipette 1 ml of 0.5 M TBA, pH 10. pH=10-10/19/99 Std. # 99076-13-16						
Pipette 2 ml of 0.25 M Na ₂ CO ₃ /0.25 M NaHCO ₃ buffer Std. # 99076-11-8						
Dispense 5 ml of methyl-t-butyl ether TN-A-2946						
Shake 20 min Shaker speed: 300						
Centrifuge 20-25 min. Centrifuge speed: 3500						
Remove a 4 ml aliquot of organic layer						
Put on Nitrogen Evaporator to dryness Temperature: Room Temp.						
Add methanol Volume 1.0 TN-A-2945						
Vortex 30 sec.						
Filter using a 3cc B-D syringe with a 0.2 um filter into an autosampler vial						
Cont. Cal. Verifications used same matrix as for std curve.						

(E) 10-19-99/SC

FN-1: The rat serum blanks + ms/D were from control rat serum 99062/88 10-17-99/SC

Extraction Worksheet ETS-8-4.1

[illegible]

000551

Sample Weights/ Volumes

Prep Date(s): 10/19/99

Analyst(s): SEE

Sample Matrix: Rat Serum

Method/Revision: ETS-8-4.1

Target Analyte(s): PFOS, PFOSA

Study Number: TOX-145

Box: 99-206

Final Solvent & TN Number: MeOH/TN-A-2945

Matrix Blank/Identifier: Rabbit Serum/99062-39

Rat Serum/99062-88

Sample ID	Initial Vol. mL		Verified By:	Comments
9R03230M,Dy1,Control	1.0 mL	N/A	N/A	N/A
9R03231M,Dy1,Control				
9R03232M,Dy1,Control				
9R03233M,Dy1,Control				
9R03234M,Dy1,Control				
9R03245M,Dy1,5mg/kg				
9R03246M,Dy1,5mg/kg	-			
9R03247M,Dy1,5mg/kg				
9R03248M,Dy1,5mg/kg				
9R03249M,Dy1,5mg/kg				
9R03235M,Dy4,Control				
9R03236M,Dy4,Control				
9R03237M,Dy4,Control				
9R03238M,Dy4,Control				
9R03239M,Dy4,Control				
9R03250M,Dy4,5mg/kg				
9R03251M,Dy4,5mg/kg				
9R03252M,Dy4,5mg/kg				
9R03254-1M,Dy4,5mg/kg				
9R03254-2M,Dy4,5mg/kg				
		10-19-99 <i>[Signature]</i>		

Summary of method:

Notes:

Review Date

Rts10199

Form Completion
Verified By: MM 10/19/99

000562

Printed 10/19/99
sc

Ion Pair Standard Curves--Fluids

Prep Date(s): 10/19/99
Analyst(s): SEE
Sample Matrix: Rat Serum
Method/Revision: ETS-8-1
Target Analyte(s): PFOS, PFOSA
Box Number: 99-206

Study Number: TOX-145
Equipment Number:
Final Solvent & TN Number: MeOH/TN-A-2945
Blank Tissue/Identifier: Rabbit Serum/99062-39
Rat Serum/99062-88

FC Mix Std Approx. 0.500 ppm: 99086-47-2
FC Mix Std Approx. 5.00 ppm: 99086-47-1
FC Mix Std Approx. 50.0 ppm: 99095-33
Surrogate Std Approx. 100 ppm: 99086-13-11

Blank serum initial volume (mL) for curves: 1.00
Final solvent volume (mL) for curves: 1.00

Actual Concentrations of Standards in the FC Mix

PFOS Std Conc. ug/mL	PFOSA Std Conc. ug/mL	PFOSEA Std Conc. ug/mL	PFOSAA Std Conc. ug/mL	EtFOSE-OH Std Conc. ug/mL	M556 Std Conc. ug/mL	All Am't Spiked mL	All initial Volum mL	All Final Vol. mL
0.5000	0.5005	0.4997	0.4999	0.5005	0.5010	0.010	1.015	1.00
0.5000	0.5005	0.4997	0.4999	0.5005	0.5010	0.020	1.025	1.00
5.000	5.005	4.997	4.999	5.005	5.010	0.005	1.010	1.00
5.000	5.005	4.997	4.999	5.005	5.010	0.010	1.015	1.00
5.000	5.005	4.997	4.999	5.005	5.010	0.020	1.025	1.00
50.00	50.05	49.97	49.99	50.05	50.10	0.005	1.010	1.00
50.00	50.05	49.97	49.99	50.05	50.10	0.010	1.015	1.00
50.00	50.05	49.97	49.99	50.05	50.10	0.015	1.020	1.00
50.00	50.05	49.97	49.99	50.05	50.10	0.020	1.025	1.00

Calculated Concentrations of Standards in the sample matrix

PFOS Std Conc. ng/mL	PFOSA Std Conc. ng/mL	PFOSEA Std Conc. ng/mL	PFOSAA Std Conc. ng/mL	EtFOSE-OH Std Conc. ng/mL	M556 Std Conc. ng/mL	Surrogate Std Conc. ug/mL	All Am't Spiked mL
4.93	4.93	4.92	4.93	4.93	4.94	101	0.005
9.76	9.77	9.75	9.75	9.77	9.78		
24.8	24.8	24.7	24.7	24.8	24.8		
49.3	49.3	49.2	49.3	49.3	49.4		
97.6	97.7	97.5	97.5	97.7	97.8		
248	248	247	247	248	248		
493	493	492	493	493	494		
735	736	735	735	736	737		
976	977	975	975	977	978		

Calculated Concentrations of Standards in the 1.0 mL final solvent volume

PFOS Final Conc. ng/mL	PFOSA Final Conc. ng/mL	PFOSEA Final Conc. ng/mL	PFOSAA Final Conc. ng/mL	EtFOSE-OH Final Conc. ng/mL	M556 Std Conc. ng/mL	Surrogate Std Conc. ug/mL	All Am't Spiked mL
5.00	5.01	5.00	5.00	5.01	5.01	101	0.005
10.0	10.0	10.0	10.0	10.0	10.0		
25.0	25.0	25.0	25.0	25.0	25.1		
50.0	50.1	50.0	50.0	50.1	50.1		
100	100	100	100	100	100		
250	250	250	250	250	251		
500	501	500	500	501	501		
750	751	750	750	751	752		
1000	1001	999	1000	1001	1002		

Sample used for MS/MSD: Rat control serum/99062-88

MS/MSD Conc. in the sample matrix

Initial Volume (mL): 1.00

Approx. Std Conc.: 50.0 ug/mL

PFOS Final Conc. ng/mL	PFOSA Final Conc. ng/mL	PFOSEA Final Conc. ng/mL	PFOSAA Final Conc. ng/mL	EtFOSE-OH Final Conc. ng/mL	M556 Std Conc. ng/mL
248	248	247	247	248	248
248	248	247	247	248	248

Am't Spiked
Vol. (mL)
0.005

Sample used for MS/MSD: Rat control serum/99062-88

MS/MSD Conc. in the final solvent

Initial Volume (mL): 1.00

Approx. Std Conc.: 50.0 ug/mL

PFOS Std Conc. ug/mL	PFOSA Std Conc. ug/mL	PFOSEA Std Conc. ug/mL	PFOSAA Std Conc. ug/mL	EtFOSE-OH Std Conc. ug/mL	M556 Std Conc. ug/mL
250	250	250	250	250	251
250	250	250	250	250	251

Am't Spiked
Vol. (mL)
0.005
Final Vol. (mL)
1.00

491/mL
← should read 735
731/mL

DILUTIONS SUMMARY SHEET

Study: TOX-145

Dilution Date/Analyst: MCH, 10/21/99 *MCH*
10/21/99

Box #: 99-206

Solvent/TN Number: MeOH, TN-A-2945

Extraction Date/Analyst: SEE, 10/19/99

Matrix/Timepoint: Rat Sera Days 1&4

sample number or description	DILUTIONS							Comments	Verified By
	1:50	1:	1:	1:	1:	1:	1:		
9R03245M, Day 1, 5mg/kg	D50	-	-	-	-	-	-	N/A	GML 10/21/99
9R03246M, Day 1, 5mg/kg	D50	-	-	-	-	-	-		N/A
9R03247M, Day 1, 5mg/kg	D50	-	-	-	-	-	-		
9R03248M, Day 1, 5mg/kg	D50	-	-	-	-	-	-		
9R03249M, Day 1, 5mg/kg	D50	-	-	-	-	-	-		
9R03250M, Day 4, 5mg/kg	D50	-	-	-	-	-	-		
9R03251M, Day 4, 5mg/kg	D50	-	-	-	-	-	-		
9R03252M, Day 4, 5mg/kg	D50	-	-	-	-	-	-		
9R03254-1M, Day 4, 5mg/kg	D50	-	-	-	-	-	-		
9R03254-2M, Day 4, 5mg/kg	D50	-	-	-	-	-	-		
 over 10/21/99									

Notes: D50=20 uL sample + 980 uL MeOH. *ms 10/21/99*

Form Completion
Verified By: GML 10/24/99

000564

Extraction Worksheet ETS-8-6.0

① Rwn - 11/19/99

Study #	Surrogate Std	FC Mix	FC Mix	FC Mix	Comments	Verified By
Matrix	Approx. ppm	approx. 0.5 ppm	approx. 5 ppm	approx. 50 ppm		
Box #	Actual 101 ppm	actual ppm	actual ppm	actual ppm		
WK/Day	#	#	#	#		
Date Spiked/Analyst						
RBL 10229 - H2O Blk -1	5.0 ul					
-Liver Blk -1	5.0 ul					
-6ppb-1	5.0 ul	2 ul				
-12ppb-1	5.0 ul	4 ul				
-30ppb-1	5.0 ul	10 ul				
-60ppb-1	5.0 ul	20 ul				
-120ppb-1	5.0 ul	40 ul				
-300ppb-1	5.0 ul		10 ul			
-600ppb-1	5.0 ul		20 ul			
-900ppb-1	5.0 ul		30 ul			
-1200ppb-1	5.0 ul			4 ul		
10/22/99 Rwn						
Blank - Rabbit Liver	Std# 99086-13-5	amount = 1.0 ml	0.163 g/ml			
Liver Extraction Method	ETS-8-6.0		Date & Initials			
Vortex 15 Sec.			10/22/99 - Rwn			
Pipette Matrix, spike with appropriate surrogate or FC-Mix	Volume 1.0 ml					
Pipette 1 ml of 0.5 M TBA, pH 10. pH= 10	10/22/99 - Rwn	Std. # 99086-13-16				
Pipette 2 ml of 0.25 M Na ₂ CO ₃ /0.25 M NaHCO ₃ buffer		Std. # 99086-14-08				
Dispense 5 ml of methyl-t-butyl ether	TN-A- 2946					
Shake 20 min	Shaker speed: 300					
Centrifuge 20-25 min.	Centrifuge speed: 3500					
Remove a 4 ml aliquot of organic layer						
Put on Nitrogen Evaporator to dryness	Temperature: Room Temp.					
Add methanol	Volume 1.0 ml	TN-A- 2945	10/25/99 - Rwn			
Vortex 30 sec.						
Filter using a 3cc B-D syringe with a 0.2 um filter into an autosampler vial						
Cont. Cal. Verifications used same matrix as for std curve.						

Ion Pair Standard Curves--Tissues

Prep Date(s): 10/25/99
 Analyst(s): RWW
 Sample Matrix: Rabbit Liver
 Method/Revision: ETS-8-6.0
 Target Analyte(s): See below
 Box Number: 99-206

Study Number: TOX145
 Equipment Number:
 Final Solvent & TN Number: MeOH/TN-A-2945
 Rabbit Liver/99086-13-5

FC Mix Std Approx. 0.500 ppm: 99086-44-3
 FC Mix Std Approx. 5.00 ppm: 99086-44-2
 FC Mix Std Approx. 50.0 ppm: 99086-44-1
 Surrogate Std Approx. 101 ppm: 99086-13-11

Blank liver initial density (g) for curves: 0.163
 Final solvent volume (mL) for curves: 1.000

Actual Concentrations of Standards in the FC Mix

PFOS Std Conc. ug/mL	PFOSA Std Conc. ug/mL	EtFOSE-OH Std Conc. ug/mL	M556 Std Conc. ug/mL	M570 Std Conc. ug/mL	MeFOSE-OH Std Conc. ug/mL	POAA Std Conc. ug/mL	PFHS Std Conc. ug/mL
0.5004	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000
0.5004	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000
0.5004	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000
0.5004	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000
0.5004	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000
5.004	5.000	5.000	5.000	5.000	5.000	5.000	5.000
5.004	5.000	5.000	5.000	5.000	5.000	5.000	5.000
5.004	5.000	5.000	5.000	5.000	5.000	5.000	5.000
50.04	50.00	50.00	50.00	50.00	50.00	50.00	50.00

All Am't Spiked mL	All Density g	All Vol. mL
0.002	0.163	1.000
0.004	0.163	1.000
0.010	0.163	1.000
0.020	0.163	1.000
0.040	0.163	1.000
0.010	0.163	1.000
0.020	0.163	1.000
0.030	0.163	1.000
0.004	0.163	1.000

Calculated Concentrations of Standards in the sample matrix

PFOS Std Conc. ng/g	PFOSA Std Conc. ng/g	EtFOSE-OH Std Conc. ng/g	M556 Std Conc. ng/g	M570 Std Conc. ng/g	MeFOSE-OH Std Conc. ng/g	POAA Std Conc. ng/g	PFHS Std Conc. ng/g	Surrogate Std Conc. ug/mL	All Am't Spiked mL
6.14	6.13	6.13	6.13	6.13	6.13	6.13	6.13	101	0.005
12.3	12.3	12.3	12.3	12.3	12.3	12.3	12.3		
30.7	30.7	30.7	30.7	30.7	30.7	30.7	30.7		
61.4	61.3	61.3	61.3	61.3	61.3	61.3	61.3		
123	123	123	123	123	123	123	123		
307	307	307	307	307	307	307	307		
614	613	613	613	613	613	613	613		
921	920	920	920	920	920	920	920		
1228	1227	1227	1227	1227	1227	1227	1227		

Calculated Concentrations of Standards in the 1.0 mL final solvent volume

PFOS Final Conc. ng/mL	PFOSA Final Conc. ng/mL	EtFOSE-OH Final Conc. ng/mL	M556 Final Conc. ng/mL	M570 Final Conc. ng/mL	MeFOSE-OH Std Conc. ng/mL	POAA Std Conc. ng/mL	PFHS Std Conc. ng/mL	Surrogate Std Conc. ug/mL	All Am't Spiked mL
1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	101	0.005
2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00		
5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00		
10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0		
20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0		
50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0		
100	100	100	100	100	100	100	100		
150	150	150	150	150	150	150	150		
200	200	200	200	200	200	200	200		

Sample used for MS/MSD: No MS/MSD

MS/MSD calc. In matrix Initial Wt. (g): 1.000

Initial Volume (mL): 5.00 6.00

Approx. Std Conc.: 5.00 ug/mL

Am't Spiked (mL) Density
0.010 0.167

PFOS Final Conc. ng/g	PFOSA Final Conc. ng/g	EtFOSE-OH Final Conc. ng/g	M556 Final Conc. ng/g	M570 Final Conc. ng/g	MeFOSE-OH Std Conc. ng/g	POAA Std Conc. ng/g	PFHS Std Conc. ng/g
300	300	300	300	300	300	300	300
300	300	300	300	300	300	300	300

Sample used for MS/MSD: No MS/MSD

MS/MSD calc. In solvent Initial Wt. (g): 1.000

Initial Volume (mL): 5.00 6.00

Approx. Std Conc.: 5.00 ug/mL

Am't Spiked (mL) Final Vol.
0.010 1.000

PFOS Final Conc. ng/g	PFOSA Final Conc. ng/g	EtFOSE-OH Final Conc. ng/g	M556 Final Conc. ng/g	M570 Final Conc. ng/g	MeFOSE-OH Std Conc. ng/g	POAA Std Conc. ng/g	PFHS Std Conc. ng/g
50	50	50	50	50	50	50	50
50	50	50	50	50	50	50	50

ng/g should read
ng/mL 11/4/99

See 10/25/99

Extraction Worksheet ETS-8-4.1

Study #	Surrogate Std	FC Mix	FC Mix	FC Mix	Comments	Verified By
Matrix	Approx. ppm	approx. 0.5 ppm	approx. 5 ppm	approx. 50 ppm		
Box #	Actual ppm	actual ppm	actual ppm	actual ppm		
WK/Day	#	#	#	#		
Date Spiked/Analyst						
Study # <u>FOX 142</u>						
Matrix <u>Robert Serum</u>						
Box # <u>99-200</u>						
WK/Day <u>NA</u>						
Date Spiked/Analyst <u>10/22/99-RWW</u>						
<u>R4510229</u> -H2OBik -1	5.0 ul	N/A	N/A	N/A		
-SeraBik -1	5.0 ul					
-5ppb -1	5.0 ul	10 ul				
-10ppb -1	5.0 ul	20 ul				
-25ppb -1	5.0 ul	N/A	5 ul			
-50ppb -1	5.0 ul		10 ul			
-100ppb -1	5.0 ul		20 ul			
-250ppb -1	5.0 ul		N/A	5 ul		
-500ppb -1	5.0 ul			10 ul		
-750ppb -1	5.0 ul			15 ul		
-1000ppb -1	5.0 ul			20 ul		
<u>10/22/99</u> <u>RWW</u>						
Blank <u>Robert Serum</u>	Std# <u>TNA-2382</u>	amount = <u>1.0</u> ml				
Serum Extraction Method <u>ETS-8-4.1</u>			Date & Initials <u>10/22/99-RWW</u>			
Vortex 15 Sec.						
Pipette Matrix, spike with appropriate surrogate or FC-Mix			Volume <u>1.0</u> ml			
Pipette 1 ml of 0.5 M TBA, pH 10. pH= <u>10</u>			<u>10/22/99-RWW</u> Std. # <u>99086-13-16</u>			
Pipette 2 ml of 0.25 M Na ₂ CO ₃ /0.25 M NaHCO ₃ buffer			Std. # <u>99086-14-08</u>			
Dispense 5 ml of methyl-t-butyl ether			TN-A- <u>2946</u>			
Shake 20 min			Shaker speed: <u>300</u>			
Centrifuge 20-25 min.			Centrifuge speed: <u>3500</u>			
Remove a 4 ml aliquot of organic layer						
Put on Nitrogen Evaporator to dryness			Temperature: <u>Room Temp.</u>			
Add methanol			Volume <u>1.0 ml</u> TN-A- <u>2945</u> <u>10/25/99-RWW</u>			
Vortex 30 sec.						
Filter using a 3cc B-D syringe with a 0.2 um filter into an autosampler vial						
Cont. Cal. Verifications used same matrix as for std curve.						

Ion Pair Standard Curves--Fluids

Prep Date(s): 10/25/99
 Analyst(s): RWW *Rev-10/25/99*
 Sample Matrix: Rabbit Serum
 Method/Revision: ETS-8-4.1
 Target Analyte(s): Below
 Box Number: 99-206

Study Number: TOX145
 Equipment Number:
 Final Solvent & TN Number: MeOH/TN-A-2945
 Blank Tissue/Identifier: Rabbit Sera/TNA-2382

FC Mix Std Approx. 0.500 ppm: 99086-44-3
 FC Mix Std Approx. 5.00 ppm: 99086-44-2
 FC Mix Std Approx. 50.0 ppm: 99086-44-1
 Surrogate Std Approx. 101 ppm: 99086-13-11

Blank serum initial volume (mL) for curves: 1.00
 Final solvent volume (mL) for curves: 1.00

Actual Concentrations of Standards in the FC Mix

PFOS Std Conc. ug/mL	PFOSA Std Conc. ug/mL	EtFOSEOH Std Conc. ug/mL	M556 Std Conc. ug/mL	M570 Std Conc. ug/mL	MeFOSEOH Std Conc. ug/mL	POAA Std Conc. ug/mL	PFHS Std Conc. ug/mL	All Am't Spiked mL	All Initial Volume mL	All Final Vol. mL
0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000		#VALUE!	1.000
0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.010	1.015	1.000
0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.020	1.025	1.000
5.000	5.000	5.000	5.000	5.000	5.000	5.000	5.000	0.005	1.010	1.000
5.000	5.000	5.000	5.000	5.000	5.000	5.000	5.000	0.010	1.015	1.000
5.000	5.000	5.000	5.000	5.000	5.000	5.000	5.000	0.020	1.025	1.000
50.00	50.00	50.00	50.00	50.00	50.00	50.00	50.00	0.005	1.010	1.000
50.00	50.00	50.00	50.00	50.00	50.00	50.00	50.00	0.010	1.015	1.000
50.00	50.00	50.00	50.00	50.00	50.00	50.00	50.00	0.015	1.020	1.000
50.00	50.00	50.00	50.00	50.00	50.00	50.00	50.00	0.020	1.025	1.000

Calculated Concentrations of Standards in the sample matrix

PFOS Std Conc. ng/mL	PFOSA Std Conc. ng/mL	EtFOSEOH Std Conc. ng/mL	M556 Std Conc. ng/mL	M570 Std Conc. ng/mL	MeFOSEOH Std Conc. ng/mL	POAA Std Conc. ng/mL	PFHS Std Conc. ng/mL	Surrogate Std Conc. ug/mL	All Am't Spiked mL
#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!	101	0.005
4.93	4.93	4.93	4.93	4.93	4.93	4.93	4.93	Surrogate	
9.76	9.76	9.76	9.76	9.76	9.76	9.76	9.76	Final Conc.	
24.8	24.8	24.8	24.8	24.8	24.8	24.8	24.8	ng/mL	
49.3	49.3	49.3	49.3	49.3	49.3	49.3	49.3	498	
97.6	97.6	97.6	97.6	97.6	97.6	97.6	97.6		
248	248	248	248	248	248	248	248		
493	493	493	493	493	493	493	493		
735	735	735	735	735	735	735	735		
976	976	976	976	976	976	976	976		

Calculated Concentrations of Standards in the 1.0 mL final solvent volume

PFOS Final Conc. ng/mL	PFOSA Final Conc. ng/mL	EtFOSEOH Final Conc. ng/mL	M556 Final Conc. ng/mL	M570 Final Conc. ng/mL	MeFOSEOH Std Conc. ng/mL	POAA Std Conc. ng/mL	PFHS Std Conc. ng/mL	Surrogate Std Conc. ug/mL	All Am't Spiked mL
#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!	101	0.005
5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	Surrogate	
10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	Final Conc.	
25.0	25.0	25.0	25.0	25.0	25.0	25.0	25.0	ng/mL	
50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	305	
100	100	100	100	100	100	100	100		
250	250	250	250	250	250	250	250		
500	500	500	500	500	500	500	500		
750	750	750	750	750	750	750	750		
1000	1000	1000	1000	1000	1000	1000	1000		

Sample used for MS/MSD: No MS/MSD

MS/MSD Conc. in the sample matrix

Initial Volume (mL): 1.00 2.00

Approx. Std Conc.: 50 ug/ml

Am't Spiked
Vol. (mL)
0.005

PFOS Final Conc. ng/mL	PFOSA Final Conc. ng/mL	EtFOSEOH Final Conc. ng/mL	M556 Final Conc. ng/mL	M570 Final Conc. ng/mL	MeFOSEOH Std Conc. ng/mL	POAA Std Conc. ng/mL	PFHS Std Conc. ng/mL
248	248	248	248	248	248	248	248
248	248	248	248	248	248	248	248

Sample used for MS/MSD: No MS/MSD

MS/MSD Conc. in the final solvent

Initial Volume (mL): 1.00 2.00

Approx. Std Conc.: 50ug/ml

Am't Spiked
Vol. (mL)
0.005
Final Vol. (mL)
1.00

PFOS Std Conc. ug/mL	PFOSA Std Conc. ug/mL	EtFOSEOH Std Conc. ug/mL	M556 Std Conc. ug/mL	M570 Std Conc. ug/mL	MeFOSEOH Std Conc. ug/mL	POAA Std Conc. ug/mL	PFHS Std Conc. ug/mL
250	250	250	250	250	250	250	250
250	250	250	250	250	250	250	250

ug/ml should read ng/ml 11/4/99 sw

Sample Extraction Sheet

Prep Date(s): 11/02/99
 Analyst(s): JAS
 Sample Matrix: Tween 80
 Method/Revision:
 Target Analyte(s): PFOS/POAA

Study Number: TOX145
 Equipment Number:
 Notebook# and Page:
 Final Solvent: MeOH TH-A-2117

Procedure Detail

A PFOSA Sample, TH-A-3046, at 1mg/mL in 1% Acetone and 2% Tween 80 was prepared for analysis. Dilutions were done to get PFOS into the range of the instrument but to also limit the amount of Tween injected into the instrument. The following dilutions were made.

Stds used

POAA/PFOS - 99086-37-2, at POAA = 5.10 - exp 2/00
 PFOS = 5.18

ISTD - 99086-13-11, 101 ppm

PFOS/POAA

(amt of PFOS in sample)

Dose 6 10 ppm $\times 1000 \times \frac{50 \mu\text{L}}{25 \text{ mL}} = 20 \text{ ppb}$

Dose + MS :

10 ppm $\times 1000 \times \frac{50 \mu\text{L}}{25 \text{ mL}} = 20 \text{ ppb}$

5 ppm $\times 1000 \times \frac{0.100}{25 \text{ mL}} = 20 \text{ ppb}$

> 40 ppb

Matrix spikes & Samples were spiked with ISTD.

The spike was 125 ppb at 101 ppm to give a 505 ppb conc in the samples.

JAS 11/19/99

Sample Extraction Sheet

Prep Date(s): 10/29/99
 Analyst(s): IAS
 Sample Matrix: Tween 80
 Method/Revision:
 Target Analyte(s): PFOS/POAA
 PFOS

Study Number: TOX145
 Equipment Number:
 Notebook# and Page:
 Final Solvent: MeOH - DNA-2542

Procedure Detail

The TH-A-# of the sample is → 3046
 A PFOS sample at 1mg/mL in 1% Acetone and 2% Tween 80 was prepared for analysis. Dilutions were done in order to get PFOS, POAA, and POAA into the range of ES/MS analysis but to also limit the amount of Tween injected into the instrument. The following dilutions were done:

Std used:

PFOS = 99086-12-14 at 100.1 ppm - exp 3/00
 PFOS/POAA = 99086-37-3 at 0.5 ppm - exp 2/00

PFOS/POAA

Dose: $1000 \times 10 \text{ ppm} \times \frac{5 \mu\text{L}}{25 \text{ mL}} = 2 \text{ ppb}$

Dose + MS: $1000 \times 10 \text{ ppm} \times \frac{5 \mu\text{L}}{25 \text{ mL}} = 2 \text{ ppb}$
 $+ 1000 \times 0.100 \text{ mL} = 2 \text{ ppb}$ $\rightarrow 4 \text{ ppb}$

PFOS:

Dose: $1000 \text{ ppm} \times \frac{5 \mu\text{L}}{25 \text{ mL}} \times 1000 = 200 \text{ ppb}$

Dose + MS: $100 \text{ ppm} \times \frac{0.025 \text{ mL}}{25 \text{ mL}} \times 1000 = 100 \text{ ppb}$
 $+ 1000 \text{ ppm} \times \frac{5 \mu\text{L}}{25 \text{ mL}} \times 1000 = 200 \text{ ppb}$ $\rightarrow 300 \text{ ppb}$

Matrix Spikes IAS 10/29/99 RE

* All samples were spiked with surrogate 99086-13-11, 101 ppm.
 The spike was 50 μL of 100 ppm diluted to 10 mL to give final conc. in the MS at 202 ppb. 25 mL IAS RE 10/29/99
 The Dose was corrected with a 0.100 spike to give a final conc. of 50.5 ppb.

Spiked incorrectly
 IAS 10/29/99

IAS 10/19/99

Sample Extraction Sheet

Prep Date(s): 12/22/99

Analyst(s): IAS

Sample Matrix: Tween

Method/Revision:

Target Analyte(s): PFOSA
POAA/PFOA

Study Number: T2145

Equipment Number:

Notebook# and Page:

Final Solvent: MeOH TN-A-297

Procedure Detail

A PFOSA sample at 1 mg/mL in 1% Acetone and 2% Tween EO was prepared for analysis. Dilutions were done in order to get PFOSA, PFOA, & POAA into the range of the ES/MS but to also to limit the amount of Tween that was injected into the instrument. The following dilutions were done:

Sds used

PFOSA = 99086-12-14 at 100.1 ppm - exp 3/00

PFOA/POAA = 99086-37-3 at 0.5 ppm - exp 3/00

PFOS/POAA =

Dose: 100 ppb $\times$ (0.200 mL) = 20 ppb
10 mL

Dose + MS: 0.500 ppm $\times$ 40 μ L $\times$ 1000 = 20 ppb
10 mL

+ 100 ppb $\times$ (0.200 mL) = 20 ppb
(PFOS/POAA std) 10 mL

> 40 ppb

PFOSA =

Dose: 10 ppm $\times$ 0.200 mL $\times$ 1000 = 200 ppb
10 mL

Dose + MS: 10 ppm $\times$ 0.200 mL $\times$ 1000 = 200 ppb
10 mL

+ 100 ppm $\times$ 0.010 mL $\times$ 1000 = 100 ppb
(PFOSA std) 10 mL

IAS @ 10/20/99
> A 300 ppb

* All samples were spiked with surrogate 99086-13-11, 101 ppm.
The spike was blank at a 100 ppm diluted in 10 mL to give a final conc. of 555 ppb.

IAS 11/19/99

Form Approved by/Date:

Extraction-I Template

Review Date

Extraction.xls

1
P3

Page 1 of

000571

Extraction Worksheet ETS-8-4.1

Study #	Surrogate Std	FC Mix	FC Mix	FC Mix	Comments	Verified By
Matrix: Rat Sera	Approx. 100ppm	approx. 0.5 ppm	approx. 5 ppm	approx. 50 ppm		
Box # 99-208	Actual 100 ppm	actual 0.5 ppm	actual 5 ppm	actual 50 ppm		
WK/Day: Day 29	#99086-13-11	#99086-47-2	#99086-47-1	#99095-33		
Date Spiked/Analyst	MLA 11/03/99				MLA 11/03/99	
RBS11039-250ppb-CCV9&10	5 uL	-	-	5 uL		N/A
RTS11039-SeraBlk-9&10	5 uL	-	-	-		
RTS11039-250ppb-MS9	5 uL	-	-	5 uL		
RTS11039-250ppb-MSD10	5 uL	-	-	5 uL		
RBS11039-H2OBlk-9&10	5 uL	-	-	-		
RBS11039-SeraBlk-9&10	5 uL	-	-	-		
RBS11039-5ppb-9&10	5 uL	10 uL	-	-		
RBS11039-10ppb-9&10	5 uL	20 uL	-	-		
RBS11039-25ppb-9&10	5 uL	-	5 uL	-		
RBS11039-50ppb-9&10	5 uL	-	10 uL	-		
RBS11039-100ppb-9&10	5 uL	-	20 uL	-		
RBS11039-250ppb-9&10	5 uL	-	-	5 uL		
RBS11039-500ppb-9&10	5 uL	-	-	10 uL		
RBS11039-750ppb-9&10	5 uL	-	-	15 uL		
RBS11039-1000ppb-9&10	5 uL	-	-	20 uL		
9R03240M-0-Day 29	5 uL	-	-	-		11/3/99 Sec
9R03241M-0-Day 29	5 uL	-	-	-		
9R03242M-0-Day 29	5 uL	-	-	-		
9R03243M-0-Day 29	5 uL	-	-	-		N/A
9R03244M-0-Day 29	5 uL	-	-	-		
9R03255M-5mg/kg-Day 29	5 uL	-	-	-		
9R03256M-5mg/kg-Day 29	5 uL	-	-	-		
9R03257M-5mg/kg-Day 29	5 uL	-	-	-		
9R03258M-5mg/kg-Day 29	5 uL	-	-	-		
9R03259M-5mg/kg-Day 29	5 uL	-	-	-		
Blank: Rabbit Sera		Std# 99062-94	amount = 1.0 ml			
Serum Extraction Method ETS-8-4.1				Date & Initials		
Vortex 15 Sec.				MLA 11/03/99		
Pipette Matrix, spike with appropriate surrogate or FC-Mix Volume 1.0 ml						
Pipette 1 ml of 0.5 M TBA, pH 10. pH= 10 11/3/99 Sec				Std. #99086-14-15		11/3/99 Sec
Pipette 2 ml of 0.25 M Na ₂ CO ₃ /0.25 M NaHCO ₃ buffer				Std. #99086-14-08		
Dispense 5 ml of methyl-t-butyl ether				TN-A-2946		
Shake 20 min				Shaker speed: 300 rpm		N/A
Centrifuge 20-25 min.				Centrifuge speed: 3500 rpm		
Remove a 4 ml aliquot of organic layer						
Put on Nitrogen Evaporator to dryness				Temperature: Room Temp.		11/3/99 Sec
Add methanol				Volume 1.0 ml	TN-A-2946	N/A
Vortex 30 sec.						
Filter using a 3cc B-D syringe with a 0.2 um filter into an autosampler vial						
Cont. Cal. Verifications used same matrix as for std curve.						

FN-1: The MS/MSD and associated sera blanks were taken from control rat sera TCR 99062-30. Date 11/03/99

FN-2: RBS11039-1000ppb-9 - I noticed after adding meth that sample had not completely blown down. I put it back on dryer and finished with method as is. Date 11/03/99

Ion Pair Standard Curves--Serum

Prep Date(s): 11/03/99
 Analyst(s): MCH
 Sample Matrix: Rat Sera
 Method/Revision: ETS-8-1
 Target Analyte(s): PFOS, PFOSA
 Box Number: 99-208

Study Number: TOX145
 Equipment Number:
 Final Solvent & TN Number: MeOH TN-A-2997
 Blank Tissue/Identifier: Rabbit Sera TCR99062-94

FC Mix Std Approx. 0.500 ppm: 99086-47-2
 FC Mix Std Approx. 5.00 ppm: 99086-47-1
 FC Mix Std Approx. 50.0 ppm: 99095-33
 Surrogate Std Approx. 100 ppm: 99086-13-11

Blank serum initial volume (mL) for curves: 1.00
 Final solvent volume (mL) for curves: 1.00

Actual Concentrations of Standards in the FC Mix

PFOS Std Conc. ug/mL	PFOSA Std Conc. ug/mL	PFOSAA Std Conc. ug/mL	EtFOSE Std Conc. ug/mL	PFOSEA Std Conc. ug/mL	M556 Std Conc. ug/mL	All Am't Spiked mL	All Initial Volum mL	All Final Vol. mL
0.5000	0.5005	0.4997	0.4999	0.5005	0.5010	0.010	1.015	1.000
0.5000	0.5005	0.4997	0.4999	0.5005	0.5010	0.020	1.025	1.000
5.000	5.005	4.997	4.999	5.005	5.010	0.005	1.010	1.000
5.000	5.005	4.997	4.999	5.005	5.010	0.010	1.015	1.000
5.000	5.005	4.997	4.999	5.005	5.010	0.020	1.025	1.000
50.00	50.05	49.97	49.99	50.05	50.10	0.005	1.010	1.000
50.00	50.05	49.97	49.99	50.05	50.10	0.010	1.015	1.000
50.00	50.05	49.97	49.99	50.05	50.10	0.015	1.020	1.000
50.00	50.05	49.97	49.99	50.05	50.10	0.020	1.025	1.000

Calculated Concentrations of Standards in the sample matrix

PFOS Std Conc. ng/mL	PFOSA Std Conc. ng/mL	PFOSAA Std Conc. ng/mL	EtFOSE Std Conc. ng/mL	PFOSEA Std Conc. ng/mL	M556 Std Conc. ng/mL	Surrogate Std Conc. ug/mL	All Am't Spiked mL
4.93	4.93	4.92	4.93	4.93	4.94	101	0.005
9.76	9.77	9.75	9.75	9.77	9.78	Surrogate	
24.8	24.8	24.7	24.7	24.8	24.8	Final Conc.	
49.3	49.3	49.2	49.3	49.3	49.4	ng/mL	
97.6	97.7	97.5	97.5	97.7	97.8	498	
248	248	247	247	248	248		
493	493	492	493	493	494		
735	736	735	735	736	737		
976	977	975	975	977	978		

Calculated Concentrations of Standards in the 1.0 mL final solvent volume

PFOS Final Conc. ng/mL	PFOSA Final Conc. ng/mL	PFOSAA Final Conc. ng/mL	EtFOSE Final Conc. ng/mL	PFOSEA Final Conc. ng/mL	M556 Std Conc. ng/mL	Surrogate Std Conc. ug/mL	All Am't Spiked mL
5.00	5.01	5.00	5.00	5.01	5.01	101	0.005
10.0	10.0	10.0	10.0	10.0	10.0	Surrogate	
25.0	25.0	25.0	25.0	25.0	25.1	Final Conc.	
50.0	50.1	50.0	50.0	50.1	50.1	ng/mL	
100	100	100	100	100	100	505	
250	250	250	250	250	251		
500	501	500	500	501	501		
750	751	750	750	751	752		
1000	1001	999	1000	1001	1002		

Sample used for MS/MSD: TCR99062-30

MS/MSD Conc. in the sample matrix

PFOS Final Conc. ng/mL	PFOSA Final Conc. ng/mL	PFOSAA Final Conc. ng/mL	EtFOSE Final Conc. ng/mL	PFOSEA Final Conc. ng/mL	M556 Std Conc. ng/mL
248	248	247	247	248	248
248	248	247	247	248	248

Approx. Std Conc.: 50.0 ug/mL

Am't Spiked
Vol. (mL)
0.005

Sample used for MS/MSD: TCR99062-30

MS/MSD Conc. in the final solvent

PFOS Std Conc. ug/mL	PFOSA Std Conc. ug/mL	PFOSAA Std Conc. ug/mL	EtFOSE Std Conc. ug/mL	PFOSEA Std Conc. ug/mL	M556 Std Conc. ug/mL
250	250	250	250	250	251
250	250	250	250	250	251

Approx. Std Conc.: 50.0 ug/mL

Am't Spiked
Vol. (mL)
0.005
Final Vol. (mL)
1.00

Tox145

000573

Verified
by 11/03/99
Jew

DILUTIONS SUMMARY SHEET

Study: TOX145

Dilution Date/Analyst: 11/5/99 MCH *mm* 11/5/99

Box #: 99-208

Solvent/TN Number: MeOH TN-A-3386

Extraction Date/Analyst: MCH 11/3/99

Matrix/Timepoint: Rat Sera Day 29

[illegible]

Notes: D10=100 uL sample + 900 uL MeOH. *Wu* 11/5/99

Form Completion
Verified By: SM 11/15/99

00513

Original - 11/4/99 - RWW

Extraction Worksheet ETS-8-6.0

O RWW - 11/15/99

Study # TOX-145	Surrogate Std	FC Mix	FC Mix	FC Mix	Comments	Verified By
Matrix Rat Liver	Approx. ppm	approx. 0.5 ppm	approx. 5 ppm	approx. 50 ppm		
Box # 99-211	Actual / #1 ppm	actual ppm	actual ppm	actual ppm		
WK/Day Day 29	# 99086-13-1	# 99086-44-3	# 99086-44-2	# 99086-44-1		
Date Spiked/Analyst	11/3/99 - RWW	11/3/99 - RWW	11/3/99 - RWW	11/3/99 - RWW	11/3/99 - RWW	
RAL11049 - CCV-1-4-2	5.0 ul		10 ul			LRA
99086-240 -MS-	5.0 ul		10 ul			
IL -MSD-	5.0 ul		10 ul			
RAL11049 -H2OBik-1&2	5.0 ul					11/16/11/04/99
-LiverBik-1&2	5.0 ul					NR
-6ppb-1&2	5.0 ul	2 ul				
-12ppb-1&2	5.0 ul	4 ul				
-30ppb-1&2	5.0 ul	10 ul				
-60ppb-1&2	5.0 ul	20 ul				
-120ppb-1&2	5.0 ul	40 ul				
-300ppb-1&2	5.0 ul		10 ul			
-600ppb-1&2	5.0 ul		20 ul			
-900ppb-1&2	5.0 ul		30 ul			
-1200ppb-1&2	5.0 ul			4 ul		
Sample numbers and weights noted on a separate page - add 5.0 ul surrogate to all samples.						
RWW 11/11/99						
Blank - Rabbit Liver Std# 99086-13-5 amount = 1.0 ml 0.163 g/ml NR						
Liver Extraction Method ETS-8-6.0 : Date & Initials 11/3/99 - RWW						
Vortex 15 Sec.						
Pipette Matrix, spike with appropriate surrogate or FC-Mix Volume 1.0 ml						
Pipette 1 ml of 0.5 M TBA, pH 10. pH= Std. # 99086-14-5 11/4/99 - RWW 11/16/11/04/99						
Pipette 2 ml of 0.25 M Na ₂ CO ₃ /0.25 M NaHCO ₃ buffer Std. # 99086-14-02						
Dispense 5 ml of methyl-t-butyl ether TN-A- 2946						
Shake 20 min Shaker speed: 300						
Centrifuge 20-25 min. Centrifuge speed: 3500						
Remove a 4 ml aliquot of organic layer						
Put on Nitrogen Evaporator to dryness Temperature: Room Temp.						
Add methanol Volume 1.0 ml TN-A- 2997						
Vortex 30 sec. 11/16/11/04/99						
Filter using a 3cc B-D syringe with a 0.2 um filter into an autosampler vial						
Cont. Cal. Verifications used same matrix as for std curve.						

Original - 11/4/99

Ion Pair Standard Curves--Tissues

Prep Date(s): 11/4/99
 Analyst(s): RWW
 Sample Matrix: Rabbit Liver
 Method/Revision: ETS-8-6.0
 Target Analyte(s): See below
 Box Number: 99-211

Study Number: TOX145
 Equipment Number:
 Final Solvent & TN Number: MeOH/TN-A-2997
 Rabbit Liver/99086-13-5

FC Mix Std Approx. 0.500 ppm: 99086-44-3
 FC Mix Std Approx. 5.00 ppm: 99086-44-2
 FC Mix Std Approx. 50.0 ppm: 99086-44-1
 Surrogate Std Approx. 101 ppm: 99086-13-11

Blank liver initial density (g) for curves: 0.163
 Final solvent volume (mL) for curves: 1.000

Actual Concentrations of Standards in the FC Mix

PFOS Std Conc. ug/mL	PFOSA Std Conc. ug/mL	EtFOSE-OH Std Conc. ug/mL	M556 Std Conc. ug/mL	M570 Std Conc. ug/mL	MeFOSE-OH Std Conc. ug/mL	POAA Std Conc. ug/mL	PFHS Std Conc. ug/mL
0.5004	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000
0.5004	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000
0.5004	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000
0.5004	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000
0.5004	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000
5.004	5.000	5.000	5.000	5.000	5.000	5.000	5.000
5.004	5.000	5.000	5.000	5.000	5.000	5.000	5.000
5.004	5.000	5.000	5.000	5.000	5.000	5.000	5.000
50.04	50.00	50.00	50.00	50.00	50.00	50.00	50.00

All Am't Spiked mL	All Density g	All Vol. mL
0.002	0.163	1.000
0.004	0.163	1.000
0.010	0.163	1.000
0.020	0.163	1.000
0.040	0.163	1.000
0.010	0.163	1.000
0.020	0.163	1.000
0.030	0.163	1.000
0.004	0.163	1.000

Calculated Concentrations of Standards in the sample matrix

PFOS Std Conc. ng/g	PFOSA Std Conc. ng/g	EtFOSE-OH Std Conc. ng/g	M556 Std Conc. ng/g	M570 Std Conc. ng/g	MeFOSE-OH Std Conc. ng/g	POAA Std Conc. ng/g	PFHS Std Conc. ng/g	Surrogate Std Conc. ug/mL	All Am't Spiked mL
6.14	6.13	6.13	6.13	6.13	6.13	6.13	6.13	101	0.005
12.3	12.3	12.3	12.3	12.3	12.3	12.3	12.3		
30.7	30.7	30.7	30.7	30.7	30.7	30.7	30.7		
61.4	61.3	61.3	61.3	61.3	61.3	61.3	61.3		
123	123	123	123	123	123	123	123		
307	307	307	307	307	307	307	307		
614	613	613	613	613	613	613	613		
921	920	920	920	920	920	920	920		
1228	1227	1227	1227	1227	1227	1227	1227		

Calculated Concentrations of Standards in the 1.0 mL final solvent volume

PFOS Final Conc. ng/mL	PFOSA Final Conc. ng/mL	EtFOSE-OH Final Conc. ng/mL	M556 Final Conc. ng/mL	M570 Final Conc. ng/mL	MeFOSE-OH Std Conc. ng/mL	POAA Std Conc. ng/mL	PFHS Std Conc. ng/mL	Surrogate Std Conc. ug/mL	All Am't Spiked mL
1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	101	0.005
2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00		
5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00		
10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0		
20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0		
50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0		
100	100	100	100	100	100	100	100		
150	150	150	150	150	150	150	150		
200	200	200	200	200	200	200	200		

Sample used for MS/MSD: 9R03240

MS/MSD calc. In matrix

Initial Wt (g): 1.020

Initial Volume (mL): 5.00

6.00

Approx. Std Conc.: 5.00 ug/mL

PFOS Final Conc. ng/g	PFOSA Final Conc. ng/g	EtFOSE-OH Final Conc. ng/g	M556 Final Conc. ng/g	M570 Final Conc. ng/g	MeFOSE-OH Std Conc. ng/g	POAA Std Conc. ng/g	PFHS Std Conc. ng/g
295	295	295	295	295	295	295	295
295	295	295	295	295	295	295	295

Am't Spiked (mL) 0.010
 Density 0.169

Sample used for MS/MSD: 9R03240

MS/MSD calc. In solvent

Initial Wt (g): 1.020

Initial Volume (mL): 5.00

6.00

Approx. Std Conc.: 5.00 ug/mL

PFOS Final Conc. ng/g	PFOSA Final Conc. ng/g	EtFOSE-OH Final Conc. ng/g	M556 Final Conc. ng/g	M570 Final Conc. ng/g	MeFOSE-OH Std Conc. ng/g	POAA Std Conc. ng/g	PFHS Std Conc. ng/g
50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0
50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0

Am't Spiked (mL) 0.010
 Final Vol. 1.000

verified by:
 Mst 11/04/99

000577

DILUTIONS SUMMARY SHEET

Study: TOX-145

Dilution Date/Analyst: SAL 11/9/99

Box #: 99-211

Solvent/TN Number: MeOH TN-A- 3386

Extraction Date/Analyst: RWW 11/4/99

Matrix/Timepoint: Rat Liver Day 29, Grp 6

[illegible]

Notes: 0100 made using 10ul sample and 990ul MeOH

Form Completion
Verified By: 11/14 11/9/00

000578

FACT: DATA REVIEW SUMMARY FORM

IAS 10/20/99 p 1 of 2

Date: 10/20/99
 Data Analyst: IAS
 FACT Study #: FOX 145
 Matrix/Timepoints: Rat Sero - Day 144 - Control 1/2 dose - rat 10/19/99 SEE
 Sample List: 1019986
 Set-up Analysis
 Date/Analyst: 10/19/99 MMH
 Instrument: Soup 020199

Data Review Sign-Off

Analyst Review
 Name / Date: IAS 10/20/99

Technical Review
 Name / Date: MEE 10/20/99

Quality Assurance Review
 Name / Date: _____

Data Entry: LAC 10/21/99

Comments / Justifications: _____

Analyte: POAA (CH1) ①
 Acceptable Linear Range: NA - See comments
 LOQ: NA Extraction Blank: NA
 R²: NA Matrix Blank: NA
 Calibration Checks: NA
 MS/MSD: OK See comments
 Data Status: 10/21/99 LAC 10/21/99

Analyte: PFOS (CH2) ②
 Acceptable Linear Range: 9.76 ppb → 976 ppb
 LOQ: 9.76 ppb Extracted Blank: less than LOQ
 R²: 0.988440 Matrix Blank: less than LOQ
 Calibration Checks: OK - Range from -16% → 16%
 MS/MSD: OK
 Data Status: Dilutions needed for dosed animals 10/21/99

Analyte: PFOSA (CH3) ③
 Acceptable Linear Range: 4.93 ppb → 493 ppb
 LOQ: 4.93 ppb Extracted Blank: less than LOQ
 R²: 0.998624 Matrix Blank: less than LOQ
 Calibration Checks: OK - Range from -13% → 1%
 MS/MSD: OK
 Data Status: entered 10/21/99 10/21/99

① A curve was not run with the analysis of POAA. The data is only good for looking at area counts.
 - IAS 10/20/99

② The 4.93 ppb point was dropped because it was outside the linear range of the curve. Some carryover ~~was~~ is noticed in the dosed samples and in the MoOH blanks following the dosed samples. Many of the samples had high area counts and were outside the range of quantitation. - IAS 10/20/99

③ The 736 ppb and 977 ppb points were dropped from the curve because they were outside the linear range. The MoOH blanks had fairly high levels of PFOSA. - IAS 10/20/99

POAA results are significant. Quantitation is needed; new curves should be prepared (or old ones used for re-analysis). Also, reanalyze undiluted livers for POAA. 10/20/99

000579

FACT: DATA REVIEW SUMMARY FORM

IAS 10/20/99 p. 2 of 2

Date: 10/20/99
 Data Analyst: IAS
 FACT Study #: Tox 145
 Matrix/Timepoints: Pat Sera - Day 14 - Control & dose - ext. 7/19/99 SE
 Sample List: 101996
 Set-up Analysis
 Date/Analyst: 10/19/99 MMH
 Instrument: Soup 020199

Data Review Sign-Off

Analyst Review

Name / Date: IAS 10/20/99

Technical Review

Name / Date: See page 1 of 2 LAC 10/21/99

Quality Assurance Review

Name / Date: _____

Data Entry: LAC 10/21/99

Comments / Justifications: _____

Analyte: IS (CH4) ④
 Acceptable Linear Range: NA
 LOQ: NA Extraction Blank: NA
 R²: NA Matrix Blank: NA
 Calibration Checks: NA
 MS/MSD: —
 Data Status: —

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: IAS 10/20/99
 MS/MSD: _____
 Data Status: _____

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: _____

④ Internal std area counts look
 consistent. All samples, curves, & cal
 checks had deviations of 30% or less.
 — IAS 10/20/99

LAC 10/21/99

Las 10/20/99 p. 1 of 4

Quantify Compound Summary Report
TOX145-rat sera, day 1 & 4, control & dose- ext. 10/19/99 SEE

Sample List: C:\MSL\LYMX\soup-rats.pro\SampleDB\101999b
Last modified: Wed Oct 20 07:37:33 1999
Method: C:\MSL\LYMX\soup-rats.pro\MethDB\101999b
Last modified: Wed Oct 20 07:36:48 1999
Job Code:

Printed: Wed Oct 20 08:08:10 1999

Compound 1: POAA (Chi)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1 101999012	MeOH Blank							
2 101999013	MeOH Blank							
3 101999014	MeOH Blank							
4 101999015	TOX145-RBS10199-H2O Blank-11. SEE			7.085	37	MM	0.88	-12
5 101999016	TOX145-RBS10199-Sera Blank-11. SEE			7.123	38	MM	0.91	-9
6 101999017	TOX145-RBS10199-Sera Blank-11. SEE			7.121	34	MM	0.81	-15
7 101999018	TOX145-RBS10199-Sera Blank-12. SEE			7.112	38	MM	0.90	-10
8 101999019	TOX145-RBS10199-5ppb-11. SEE	Standard	1.00	7.084	35	bb	0.83	-17
9 101999020	TOX145-RBS10199-10ppb-11. SEE	Standard	1.00	7.111	42	bb	0.99	-1
10 101999021	TOX145-RBS10199-25ppb-11. SEE	Standard	1.00	7.109	33	bb	0.77	-23
11 101999022	TOX145-RBS10199-50ppb-11. SEE	Standard	1.00	7.100	42	bb	1.00	0
12 101999023	TOX145-RBS10199-100ppb-11. SEE	Standard	1.00	7.112	39	bb	0.92	-8
13 101999024	TOX145-RBS10199-250ppb-11. SEE	Standard	1.00	7.110	39	bb	0.93	-7
14 101999025	TOX145-RBS10199-500ppb-11. SEE	Standard	1.00	7.111	51	MM	1.21	21
15 101999026	TOX145-RBS10199-750ppb-11. SEE	Standard	1.00	7.112	42	bb	1.00	0
16 101999027	TOX145-RBS10199-1000ppb-11. SEE	Standard	1.00	7.111	60	MM	1.41	41
17 101999028	MeOH Blank							
18 101999029	TOX145-RTS. 9R03230M. Dy1. Control.-10/19/99 SEE			7.090	30	MM	0.71	-29
19 101999030	TOX145-RTS. 9R03231M. Dy1. Control.-10/19/99 SEE			7.111	37	bb	0.87	-13
20 101999031	TOX145-RTS. 9R03232M. Dy1. Control.-10/19/99 SEE			7.110	36	bb	0.85	-15
21 101999032	TOX145-RTS. 9R03233M. Dy1. Control.-10/19/99 SEE			7.086	42	MM	1.00	-0
22 101999033	TOX145-RTS. 9R03234M. Dy1. Control.-10/19/99 SEE			7.113	43	MM	1.02	2
23 101999034	TOX145-RBS10199-250ppb-CCV-11-1. SEE	QC	1.00	7.089	34	MM	0.81	-19
24 101999035	MeOH Blank							
25 101999036	TOX145-RTS. 9R03235M. Dy4. Control.-10/19/99 SEE			7.133	29	bb	0.68	-32
26 101999037	TOX145-RTS. 9R03236M. Dy4. Control.-10/19/99 SEE			7.112	37	MM	0.88	-12
27 101999038	TOX145-RTS. 9R03237M. Dy4. Control.-10/19/99 SEE			7.129	34	MM	0.81	-19
28 101999039	TOX145-RTS. 9R03238M. Dy4. Control.-10/19/99 SEE			7.112	33	bb	0.77	-23
29 101999040	TOX145-RTS. 9R03239M. Dy4. Control.-10/19/99 SEE			7.106	30	MM	0.72	-28
30 101999041	TOX145-RBS10199-250ppb-CCV-11-2. SEE	QC	1.00	7.105	40	MM	0.96	-4
31 101999042	MeOH Blank							
32 101999043	TOX145-RTS10199-250ppb-MS-11. 10/19/99 SEE			7.132	56	MM	1.32	32
33 101999044	TOX145-RTS10199-250ppb-MSD-11. 10/19/99 SEE			7.123	35	bb	0.83	-17
34 101999045	TOX145-RTS. 9R03245M. Dy1. 5 mg/kg 10/19/99 SEE			7.090	308	bb	7.28	628
35 101999046	TOX145-RTS. 9R03246M. Dy1. 5 mg/kg 10/19/99 SEE			7.111	425	bb	10.06	906
36 101999047	TOX145-RTS. 9R03247M. Dy1. 5 mg/kg 10/19/99 SEE			7.112	360	bb	8.52	752
37 101999048	TOX145-RBS10199-250ppb-CCV-11-3. SEE	QC	1.00	7.107	42	bb	1.00	-0
38 101999049	MeOH Blank							
39 101999050	TOX145-RTS. 9R03248M. Dy1. 5 mg/kg 10/19/99 SEE			7.104	291	bb	6.88	588
40 101999051	TOX145-RTS. 9R03249M. Dy1. 5 mg/kg 10/19/99 SEE			7.090	340	bb	8.04	704
41 101999052	TOX145-RTS. 9R03250M. Dy4. 5 mg/kg 10/19/99 SEE			7.108	210	bb	4.97	397
42 101999053	TOX145-RTS. 9R03251M. Dy4. 5 mg/kg 10/19/99 SEE			7.103	209	bb	4.93	393
43 101999054	TOX145-RTS. 9R03252M. Dy4. 5 mg/kg 10/19/99 SEE			7.110	260	bb	6.15	515
44 101999055	TOX145-RBS10199-250ppb-CCV-11-4. SEE	QC	1.00	7.113	31	bb	0.73	-27
45 101999056	MeOH Blank							
46 101999057	TOX145-RTS. 9R03254-1M. Dy4. 5 mg/kg 10/19/99 SEE			7.110	248	bb	5.88	488
47 101999058	TOX145-RTS. 9R03254-2M. Dy4. 5 mg/kg 10/19/99 SEE			7.113	234	bb	5.55	455
48 101999059	TOX145-RBS10199-250ppb-CCV-11-5. SEE	QC	1.00	7.111	39	bb	0.92	-8
49 101999060	MeOH Blank							
50 101999061	TOX145-RBS10199-H2O Blank-11. SEE			7.109	28	MM	0.66	-34
51 101999062	TOX145-RBS10199-Sera Blank-11. SEE			7.129	38	bb	0.89	-11
52 101999063	TOX145-RBS10199-Sera Blank-11. SEE			7.110	28	bb	0.66	-34
53 101999064	TOX145-RBS10199-Sera Blank-12. SEE			7.091	44	MM	1.04	4
54 101999065	TOX145-RBS10199-5ppb-11. SEE	Standard	1.00	7.113	31	MM	0.73	-27
55 101999066	TOX145-RBS10199-10ppb-11. SEE	Standard	1.00	7.134	37	MM	0.87	-13
56 101999067	TOX145-RBS10199-25ppb-11. SEE	Standard	1.00	7.132	34	MM	0.81	-19
57 101999068	TOX145-RBS10199-50ppb-11. SEE	Standard	1.00	7.111	40	MM	0.95	-5
58 101999069	TOX145-RBS10199-100ppb-11. SEE	Standard	1.00	7.126	37	MM	0.88	-12
59 101999070	TOX145-RBS10199-250ppb-11. SEE	Standard	1.00	7.111	45	MM	1.07	7
60 101999071	TOX145-RBS10199-500ppb-11. SEE	Standard	1.00	7.111	37	MM	0.87	-13
61 101999072	TOX145-RBS10199-750ppb-11. SEE	Standard	1.00	7.107	53	bb	1.26	26
62 101999073	TOX145-RBS10199-1000ppb-11. SEE	Standard	1.00	7.110	64	bb	1.51	51

Not a target analyte - Data not used.

LAC 10/21/99

Sample List: C:\MASSLYNK\soup-rats.pro\SampleDB\101999b
Last modified: Wed Oct 20 07:37:33 1999
Method: C:\MASSLYNK\soup-rats.pro\MethDB\101999b
Last modified: Wed Oct 20 07:36:48 1999
Job Code:

Printed: Wed Oct 20 08:08:10 1999

Compound 2: PFOS (Ch2)

#	Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1	101999012	MeOH Blank			7.274	114	MM	0.00	
2	101999013	MeOH Blank			7.269	31	bb	0.00	
3	101999014	MeOH Blank			7.268	38	bb	0.00	
4	101999015	TOX145-RBS10199-H2O Blank-11, SEE			7.247	49	MM	0.00	
5	101999016	TOX145-RBS10199-Sera Blank-11, SEE			7.260	58	MM	0.00	
6	101999017	TOX145-RBS10199-Sera Blank-11, SEE			7.260	105	bb	0.00	
7	101999018	TOX145-RBS10199-Sera Blank-12, SEE			7.250	109	MM	0.00	
8	101999019	TOX145-RBS10199-5ppb-11, SEE	Standard	4.93	7.246	178	bbX	1.74	-65
9	101999020	TOX145-RBS10199-10ppb-11, SEE	Standard	9.76	7.250	316	bb	7.83	-20
10	101999021	TOX145-RBS10199-25ppb-11, SEE	Standard	24.80	7.248	724	bb	25.90	4
11	101999022	TOX145-RBS10199-100ppb-11, SEE	Standard	49.30	7.240	1354	bb	53.82	9
12	101999023	TOX145-RBS10199-250ppb-11, SEE	Standard	97.60	7.252	2251	bb	93.57	-4
13	101999024	TOX145-RBS10199-500ppb-11, SEE	Standard	248.00	7.249	6230	bb	269.87	9
14	101999025	TOX145-RBS10199-750ppb-11, SEE	Standard	493.00	7.250	12933	bd	566.87	15
15	101999026	TOX145-RBS10199-1000ppb-11, SEE	Standard	735.00	7.251	16324	bb	717.14	-2
16	101999027	TOX145-RBS10199-1000ppb-11, SEE	Standard	976.00	7.251	20800	bb	915.47	-6
17	101999028	MeOH Blank			7.293	84	MM	0.00	
18	101999029	TOX145-RTS, 9R03230M, Dy1, Control, -10/19/99 SEE			7.273	55	bb	0.00	
19	101999030	TOX145-RTS, 9R03231M, Dy1, Control, -10/19/99 SEE			7.272	45	MM	0.00	
20	101999031	TOX145-RTS, 9R03232M, Dy1, Control, -10/19/99 SEE			7.270	78	MM	0.00	
21	101999032	TOX145-RTS, 9R03233M, Dy1, Control, -10/19/99 SEE			7.247	49	MM	0.00	
22	101999033	TOX145-RTS, 9R03234M, Dy1, Control, -10/19/99 SEE			7.274	44	bb	0.00	
23	101999034	TOX145-RBS10199-250ppb-CCV-11-1, SEE	QC	248.00	7.250	6337	bb	274.63	11
24	101999035	MeOH Blank			7.292	36	MM	0.00	
25	101999036	TOX145-RTS, 9R03235M, Dy4, Control, -10/19/99 SEE			7.271	40	bb	0.00	
26	101999037	TOX145-RTS, 9R03236M, Dy4, Control, -10/19/99 SEE			7.274	49	bb	0.00	
27	101999038	TOX145-RTS, 9R03237M, Dy4, Control, -10/19/99 SEE			7.268	32	bb	0.00	
28	101999039	TOX145-RTS, 9R03238M, Dy4, Control, -10/19/99 SEE			7.273	37	bb	0.00	
29	101999040	TOX145-RTS, 9R03239M, Dy4, Control, -10/19/99 SEE			7.267	41	bb	0.00	
30	101999041	TOX145-RBS10199-250ppb-CCV-11-2, SEE	QC	248.00	7.266	4840	bb	208.29	-16
31	101999042	MeOH Blank			7.292	34	MM	0.00	
32	101999043	TOX145-RTS10199-250ppb-MS-11, 10/19/99 SEE			7.250	5358	bb	231.24	
33	101999044	TOX145-RTS10199-250ppb-MSD-11, 10/19/99 SEE			7.263	5626	bb	243.11	
34	101999045	TOX145-RTS, 9R03240M, Dy1, 5 mg/kg 10/19/99 SEE			7.250	69678	bb	3081.19	
35	101999046	TOX145-RTS, 9R03241M, Dy1, 5 mg/kg 10/19/99 SEE			7.250	111341	bb	4927.21	
36	101999047	TOX145-RTS, 9R03242M, Dy1, 5 mg/kg 10/19/99 SEE			7.251	98608	bb	4363.05	
37	101999048	TOX145-RBS10199-250ppb-CCV-11-3, SEE	QC	248.00	7.247	6344	bd	274.96	11
38	101999049	MeOH Blank			7.297	136	bb	0.00	
39	101999050	TOX145-RTS, 9R03243M, Dy1, 5 mg/kg 10/19/99 SEE			7.243	71589	bb	3165.86	
40	101999051	TOX145-RTS, 9R03244M, Dy1, 5 mg/kg 10/19/99 SEE			7.251	76176	bb	3369.10	
41	101999052	TOX145-RTS, 9R03245M, Dy4, 5 mg/kg 10/19/99 SEE			7.248	78647	bb	3478.58	
42	101999053	TOX145-RTS, 9R03246M, Dy4, 5 mg/kg 10/19/99 SEE			7.242	76820	bb	3397.64	
43	101999054	TOX145-RTS, 9R03247M, Dy4, 5 mg/kg 10/19/99 SEE			7.250	113380	bb	5017.56	
44	101999055	TOX145-RBS10199-250ppb-CCV-11-4, SEE	QC	248.00	7.252	6149	bb	266.30	7
45	101999056	MeOH Blank			7.294	145	MM	0.27	
46	101999057	TOX145-RTS, 9R03248M, Dy4, 5 mg/kg 10/19/99 SEE			7.250	90296	bb	3994.74	
47	101999058	TOX145-RTS, 9R03249M, Dy4, 5 mg/kg 10/19/99 SEE			7.252	81755	bb	3616.28	
48	101999059	TOX145-RBS10199-250ppb-CCV-11-5, SEE	QC	248.00	7.271	6641	bb	288.09	16
49	101999060	MeOH Blank			7.295	124	bb	0.00	
50	101999061	TOX145-RBS10199-H2O Blank-11, SEE			7.292	99	MM	0.00	
51	101999062	TOX145-RBS10199-Sera Blank-11, SEE			7.289	82	bb	0.00	
52	101999063	TOX145-RBS10199-Sera Blank-11, SEE			7.249	126	MM	0.00	
53	101999064	TOX145-RBS10199-Sera Blank-12, SEE			7.252	130	MM	0.00	
54	101999065	TOX145-RBS10199-5ppb-11, SEE	Standard	4.93	7.252	213	MMX	3.29	-33
55	101999066	TOX145-RBS10199-10ppb-11, SEE	Standard	9.76	7.251	330	bb	8.48	-13
56	101999067	TOX145-RBS10199-25ppb-11, SEE	Standard	24.80	7.249	685	bb	24.19	-2
57	101999068	TOX145-RBS10199-50ppb-11, SEE	Standard	49.30	7.251	1351	bb	53.71	9
58	101999069	TOX145-RBS10199-100ppb-11, SEE	Standard	97.60	7.265	2168	bb	89.88	-8
59	101999070	TOX145-RBS10199-250ppb-11, SEE	Standard	248.00	7.251	6116	bb	264.84	7
60	101999071	TOX145-RBS10199-500ppb-11, SEE	Standard	493.00	7.250	12734	bd	558.05	13
61	101999072	TOX145-RBS10199-750ppb-11, SEE	Standard	735.00	7.246	16227	bb	712.83	-3
62	101999073	TOX145-RBS10199-1000ppb-11, SEE	Standard	976.00	7.249	20551	MM	904.45	-7

Entered all except
those needing a
dilution.
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10/21/99

Need 1:50 dilution

Need a 1:50 dilution

Need a 1:50 dilution

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10/21/99

Quantify Compound Summary Report
TOX145-rat sera, day 1 & 4, control & dose- ext. 10/19/99 SEE

Page 3

Sample List: C:\MASSLYNX\sour-rata.pro\SampleDB\101999b
Last modified: Wed Oct 20 07:37:33 1999
Method: C:\MASSLYNX\sour-rata.pro\MethodDB\101999b
Last modified: Wed Oct 20 07:34:48 1999
Job Code:

Printed: Wed Oct 20 08:08:10 1999

Compound 3: PFOA (Ch3)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1 101999012	MeOH Blank			7.816	2725	MM	13.20	
2 101999013	MeOH Blank			7.806	539	MM	2.28	
3 101999014	MeOH Blank			7.853	294	MM	1.06	
4 101999015	TOX145-RBS10159-MSD Blank-11, SEE			7.833	171	MM	0.45	
5 101999016	TOX145-RBS10159-Sera Blank-11, SEE			7.839	154	MM	0.36	
6 101999017	TOX145-RBS10159-Sera Blank-12, SEE			7.822	159	MM	0.39	
7 101999018	TOX145-RBS10159-50ppb-11, SEE			7.834	145	MM	0.32	
8 101999019	TOX145-RBS10159-100ppb-11, SEE	Standard	4.93	7.808	1074	MM	4.96	1
9 101999020	TOX145-RBS10159-250ppb-11, SEE	Standard	9.77	7.813	2036	MM	9.76	-0
10 101999021	TOX145-RBS10159-500ppb-11, SEE	Standard	24.80	7.810	5196	MM	25.54	3
11 101999022	TOX145-RBS10159-1000ppb-11, SEE	Standard	49.30	7.802	9515	MM	47.10	-4
12 101999023	TOX145-RBS10159-2500ppb-11, SEE	Standard	97.70	7.814	16241	MM	80.69	-17
13 101999024	TOX145-RBS10159-5000ppb-11, SEE	Standard	248.00	7.811	49679	MM	247.65	-0
14 101999025	TOX145-RBS10159-7500ppb-11, SEE	Standard	493.00	7.809	100839	MM	503.11	2
15 101999026	TOX145-RBS10159-10000ppb-11, SEE	Standard	736.00	7.814	117401	MM	585.80	-20
16 101999027	TOX145-RBS10159-10000ppb-11, SEE	Standard	977.00	7.813	149225	MM	744.71	-24
17 101999028	MeOH Blank			7.877	993	MM	4.55	
18 101999029	TOX145-RTS, 9R03213M, Dy1, Control, -10/19/99 SEE			7.836	363	MM	1.41	
19 101999030	TOX145-RTS, 9R03213M, Dy1, Control, -10/19/99 SEE			7.834	226	MM	0.72	
20 101999031	TOX145-RTS, 9R03213M, Dy1, Control, -10/19/99 SEE			7.808	147	MM	0.33	
21 101999032	TOX145-RTS, 9R03213M, Dy1, Control, -10/19/99 SEE			7.832	150	MM	0.34	
22 101999033	TOX145-RTS, 9R03214M, Dy1, Control, -10/19/99 SEE			7.837	142	MM	0.30	
23 101999034	TOX145-RBS10159-250ppb-CCV-11-1, SEE	QC	248.00	7.812	49041	MM	244.47	-1
24 101999035	MeOH Blank			7.854	312	MM	1.15	
25 101999036	TOX145-RTS, 9R03213M, Dy4, Control, -10/19/99 SEE			7.856	168	MM	0.43	
26 101999037	TOX145-RTS, 9R03213M, Dy4, Control, -10/19/99 SEE			7.837	139	MM	0.29	
27 101999038	TOX145-RTS, 9R03217M, Dy4, Control, -10/19/99 SEE			7.852	167	MM	0.43	
28 101999039	TOX145-RTS, 9R03217M, Dy4, Control, -10/19/99 SEE			7.858	146	MM	0.32	
29 101999040	TOX145-RTS, 9R03213M, Dy4, Control, -10/19/99 SEE			7.852	136	MM	0.27	
30 101999041	TOX145-RBS10159-250ppb-CCV-11-2, SEE	QC	248.00	7.806	43165	MM	215.13	-13
31 101999042	MeOH Blank			7.854	315	MM	1.17	
32 101999043	TOX145-RTS10159-250ppb-MS-11, 10/19/99 SEE			7.789	48811	MM	243.32	
33 101999044	TOX145-RTS10159-250ppb-MSD-11, 10/19/99 SEE			7.803	52881	MM	263.64	
34 101999045	TOX145-RTS, 9R03213M, Dy1, 5 mg/kg 10/19/99 SEE			7.791	52155	MM	260.01	
35 101999046	TOX145-RTS, 9R03213M, Dy1, 5 mg/kg 10/19/99 SEE			7.790	87504	MM	436.52	
36 101999047	TOX145-RTS, 9R03217M, Dy1, 5 mg/kg 10/19/99 SEE			7.814	51116	dd	254.81	
37 101999048	TOX145-RBS10159-250ppb-CCV-11-3, SEE	QC	248.00	7.809	49904	MM	248.78	0
38 101999049	MeOH Blank			7.881	384	MM	1.51	
39 101999050	TOX145-RTS, 9R03213M, Dy1, 5 mg/kg 10/19/99 SEE			7.805	67032	db	334.30	
40 101999051	TOX145-RTS, 9R03213M, Dy1, 5 mg/kg 10/19/99 SEE			7.791	55570	MM	277.07	
41 101999052	TOX145-RTS, 9R03213M, Dy4, 5 mg/kg 10/19/99 SEE			7.810	12358	MM	61.30	
42 101999053	TOX145-RTS, 9R03213M, Dy4, 5 mg/kg 10/19/99 SEE			7.804	11759	dd	58.31	
43 101999054	TOX145-RTS, 9R03213M, Dy4, 5 mg/kg 10/19/99 SEE			7.789	15158	MM	75.28	
44 101999055	TOX145-RBS10159-250ppb-CCV-11-4, SEE	QC	248.00	7.814	46625	MM	232.41	-6
45 101999056	MeOH Blank			7.856	307	MM	1.13	
46 101999057	TOX145-RTS, 9R03214M, Dy4, 5 mg/kg 10/19/99 SEE			7.813	12235	bd	60.69	
47 101999058	TOX145-RTS, 9R03214M, Dy4, 5 mg/kg 10/19/99 SEE			7.792	11895	MM	58.99	
48 101999059	TOX145-RBS10159-250ppb-CCV-11-1, SEE	QC	248.00	7.811	50283	MM	250.67	1
49 101999060	MeOH Blank			7.857	348	MM	1.33	
50 101999061	TOX145-RBS10159-MSD Blank-11, SEE			7.854	145	MM	0.32	
51 101999062	TOX145-RBS10159-Sera Blank-11, SEE			7.851	126	MM	0.23	
52 101999063	TOX145-RBS10159-Sera Blank-12, SEE			7.833	120	MM	0.19	
53 101999064	TOX145-RBS10159-50ppb-11, SEE			7.859	148	MM	0.34	
54 101999065	TOX145-RBS10159-100ppb-11, SEE	Standard	4.93	7.814	1139	MM	5.28	7
55 101999066	TOX145-RBS10159-250ppb-11, SEE	Standard	9.77	7.813	2063	MM	9.90	1
56 101999067	TOX145-RBS10159-500ppb-11, SEE	Standard	24.80	7.811	5615	MM	27.63	11
57 101999068	TOX145-RBS10159-1000ppb-11, SEE	Standard	49.30	7.813	10041	MM	49.73	1
58 101999069	TOX145-RBS10159-2500ppb-11, SEE	Standard	97.70	7.805	18028	MM	89.61	-8
59 101999070	TOX145-RBS10159-5000ppb-11, SEE	Standard	248.00	7.813	51069	MM	254.60	3
60 101999071	TOX145-RBS10159-7500ppb-11, SEE	Standard	493.00	7.791	100103	MM	499.43	1
61 101999072	TOX145-RBS10159-10000ppb-11, SEE	Standard	736.00	7.786	122134	MM	609.44	-17
62 101999073	TOX145-RBS10159-10000ppb-11, SEE	Standard	977.00	7.811	153314	MM	765.23	-22

Entered
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Soup 020199: Quantified and printed by las on 10/19/99

000583

Quantify Compound Summary Report:
 TOK145-rat sera, day 1 & 4, control & dose- ext. 10/19/99 SEE

Page 4

Sample List: C:\MASSLYNK\source-rats.pro\SampleDB\101999b
 Last modified: Wed Oct 20 07:37:33 1999
 Method: C:\MASSLYNK\source-rats.pro\MethDB\101999b
 Last modified: Wed Oct 20 07:37:48 1999
 Job Code:

Printed: Wed Oct 20 08:08:10 1999

Compound 4: IS (Ch4)

# Name	SampleList Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1 101999012	MeOH Blank							
2 101999013	MeOH Blank							
3 101999014	MeOH Blank							
4 101999015	TOK145-RBS100-9-RSD Blank-11, SEE			7.080	1064	bb	0.70	-30
5 101999016	TOK145-RBS100-9-Sera Blank-11, SEE			7.094	1185	bb	0.78	-22
6 101999017	TOK145-RBS100-9-Sera Blank-11, SEE			7.093	1198	bb	0.79	-21
7 101999018	TOK145-RBS100-9-Sera Blank-12, SEE			7.083	1222	bb	0.81	-19
8 101999019	TOK145-RBS100-9-5ppb-11, SEE	Standard	1.00	7.079	1311	bb	0.87	-13
9 101999020	TOK145-RBS100-9-10ppb-11, SEE	Standard	1.00	7.106	1493	bd	0.99	-1
10 101999021	TOK145-RBS100-9-50ppb-11, SEE	Standard	1.00	7.104	1689	bb	1.12	12
11 101999022	TOK145-RBS100-9-50ppb-11, SEE	Standard	1.00	7.095	1526	bb	1.01	1
12 101999023	TOK145-RBS100-9-100ppb-11, SEE	Standard	1.00	7.107	1402	MM	0.93	-7
13 101999024	TOK145-RBS100-9-250ppb-11, SEE	Standard	1.00	7.105	1774	bb	1.17	17
14 101999025	TOK145-RBS100-9-500ppb-11, SEE	Standard	1.00	7.105	1625	bb	1.08	8
15 101999026	TOK145-RBS100-9-750ppb-11, SEE	Standard	1.00	7.106	1335	bb	0.88	-12
16 101999027	TOK145-RBS100-9-1000ppb-11, SEE	Standard	1.00	7.106	1369	bb	0.91	-9
17 101999028	MeOH Blank							
18 101999029	TOK145-RTS, 9-03230M, Dyl, Control, -10/19/99 SEE			7.084	1680	bb	1.11	11
19 101999030	TOK145-RTS, 9-03231M, Dyl, Control, -10/19/99 SEE			7.105	1602	bb	1.06	6
20 101999031	TOK145-RTS, 9-03232M, Dyl, Control, -10/19/99 SEE			7.103	1633	bb	1.08	8
21 101999032	TOK145-RTS, 9-03233M, Dyl, Control, -10/19/99 SEE			7.104	1598	bb	1.06	6
22 101999033	TOK145-RTS, 9-03234M, Dyl, Control, -10/19/99 SEE			7.107	1543	bb	1.02	2
23 101999034	TOK145-RBS100-9-250ppb-CCV-11-1, SEE	QC	1.00	7.105	1570	bb	1.04	4
24 101999035	MeOH Blank							
25 101999036	TOK145-RTS, 9-03235M, Dy4, Control, -10/19/99 SEE			7.104	1324	bb	0.88	-12
26 101999037	TOK145-RTS, 9-03236M, Dy4, Control, -10/19/99 SEE			7.107	1367	bb	0.90	-10
27 101999038	TOK145-RTS, 9-03237M, Dy4, Control, -10/19/99 SEE			7.101	1577	bb	1.04	4
28 101999039	TOK145-RTS, 9-03238M, Dy4, Control, -10/19/99 SEE			7.106	1301	bb	0.86	-14
29 101999040	TOK145-RTS, 9-03239M, Dy4, Control, -10/19/99 SEE			7.100	1102	bb	0.73	-27
30 101999041	TOK145-RBS100-9-250ppb-CCV-11-2, SEE	QC	1.00	7.099	1422	bb	0.94	-6
31 101999042	MeOH Blank							
32 101999043	TOK145-RTS100-9-250ppb-MS-11, 10/19/99 SEE			7.105	1547	bb	1.02	2
33 101999044	TOK145-RTS100-9-250ppb-MSD-11, 10/19/99 SEE			7.117	1588	bb	1.05	5
34 101999045	TOK145-RTS, 9-03240M, Dyl, 5 mg/kg 10/19/99 SEE			7.083	1497	bb	0.99	-1
35 101999046	TOK145-RTS, 9-03241M, Dyl, 5 mg/kg 10/19/99 SEE			7.105	1433	bb	0.95	-5
36 101999047	TOK145-RTS, 9-03242M, Dyl, 5 mg/kg 10/19/99 SEE			7.106	1329	bb	0.88	-12
37 101999048	TOK145-RBS100-9-250ppb-CCV-11-3, SEE	QC	1.00	7.102	1570	bb	1.04	4
38 101999049	MeOH Blank							
39 101999050	TOK145-RTS, 9-03243M, Dyl, 5 mg/kg 10/19/99 SEE			7.098	1180	MM	0.78	-22
40 101999051	TOK145-RTS, 9-03244M, Dyl, 5 mg/kg 10/19/99 SEE			7.105	1165	bb	0.77	-23
41 101999052	TOK145-RTS, 9-03245M, Dy4, 5 mg/kg 10/19/99 SEE			7.103	1142	bb	0.76	-24
42 101999053	TOK145-RTS, 9-03246M, Dy4, 5 mg/kg 10/19/99 SEE			7.097	1172	bb	0.78	-22
43 101999054	TOK145-RTS, 9-03247M, Dy4, 5 mg/kg 10/19/99 SEE			7.083	1254	bb	0.83	-17
44 101999055	TOK145-RBS100-9-250ppb-CCV-11-4, SEE	QC	1.00	7.107	1502	bb	0.99	-1
45 101999056	MeOH Blank							
46 101999057	TOK145-RTS, 9-03248M, Dy4, 5 mg/kg 10/19/99 SEE			7.105	1318	bb	0.87	-13
47 101999058	TOK145-RTS, 9-03249M, Dy4, 5 mg/kg 10/19/99 SEE			7.085	1238	bb	0.82	-18
48 101999059	TOK145-RBS100-9-250ppb-CCV-11-5, SEE	QC	1.00	7.105	1488	bb	0.99	-1
49 101999060	MeOH Blank							
50 101999061	TOK145-RBS100-9-RSD Blank-11, SEE			7.102	1192	bb	0.79	-21
51 101999062	TOK145-RBS100-9-Sera Blank-11, SEE			7.100	1485	bb	0.98	-2
52 101999063	TOK145-RBS100-9-Sera Blank-11, SEE			7.105	1543	bb	1.02	2
53 101999064	TOK145-RBS100-9-Sera Blank-12, SEE			7.085	1541	bb	1.02	2
54 101999065	TOK145-RBS100-9-5ppb-11, SEE	Standard	1.00	7.107	1540	bb	1.02	2
55 101999066	TOK145-RBS100-9-10ppb-11, SEE	Standard	1.00	7.106	1561	bb	1.03	3
56 101999067	TOK145-RBS100-9-50ppb-11, SEE	Standard	1.00	7.104	1595	bb	1.06	6
57 101999068	TOK145-RBS100-9-50ppb-11, SEE	Standard	1.00	7.083	1499	bb	0.99	-1
58 101999069	TOK145-RBS100-9-100ppb-11, SEE	Standard	1.00	7.098	1357	bb	0.90	-10
59 101999070	TOK145-RBS100-9-250ppb-11, SEE	Standard	1.00	7.106	1689	bb	1.12	12
60 101999071	TOK145-RBS100-9-500ppb-11, SEE	Standard	1.00	7.106	1574	bb	1.04	4
61 101999072	TOK145-RBS100-9-750ppb-11, SEE	Standard	1.00	7.101	1415	bb	0.94	-6
62 101999073	TOK145-RBS100-9-1000ppb-11, SEE	Standard	1.00	7.104	1441	bb	0.95	-5

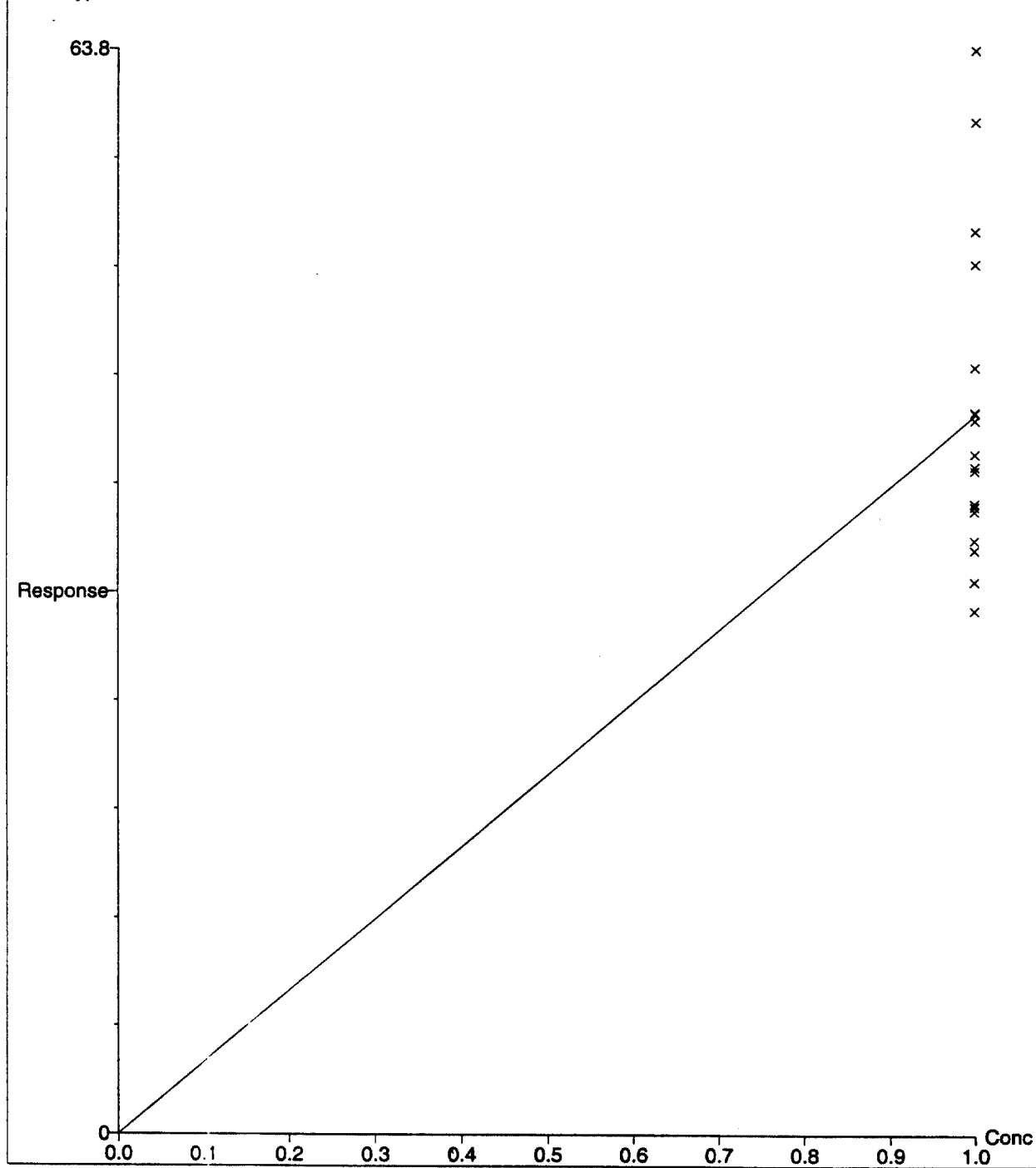
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Quantify Calibration Report
TOX145-rat sera, day 1 & 4, control & dose- ext. 10/19/99 SKE

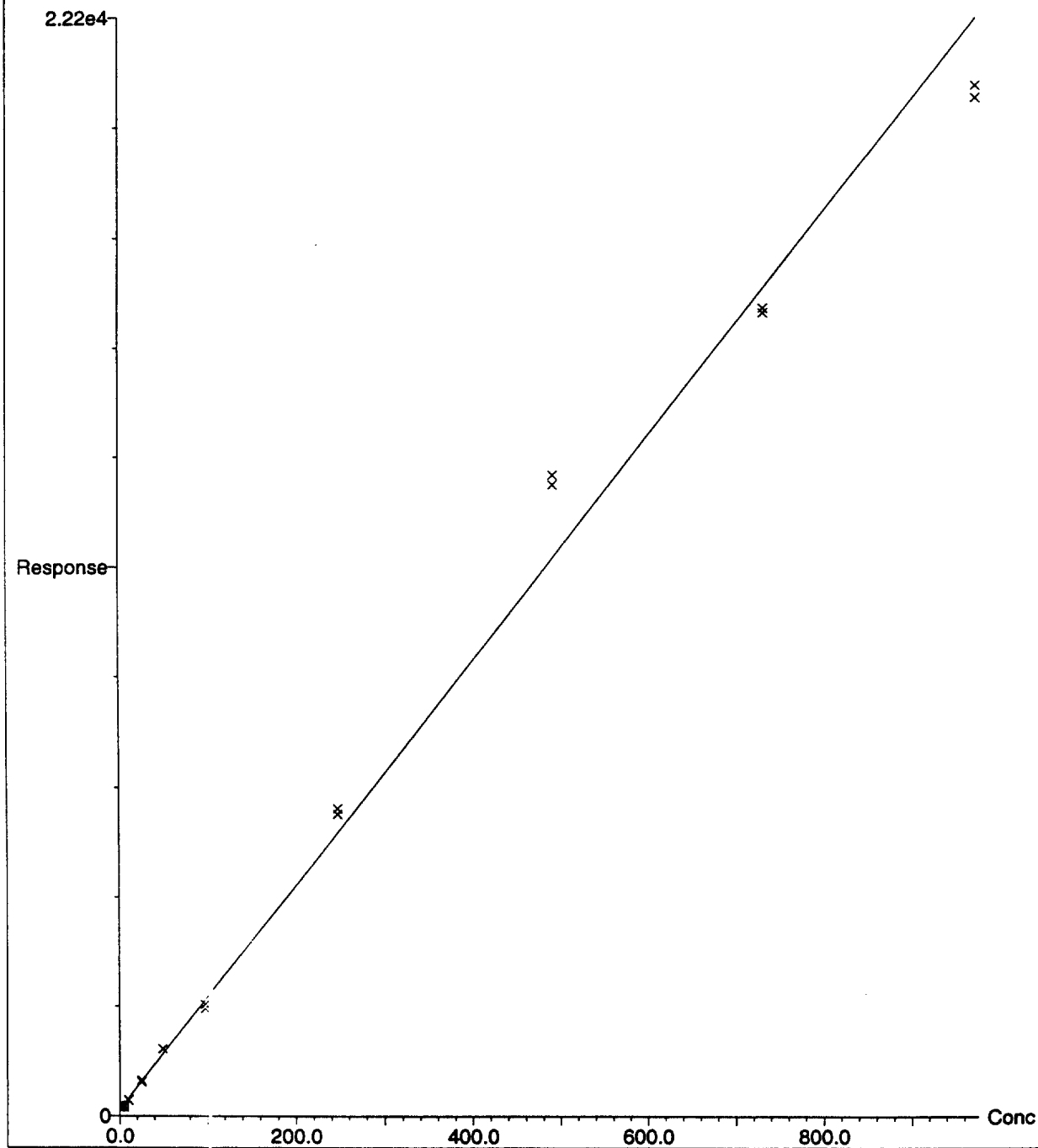
Compound 1 name: POAA (Ch1)
Response Factor: 42.2698
Response type: External Std, Area
Curve type: RF



Soup 020199: Quantified and printed by ias on 10/19/99

000585

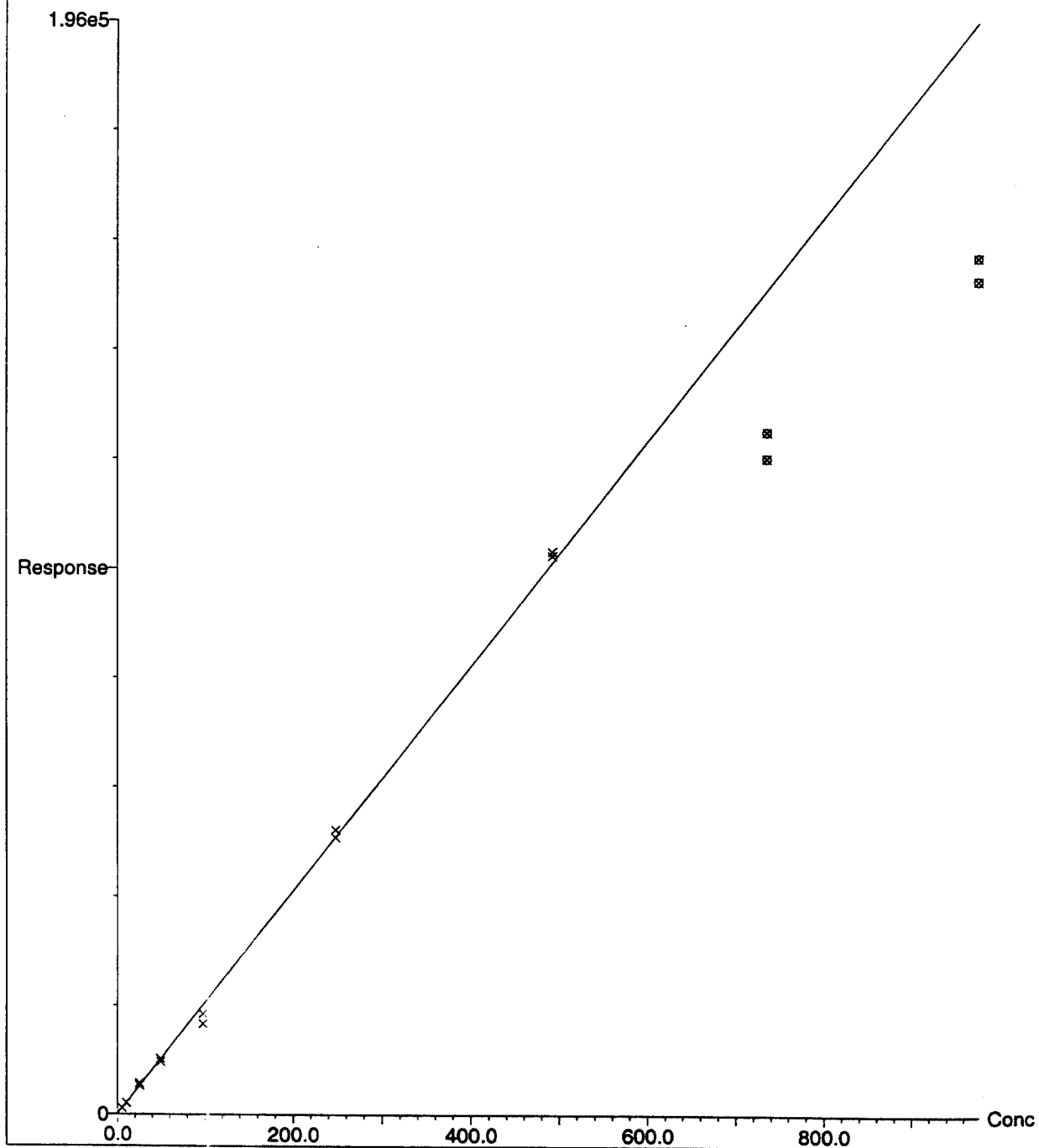
Compound 2 name: PFOS (Ch2)
Coefficient of Determination: 0.988440
Calibration curve: $22.5689 * x + 138.989$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Soup 020199: Quantified and printed by ias on 10/19/99

000586

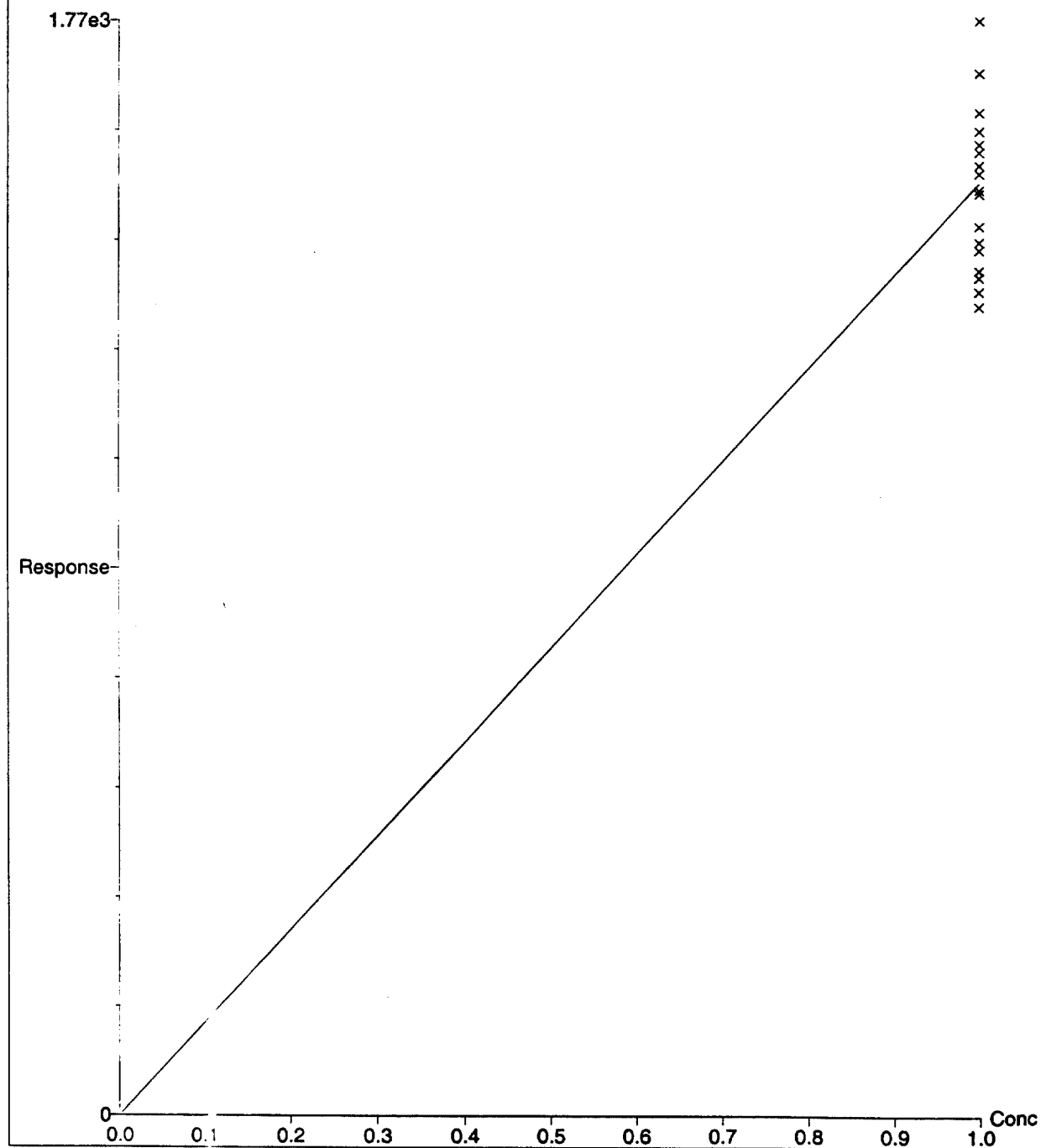
Compound 3 name: PFOSA (Ch3)
Coefficient of Determination: 0.998624
Calibration curve: $200.271 \cdot x + 81.0911$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Soup 020199: Quantified and printed by las on 10/19/99

000587

Compound 4 name: IS (Ch4)
Response Factor: 1510.76
Response type: External Std, Area
Curve type: RF



Soup 020199: Quantified and printed by ias on 10/19/99

000588

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IAS 10/20/99 p. 1 of 4

Page 1

Method Report

Method file: C:\MSL\Soup-rats.pro\MethDB\101999b.mdb
Last modified: Wed Oct 20 13:36:48 1999

Component: 101999A (C)

Printed: Wed Oct 20 13:10:55 1999

Soup 020199 IAS 10/20/99

Component Name: QAA (Ch1)
Internal Ref.: None
Quantity Trace: TIC
Conc. of Standards: Fixed
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 0.000
Chromatogram mass window: 1.00000Da

Retention Time (min): 7.133, Enabled
Relative Retention Time: 1.000, Disabled
RT Resolution: None
Time Window (min): 0.134

Peak Detection: nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 7

Smoothing: enabled
Window size (scans):
Number of smooths:
Smoothing method:

Join valley threshold: 0.00
Reduce peak tailing: 10.00
Raise baseline maximum: 100

Draw original threshold: 1.00
Detect outliers: Disabled
Shoulder threshold: 10.00

Relative height: 1.00, Disabled
Absolute height: 1.00, Disabled
Relative area: 1.00, Disabled
Absolute area: 1.00, Disabled

000589

Method Report

Page 2

Method file: C:\MASSLYM\sup-rats.pro\MethDB\101999b.mdb
 Last modified: Wed Oct 20 10:36:48 1999

Compounds: 2: PFOS (C)

Printed: Wed Oct 20 10:55 1999

Soup 020199 JMS 10/20/99

Component Name: FOS (Ch2)
 Internal Ref: None
 Quantify Trace: IC
 Control Standards: Conc. B
 Used RF Value: 0.000000, Disabled
 Used Peak Factors: 0.000000
 Integrate Window Factors: 0.000
 Chromatogram mass window: 0.0000Da

Retention Time (min): 0.106, Enabled
 Relative Retention Time: 0.020, Disabled
 RT Reference: None
 Time Window (min): 0.411

Peak Selection: Nearest

Response Type: External
 Response: Mass

Polynomial Type: Linear
 Point of Origin: Exclude
 Fit Weighting: 1/X
 Axis Transformation: None

Units:

Peak-to-peak Amplitude: 2

Smoothing: Enabled
 Window size (points): 1
 Number of smooths: 1
 Smoothing method:

Join valley threshold: 0.00
 Relative peak height: 0.00
 Relative peak width: 0.00

Draw external threshold: 0.00
 Detect threshold: Enabled
 Shoulder threshold: 0.00

Relative height: 0.50, Disabled
 Absolute height: 0.00, Disabled
 Relative area: 0.00, Disabled
 Absolute area: 0.00, Disabled

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000590

Method Report

Page 3

Method Name: C:\MASSLYN\Soup-rats.pro\MethDB\101999b.mdb
 Last Modified: Sat Oct 2 10:38:48 1999

Compound: 10 PFOSA

Printed: Sat Oct 2 10:56 1999

Soup 020199 IRS 10/22/99

Compound Name: PFOSA (Ch3)
 Internal File: None
 Quantity Traced: TIC
 Conc. of Standards: Conc. C
 Use of RF Values: 1.000000, Disabled
 Use of Peak Ratios: 1.000000
 Integrate Under Peaks: 1.000
 Chromatogram Windows: 1.000000s

Retention Time (min): 1.357, Enabled
 Retention Duration Time: 1.000, Disabled
 RT Toler. (min): None
 Toler. (min): 0.735

Peak Selection: Nearest

Response Type: External
 Response: Areas

Polynomial Type: Linear
 Point of Origin: Exclude
 Fit Weights: 1/X
 Axis Intersections: None

Units:

Peak Response Amplitude: 13

Smoothing: Enabled
 Window Size (Samples): 1
 Number of Smoothing: 1
 Smoothing Method:

Joining Threshold: 0.00
 Retention Threshold: 0.00
 Retention Minimum: 0.00

Detection Threshold: 0.00
 Detection: Disabled
 Signal-to-Noise: 0.00

Retention Time: 0.50, Disabled
 Absolute Area: 0.00, Disabled
 Relative Area: 0.00, Disabled
 Absolute Area: 0.00, Disabled

000591

Method Report

Page 4

Method file: C:\MASSLY\Group-rats.pro\MethDB\101999b.mdb
 Last modified: Wed Oct 20 17:36:48 1999

Component: 1: IS (Ch4)

Printed: Wed Oct 20 17:10:56 1999

Soap 320199 Ins 10/20/99

Component Name: IS (Ch4)
 Internal Label: [None]
 Quantify Label: TIC
 Control Standards: Fixed

User RF Value: 1.000000, Disabled
 User Peak Factor: 1.000000
 Integrate Window Factor: 1.000
 Chromatogram Mass Window: 1.0000Da

Retention Time (min): 7.162, Enabled
 Relative Retention Time: 1.000, Disabled
 RT Tolerance: [None]
 Time to Peak (min): 7.123

Peak Selection: Nearest

Response Type: External
 Response: Area

Polynomial Type: Linear
 Point of Origin: Exclude
 Fit Weights: 1/X
 Axis Transformation: None

Units:

Peak Amplitude: 79

Smoothing: Enabled
 Window (Scans): 2
 Number of Points: 1
 Smoothing Method:

Joining Filter Threshold: 0.00
 Reduce Peak Splitting: 00.00
 Reduce Line Maximum: 0.00

Draw Vertical Threshold: 0.00
 Detect Peaks: Disabled
 Show Peak Selection: 00.00

Retention Time: 7.162, Disabled
 Absolute Peak: 7.162, Disabled
 Relative Peak: 7.162, Disabled
 Absolute Peak: 7.162, Disabled

000592

mmh
10/19/99

SOUP 020199: FACT Equipment
Log 99092 PAGE 1/1

Quattro II, #6414E

DATE: 10/19/99

ANALYST: mmh

STUDY NUMBER: TOX145 MATRIX: rat sera DESCRIPTION: Days 1,4

FACT Method: ETS-8-5.1

Injection volume: 10 uL Cycle time: 10 min. Electrode: Z-Spray

Mobile Phase: (A) 2.0 mM NH₄CH₃CO₂ (B) MeOH

TN-A- or stdrd number (A) 99085-10-14 (B) TN-A-2997

Prepared (date/by): (A) 10/16/99 mmh (B) 10/16/99 sah

Expires: (A) 04/00 (B) 2010

Store at room temp? ☒ Y or N Other: _____

Guard column: None

Part number: na Lot number: na

Analytical column: Keystone Betasil

Size: 2 x 50 mm Type: C18 Sphere size: 5 um

Part number: 055-701-2 Lot number: P9E11 Serial number: 0591804Z

Analytical parameters:

Drying gas (L/hr): 350 ESI nebulizing gas (L/hr) 13 Sheath gas (L/hr) 0

LC Flow rate: 300 uL/min. Back pressure: 65 bar

HPLC solvent gradient: 10min-10-95

Time (min.)	%B
1) 0.00	10.0
2) 1.00	10.0
3) 5.50	95.0
4) 7.50	95.0
5) 8.00	10.0

000593

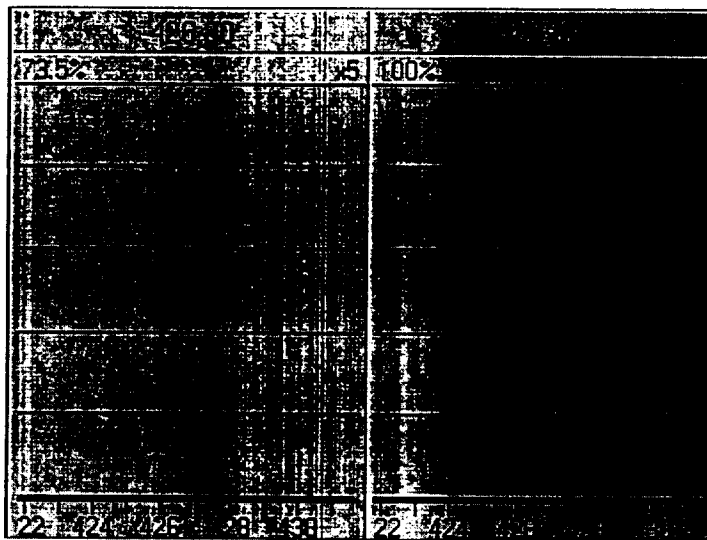
1777H
107999

Tuning Method Report

Page 1

Method: C:\MASSLYNK\SOUP-RATS.PRO\ACQUDE\PFOS3

Printed: Tue Oct 19 15:40:03 1999



SOURCE (ESP-)

	Set	Rdbk
Capillary	3.00	3.01
Cone	40	-41
Extractor	5	-8
RF Lens	0.2	
Source Block Temp.	150	150
Desolvation Temp.	250	251

MS1

	Set	Rdbk
LM Resolution	14.0	
HM Resolution	14.0	
Ion Energy	0.9	
Ion Energy Ramp	0.0	
Lens 6	4	3
Multiplier	650	

Pressures

	Rdbk
Analysar Vacuum	5.5e-5
Gas Cell	4.1e-3

MS2

	Set	Rdbk
LM Resolution	14.0	
HM Resolution	14.0	
Collision	10	8
Ion Energy	2.0	
Ion Energy Ramp	0.0	
Lens 8	40	38
Lens 9	0	1
Multiplier	650	

000594

17 PMV
10/19/95

Scanning Method Report

Page 1

Method: C:\MASSLIVE\SOUP-RATE.PRO\ACQDS\10MIN-PP08-POAA-PP08A-12
Last Modified: Fri Oct 15 11:35:03 1999

Printed: Tue Oct 19 15:40:29 1999

Solvent Delay (mins) : 0.00

Function : 1 SUM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	413.00	169.00	0.30	30	15

Function : 2 SUM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	499.00	99.00	0.30	45	60

Function : 3 SUM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	498.00	78.00	0.30	45	60

Function : 4 SUM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	427.00	80.00	0.30	40	60

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10/19/99

Masslynx - Sample List

Page 1

Sample List: C:\MASSLYNX\soup-rats.pro\SampleDB\101999b.SPL
Printed: Tue Oct 19 15:42:35 1999

Page Position: (1, 1)

	File Name	File Text	MS File
1	101999012	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
2	101999013	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
3	101999014	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
4	101999015	TOX145-RBS10199-H2O Blank-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
5	101999016	TOX145-RBS10199-Sera Blank-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
6	101999017	TOX145-RTS10199-Sera Blank-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
7	101999018	TOX145-RTS10199-Sera Blank-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
8	101999019	TOX145-RBS10199-5ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
9	101999020	TOX145-RBS10199-10ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
10	101999021	TOX145-RBS10199-25ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
11	101999022	TOX145-RBS10199-50ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
12	101999023	TOX145-RBS10199-100ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
13	101999024	TOX145-RBS10199-250ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
14	101999025	TOX145-RBS10199-500ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
15	101999026	TOX145-RBS10199-750ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
16	101999027	TOX145-RBS10199-1000ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
17	101999028	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
18	101999029	TOX145-RTS, 9R03230M, Dy1, Control,-10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
19	101999030	TOX145-RTS, 9R03231M, Dy1, Control,-10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
20	101999031	TOX145-RTS, 9R03232M, Dy1, Control,-10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
21	101999032	TOX145-RTS, 9R03233M, Dy1, Control,-10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
22	101999033	TOX145-RTS, 9R03234M, Dy1, Control,-10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
23	101999034	TOX145-RBS10199-250ppb-CCV-11-1, SEE	10MIN-PFOS-POAA-PFOSA-IS
24	101999035	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
25	101999036	TOX145-RTS, 9R03235M, Dy4, Control,-10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
26	101999037	TOX145-RTS, 9R03236M, Dy4, Control,-10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
27	101999038	TOX145-RTS, 9R03237M, Dy4, Control,-10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
28	101999039	TOX145-RTS, 9R03238M, Dy4, Control,-10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
29	101999040	TOX145-RTS, 9R03239M, Dy4, Control,-10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
30	101999041	TOX145-RBS10199-250ppb-CCV-11-2, SEE	10MIN-PFOS-POAA-PFOSA-IS
31	101999042	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
32	101999043	TOX145-RTS10199-250ppb-MS-11, 10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
33	101999044	TOX145-RTS10199-250ppb-MSD-11, 10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
34	101999045	TOX145-RTS, 9R03245M, Dy1, 5 mg/kg 10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
35	101999046	TOX145-RTS, 9R03246M, Dy1, 5 mg/kg 10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
36	101999047	TOX145-RTS, 9R03247M, Dy1, 5 mg/kg 10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
37	101999048	TOX145-RBS10199-250ppb-CCV-11-3, SEE	10MIN-PFOS-POAA-PFOSA-IS
38	101999049	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
39	101999050	TOX145-RTS, 9R03248M, Dy1, 5 mg/kg 10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
40	101999051	TOX145-RTS, 9R03249M, Dy1, 5 mg/kg 10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
41	101999052	TOX145-RTS, 9R03250M, Dy4, 5 mg/kg 10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
42	101999053	TOX145-RTS, 9R03251M, Dy4, 5 mg/kg 10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
43	101999054	TOX145-RTS, 9R03252M, Dy4, 5 mg/kg 10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
44	101999055	TOX145-RBS10199-250ppb-CCV-11-4, SEE	10MIN-PFOS-POAA-PFOSA-IS
45	101999056	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
46	101999057	TOX145-RTS, 9R03254-1M, Dy4, 5 mg/kg 10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
47	101999058	TOX145-RTS, 9R03254-2M, Dy4, 5 mg/kg 10/19/99 SEE	10MIN-PFOS-POAA-PFOSA-IS
48	101999059	TOX145-RBS10199-250ppb-CCV-11-1, SEE	10MIN-PFOS-POAA-PFOSA-IS
49	101999060	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
50	101999061	TOX145-RBS10199-H2O Blank-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
51	101999062	TOX145-RBS10199-Sera Blank-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
52	101999063	TOX145-RTS10199-Sera Blank-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
53	101999064	TOX145-RTS10199-Sera Blank-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
54	101999065	TOX145-RBS10199-5ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
55	101999066	TOX145-RBS10199-10ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
56	101999067	TOX145-RBS10199-25ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
57	101999068	TOX145-RBS10199-50ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
58	101999069	TOX145-RBS10199-100ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
59	101999070	TOX145-RBS10199-250ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS

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Masslynx - Sample List

Page 2

Sample List: C:\MASSLYNX\soup-rats.pro\SampleDB\101999b.SPL
Printed: Tue Oct 19 15:42:35 1999

Page Position: (2, 1)

	Inlet File	Bottle	Inject Volume	Sample Type	Conc A
1	10min-10-95	91	10	---	---
2	10min-10-95	91	10	---	---
3	10min-10-95	91	10	---	---
4	10min-10-95	1	10	---	---
5	10min-10-95	2	10	---	---
6	10min-10-95	3	10	---	---
7	10min-10-95	4	10	---	---
8	10min-10-95	5	10	---	---
9	10min-10-95	6	10	---	---
10	10min-10-95	7	10	---	---
11	10min-10-95	8	10	---	---
12	10min-10-95	9	10	---	---
13	10min-10-95	10	10	---	---
14	10min-10-95	11	10	---	---
15	10min-10-95	12	10	---	---
16	10min-10-95	13	10	---	---
17	10min-10-95	92	10	---	---
18	10min-10-95	14	10	---	---
19	10min-10-95	15	10	---	---
20	10min-10-95	16	10	---	---
21	10min-10-95	17	10	---	---
22	10min-10-95	18	10	---	---
23	10min-10-95	41	10	---	---
24	10min-10-95	92	10	---	---
25	10min-10-95	19	10	---	---
26	10min-10-95	20	10	---	---
27	10min-10-95	21	10	---	---
28	10min-10-95	22	10	---	---
29	10min-10-95	23	10	---	---
30	10min-10-95	42	10	---	---
31	10min-10-95	92	10	---	---
32	10min-10-95	24	10	---	---
33	10min-10-95	25	10	---	---
34	10min-10-95	26	10	---	---
35	10min-10-95	27	10	---	---
36	10min-10-95	28	10	---	---
37	10min-10-95	43	10	---	---
38	10min-10-95	93	10	---	---
39	10min-10-95	29	10	---	---
40	10min-10-95	30	10	---	---
41	10min-10-95	31	10	---	---
42	10min-10-95	32	10	---	---
43	10min-10-95	33	10	---	---
44	10min-10-95	44	10	---	---
45	10min-10-95	93	10	---	---
46	10min-10-95	34	10	---	---
47	10min-10-95	35	10	---	---
48	10min-10-95	41	10	---	---
49	10min-10-95	93	10	---	---
50	10min-10-95	1	10	---	---
51	10min-10-95	2	10	---	---
52	10min-10-95	3	10	---	---
53	10min-10-95	4	10	---	---
54	10min-10-95	5	10	---	---
55	10min-10-95	6	10	---	---
56	10min-10-95	7	10	---	---
57	10min-10-95	8	10	---	---
58	10min-10-95	9	10	---	---
59	10min-10-95	10	10	---	---

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Masslynx - Sample List

Page 3

Sample List: C:\MASSLYNX\soup-rats.pro\SampleDB\101999b.SPL'
Printed: Tue Oct 19 15:42:35 1999

Page Position: (1, 2)

	File Name	File Text	MS File
60	101999071	TOX145-RBS10199-500ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
61	101999072	TOX145-RBS10199-750ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
62	101999073	TOX145-RBS10199-1000ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS

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RMX
10-19-99

Masslynx - Sample List

Page 4

Sample List: C:\MASSLYNX\soup-rats.pro\SampleDB\101999b.SPL
Printed: Tue Oct 19 15:42:35 1999

Page Position: (2, 2)

	Inlet File	Bottle	Inject Volume	Sample Type	Conc A
60	10min-10-95	11	10 ---	---	---
61	10min-10-95	12	10 ---	---	---
62	10min-10-95	13	10 ---	---	---

000599

FACT: DATA REVIEW SUMMARY FORM

page 1 of 2
UC
10/25/99

Date: 10/25/99
Data Analyst: JAS
FACT Study #: Tox 145
Matrix/Timepoints: Kat Sera Dilutions - Day 1 & 4, ext 10/19/99
Sample List: 102299a
Set-up Analysis Date/Analyst: 10/22/99 JAS
Instrument: Amelica 262488

Data Review Sign-Off

Analyst Review
Name / Date: JAS 10/25/99

Technical Review
Name / Date: MEG 10/25/99

Quality Assurance Review
Name / Date: _____

Data Entry: Not entered need to reanalyze UC
10/25/99

Comments / Justifications: _____

Analyte: POAA (CH1) ①
Acceptable Linear Range: NA
LOQ: NA Extraction Blank: NA
R²: NA Matrix Blank: NA
Calibration Checks: NA
MS/MSD: Not entered
Data Status: Not entered

Analyte: PFOS (CH2) ②
Acceptable Linear Range: 4.93 ppb → 493 ppb
LOQ: 4.93 ppb Extracted Blank: less than LOQ
R²: 0.995295 Matrix Blank: greater than LOQ
Calibration Checks: failed
MS/MSD: Need to reanalyze UC 10/25/99
Data Status: ↓

Analyte: PFOSA (CH4) ③
Acceptable Linear Range: 4.93 ppb → 248 ppb
LOQ: 4.93 ppb Extracted Blank: less than LOQ
R²: 0.989156 Matrix Blank: less than LOQ
Calibration Checks: failed
MS/MSD: Need to reanalyze UC 10/25/99
Data Status: ↓

① The was no curve run with the sample set for POAA. Data is useful for Area counts and retention times and conc. data should not be used. Other peaks frequently were seen in the POAA channel. Peaks should be investigated. - JAS 10/25/99

② Calibration checks for PFOS failed. It is suspected that injections failed for cal checks due to lack of sample. The 735ppb & 974ppb curve points were dropped to provide a good linear fit to the sample range and to limit curve points to 30% or less deviation. It was attempted to drop the lower points so that the matrix blank would be less than the LOQ. Taking out the lower points led to 30% or more deviations for other points in the curve.

④ SUGGEST DETERMINING REASON FOR CAL CHECK FAILURE AND THEN REPEAT ANALYSIS OF THE SAMPLE SET. MEG 10/25/99.

⑦ I would suggest reanalyzing sample extracts + using mid points from std curve as the continuing calibration verifications. Also, the 4 CCV's extracted with these samples analyze prior to bracketing curve to verify Conc. UC 10/25/99.

000600

FACT: DATA REVIEW SUMMARY FORM

Page 2 of 2

LAC

10/25/99

Date: 10/25/99
 Data Analyst: Jas
 FACT Study #: TOX145
 Matrix/Timepoints: Pt Sera Dilutions, Day 1 & 4, ext 10/19/99
 Sample List: 102299a (5)
 Set-up Analysis Date/Analyst: 10/22/99 Jas
 Instrument: Amelia 062498

Data Review Sign-Off

Analyst Review

Name / Date: Jas 10/25/99

Technical Review

Name / Date: Refer to page 1-2

Quality Assurance Review

Name / Date: _____

Data Entry: Not entered - need to reanalyze LAC

Comments / Justifications: _____

10/25/99

Analyte: IS (CH3) (8)
 Acceptable Linear Range: NA
 LOQ: NA Extraction Blank: NA
 R²: NA Matrix Blank: NA
 Calibration Checks: NA
 MS/MSD: _____
 Data Status: _____

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: Jas 10/25/99
 Data Status: _____

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: _____

(2) Other peaks were frequently seen in the ~~PTOSA~~ ^{PTOSA} channel. Peaks should be investigated. -Jas 10/25/99

(3) The 493 ppb, 736 ppb, and 997 ppb calix points were dropped to provide a good linear curve fit to the sample set. The Cal checks failed; probably due to lack of sample.

Other peaks were frequently seen in the PTOSA channel and should be investigated -Jas 10/25/99

(4) IS area counts looked consistent in the curves & cal checks. Sample dilutions were not spiked with IS. -Jas 10/25/99

(5) Samples should be rerun, maybe(?) due to the failed cal checks. The samples are bracketed by the curves. In comments (2) & (3) cal check deviations were thought to have resulted from lack of sample but IS area counts look good(?). Jas 10/25/99

000601

Isa 10/25/99 7 lot 4

Quantify Compound Summary Report
TOX145-rat serum dilutions, day 1 & 4- ext. 10/19/99 SEE

Sample List: C:\MSASLT\TOX\amelia-rats.FPO\Sample08\102299a
Last modified: Mon Oct 25 06:43:56 1999
Method: C:\MSASLT\TOX\amelia-rats.FPO\Meth08\102299a
Last modified: Mon Oct 25 06:39:55 1999
Job Code:

Printed: Mon Oct 25 06:59:42 1999

Compound 1: POAA (Chl)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Conc.	Flags	%Dev
1 102299001	MeOH Blank							
2 102299002	MeOH Blank							
3 102299003	TOX145-RBS10199-H2O Blk-12, SEE			6.990	120	1.04 bd		4
4 102299004	TOX145-RBS10199-Serum Blk-12, SEE			6.997	109	0.94 bb		-6
5 102299005	TOX145-RBS10199-Serum Blk-12, SEE			7.015	115	0.99 MM		-1
6 102299006	TOX145-RBS10199-5ppb-12, SEE	Standard	1.00	7.000	110	0.95 bb		-5
7 102299007	TOX145-RBS10199-10ppb-12, SEE	Standard	1.00	7.027	125	1.08 bb		8
8 102299008	TOX145-RBS10199-25ppb-12, SEE	Standard	1.00	7.010	110	0.95 bb		-5
9 102299009	TOX145-RBS10199-50ppb-12, SEE	Standard	1.00	6.998	97	0.84 db		-16
10 102299010	TOX145-RBS10199-100ppb-12, SEE	Standard	1.00	6.989	75	0.65 bb		-35
11 102299011	TOX145-RBS10199-250ppb-12, SEE	Standard	1.00	6.995	119	1.02 bb		2
12 102299012	TOX145-RBS10199-500ppb-12, SEE	Standard	1.00	6.998	142	1.22 bb		22
13 102299013	TOX145-RBS10199-750ppb-12, SEE	Standard	1.00	6.996	76	0.66 bb		-34
14 102299014	TOX145-RBS10199-1000ppb-12, SEE	Standard	1.00	7.006	158	1.37 bb		37
15 102299015	MeOH Blank							
16 102299016	TOX145-RTS-9R03245M, Day1, 5mg/kg, D50-ext10/19/99			6.997	24	0.21 bb		-79
17 102299017	TOX145-RTS-9R03246M, Day1, 5mg/kg, D50-ext10/19/99			6.974	34	0.29 bb		-71
18 102299018	TOX145-RTS-9R03247M, Day1, 5mg/kg, D50-ext10/19/99			6.994	36	0.31 bb		-69
19 102299019	TOX145-RTS-9R03248M, Day1, 5mg/kg, D50-ext10/19/99			6.995	29	0.25 bb		-75
20 102299020	TOX145-RTS-9R03249M, Day1, 5mg/kg, D50-ext10/19/99			6.965	26	0.23 bb		-77
21 102299021	TOX145-RBS10199-250ppb-CCV-11-1, SEE	QC	1.00	6.996	77	0.66 bb		-34
22 102299022	MeOH Blank							
23 102299023	TOX145-RTS-9R03250M, Day4, 5mg/kg, D50-ext10/19/99			7.018	20	0.17 db		-83
24 102299024	TOX145-RTS-9R03251M, Day4, 5mg/kg, D50-ext10/19/99			7.000	25	0.21 bb		-79
25 102299025	TOX145-RTS-9R03252M, Day4, 5mg/kg, D50-ext10/19/99			6.972	26	0.23 bb		-77
26 102299026	TOX145-RTS-9R03254-1M, Day4, 5mg/kg, D50- 10/19/99			6.992	22	0.19 bb		-81
27 102299027	TOX145-RTS-9R03254-2M, Day4, 5mg/kg, D50- 10/19/99							
28 102299028	TOX145-RBS10199-250ppb-CCV-11-2, SEE	QC	1.00	6.997	52	0.45 bb		-55
29 102299029	MeOH Blank							
30 102299030	TOX145-RBS10199-H2O Blk-12, SEE			7.094	11	0.10 bb		-90
31 102299031	TOX145-RBS10199-Serum Blk-12, SEE			6.993	117	1.01 bb		1
32 102299032	TOX145-RBS10199-Serum Blk-12, SEE			6.999	119	1.03 bb		3
33 102299033	TOX145-RBS10199-5ppb-12, SEE	Standard	1.00	6.990	94	0.81 bb		-19
34 102299034	TOX145-RBS10199-10ppb-12, SEE	Standard	1.00	6.996	105	0.91 bb		-9
35 102299035	TOX145-RBS10199-25ppb-12, SEE	Standard	1.00	7.001	138	1.19 bd		19
36 102299036	TOX145-RBS10199-50ppb-12, SEE	Standard	1.00	7.022	117	1.01 bb		1
37 102299037	TOX145-RBS10199-100ppb-12, SEE	Standard	1.00	6.996	104	0.90 bd		-10
38 102299038	TOX145-RBS10199-250ppb-12, SEE	Standard	1.00	7.001	73	0.63 db		-37
39 102299039	TOX145-RBS10199-500ppb-12, SEE	Standard	1.00	7.016	138	1.19 bb		19
40 102299040	TOX145-RBS10199-750ppb-12, SEE	Standard	1.00	6.998	160	1.38 bb		38
41 102299041	TOX145-RBS10199-1000ppb-12, SEE	Standard	1.00	6.997	95	0.82 bb		-18
				6.980	143	1.24 bb		24

Quantify Compound Summary Report
 TOX145-rat serum dilutions, day 1 & 4- ext. 10/19/99 SEE

Sample List: C:\MASSLYNK\amelia-rats.PPO\SampleDB\102299a
 Last modified: Mon Oct 25 06:43:56 1999
 Method: C:\MASSLYNK\amelia-rats.PPO\MethDB\102299a
 Last modified: Mon Oct 25 06:39:55 1999
 Job Code:

Printed: Mon Oct 25 06:59:42 1999

Compound 2: PFOS (CH2)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Conc. Flags	%Dev
1 102299001	MeOH Blank			7.190	119	0.24 MM	
2 102299002	MeOH Blank			7.183	82	0.00 MM	
3 102299003	TOX145-RBS10199-H2O Blk-12, SEE			7.186	112	0.08 MM	
4 102299004	TOX145-RBS10199-Serum Blk-12, SEE			7.170	88	0.00 MM	
5 102299005	TOX145-RBS10199-Serum Blk-12, SEE			7.162	263	3.44 bb	
6 102299006	TOX145-RBS10199-5ppb-12, SEE	Standard	4.93	7.171	330	4.94 bb	0
7 102299007	TOX145-RBS10199-10ppb-12, SEE	Standard	9.76	7.176	528	9.34 bb	-4
8 102299008	TOX145-RBS10199-25ppb-12, SEE	Standard	24.80	7.184	1195	24.22 bb	-2
9 102299009	TOX145-RBS10199-50ppb-12, SEE	Standard	49.30	7.173	1909	40.12 bd	-19
10 102299010	TOX145-RBS10199-100ppb-12, SEE	Standard	97.60	7.164	5512	120.40 bb	23
11 102299011	TOX145-RBS10199-250ppb-12, SEE	Standard	248.00	7.166	11228	247.73 bb	-0
12 102299012	TOX145-RBS10199-500ppb-12, SEE	Standard	493.00	7.169	22030	488.41 bb	-1
13 102299013	TOX145-RBS10199-750ppb-12, SEE	Standard	735.00	7.169	28219	626.31 bbX	-15
14 102299014	TOX145-RBS10199-1000ppb-12, SEE	Standard	976.00	7.179	36823	818.00 bbX	-16
15 102299015	MeOH Blank			7.172	109	0.01 MM	
16 102299016	TOX145-RTS-9R03245M, Day1, 5mg/kg, D50-ext10/19/99			7.169	4125	89.49 bb	
17 102299017	TOX145-RTS-9R03246M, Day1, 5mg/kg, D50-ext10/19/99			7.173	6459	141.49 bb	
18 102299018	TOX145-RTS-9R03247M, Day1, 5mg/kg, D50-ext10/19/99			7.143	5637	123.18 bb	
19 102299019	TOX145-RTS-9R03248M, Day1, 5mg/kg, D50-ext10/19/99			7.168	3710	80.23 bb	
20 102299020	TOX145-RTS-9R03249M, Day1, 5mg/kg, D50-ext10/19/99			7.161	4380	95.16 bb	
21 102299021	TOX145-RBS10199-250ppb-CCV-11-1, SEE	QC	248.00	7.168	168	1.33 bb	-99
22 102299022	MeOH Blank			7.165	40	0.00 MM	
23 102299023	TOX145-RTS-9R03250M, Day4, 5mg/kg, D50-ext10/19/99			7.168	4350	94.50 bb	
24 102299024	TOX145-RTS-9R03251M, Day4, 5mg/kg, D50-ext10/19/99			7.172	4238	92.01 bb	
25 102299025	TOX145-RTS-9R03252M, Day4, 5mg/kg, D50-ext10/19/99			7.168	6453	141.35 bb	
26 102299026	TOX145-RTS-9R03254-1M, Day4, 5mg/kg, D50- 10/19/99			7.164	5257	114.70 bb	
27 102299027	TOX145-RTS-9R03254-2M, Day4, 5mg/kg, D50- 10/19/99			7.169	4193	91.01 bb	
28 102299028	TOX145-RBS10199-250ppb-CCV-11-2, SEE	QC	248.00	7.170	217	2.42 bb	-99
29 102299029	MeOH Blank			7.171	23	0.00 MM	
30 102299030	TOX145-RBS10199-H2O Blk-12, SEE			7.165	75	0.00 MM	
31 102299031	TOX145-RBS10199-Serum Blk-12, SEE			7.172	96	0.00 MM	
32 102299032	TOX145-RBS10199-Serum Blk-12, SEE			7.163	262	3.43 bb	
33 102299033	TOX145-RBS10199-5ppb-12, SEE	Standard	4.93	7.169	336	5.07 bb	3
34 102299034	TOX145-RBS10199-10ppb-12, SEE	Standard	9.76	7.173	521	9.20 bb	-6
35 102299035	TOX145-RBS10199-25ppb-12, SEE	Standard	24.80	7.194	1235	25.11 bb	1
36 102299036	TOX145-RBS10199-50ppb-12, SEE	Standard	49.30	7.169	2005	42.26 bb	-14
37 102299037	TOX145-RBS10199-100ppb-12, SEE	Standard	97.60	7.173	5514	120.43 bb	23
38 102299038	TOX145-RBS10199-250ppb-12, SEE	Standard	248.00	7.189	11233	247.86 bb	-0
39 102299039	TOX145-RBS10199-500ppb-12, SEE	Standard	493.00	7.171	21189	469.68 bb	-5
40 102299040	TOX145-RBS10199-750ppb-12, SEE	Standard	735.00	7.169	29094	645.81 bbX	-12
41 102299041	TOX145-RBS10199-1000ppb-12, SEE	Standard	976.00	7.175	37681	837.11 bbX	-14

Quantify Compound Summary Report
TOX145-rat serum dilutions, day 1 & 4- ext. 10/19/99 SEE

Page 3

Sample List: C:\MASSLYNK\amalia-rats.PRO\SampleDB\102299a
Last modified: Mon Oct 25 06:43:56 1999
Method: C:\MASSLYNK\amalia-rats.PRO\MethDB\102299a
Last modified: Mon Oct 25 06:39:55 1999
Job Code:

Printed: Mon Oct 25 06:59:42 1999

Compound 3: PFOSA (Ch4)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Conc.	Flags	dev
1 102299001	MeOH Blank			7.846	465	4.23	bb	
2 102299002	MeOH Blank			7.840	331	3.42	bd	
3 102299003	TOX145-RBS10199-H2O Blk-12, SEE			7.843	152	2.33	MM	
4 102299004	TOX145-RBS10199-Serum Blk-12, SEE			7.850	151	2.33	MM	
5 102299005	TOX145-RBS10199-Serum Blk-12, SEE			7.843	95	1.99	MM	
6 102299006	TOX145-RBS10199-5ppb-12, SEE	Standard	4.93	7.826	815	6.36	bb	29
7 102299007	TOX145-RBS10199-10ppb-12, SEE	Standard	9.77	7.834	1344	9.57	bb	-2
8 102299008	TOX145-RBS10199-25ppb-12, SEE	Standard	24.80	7.840	3615	23.35	bb	-6
9 102299009	TOX145-RBS10199-50ppb-12, SEE	Standard	49.30	7.828	5624	35.54	bb	-28
10 102299010	TOX145-RBS10199-100ppb-12, SEE	Standard	97.70	7.821	18036	110.88	bb	13
11 102299011	TOX145-RBS10199-250ppb-12, SEE	Standard	248.00	7.820	39992	244.14	bb	-2
12 102299012	TOX145-RBS10199-500ppb-12, SEE	Standard	493.00	7.826	88286	537.25	bbX	9
13 102299013	TOX145-RBS10199-750ppb-12, SEE	Standard	736.00	7.826	126147	767.03	bbX	4
14 102299014	TOX145-RBS10199-1000ppb-12, SEE	Standard	977.00	7.835	170989	1039.19	bbX	6
15 102299015	MeOH Blank			7.853	424	3.98	bb	
16 102299016	TOX145-RTS-9R03245M, Day1, 5mg/kg, D50-ext10/19/99			7.825	1404	9.94	bb	
17 102299017	TOX145-RTS-9R03246M, Day1, 5mg/kg, D50-ext10/19/99			7.828	2212	14.83	bb	
18 102299018	TOX145-RTS-9R03247M, Day1, 5mg/kg, D50-ext10/19/99			7.825	1199	8.69	bb	
19 102299019	TOX145-RTS-9R03248M, Day1, 5mg/kg, D50-ext10/19/99			7.825	1612	11.19	bb	
20 102299020	TOX145-RTS-9R03249M, Day1, 5mg/kg, D50-ext10/19/99			7.842	1370	9.72	bb	
21 102299021	TOX145-RBS10199-250ppb-CCV-11-1, SEE	QC	248.00	7.824	218	2.73	bb	-99
22 102299022	MeOH Blank			7.868	37	1.63	bb	
23 102299023	TOX145-RTS-9R03250M, Day4, 5mg/kg, D50-ext10/19/99			7.848	330	3.42	MM	
24 102299024	TOX145-RTS-9R03251M, Day4, 5mg/kg, D50-ext10/19/99			7.833	316	3.33	bb	
25 102299025	TOX145-RTS-9R03252M, Day4, 5mg/kg, D50-ext10/19/99			7.848	398	3.82	bb	
26 102299026	TOX145-RTS-9R03254-1M, Day4, 5mg/kg, D50- 10/19/99			7.845	422	3.97	MM	
27 102299027	TOX145-RTS-9R03254-2M, Day4, 5mg/kg, D50- 10/19/99			7.850	337	3.46	bb	
28 102299028	TOX145-RBS10199-250ppb-CCV-11-2, SEE	QC	248.00	7.828	283	3.13	bb	-99
29 102299029	MeOH Blank			7.852	25	1.56	bb	
30 102299030	TOX145-RBS10199-H2O Blk-12, SEE			7.846	61	1.78	MM	
31 102299031	TOX145-RBS10199-Serum Blk-12, SEE			7.853	47	1.70	MM	
32 102299032	TOX145-RTS10199-Serum Blk-12, SEE			7.843	56	1.75	MM	
33 102299033	TOX145-RBS10199-5ppb-12, SEE	Standard	4.93	7.826	730	5.84	bb	19
34 102299034	TOX145-RBS10199-10ppb-12, SEE	Standard	9.77	7.827	1319	9.41	bb	-4
35 102299035	TOX145-RBS10199-25ppb-12, SEE	Standard	24.80	7.851	3425	22.20	bb	-10
36 102299036	TOX145-RBS10199-50ppb-12, SEE	Standard	49.30	7.828	5469	34.60	bb	-30
37 102299037	TOX145-RBS10199-100ppb-12, SEE	Standard	97.70	7.830	18963	116.50	bb	19
38 102299038	TOX145-RBS10199-250ppb-12, SEE	Standard	248.00	7.846	41057	250.60	bb	1
39 102299039	TOX145-RBS10199-500ppb-12, SEE	Standard	493.00	7.828	88564	538.93	bbX	9
40 102299040	TOX145-RBS10199-750ppb-12, SEE	Standard	736.00	7.827	125699	764.31	bbX	4
41 102299041	TOX145-RBS10199-1000ppb-12, SEE	Standard	977.00	7.832	168896	1026.49	bbX	5

Amelia 062498: Quantitated and Printed by ias 10/25/99

000604

Sample List: C:\MSL\TOX\Amelia-rats.PRO\SampleDB\102299a
 Last modified: Mon Oct 25 06:43:56 1999
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 Last modified: Mon Oct 25 06:39:55 1999
 Job Code:

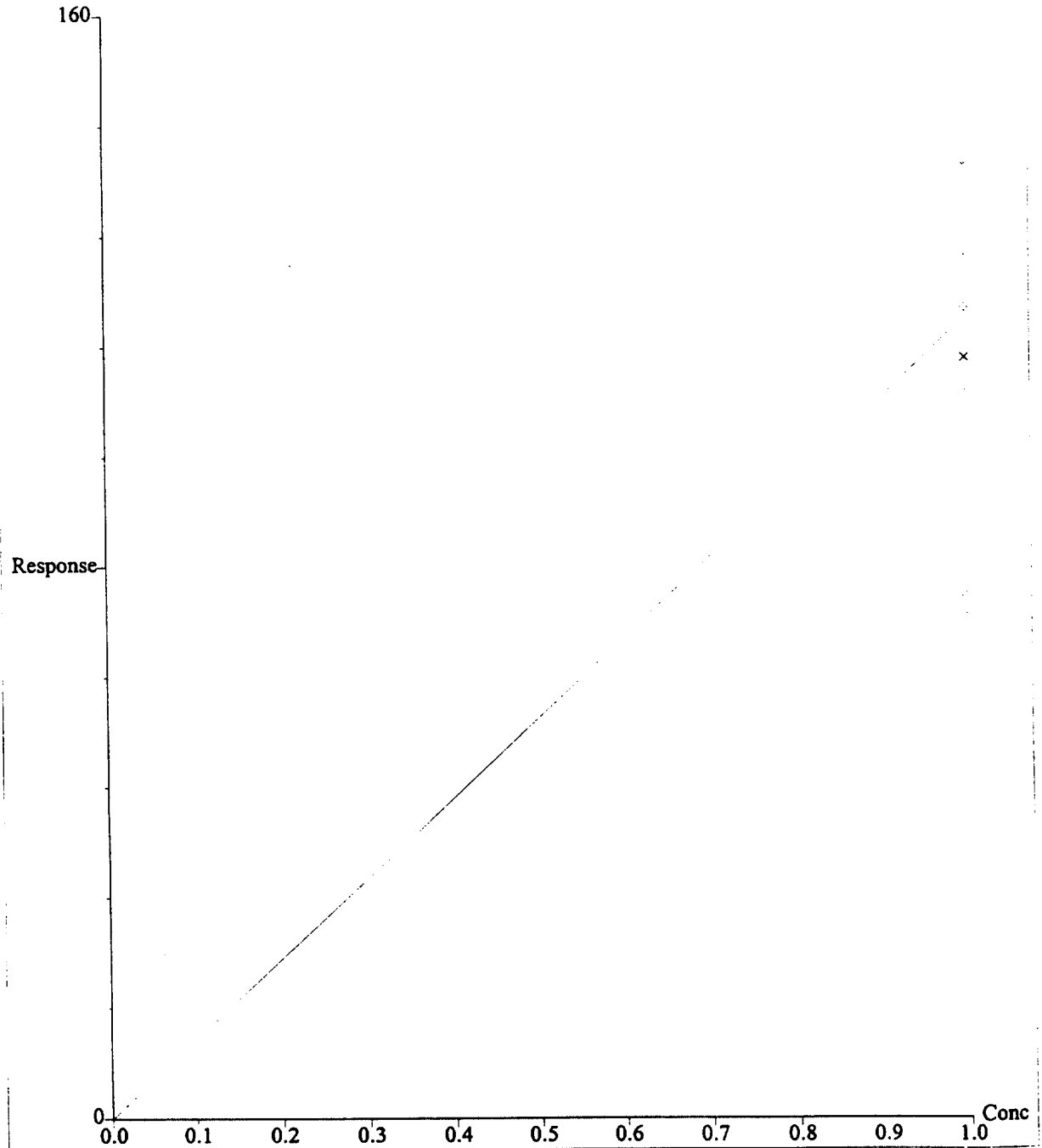
Printed: Mon Oct 25 06:59:42 1999

Compound 4: IS (Ch3)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Conc.	Flags	StDev
1 102299001	MeOH Blank							
2 102299002	MeOH Blank							
3 102299003	TOX145-RBS10199-H2O Blk-12, SEE			7.001	3036	1.13	bd	13
4 102299004	TOX145-RBS10199-Serum Blk-12, SEE			6.984	2591	0.96	bb	-4
5 102299005	TOX145-RBS10199-Serum Blk-12, SEE			7.002	2851	1.06	bb	6
6 102299006	TOX145-RBS10199-5ppb-12, SEE	Standard	1.00	6.988	2705	1.00	bb	0
7 102299007	TOX145-RBS10199-10ppb-12, SEE	Standard	1.00	7.014	2805	1.04	bb	4
8 102299008	TOX145-RBS10199-25ppb-12, SEE	Standard	1.00	6.997	2491	0.93	bb	-7
9 102299009	TOX145-RBS10199-50ppb-12, SEE	Standard	1.00	7.009	2154	0.80	bb	-20
10 102299010	TOX145-RBS10199-100ppb-12, SEE	Standard	1.00	7.000	2852	1.06	bb	6
11 102299011	TOX145-RBS10199-250ppb-12, SEE	Standard	1.00	6.983	2809	1.04	bb	4
12 102299012	TOX145-RBS10199-500ppb-12, SEE	Standard	1.00	6.985	3031	1.13	dd	13
13 102299013	TOX145-RBS10199-750ppb-12, SEE	Standard	1.00	6.983	3075	1.14	bd	14
14 102299014	TOX145-RBS10199-1000ppb-12, SEE	Standard	1.00	6.994	2977	1.11	MM	11
15 102299015	MeOH Blank							
16 102299016	TOX145-RTS-9R03245M, Day1, 5mg/kg, D50-ext10/19/99			6.984	89	0.03	MM	-97
17 102299017	TOX145-RTS-9R03246M, Day1, 5mg/kg, D50-ext10/19/99			6.985	83	0.03	MM	-97
18 102299018	TOX145-RTS-9R03247M, Day1, 5mg/kg, D50-ext10/19/99			6.982	76	0.03	bd	-97
19 102299019	TOX145-RTS-9R03248M, Day1, 5mg/kg, D50-ext10/19/99			6.982	76	0.03	bd	-97
20 102299020	TOX145-RTS-9R03249M, Day1, 5mg/kg, D50-ext10/19/99			6.976	69	0.03	bb	-97
21 102299021	TOX145-RBS10199-250ppb-CCV-11-1, SEE	QC	1.00	6.983	2290	0.85	bb	-15
22 102299022	MeOH Blank							
23 102299023	TOX145-RTS-9R03250M, Day4, 5mg/kg, D50-ext10/19/99			6.982	84	0.03	bb	-97
24 102299024	TOX145-RTS-9R03251M, Day4, 5mg/kg, D50-ext10/19/99			6.987	58	0.02	bd	-98
25 102299025	TOX145-RTS-9R03252M, Day4, 5mg/kg, D50-ext10/19/99			6.983	80	0.03	MM	-97
26 102299026	TOX145-RTS-9R03254-1M, Day4, 5mg/kg, D50- 10/19/99			7.003	66	0.02	bd	-98
27 102299027	TOX145-RTS-9R03254-2M, Day4, 5mg/kg, D50- 10/19/99			6.983	70	0.03	bd	-97
28 102299028	TOX145-RBS10199-250ppb-CCV-11-2, SEE	QC	1.00	6.985	1889	0.70	bb	-30
29 102299029	MeOH Blank							
30 102299030	TOX145-RBS10199-H2O Blk-12, SEE			6.981	2798	1.04	bd	4
31 102299031	TOX145-RBS10199-Serum Blk-12, SEE			6.987	2329	0.86	bb	-14
32 102299032	TOX145-RTS10199-Serum Blk-12, SEE			7.001	2521	0.94	bb	-6
33 102299033	TOX145-RBS10199-5ppb-12, SEE	Standard	1.00	6.983	2515	0.93	bd	-7
34 102299034	TOX145-RBS10199-10ppb-12, SEE	Standard	1.00	6.988	2308	0.86	MM	-14
35 102299035	TOX145-RBS10199-25ppb-12, SEE	Standard	1.00	7.009	2391	0.89	bd	-11
36 102299036	TOX145-RBS10199-50ppb-12, SEE	Standard	1.00	7.007	2192	0.81	bd	-19
37 102299037	TOX145-RBS10199-100ppb-12, SEE	Standard	1.00	7.012	2855	1.06	bb	6
38 102299038	TOX145-RBS10199-250ppb-12, SEE	Standard	1.00	7.004	2958	1.10	bd	10
39 102299039	TOX145-RBS10199-500ppb-12, SEE	Standard	1.00	7.009	2896	1.08	bd	8
40 102299040	TOX145-RBS10199-750ppb-12, SEE	Standard	1.00	7.008	2813	1.04	bd	4
41 102299041	TOX145-RBS10199-1000ppb-12, SEE	Standard	1.00	6.991	2853	1.06	bb	6

Quantify Calibration Report
TOX145-rat serum dilutions, day 1 & 4- ext. 10/19/99 SEE

Compound 1 name: POAA (Ch1)
Response Factor: 115.846
Response type: External Std, Area
Curve type: RF



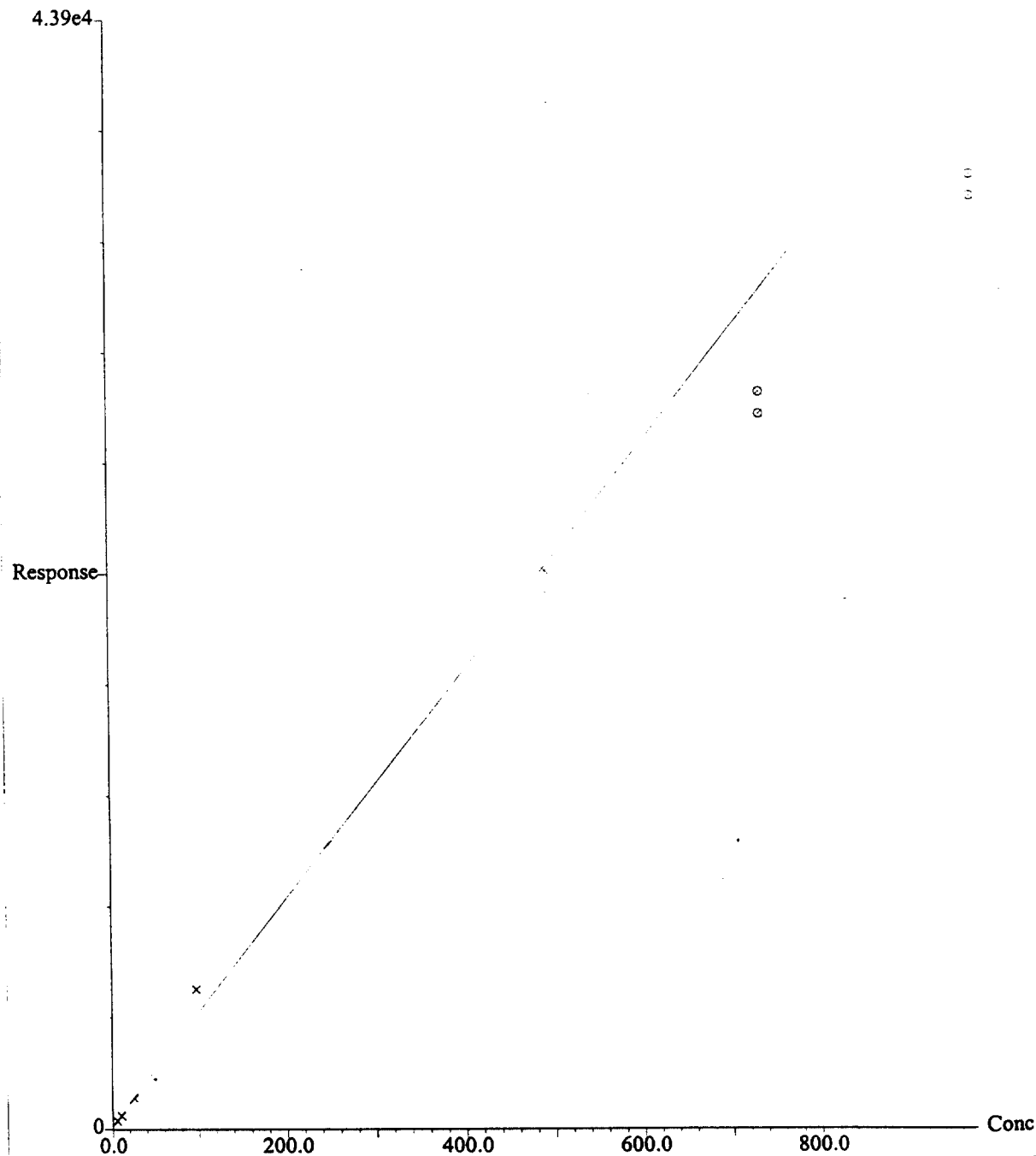
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000606

Quantify Calibration Report
TOX145-rat serum dilutions, day 1 & 4- ext. 10/19/99 SEE

Page 2

Compound 2 name: PFOS (Ch2)
Coefficient of Determination: 0.995295
Calibration curve: $44.8833 * x + 108.462$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



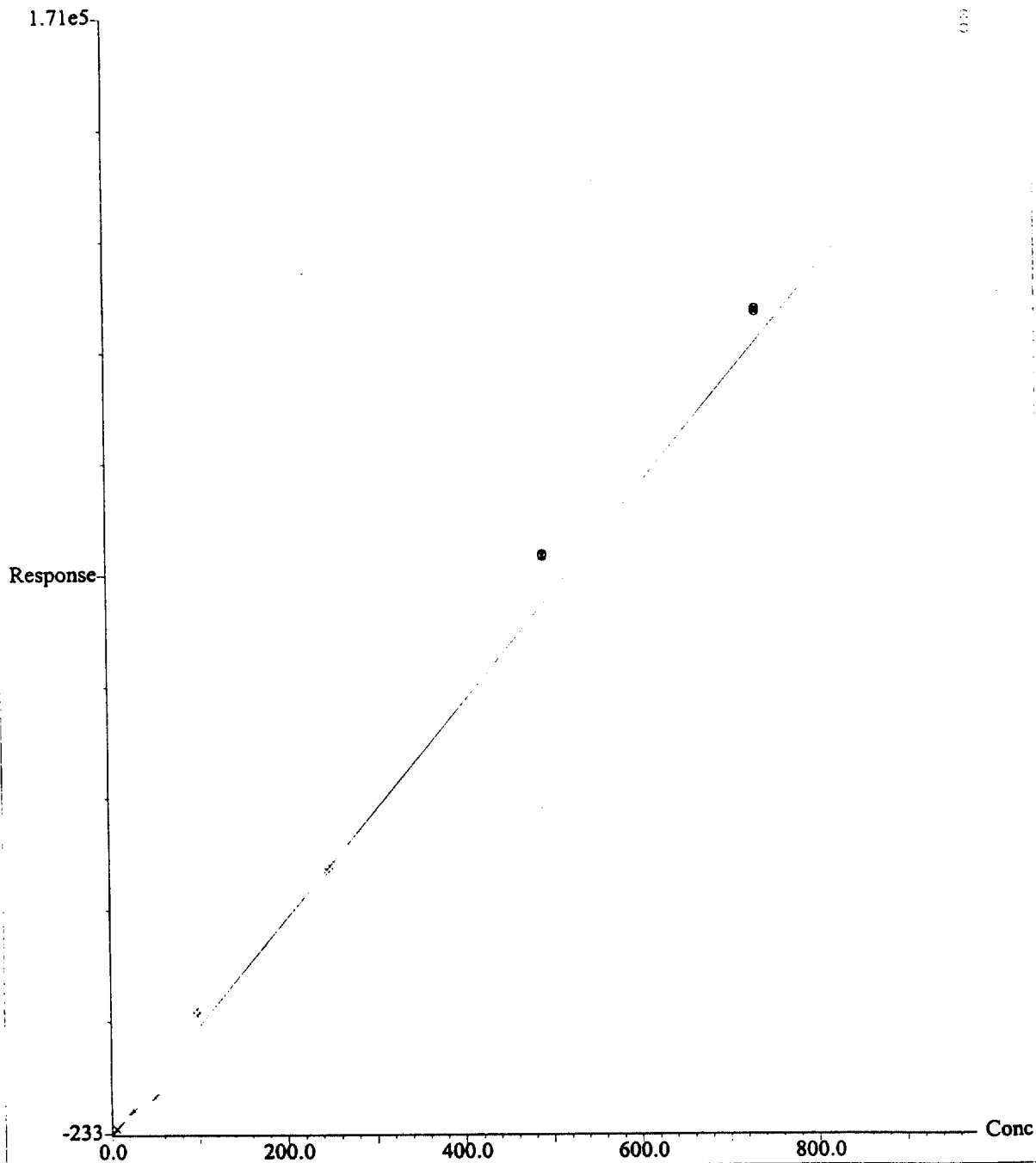
Amelia 062498: Quantitated and Printed by las 10/25/99

000607

Quantify Calibration Report
TOX145-rat serum dilutions, day 1 & 4- ext. 10/19/99 BEE

Page 3

Compound 3 name: PFOSA (Ch4)
Coefficient of Determination: 0.989156
Calibration curve: $164.764 * x + -232.515$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None

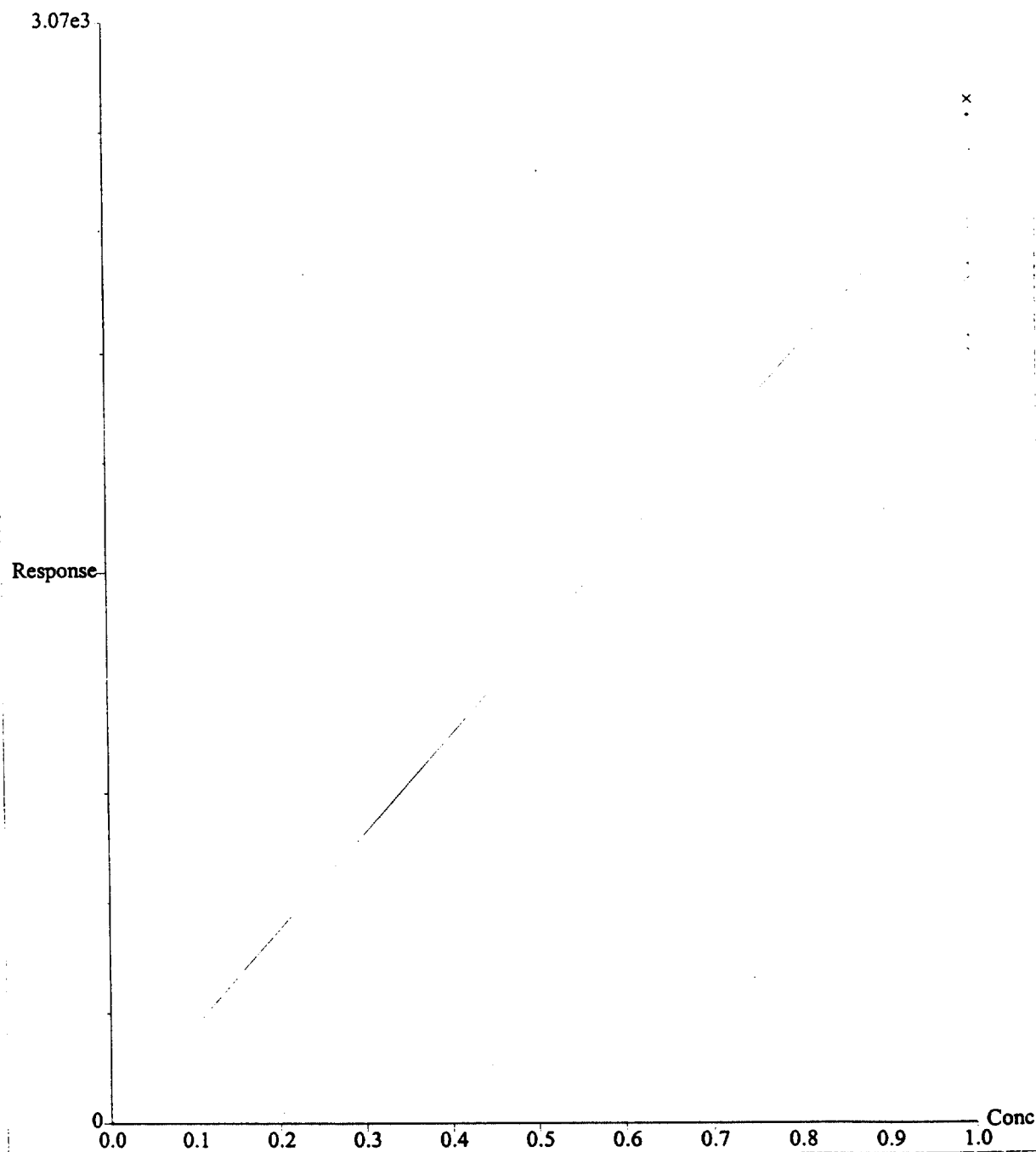


Amelia 062498: Quantitated and Printed by iac 10/25/99

Quantify Calibration Report
TOX145-rat serum dilutions, day 1 & 4- ext. 10/19/99 SEE

Page 4

Compound 4 name: IS (Ch3)
Response Factor: 2692.44
Response type: External Std, Area
Curve type: RF



Amelia 062490: Quantitated and Printed by las 10/25/99

000609

IAS 10/25/99 p. 1 of 4

Page 1

Method Report

Method file: C:\MASSLYNX\amelia-rats.PRO\MethDB\102299a.mdb
Last modified: Mon Oct 25 06:39:55 1999C
Compound: 1: POAA (Ch1)
Printed: Mon Oct 25 07:03:23 1999

Amelia 062498 IAS 10/25/99

Compound Name: POAA (Ch1)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Fixed

User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da
Peak Location

Retention Time (mins): 6.994, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.203

Peak Selection: Nearest
Response

Response Type: External
Response: Areas
Calibration Curves

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None
Concentration

Units:
Noise

Peak-to-peak amplitude: 95
Smooth

Smoothing: Disabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:
Baselines

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00
Peak Separation

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00
Response Threshold

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000610

Method Report

Method file: C:\MASSL\YOK\amelia-rata.PRO\MethDB\102299a.mdb
 Last modified: Mon Oct 25 06:39:55 1999C
 Compound: 2: PFOS (Ch2)
 Printed: Mon Oct 25 07:03:23 1999

Amelia 062498 IN 10/25/99

Compound Name: PFOS (Ch2)
 Internal Ref.: [None]
 Quantify Trace: TIC
 Conc. of Standards: Conc. A

User RF Value: 1.000000, Disabled
 User Peak Factor: 1.000000
 Integrate Window Factor: 2.000
 Chromatogram mass window: 1.0000Da
Peak Location

Retention Time (mins): 7.178, Enabled
 Relative Retention Time: 0.000, Disabled
 RT Reference: [None]
 Time Window (mins): 0.262

Peak Selection: Nearest
Response

Response Type: External
 Response: Areas
Calibration Curves

Polynomial Type: Linear
 Point of Origin: Exclude
 Fit Weighting: 1/X
 Axis Transformation: None
Concentration

Units:
Noise

Peak-to-peak amplitude: 2300
Smooth

Smoothing: Disabled
 Window size (scans): 2
 Number of smooths: 1
 Smoothing method:
Baselines

Join valley threshold: 30.00
 Reduce peak tailing: 200.00
 Raise baseline maximum: 5.00
Peak Separation

Draw vertical threshold: 90.00
 Detect shoulders: Disabled
 Shoulder threshold: 30.00
Response Threshold

Relative height: 1.50, Disabled
 Absolute height: 0.00, Disabled
 Relative area: 2.00, Disabled
 Absolute area: 0.00, Disabled

000611

Method Report

Page 3

Method file: C:\MASSLYNX\amelia-rats.PRO\MethDB\102299a.mdb
Last modified: Mon Oct 25 06:39:55 1999

Compound: 3: PFOSA (Ch4)

Printed: Mon Oct 25 07:03:23 1999

Amelia 062498 Inc 10/26/99

Compound Name: PFOSA (Ch4)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. B

User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da
Peak Location

Retention Time (mins): 7.846, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.273

Peak Selection: Nearest
Response

Response Type: External
Response: Areas
Calibration Curves

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None
Concentration

Units:
Noise

Peak-to-peak amplitude: 161
Smooth

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:
Baselines

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00
Peak Separation

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00
Response Threshold

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000612

Method Report

Method file: C:\MASSLYNX\amelia-rats.PRO\MethDB\102299a.mdb
 Last modified: Mon Oct 25 06:39:55 1999

Compound: 4: IS (Ch3)

Printed: Mon Oct 25 07:03:23 1999

Amelia 062498 JMS 10/25/99

Compound Name: IS (Ch3)
 Internal Ref.: [None]
 Quantify Trace: TIC
 Conc. of Standards: Fixed

User RF Value: 1.000000, Disabled
 User Peak Factor: 1.000000
 Integrate Window Factor: 2.000
 Chromatogram mass window: 1.0000Da
Peak Location

Retention Time (mins): 7.040, Enabled
 Relative Retention Time: 0.000, Disabled
 RT Reference: [None]
 Time Window (mins): 0.239

Peak Selection: Nearest
Response

Response Type: External
 Response: Areas
Calibration Curves

Polynomial Type: Linear
 Point of Origin: Exclude
 Fit Weighting: 1/X
 Axis Transformation: None
Concentration

Units:
Noise

Peak-to-peak amplitude: 89
Smooth

Smoothing: Disabled
 Window size (scans): 2
 Number of smooths: 1
 Smoothing method:
Baselines

Join valley threshold: 30.00
 Reduce peak tailing: 200.00
 Raise baseline maximum: 5.00
Peak Separation

Draw vertical threshold: 90.00
 Detect shoulders: Disabled
 Shoulder threshold: 30.00
Response Threshold

Relative height: 1.50, Disabled
 Absolute height: 0.00, Disabled
 Relative area: 2.00, Disabled
 Absolute area: 0.00, Disabled

000613

10/22/99

AMELIA 062498: FACT Equipment Log 99114
Quattro II, #6448E

PAGE _____

DATE: 10/22/99

ANALYST: ias

STUDY NUMBER: TOX145 MATRIX: Rat sera DESCRIPTION: Day 1&4 dilutions

FACT Method: ETS-8-5.1

Injection volume: 10 uL Cycle time: 10 min Electrode: Z-Spray

Mobile Phase: (A) 2.0 mM NH₄CH₃CO₂ (B) MeOH

TN-A- or stdrd number (A) 99085-10-14 (B) TN-A-2945

Prepared (date/by): (A) 10/16/99 sah (B) 10/20/99 ias

Expires: (A) 04/00 (B) 04/00

Store at room temp? ☒ Y or N Other: _____

Guard column: None

Part number: na Lot number: na

Analytical column: Keystone Betasil

Size: 2 x 50 mm Type: C18 Sphere size: 5 um

Part number: 055-701-2 Lot number: P9E11 Serial number: 0591816Z

Analytical parameters:

Drying gas (L/hr): 300 ESI nebulizing gas (L/hr) 10 Sheath gas (L/hr) 0

LC Flow rate: 300 uL/min Back pressure: 62 bar

HPLC solvent gradient: 10min-10-95

Time (min.)	%B
1) <u>0.00</u>	<u>10.0</u>
2) <u>1.00</u>	<u>10.0</u>
3) <u>5.50</u>	<u>95.0</u>
4) <u>7.50</u>	<u>95.0</u>
5) <u>8.00</u>	<u>10.0</u>

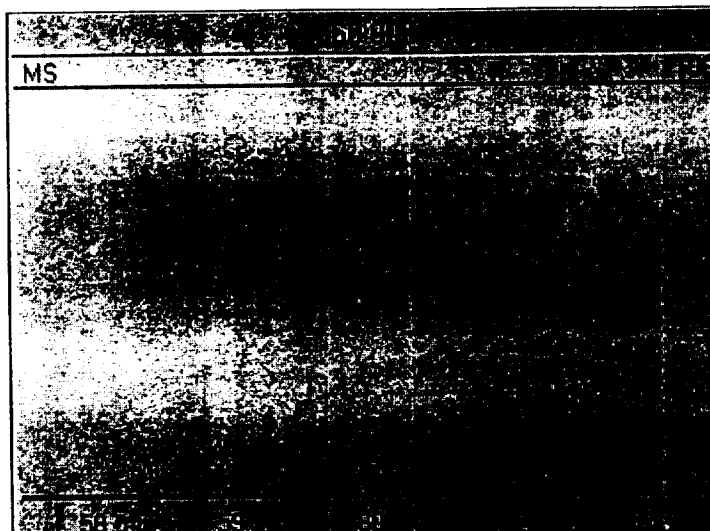
000614

Tuning Method Report

Page 1

Method: C:\MASSLYNX\AMELIA-RATS.PRO\ACQUDE\PFOS1-6KV

Printed: Fri Oct 22 09:58:58 1999



MS

SOURCE (ESP-)	Set	Rdbk	MS1	Set	Rdbk
Capillary	1.60	1.60	LM Resolution	15.0	
Cone	40	-14	HM Resolution	15.0	
Extractor	5	-4	Ion Energy	2.0	
RF Lens	0.2		Ion Energy Ramp	0.0	
Source Block Temp.	150	149	Lens 6	4	7
Desolvation Temp.	250	253	Multiplier	650	

Pressures	Rdbk	MS2	Set	Rdbk
Analysar Vacuum	1.2e-4	LM Resolution	14.0	
		HM Resolution	14.0	
		Collision	10	41
		Ion Energy	2.0	
		Ion Energy Ramp	0.0	
		Lens 8	40	38
		Lens 9	0	40
		Multiplier	650	

000615

Scanning Method Report

Page 1

Method: C:\MSBLYNK\NELIA-RATS.PRO\ACQUS\10MIN-PT06-FOAM-IS-PT06A
Last Modified: Mon Oct 11 08:22:17 1999

Printed: Fri Oct 22 09:59:08 1999

Solvent Delay (mins) : 0.00

Function : 1 MM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	413.00	169.00	0.30	30	15

Function : 2 MM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	499.00	99.00	0.30	45	60

Function : 3 MM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	427.00	80.00	0.30	40	60

Function : 4 MM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	498.00	78.00	0.40	45	60

000616

Method Report

Page 1

Method File: c:\masslynx\amelia-rats.pro\acqudb\10min-10-95
 Last Modified: Friday, October 22, 1999 09:56:36
 Printed: Friday, October 22, 1999 09:59:25

HP1100 LC Pump Initial Conditions

Solvents
 A% 90.0
 B% 10.0
 C% 0.0
 D% 0.0
 Flow (ml/min) 0.300
 Stop Time (mins) 10.0
 Min Pressure (bar) 0
 Max Pressure (bar) 400
 Oven Temperature Left(°C) 30.0
 Oven Temperature Right(°C) 30.0

HP1100 LC Pump Gradient Timetable

The gradient Timetable contains 5 entries which are :

Time	A%	B%	C%	D%	Flow	Pressure
0.00	90.0	10.0	0.0	0.0	0.300	400
1.00	90.0	10.0	0.0	0.0	0.300	400
5.50	5.0	95.0	0.0	0.0	0.300	400
7.50	5.0	95.0	0.0	0.0	0.300	400
8.00	90.0	10.0	0.0	0.0	0.300	400

HP1100 LC Pump External Event Timetable

The Timetable contains 3 entries which are :

Time	Column Switch	Contact1	Contact2	Contact3	Contact4
Initial	Off	Off	Off	Off	Off
0.00	Off	On	On	Off	On
0.10	Off	Off	Off	Off	Off

HP1100 Autosampler Initial Conditions

Draw Speed 200.0
 Eject Speed (μl/min) 200
 Draw Position (mm) 0.00
 Stop Time (mins) 9.00
 Injection Volume(μl) 10.0
 Vial Number 13

000617

Sample List: C:\MASSLYNX\amelia-rats.PRO\SampleDB\102299a.SPL
Printed: Fri Oct 22 09:58:46 1999

Page Position: (1, 1)

	File Name	File Text	MS File
1	102299001	MeOH Blank	10MIN-PFOS-POAA-IS-PFOSA
2	102299002	MeOH Blank	10MIN-PFOS-POAA-IS-PFOSA
3	102299003	TOX145-RBS10199-H2O Blk-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
4	102299004	TOX145-RBS10199-Serum Blk-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
5	102299005	TOX145-RTS10199-Serum Blk-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
6	102299006	TOX145-RBS10199-5ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
7	102299007	TOX145-RBS10199-10ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
8	102299008	TOX145-RBS10199-25ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
9	102299009	TOX145-RBS10199-50ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
10	102299010	TOX145-RBS10199-100ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
11	102299011	TOX145-RBS10199-250ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
12	102299012	TOX145-RBS10199-500ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
13	102299013	TOX145-RBS10199-750ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
14	102299014	TOX145-RBS10199-1000ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
15	102299015	MeOH Blank	10MIN-PFOS-POAA-IS-PFOSA
16	102299016	TOX145-RTS-9R03245M, Day1, 5mg/kg, D50-ext10/19/99	10MIN-PFOS-POAA-IS-PFOSA
17	102299017	TOX145-RTS-9R03246M, Day1, 5mg/kg, D50-ext10/19/99	10MIN-PFOS-POAA-IS-PFOSA
18	102299018	TOX145-RTS-9R03247M, Day1, 5mg/kg, D50-ext10/19/99	10MIN-PFOS-POAA-IS-PFOSA
19	102299019	TOX145-RTS-9R03248M, Day1, 5mg/kg, D50-ext10/19/99	10MIN-PFOS-POAA-IS-PFOSA
20	102299020	TOX145-RTS-9R03249M, Day1, 5mg/kg, D50-ext10/19/99	10MIN-PFOS-POAA-IS-PFOSA
21	102299021	TOX145-RBS10199-250ppb-CCV-11-1, SEE	10MIN-PFOS-POAA-IS-PFOSA
22	102299022	MeOH Blank	10MIN-PFOS-POAA-IS-PFOSA
23	102299023	TOX145-RTS-9R03250M, Day4, 5mg/kg, D50-ext10/19/99	10MIN-PFOS-POAA-IS-PFOSA
24	102299024	TOX145-RTS-9R03251M, Day4, 5mg/kg, D50-ext10/19/99	10MIN-PFOS-POAA-IS-PFOSA
25	102299025	TOX145-RTS-9R03252M, Day4, 5mg/kg, D50-ext10/19/99	10MIN-PFOS-POAA-IS-PFOSA
26	102299026	TOX145-RTS-9R03254-1M, Day4, 5mg/kg, D50- 10/19/99	10MIN-PFOS-POAA-IS-PFOSA
27	102299027	TOX145-RTS-9R03254-2M, Day4, 5mg/kg, D50- 10/19/99	10MIN-PFOS-POAA-IS-PFOSA
28	102299028	TOX145-RBS10199-250ppb-CCV-11-2, SEE	10MIN-PFOS-POAA-IS-PFOSA
29	102299029	MeOH Blank	10MIN-PFOS-POAA-IS-PFOSA
30	102299030	TOX145-RBS10199-H2O Blk-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
31	102299031	TOX145-RBS10199-Serum Blk-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
32	102299032	TOX145-RTS10199-Serum Blk-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
33	102299033	TOX145-RBS10199-5ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
34	102299034	TOX145-RBS10199-10ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
35	102299035	TOX145-RBS10199-25ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
36	102299036	TOX145-RBS10199-50ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
37	102299037	TOX145-RBS10199-100ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
38	102299038	TOX145-RBS10199-250ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
39	102299039	TOX145-RBS10199-500ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
40	102299040	TOX145-RBS10199-750ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA
41	102299041	TOX145-RBS10199-1000ppb-12, SEE	10MIN-PFOS-POAA-IS-PFOSA

Masslynx - Sample List

Page 2

Sample List: C:\MASSLYNX\amelia-rats.PRO\SampleDB\102299a.SPL
Printed: Fri Oct 22 09:58:46 1999

Page Position: (2, 1)

	Inlet File	Bottle	Inject Volume	Sample Type	Conc A	Conc B	Conc C	Conc D	Conc E	Conc F
1	10min-10-95	91	10	---	---	---	---	---	---	---
2	10min-10-95	91	10	---	---	---	---	---	---	---
3	10min-10-95	1	10	---	---	---	---	---	---	---
4	10min-10-95	2	10	---	---	---	---	---	---	---
5	10min-10-95	3	10	---	---	---	---	---	---	---
6	10min-10-95	4	10	---	---	---	---	---	---	---
7	10min-10-95	5	10	---	---	---	---	---	---	---
8	10min-10-95	6	10	---	---	---	---	---	---	---
9	10min-10-95	7	10	---	---	---	---	---	---	---
10	10min-10-95	8	10	---	---	---	---	---	---	---
11	10min-10-95	9	10	---	---	---	---	---	---	---
12	10min-10-95	10	10	---	---	---	---	---	---	---
13	10min-10-95	11	10	---	---	---	---	---	---	---
14	10min-10-95	12	10	---	---	---	---	---	---	---
15	10min-10-95	92	10	---	---	---	---	---	---	---
16	10min-10-95	13	10	---	---	---	---	---	---	---
17	10min-10-95	14	10	---	---	---	---	---	---	---
18	10min-10-95	15	10	---	---	---	---	---	---	---
19	10min-10-95	16	10	---	---	---	---	---	---	---
20	10min-10-95	17	10	---	---	---	---	---	---	---
21	10min-10-95	33	10	---	---	---	---	---	---	---
22	10min-10-95	92	10	---	---	---	---	---	---	---
23	10min-10-95	18	10	---	---	---	---	---	---	---
24	10min-10-95	19	10	---	---	---	---	---	---	---
25	10min-10-95	20	10	---	---	---	---	---	---	---
26	10min-10-95	21	10	---	---	---	---	---	---	---
27	10min-10-95	22	10	---	---	---	---	---	---	---
28	10min-10-95	34	10	---	---	---	---	---	---	---
29	10min-10-95	93	10	---	---	---	---	---	---	---
30	10min-10-95	1	10	---	---	---	---	---	---	---
31	10min-10-95	2	10	---	---	---	---	---	---	---
32	10min-10-95	3	10	---	---	---	---	---	---	---
33	10min-10-95	4	10	---	---	---	---	---	---	---
34	10min-10-95	5	10	---	---	---	---	---	---	---
35	10min-10-95	6	10	---	---	---	---	---	---	---
36	10min-10-95	7	10	---	---	---	---	---	---	---
37	10min-10-95	8	10	---	---	---	---	---	---	---
38	10min-10-95	9	10	---	---	---	---	---	---	---
39	10min-10-95	10	10	---	---	---	---	---	---	---
40	10min-10-95	11	10	---	---	---	---	---	---	---
41	10min-10-95	12	10	---	---	---	---	---	---	---

000619

FACT: DATA REVIEW SUMMARY FORM

Date: 10-26-99
 Data Analyst: MMN
 FACT Study #: 10x145
 Matrix/Timepoints: Bot Ser., Day 1+4 Dilutions
 Sample List: 1025996
 Set-up Analysis Date/Analyst: 10-25-99 MMN
 Instrument: SUP 120199

Data Review Sign-Off

Analyst Review
 Name / Date: 10-26-99 MMN

Technical Review
 Name / Date: MEE 10/26/99 | kh 10/27/99

Quality Assurance Review
 Name / Date: _____

Data Entry: LAC 10/29/99

Comments / Justifications: _____

Analyte: PROD 4.93 at 2.1 mg/2799
 Acceptable Linear Range: 4.93 - 976 ppb
 LOQ: 4.93 ppb Extraction Blank: < 1/2 LOQ
 R²: 0.9971 Matrix Blank: < 1/2 LOQ
 Calibration Checks: OK
 MS/MSD: N/A - none were analyzed with this sample.
 Data Status: ① ② ③ Entered LAC 10/29/99

Analyte: PROD A
 Acceptable Linear Range: 9.77 - 9770 ppb
 LOQ: 9.77 ppb Extracted Blank: < 1/2 LOQ
 R²: 0.9926 Matrix Blank: < 1/2 LOQ
 Calibration Checks: OK
 MS/MSD: Data not used since dil.
 Data Status: ③ were not needed. LAC 10/29/99

Analyte: SUR
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: _____

- ① excluded 2nd run since it was divergent from the 1st MMN 10/26/99
- ② lowest ex point dropped, it was not 2x matrix blank. MMN 10/26/99
- ③ excluded lowest point to meet less than 30% deviation criteria MMN 10/26/99
- ④ did not set up method to screen for PROD since only dilutions of samples were analyzed MMN 10/26/99
- ② lowest point can be included since it is 2x RBS blank so LOQ is 4.93 ppb MMN 10/27/99

LAC 10/29/99

mmh
10-26-99
P1/3
Page 1

Quantify Compound Summary Report
Tox 145: Rat Sera, Day 144 dilutions, Ext 10/19/99

Sample List: C:\MMS\LYNK\scup020199.pro\SampleDB\102599b
Last modified: Mon Oct 25 16:32:42 1999
Method: C:\MMS\LYNK\scup020199.pro\MethDB\102599b
Last modified: Tue Oct 26 09:11:31 1999
Job Code:

Printed: Tue Oct 26 10:51:19 1999

Compound 1: PFOS

#	Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1	102599039	MeOH Blank			7.313	40	bb	2.73	
2	102599040	MeOH Blank			7.416	50	bb	3.09	
3	102599041	TOXI45-RBS10199-H2O Blk-12, SEE			7.424	54	bb	3.23	
4	102599042	TOXI45-RBS10199-Serum Blk-12, SEE			7.402	43	MM	2.84	
5	102599043	TOXI45-RBS10199-Serum Blk-12, SEE			7.417	86	bb	4.42	
6	102599044	TOXI45-RBS10199-5ppb-12, SEE	Standard	4.93	7.416	134	MM	6.17	25
7	102599045	TOXI45-RBS10199-10ppb-12, SEE	Standard	9.76	7.442	258	MM	10.76	10
8	102599046	TOXI45-RBS10199-25ppb-12, SEE	Standard	24.80	7.390	578	bb	22.57	-9
9	102599047	TOXI45-RBS10199-50ppb-12, SEE	Standard	49.30	7.398	943	bb	36.04	-27
10	102599048	TOXI45-RBS10199-100ppb-12, SEE	Standard	97.60	7.420	2493	bb	100.56	3
11	102599049	TOXI45-RBS10199-250ppb-12, SEE	Standard	248.00	7.420	6270	bb	232.47	-6
12	102599050	TOXI45-RBS10199-500ppb-12, SEE	Standard	493.00	7.418	13890	bb	513.46	4
13	102599051	TOXI45-RBS10199-750ppb-12, SEE	Standard	735.00	7.394	19051	bb	703.80	-4
14	102599052	TOXI45-RBS10199-1000ppb-12, SEE	Standard	976.00	7.423	27424	bb	1012.57	4
15	102599053	MeOH Blank			7.376	202	bb	8.70	
16	102599054	TOXI45-RTS-9R03245M, Day1, 5mg/kg, D50-ext10/19/99			7.330	3728	bb	138.73	
17	102599055	TOXI45-RTS-9R03246M, Day1, 5mg/kg, D50-ext10/19/99			7.441	5832	bb	216.30	
18	102599056	TOXI45-RTS-9R03247M, Day1, 5mg/kg, D50-ext10/19/99			7.378	5036	bb	186.95	
19	102599057	TOXI45-RTS-9R03248M, Day1, 5mg/kg, D50-ext10/19/99			7.360	3572	bb	132.99	
20	102599058	TOXI45-RTS-9R03249M, Day1, 5mg/kg, D50-ext10/19/99			7.313	3945	bb	146.71	
21	102599059	TOXI45-RBS10199-250ppb-CCV-11-1, SEE	QC	248.00	7.350	6204	bb	230.03	-7
22	102599060	TOXI45-RBS10199-250ppb-12, SEE	QC	248.00	7.337	5979	bb	221.72	-11
23	102599061	MeOH Blank			7.347	47	MM	2.99	
24	102599062	TOXI45-RTS-9R03250M, Day4, 5mg/kg, D50-ext10/19/99			7.352	4185	bd	155.58	
25	102599063	TOXI45-RTS-9R03251M, Day4, 5mg/kg, D50-ext10/19/99			7.285	4024	bb	149.65	
26	102599064	TOXI45-RTS-9R03252M, Day4, 5mg/kg, D50-ext10/19/99			7.219	6096	bd	226.05	
27	102599065	TOXI45-RTS-9R03254-1M, Day4, 5mg/kg, D50- 10/19/99			7.238	4634	bd	172.14	
28	102599066	TOXI45-RTS-9R03254-2M, Day4, 5mg/kg, D50- 10/19/99			7.219	4255	bb	158.17	
29	102599067	TOXI45-RBS10199-250ppb-CCV-11-2, SEE	QC	248.00	7.199	4647	MM	172.63	-30
30	102599068	TOXI45-RBS10199-250ppb-12, SEE	QC	248.00	7.221	6747	bb	250.05	1
31	102599069	TOXI45-RBS10199-250ppb-CCV-11-3, SEE	QC	248.00	7.247	7505	bb	278.00	12
32	102599070	TOXI45-RBS10199-250ppb-CCV-11-4, SEE	QC	248.00	7.224	8184	bb	303.05	22
33	102599071	MeOH Blank			7.267	66	bb	3.67	
34	102599072	TOXI45-RBS10199-H2O Blk-12, SEE			7.245	76	bb	4.04	
35	102599073	TOXI45-RBS10199-Serum Blk-12, SEE			7.257	69	bb	3.80	
36	102599074	TOXI45-RBS10199-Serum Blk-12, SEE			7.234	181	MM	7.94	
37	102599075	TOXI45-RBS10199-5ppb-12, SEE	Standard	4.93	7.268	233	bbX	9.84	100
38	102599076	TOXI45-RBS10199-10ppb-12, SEE	Standard	9.76	7.259	369	bbX	14.86	52
39	102599077	TOXI45-RBS10199-25ppb-12, SEE	Standard	24.80	7.266	985	bdX	37.57	51
40	102599078	TOXI45-RBS10199-50ppb-12, SEE	Standard	49.30	7.259	1480	bbX	55.83	13
41	102599079	TOXI45-RBS10199-100ppb-12, SEE	Standard	97.60	7.244	4608	MMX	171.16	75
42	102599080	TOXI45-RBS10199-250ppb-12, SEE	Standard	248.00	7.247	10314	MMX	381.61	54
43	102599081	TOXI45-RBS10199-500ppb-12, SEE	Standard	493.00	7.227	21815	MMX	805.73	63
44	102599082	TOXI45-RBS10199-750ppb-12, SEE	Standard	735.00	7.246	31783	MMX	1173.29	60
45	102599083	TOXI45-RBS10199-1000ppb-12, SEE	Standard	976.00	7.242	42982	MMX	1586.30	63

} Entered
lac
10/29/99

Quantify Compound Summary Report
Tox 145: Rat Sera, Day 144 dilutions, Ext 10/19/99

Page 2

Sample List: C:\MSLSL\YEX\scup020199.pro\SampleDB\102599b
Last modified: Mon Oct 25 16:32:42 1999
Method: C:\MSLSL\YEX\scup020199.pro\MethDB\102599b
Last modified: Tue Oct 26 09:11:31 1999
Job Code:

Printed: Tue Oct 26 10:51:19 1999

Compound 2: PFOSA

# Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1 102599039	MeOH Blank			8.165	101	bb	3.13	
2 102599040	MeOH Blank			8.175	153	bb	3.28	
3 102599041	TOX145-RBS10199-H2O Blk-12, SEE			8.230	178	bb	3.35	
4 102599042	TOX145-RBS10199-Serum Blk-12, SEE			8.252	194	MM	3.40	
5 102599043	TOX145-RBS10199-Serum Blk-12, SEE			8.240	245	db	3.55	
6 102599044	TOX145-RBS10199-5ppb-12, SEE	Standard	4.93	8.246	1430	MMX	6.97	41
7 102599045	TOX145-RBS10199-10ppb-12, SEE	Standard	9.77	8.248	2534	MM	10.17	4
8 102599046	TOX145-RBS10199-25ppb-12, SEE	Standard	24.80	8.195	6861	MM	22.68	-9
9 102599047	TOX145-RBS10199-50ppb-12, SEE	Standard	49.30	8.229	11166	MM	35.12	-29
10 102599048	TOX145-RBS10199-100ppb-12, SEE	Standard	97.70	8.221	38368	MM	113.77	16
11 102599049	TOX145-RBS10199-250ppb-12, SEE	Standard	248.00	8.201	83269	MM	243.59	-2
12 102599050	TOX145-RBS10199-500ppb-12, SEE	Standard	493.00	8.202	161220	MM	468.97	-5
13 102599051	TOX145-RBS10199-750ppb-12, SEE	Standard	736.00	8.178	236588	MM	686.89	-7
14 102599052	TOX145-RBS10199-1000ppb-12, SEE	Standard	977.00	8.185	313964	MM	910.61	-7
15 102599053	MeOH Blank			8.138	1523	MM	7.24	
16 102599054	TOX145-RTS-9R03245M, Day1, 5mg/kg, D50-ext10/19/99			8.135	2311	MM	9.52	
17 102599055	TOX145-RTS-9R03246M, Day1, 5mg/kg, D50-ext10/19/99			8.247	3545	MM	13.09	
18 102599056	TOX145-RTS-9R03247M, Day1, 5mg/kg, D50-ext10/19/99			8.163	1989	MM	8.59	
19 102599057	TOX145-RTS-9R03248M, Day1, 5mg/kg, D50-ext10/19/99			8.143	2622	MM	10.42	
20 102599058	TOX145-RTS-9R03249M, Day1, 5mg/kg, D50-ext10/19/99			8.097	2129	MM	8.99	
21 102599059	TOX145-RBS10199-250ppb-CCV-11-1, SEE	QC	248.00	8.157	86047	MM	251.63	1
22 102599060	TOX145-RBS10199-250ppb-12, SEE	QC	248.00	8.143	84647	MM	247.58	-0
23 102599061	MeOH Blank			8.133	442	MM	4.12	
24 102599062	TOX145-RTS-9R03250M, Day4, 5mg/kg, D50-ext10/19/99			8.159	677	db	4.79	
25 102599063	TOX145-RTS-9R03251M, Day4, 5mg/kg, D50-ext10/19/99			8.092	633	MM	4.67	
26 102599064	TOX145-RTS-9R03252M, Day4, 5mg/kg, D50-ext10/19/99			7.804	828	db	5.23	
27 102599065	TOX145-RTS-9R03254-1M, Day4, 5mg/kg, D50- 10/19/99			7.822	599	MM	4.57	
28 102599066	TOX145-RTS-9R03254-2M, Day4, 5mg/kg, D50- 10/19/99			7.803	607	dd	4.59	
29 102599067	TOX145-RBS10199-250ppb-CCV-11-2, SEE	QC	248.00	7.759	81385	MM	238.15	-4
30 102599068	TOX145-RBS10199-250ppb-12, SEE	QC	248.00	7.782	91754	MM	268.13	8
31 102599069	TOX145-RBS10199-250ppb-CCV-11-3, SEE	QC	248.00	7.786	89388	MM	261.29	5
32 102599070	TOX145-RBS10199-250ppb-CCV-11-4, SEE	QC	248.00	7.788	86953	MM	254.25	3
33 102599071	MeOH Blank			7.806	344	bb	3.83	
34 102599072	TOX145-RBS10199-H2O Blk-12, SEE			7.808	302	MM	3.71	
35 102599073	TOX145-RBS10199-Serum Blk-12, SEE			7.820	233	MM	3.51	
36 102599074	TOX145-RTS10199-Serum Blk-12, SEE			7.795	298	MM	3.70	
37 102599075	TOX145-RBS10199-5ppb-12, SEE	Standard	4.93	7.808	1467	MMX	7.66	55
38 102599076	TOX145-RBS10199-10ppb-12, SEE	Standard	9.77	7.776	2861	MM	11.11	14
39 102599077	TOX145-RBS10199-25ppb-12, SEE	Standard	24.80	7.828	7460	MM	24.41	-2
40 102599078	TOX145-RBS10199-50ppb-12, SEE	Standard	49.30	7.799	11958	MM	37.41	-24
41 102599079	TOX145-RBS10199-100ppb-12, SEE	Standard	97.70	7.783	42260	MM	125.02	28
42 102599080	TOX145-RBS10199-250ppb-12, SEE	Standard	248.00	7.787	89431	MM	261.41	5
43 102599081	TOX145-RBS10199-500ppb-12, SEE	Standard	493.00	7.789	179977	MM	521.21	6
44 102599082	TOX145-RBS10199-750ppb-12, SEE	Standard	736.00	7.808	262487	MM	761.77	4
45 102599083	TOX145-RBS10199-1000ppb-12, SEE	Standard	977.00	7.801	356987	MM	1035.00	6

Dilutions were not needed for PFOSA, undil. values reported from 10/19/99 analyses. These data not used.

UC 10/29/99

Quantify Compound Summary Report
Tox 145: Rat Sera, Day 144 dilutions, Ext 10/19/99

Page 3

Sample List: C:\MSASLYNK\soup020199.pro\SampleDB\102599b
Last modified: Mon Oct 25 16:32:42 1999
Method: C:\MSASLYNK\soup020199.pro\MethDB\102599b
Last modified: Tue Oct 26 09:11:31 1999
Job Code:

Printed: Tue Oct 26 10:51:19 1999

Compound 3: Surr

# Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1 102599039	MeOH Blank			7.101	14	bb	0.00	-100
2 102599040	MeOH Blank							
3 102599041	TOXI45-RBS10199-M2O Blk-12, SEE			7.213	3491	bb	1.11	11
4 102599042	TOXI45-RBS10199-Serum Blk-12, SEE			7.213	2874	bb	0.91	-9
5 102599043	TOXI45-RBS10199-Serum Blk-12, SEE			7.230	2230	bb	0.71	-29
6 102599044	TOXI45-RBS10199-5ppb-12, SEE	Standard	1.00	7.227	3080	bb	0.98	-2
7 102599045	TOXI45-RBS10199-10ppb-12, SEE	Standard	1.00	7.255	2937	bb	0.93	-7
8 102599046	TOXI45-RBS10199-25ppb-12, SEE	Standard	1.00	7.201	2890	bb	0.92	-8
9 102599047	TOXI45-RBS10199-50ppb-12, SEE	Standard	1.00	7.211	2576	bb	0.82	-18
10 102599048	TOXI45-RBS10199-100ppb-12, SEE	Standard	1.00	7.230	2965	bb	0.94	-6
11 102599049	TOXI45-RBS10199-250ppb-12, SEE	Standard	1.00	7.253	3053	MM	0.97	-3
12 102599050	TOXI45-RBS10199-500ppb-12, SEE	Standard	1.00	7.229	3171	bb	1.01	1
13 102599051	TOXI45-RBS10199-750ppb-12, SEE	Standard	1.00	7.205	3038	bb	0.96	-4
14 102599052	TOXI45-RBS10199-1000ppb-12, SEE	Standard	1.00	7.234	3041	MM	0.96	-4
15 102599053	MeOH Blank			7.231	9	bb	0.00	-100
16 102599054	TOXI45-RTS-9R0324SM, Day1, 5mg/kg, D50-ext10/19/99			7.163	69	bb	0.02	-98
17 102599055	TOXI45-RTS-9R0324SM, Day1, 5mg/kg, D50-ext10/19/99			7.251	66	MM	0.02	-98
18 102599056	TOXI45-RTS-9R0324SM, Day1, 5mg/kg, D50-ext10/19/99			7.211	71	bb	0.02	-98
19 102599057	TOXI45-RTS-9R0324SM, Day1, 5mg/kg, D50-ext10/19/99			7.171	42	bb	0.01	-99
20 102599058	TOXI45-RTS-9R0324SM, Day1, 5mg/kg, D50-ext10/19/99			7.146	45	bb	0.02	-98
21 102599059	TOXI45-RBS10199-250ppb-CCV-11-1, SEE	QC	1.00	7.183	2885	bb	0.91	-9
22 102599060	TOXI45-RBS10199-250ppb-12, SEE	QC	1.00	7.148	2800	bb	0.89	-11
23 102599061	MeOH Blank			7.180	3	bb	0.00	-100
24 102599062	TOXI45-RTS-9R03250M, Day4, 5mg/kg, D50-ext10/19/99			7.163	47	bb	0.02	-98
25 102599063	TOXI45-RTS-9R03251M, Day4, 5mg/kg, D50-ext10/19/99			7.119	45	bb	0.01	-99
26 102599064	TOXI45-RTS-9R03252M, Day4, 5mg/kg, D50-ext10/19/99			7.074	60	bb	0.02	-98
27 102599065	TOXI45-RTS-9R03254-1M, Day4, 5mg/kg, D50- 10/19/99			7.093	60	MM	0.02	-98
28 102599066	TOXI45-RTS-9R03254-2M, Day4, 5mg/kg, D50- 10/19/99			7.074	55	MM	0.02	-98
29 102599067	TOXI45-RBS10199-250ppb-CCV-11-2, SEE	QC	1.00	7.056	1907	MM	0.60	-40
30 102599068	TOXI45-RBS10199-250ppb-12, SEE	QC	1.00	7.076	2828	bd	0.90	-10
31 102599069	TOXI45-RBS10199-250ppb-CCV-11-3, SEE	QC	1.00	7.102	2949	bb	0.93	-7
32 102599070	TOXI45-RBS10199-250ppb-CCV-11-4, SEE	QC	1.00	7.080	2911	bb	0.92	-8
33 102599071	MeOH Blank			7.100	8	MM	0.00	-100
34 102599072	TOXI45-RBS10199-M2O Blk-12, SEE			7.102	3618	bb	1.15	15
35 102599073	TOXI45-RBS10199-Serum Blk-12, SEE			7.114	3157	bd	1.00	0
36 102599074	TOXI45-RBS10199-Serum Blk-12, SEE			7.088	2944	bd	0.93	-7
37 102599075	TOXI45-RBS10199-5ppb-12, SEE	Standard	1.00	7.101	3177	bd	1.01	1
38 102599076	TOXI45-RBS10199-10ppb-12, SEE	Standard	1.00	7.092	3076	MM	0.98	-2
39 102599077	TOXI45-RBS10199-25ppb-12, SEE	Standard	1.00	7.121	2980	MM	0.94	-6
40 102599078	TOXI45-RBS10199-50ppb-12, SEE	Standard	1.00	7.092	2748	bb	0.87	-13
41 102599079	TOXI45-RBS10199-100ppb-12, SEE	Standard	1.00	7.099	3441	bd	1.09	9
42 102599080	TOXI45-RBS10199-250ppb-12, SEE	Standard	1.00	7.102	3426	bb	1.18	18
43 102599081	TOXI45-RBS10199-500ppb-12, SEE	Standard	1.00	7.082	3712	bb	1.20	20
44 102599082	TOXI45-RBS10199-750ppb-12, SEE	Standard	1.00	7.101	3771	bb	1.20	20
45 102599083	TOXI45-RBS10199-1000ppb-12, SEE	Standard	1.00	7.098	3695	MM	1.17	17

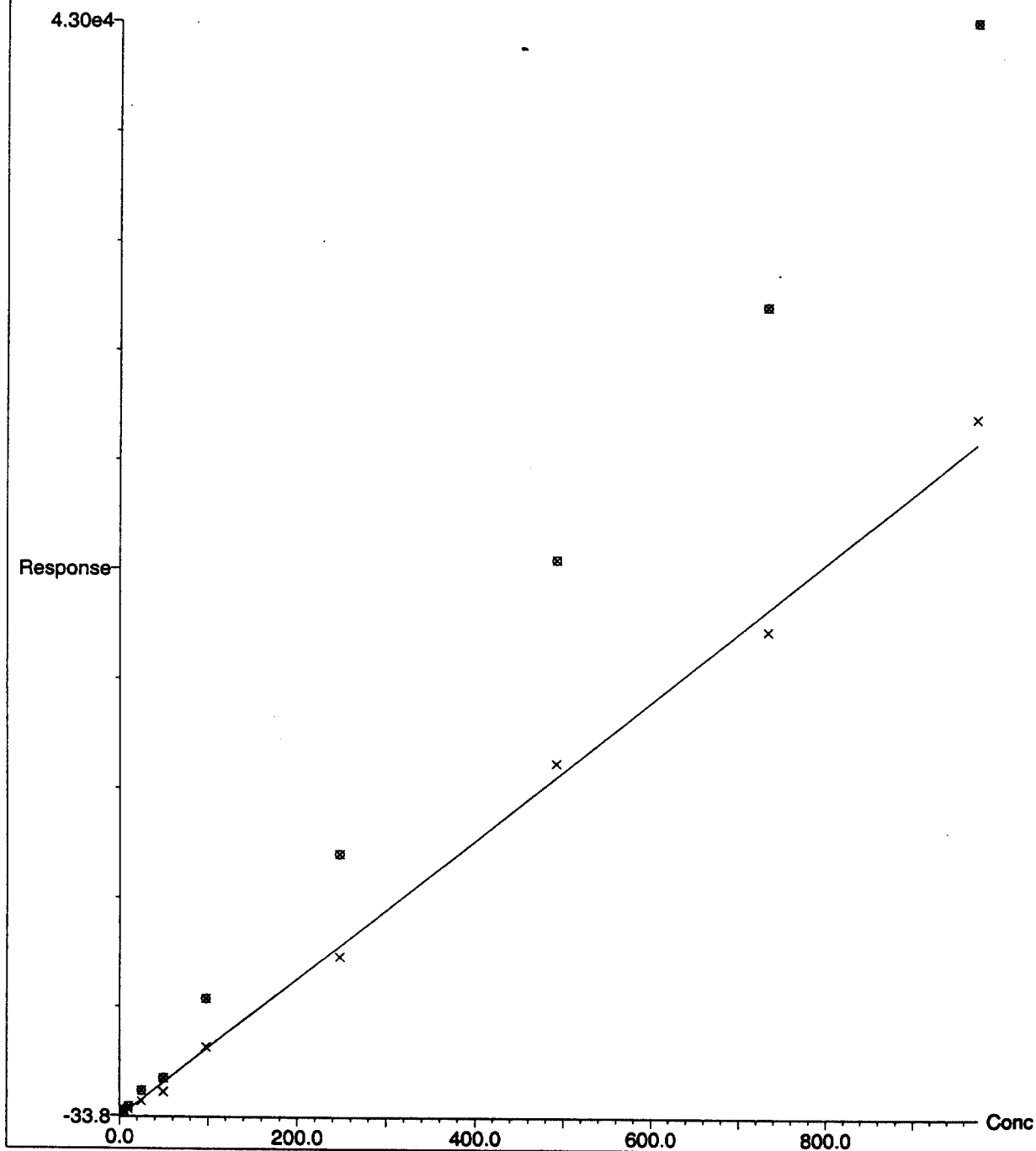
Not a target analyte - data not used.

LAC 10/29/99

MMN
10-26-99
P1/B
Page 1

Quantify Calibration Report
Tox 145: Rat Sera, Day 144 dilutions, Ret 10/19/99

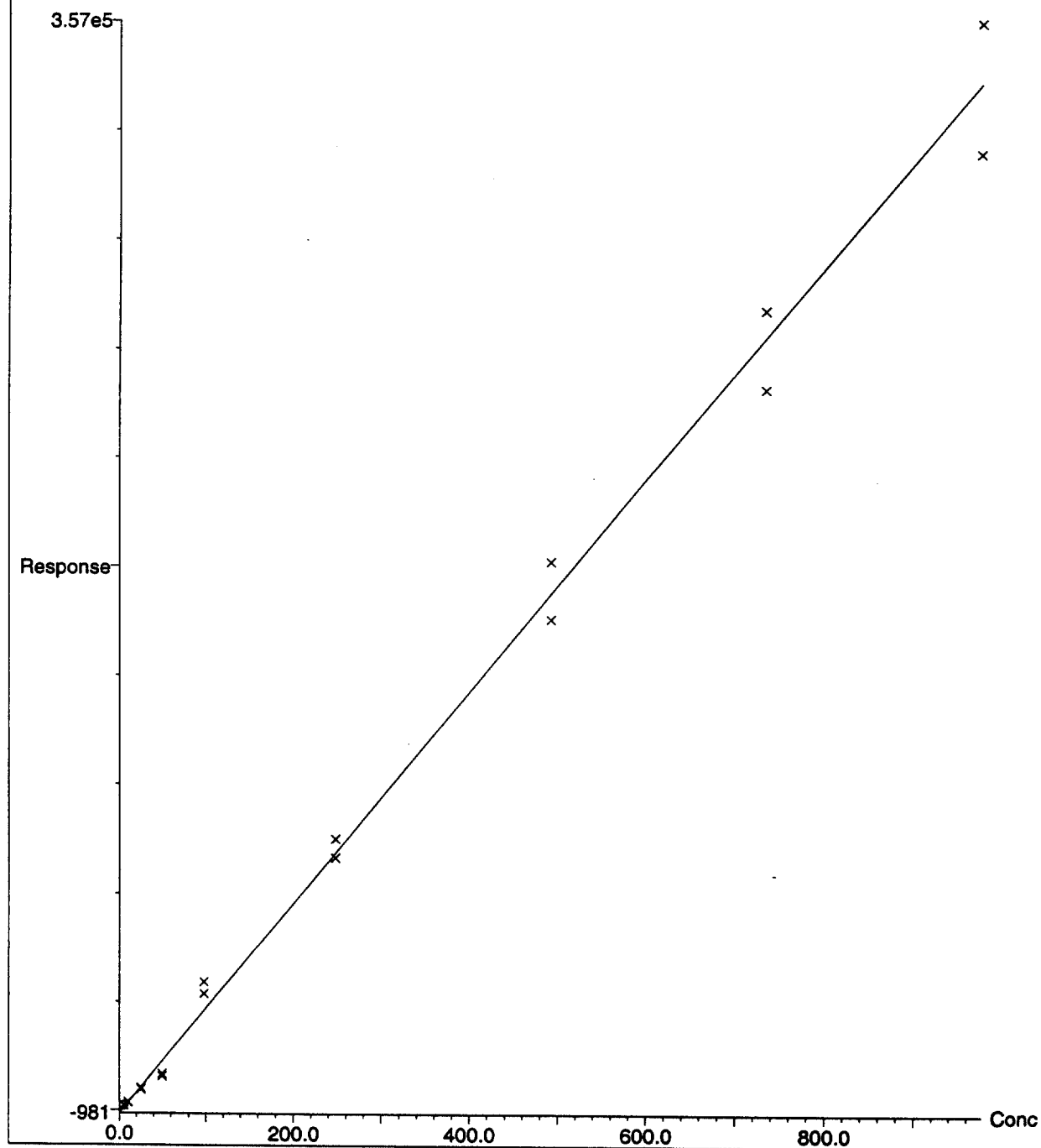
Compound 1 name: PFOS
Coefficient of Determination: 0.997063
Calibration curve: $27.1171 * x + -33.7747$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



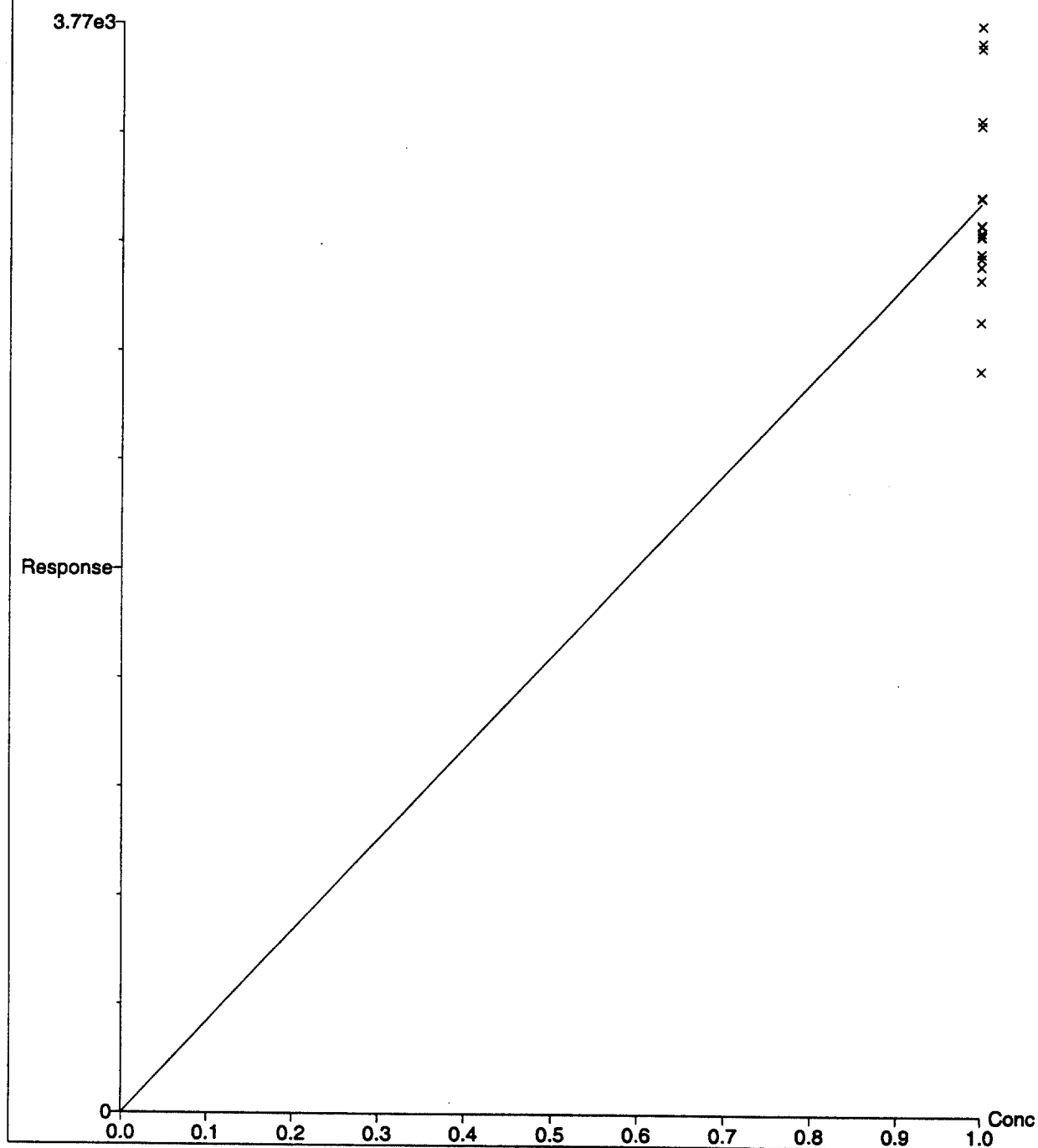
Scup 020199: Quantified and printed by MMN on 10/26/99

000624

Compound 2 name: PFOSA
Coefficient of Determination: 0.992571
Calibration curve: $345.863 * x + -981.494$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound 3 name: Surr
Response Factor: 3154.38
Response type: External Std, Area
Curve type: RF



mm
10/26/99
P1/3

Method Report

Page 1

Method file: C:\MASSLYMX\scup020199.pro\MethDB\102599b.mdb
Last modified: Tue Oct 26 09:11:31 1999

Compound: 1: PFOS

Printed: Tue Oct 26 10:48:14 1999

Compound Name: PFOS
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. A
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.490, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.312

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 76

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000627

Method Report

Page 2

Method file: C:\MASSLYNK\soup020199.pro\MethDB\102599b.mdb
Last modified: Tue Oct 26 09:11:31 1999

Compound: 2: PFOSA

Printed: Tue Oct 26 10:48:14 1999

Compound Name: PFOSA
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. B
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.960, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.400

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 76

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000628

Method Report

Page 3

Method file: C:\MASSLYNK\scup020199.pro\MethDB\102599b.mdb
Last modified: Tue Oct 26 09:11:31 1999

Compound: 3: Surr

Printed: Tue Oct 26 10:48:14 1999

Compound Name: Surr
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Fixed
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.245, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.301

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 65

Smoothing: Enabled
Window size (scans): 3
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000629

mmh
10-25-99

SOUP 020199: FACT Equipment
Log 99092 PAGE 1/1

Quattro II, #6414E

DATE: 10-25-99

ANALYST: mmmh

STUDY NUMBER: TOX145 MATRIX: Rat sera DESCRIPTION: Dilutions

FACT Method: ETS-8-5.1

Injection volume: 10 uL Cycle time: 10 min. Electrode: Z-Spray

Mobile Phase: (A) 2.0 mM NH₄CH₃CO₂ (B) MeOH

TN-A- or stdrd number (A) 99085-11-3 (B) TN-A-2997

Prepared (date/by): (A) 10/21/99 mmh (B) 10/16/99 sah

Expires: (A) 04/00 (B) 2010

Store at room temp? ☒ Y or N Other: _____

Guard column: None

Part number: na Lot number: na

Analytical column: Keystone Betasil

Size: 2 x 50 mm Type: C18 Sphere size: 5 um

Part number: 055-701-2 Lot number: P9E11 Serial number: 0591804Z

Analytical parameters:

Drying gas (L/hr): 350 ESI nebulizing gas (L/hr) 13 Sheath gas (L/hr) 0

LC Flow rate: 300 uL/min. Back pressure: 65 bar

HPLC solvent gradient: 10min-10-95

Time (min.)	%B
1) <u>0.00</u>	<u>10.0</u>
2) <u>1.00</u>	<u>10.0</u>
3) <u>5.50</u>	<u>95.0</u>
4) <u>7.50</u>	<u>95.0</u>
5) <u>8.00</u>	<u>10.0</u>

000630

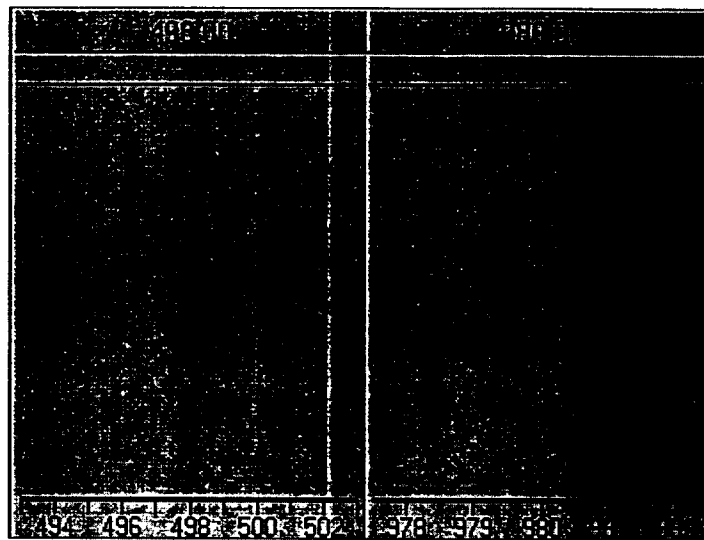
1770V
10-25-99

Tuning Method Report

Page 1

Method: C:\MASSLYNX\SOUP-RATS.PRO\ACQUDB\PFOS3

Printed: Mon Oct 25 16:37:32 1999



MS

MS

SOURCE (ESP-)	Set	Rdbk	MS1	Set	Rdbk
Capillary	1.60	1.60	LM Resolution	14.0	
Cone	60	-60	HM Resolution	14.0	
Extractor	5	-6	Ion Energy	0.9	
RF Lens	0.2		Ion Energy Ramp	0.0	
Source Block Temp.	150	151	Lens 6	4	3
Desolvation Temp.	250	250	Multiplier	650	

Pressures	Rdbk	MS2	Set	Rdbk
Analysers Vacuum	5.9e-5	LM Resolution	14.0	
		HM Resolution	14.0	
Gas Cell	4.1e-3	Collision	60	42
		Ion Energy	2.0	
		Ion Energy Ramp	0.0	
		Lens 8	40	38
		Lens 9	0	44
		Multiplier	650	

000631

MRN
10-25-99

Scanning Method Report

Page 1

Method: C:\MASSL\H\SOUP-RATE.PRO\ACQ\DS\10MIN-PP08-POAA-PP08A-IS
Last Modified: Fri Oct 15 11:35:03 1999
Printed: Mon Oct 25 16:28:24 1999

Solvent Delay (mins) : 0.00

Function : 1 MEM of 1 Mass Pair (MSP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	413.00	169.00	0.30	30	15

Function : 2 MEM of 1 Mass Pair (MSP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	499.00	99.00	0.30	45	60

Function : 3 MEM of 1 Mass Pair (MSP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	498.00	78.00	0.30	45	60

Function : 4 MEM of 1 Mass Pair (MSP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	427.00	80.00	0.30	40	60

000632

Method Report

Page 1

Method File:
Last Modified:c:\masslynx\soup-rats.pro\acqddb\10min-10-95
Monday, October 25, 1999 16:29:58

Printed:

Monday, October 25, 1999 16:34:46

HP1100 LC Pump Initial Conditions**Solvents**

A%	90.0
B%	10.0
C%	0.0
D%	0.0

Flow (ml/min)	0.300
Stop Time (mins)	10.0
Min Pressure (bar)	0
Max Pressure (bar)	400
Oven Temperature Left(°C)	30.0
Oven Temperature Right(°C)	30.0

HP1100 LC Pump Gradient Timetable

The gradient Timetable contains 5 entries which are :

Time	A%	B%	C%	D%	Flow	Pressure
0.00	90.0	10.0	0.0	0.0	0.300	400
1.00	90.0	10.0	0.0	0.0	0.300	400
5.50	5.0	95.0	0.0	0.0	0.300	400
7.50	5.0	95.0	0.0	0.0	0.300	400
8.00	90.0	10.0	0.0	0.0	0.300	400

HP1100 LC Pump External Event Timetable

The Timetable contains 3 entries which are :

Time	Column Switch	Contact1	Contact2	Contact3	Contact4
Initial	Off	Off	Off	Off	Off
0.00	Off	On	On	Off	On
0.10	Off	Off	Off	Off	Off

HP1100 Autosampler Initial Conditions

Draw Speed	200.0
Eject Speed (µl/min)	200
Draw Position (mm)	0.00
Stop Time (mins)	9.00
Injection Volume(µl)	10.0
Vial Number	9

000633

MPX
10-25-99

Masslynx - Sample List

Page 1

Sample List: C:\MASSLYNX\ soup-rats.pro\SampleDB\102599b.SPL
Printed: Mon Oct 25 16:32:57 1999

Page Position: (1, 1)

	File Name	File Text	MS File
1	102599039	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
2	102599040	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
3	102599041	TOX145-RBS10199-H2O Blk-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
4	102599042	TOX145-RBS10199-Serum Blk-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
5	102599043	TOX145-RTS10199-Serum Blk-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
6	102599044	TOX145-RBS10199-5ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
7	102599045	TOX145-RBS10199-10ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
8	102599046	TOX145-RBS10199-25ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
9	102599047	TOX145-RBS10199-50ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
10	102599048	TOX145-RBS10199-100ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
11	102599049	TOX145-RBS10199-250ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
12	102599050	TOX145-RBS10199-500ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
13	102599051	TOX145-RBS10199-750ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
14	102599052	TOX145-RBS10199-1000ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
15	102599053	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
16	102599054	TOX145-RTS-9R03245M, Day1, 5mg/kg, D50-ext10/19/99	10MIN-PFOS-POAA-PFOSA-IS
17	102599055	TOX145-RTS-9R03246M, Day1, 5mg/kg, D50-ext10/19/99	10MIN-PFOS-POAA-PFOSA-IS
18	102599056	TOX145-RTS-9R03247M, Day1, 5mg/kg, D50-ext10/19/99	10MIN-PFOS-POAA-PFOSA-IS
19	102599057	TOX145-RTS-9R03248M, Day1, 5mg/kg, D50-ext10/19/99	10MIN-PFOS-POAA-PFOSA-IS
20	102599058	TOX145-RTS-9R03249M, Day1, 5mg/kg, D50-ext10/19/99	10MIN-PFOS-POAA-PFOSA-IS
21	102599059	TOX145-RBS10199-250ppb-CCV-11-1, SEE	10MIN-PFOS-POAA-PFOSA-IS
22	102599060	TOX145-RBS10199-250ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
23	102599061	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
24	102599062	TOX145-RTS-9R03250M, Day4, 5mg/kg, D50-ext10/19/99	10MIN-PFOS-POAA-PFOSA-IS
25	102599063	TOX145-RTS-9R03251M, Day4, 5mg/kg, D50-ext10/19/99	10MIN-PFOS-POAA-PFOSA-IS
26	102599064	TOX145-RTS-9R03252M, Day4, 5mg/kg, D50-ext10/19/99	10MIN-PFOS-POAA-PFOSA-IS
27	102599065	TOX145-RTS-9R03254-1M, Day4, 5mg/kg, D50- 10/19/99	10MIN-PFOS-POAA-PFOSA-IS
28	102599066	TOX145-RTS-9R03254-2M, Day4, 5mg/kg, D50- 10/19/99	10MIN-PFOS-POAA-PFOSA-IS
29	102599067	TOX145-RBS10199-250ppb-CCV-11-2, SEE	10MIN-PFOS-POAA-PFOSA-IS
30	102599068	TOX145-RBS10199-250ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
31	102599069	TOX145-RBS10199-250ppb-CCV-11-3, SEE	10MIN-PFOS-POAA-PFOSA-IS
32	102599070	TOX145-RBS10199-250ppb-CCV-11-4, SEE	10MIN-PFOS-POAA-PFOSA-IS
33	102599071	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
34	102599072	TOX145-RBS10199-H2O Blk-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
35	102599073	TOX145-RBS10199-Serum Blk-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
36	102599074	TOX145-RTS10199-Serum Blk-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
37	102599075	TOX145-RBS10199-5ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
38	102599076	TOX145-RBS10199-10ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
39	102599077	TOX145-RBS10199-25ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
40	102599078	TOX145-RBS10199-50ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
41	102599079	TOX145-RBS10199-100ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
42	102599080	TOX145-RBS10199-250ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
43	102599081	TOX145-RBS10199-500ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
44	102599082	TOX145-RBS10199-750ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS
45	102599083	TOX145-RBS10199-1000ppb-12, SEE	10MIN-PFOS-POAA-PFOSA-IS

000634

11/25/99

Masslynx - Sample List

Page 2

Sample List: C:\MASSLYNX\soup-rats.pro\SampleDB\102599b.SPL
Printed: Mon Oct 25 16:32:57 1999

Page Position: (2, 1)

	Inlet File	Bottle	Inject Volume	Sample Type	Conc A	Conc B	Conc C
1	10min-10-95	91	10	--	--	--	--
2	10min-10-95	91	10	--	--	--	--
3	10min-10-95	21	10	--	--	--	--
4	10min-10-95	22	10	--	--	--	--
5	10min-10-95	23	10	--	--	--	--
6	10min-10-95	24	10	Standard	4.93	4.93	--
7	10min-10-95	25	10	Standard	9.76	9.77	--
8	10min-10-95	26	10	Standard	24.8	24.8	--
9	10min-10-95	27	10	Standard	49.3	49.3	--
10	10min-10-95	28	10	Standard	97.6	97.7	--
11	10min-10-95	29	10	Standard	248	248	--
12	10min-10-95	30	10	Standard	493	493	--
13	10min-10-95	31	10	Standard	735	736	--
14	10min-10-95	32	10	Standard	976	977	--
15	10min-10-95	92	10	--	--	--	--
16	10min-10-95	33	10	--	--	--	--
17	10min-10-95	34	10	--	--	--	--
18	10min-10-95	35	10	--	--	--	--
19	10min-10-95	36	10	--	--	--	--
20	10min-10-95	37	10	--	--	--	--
21	10min-10-95	43	10	QC	248	248	--
22	10min-10-95	29	10	QC	248	248	--
23	10min-10-95	92	10	--	--	--	--
24	10min-10-95	38	10	--	--	--	--
25	10min-10-95	39	10	--	--	--	--
26	10min-10-95	40	10	--	--	--	--
27	10min-10-95	41	10	--	--	--	--
28	10min-10-95	42	10	--	--	--	--
29	10min-10-95	44	10	QC	248	248	--
30	10min-10-95	29	10	QC	248	248	--
31	10min-10-95	45	10	QC	248	248	--
32	10min-10-95	46	10	QC	248	248	--
33	10min-10-95	93	10	--	--	--	--
34	10min-10-95	21	10	--	--	--	--
35	10min-10-95	22	10	--	--	--	--
36	10min-10-95	23	10	--	--	--	--
37	10min-10-95	24	10	Standard	4.93	4.93	--
38	10min-10-95	25	10	Standard	9.76	9.77	--
39	10min-10-95	26	10	Standard	24.8	24.8	--
40	10min-10-95	27	10	Standard	49.3	49.3	--
41	10min-10-95	28	10	Standard	97.6	97.7	--
42	10min-10-95	29	10	Standard	248	248	--
43	10min-10-95	30	10	Standard	493	493	--
44	10min-10-95	31	10	Standard	735	736	--
45	10min-10-95	32	10	Standard	976	977	--

000635

FACT: DATA REVIEW SUMMARY FORM

Date: 10-28-99
 Data Analyst: mm
 FACT Study #: TOX145
 Matrix/Timepoints: Rot Liver, day 1 & 4
 Sample List: 102799.6
 Set-up Analysis Date/Analyst: 10-27-99 mm
 Instrument: DAVEY 070799

Data Review Sign-Off

Analyst Review Name / Date: mm 10-28-99

Technical Review Name / Date: MEC 10/28/99 / gh 10/28/99

Quality Assurance Review Name / Date: _____

Data Entry: Entered LAC 10/29/99

Comments / Justifications: _____

Analyte: POPA
 Acceptable Linear Range: 49.30 - 976.0 ppb
 LOQ: 49.3 ppb Extraction Blank: <1/2 LOQ
 R²: 0.9952 Matrix Blank: <1/2 LOQ
 Calibration Checks: OK
 MS/MSD: Not spiked with POPA
 Data Status: ① ② Entered LAC 10/29/99

Analyte: 980
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: ③ ④

Analyte: SURV
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: _____

- ① excluded and curve due to divergence mm 10-28-99
- ② excluded bottom 4 points to meet 30% Deviation Criteria and bottom 3 points did not have area counts that were 2x matrix blank. mm 10-28-99
- ③ 980 was not in the standards, used the concentration of POPA as a guide to check linearity of 980 in the standards. mm 10-28-99
- ④ the analysis of 980 was for screening purposes only. mm 10-28-99
- ⑤ INTERESTING m/z = 980 RESPONSE. OBSERVED CONCENTRATION GRADIENT IN CURVE (RESIDUAL?). NONE PRESENT IN CONTROL GROUP BUT 5mg/kg dose group has significant amount. MEC 10/25/99

LAC 10/29/99

mmh
10-28-99
Page 1 P1/3

Quantify Compound Summary Report
Tox 145: Rat Sera and Liver, Day 1 & 4

Sample List: C:\MASSLYNK\Davey070799.PRO\SampleDB\102799b
Last modified: Thu Oct 28 09:14:03 1999
Method: C:\MASSLYNK\Davey070799.PRO\MethDB\102799b
Last modified: Thu Oct 28 08:57:32 1999
Job Code:

Printed: Thu Oct 28 09:27:15 1999

Compound 1: POAA (fume 1)

# Name	Samplelist Text	Std Conc	RT	Area	Conc. (ppt)	Flags	%Dev
1 102799013	MeOH Blank		6.822	557	0.00	bd	
2 102799014	MeOH Blank		6.793	315	0.00	bb	
3 102799015	TOX145-RBS10229-H2O Blk-1, RMW		6.821	5022	0.00	MM	
4 102799016	TOX145-RBS10229-Serum Blk-1, RMW		6.825	4609	0.00	MM	
5 102799017	TOX145-RBS10229-1ppb-1, RMW	0.99	6.818	1615	0.00	MMX	-100
6 102799018	TOX145-RBS10229-5ppb-1, RMW	4.93	6.813	6515	0.00	MMX	-100
7 102799019	TOX145-RBS10229-10ppb-1, RMW	9.76	6.826	7039	0.00	MMX	-100
8 102799020	TOX145-RBS10229-25ppb-1, RMW	24.80	6.815	12929	2.95	MMX	-88
9 102799021	TOX145-RBS10229-50ppb-1, RMW	49.30	6.809	21414	39.07	bb	-21
10 102799022	TOX145-RBS10229-100ppb-1, RMW	97.60	6.817	38054	109.91	MM	13
11 102799023	TOX145-RBS10229-250ppb-1, RMW	248.00	6.811	77474	277.72	MM	12
12 102799024	TOX145-RBS10229-500ppb-1, RMW	493.00	6.806	125236	481.04	bb	-2
13 102799025	TOX145-RBS10229-750ppb-1, RMW	735.00	6.807	189194	753.30	bb	2
14 102799026	TOX145-RBS10229-1000ppb-1, RMW	976.00	6.809	232549	937.86	bb	-4
15 102799027	MeOH Blank		6.844	362	0.00	bb	
16 102799028	TOX145-RTS-9R03230M, Day1, control, -ext10/19/99		6.813	3894	0.00	bb	
17 102799029	TOX145-RTS-9R03231M, Day1, control, -ext10/19/99		6.811	4119	0.00	bb	
18 102799030	TOX145-RTS-9R03232M, Day1, control, -ext10/19/99		6.822	4255	0.00	bb	
19 102799031	TOX145-RTS-9R03233M, Day1, control, -ext10/19/99		6.820	5144	0.00	bb	
20 102799032	TOX145-RTS-9R03234M, Day1, control, -ext10/19/99		6.818	5636	0.00	bb	
21 102799033	TOX145-RBS10229-250ppb-1, RMW	248.00	6.821	79666	287.05	bb	16
22 102799034	MeOH Blank		6.820	270	0.00	bb	
23 102799035	TOX145-RTS-9R03235M, Day4, control, -ext10/19/99		6.811	3472	0.00	bb	
24 102799036	TOX145-RTS-9R03236M, Day4, control, -ext10/19/99		6.818	4590	0.00	bb	
25 102799037	TOX145-RTS-9R03237M, Day4, control, -ext10/19/99		6.815	4364	0.00	bb	
26 102799038	TOX145-RTS-9R03238M, Day4, control, -ext10/19/99		6.808	4614	0.00	bb	
27 102799039	TOX145-RTS-9R03239M, Day4, control, -ext10/19/99		6.811	3782	0.00	bb	
28 102799040	TOX145-RBS10229-250ppb-1, RMW	248.00	6.814	77581	278.17	bb	12
29 102799041	MeOH Blank		7.100	150	0.00	bb	
30 102799042	TOX145-RTS-9R03245M, Day1, 5mg/kg, ext10/19/99		6.811	37594	107.95	bb	
31 102799043	TOX145-RTS-9R03246M, Day1, 5mg/kg, ext10/19/99		6.806	46851	147.35	bb	
32 102799044	TOX145-RTS-9R03247M, Day1, 5mg/kg, ext10/19/99		6.810	42376	128.31	bb	
33 102799045	TOX145-RTS-9R03248M, Day1, 5mg/kg, ext10/19/99		6.822	36393	102.84	bb	
34 102799046	TOX145-RTS-9R03249M, Day1, 5mg/kg, ext10/19/99		6.810	40408	119.93	bb	
35 102799047	TOX145-RBS10229-250ppb-1, RMW	248.00	6.818	80830	292.00	bb	18
36 102799048	MeOH Blank		6.839	181	0.00	bb	
37 102799049	TOX145-RTS-9R03250M, Day4, 5mg/kg, ext10/19/99		6.811	28217	68.03	bb	
38 102799050	TOX145-RTS-9R03251M, Day4, 5mg/kg, ext10/19/99		6.819	28068	67.39	bb	
39 102799051	TOX145-RTS-9R03252M, Day4, 5mg/kg, ext10/19/99		6.810	36897	104.98	bb	
40 102799052	TOX145-RTS-9R03254-LM, Day4, 5mg/kg, ext 10/19/99		6.821	35914	100.80	bb	
41 102799053	TOX145-RTS-9R03254-ZM, Day4, 5mg/kg, ext 10/19/99		6.815	31360	81.41	bb	
42 102799054	TOX145-RBS10229-250ppb-1, RMW	248.00	6.810	82686	299.90	bb	21
43 102799055	MeOH Blank		7.173	79	0.00	bb	
44 102799056	TOX145-RTL-9R03247-M, Day1, 5mg/kg, ext 10/13/99		6.808	8814	0.00	bb	
45 102799057	TOX145-RTL-9R03253-M, Day1, 5mg/kg, ext 10/13/99		6.796	5855	0.00	bb	
46 102799058	TOX145-RBS10229-250ppb-1, RMW	248.00	6.808	87005	318.29	MM	28
47 102799059	MeOH Blank		6.823	225	0.00	db	
48 102799060	TOX145-RBS10229-H2O Blk-1, RMW		6.806	5847	0.00	bb	
49 102799061	TOX145-RBS10229-Serum Blk-1, RMW		6.814	5385	0.00	bb	
50 102799062	TOX145-RBS10229-1ppb-1, RMW	0.99	6.791	2075	0.00	bdX	-100
51 102799063	TOX145-RBS10229-5ppb-1, RMW	4.93	6.807	7285	0.00	bbX	-100
52 102799064	TOX145-RBS10229-10ppb-1, RMW	9.76	6.811	8322	0.00	bbX	-100
53 102799065	TOX145-RBS10229-25ppb-1, RMW	24.80	6.815	15843	15.35	bbX	-38
54 102799066	TOX145-RBS10229-50ppb-1, RMW	49.30	6.799	29105	71.81	bbX	46
55 102799067	TOX145-RBS10229-100ppb-1, RMW	97.60	6.815	56970	190.43	bbX	95
56 102799068	TOX145-RBS10229-250ppb-1, RMW	248.00	6.806	117936	449.96	bbX	81
57 102799069	TOX145-RBS10229-500ppb-1, RMW	493.00	6.826	203783	815.41	bbX	65
58 102799070	TOX145-RBS10229-750ppb-1, RMW	735.00	6.812	306442	1252.42	bbX	70
59 102799071	TOX145-RBS10229-1000ppb-1, RMW	976.00	6.812	374817	1543.49	bbX	58

Entered, except RTL
conc.
LAC
10/29/99

Quantify Compound Summary Report
Tox 145: Rat Sera and Liver, Day 1 & 4

Page 2

Sample List: C:\MASSLYNK\Davey070799.PRO\SampleDB\102799b
Last modified: Thu Oct 28 09:14:03 1999
Method: C:\MASSLYNK\Davey070799.PRO\MethDB\102799b
Last modified: Thu Oct 28 08:57:32 1999
Job Code:

Printed: Thu Oct 28 09:27:15 1999

Compound 2: 980 (func 2)

# Name	Samplelist Text	Std Conc	RT	Area	Conc. (ppt)	Flags	%Dev
1 102799013	MeOH Blank						
2 102799014	MeOH Blank						
3 102799015	TOX145-RBS10229-H2O Blk-1, RWV						
4 102799016	TOX145-RBS10229-Serum Blk-1, RWV						
5 102799017	TOX145-RBS10229-1ppb-1, RWV	0.99	7.542	24	0.99 MCK		1
6 102799018	TOX145-RBS10229-5ppb-1, RWV	4.93	7.585	84	4.60 bb		-7
7 102799019	TOX145-RBS10229-10ppb-1, RWV	9.76	7.594	173	9.95 bb		2
8 102799020	TOX145-RBS10229-25ppb-1, RWV	24.80	7.584	396	23.34 MCK		-6
9 102799021	TOX145-RBS10229-50ppb-1, RWV	49.30	7.598	849	50.50 bb		2
10 102799022	TOX145-RBS10229-100ppb-1, RWV	97.60	7.586	1636	97.73 bb		0
11 102799023	TOX145-RBS10229-250ppb-1, RWV	248.00	7.598	3918	234.77 bb		-5
12 102799024	TOX145-RBS10229-500ppb-1, RWV	493.00	7.589	6110	366.38 MCK		-26
13 102799025	TOX145-RBS10229-750ppb-1, RWV	735.00	7.594	9343	560.46 bbX		-24
14 102799026	TOX145-RBS10229-1000ppb-1, RWV	976.00	7.595	10952	657.07 bbX		-33
15 102799027	MeOH Blank						
16 102799028	TOX145-RTS-9R03230M, Day1, control, -ext10/19/99						
17 102799029	TOX145-RTS-9R03231M, Day1, control, -ext10/19/99						
18 102799030	TOX145-RTS-9R03232M, Day1, control, -ext10/19/99						
19 102799031	TOX145-RTS-9R03233M, Day1, control, -ext10/19/99						
20 102799032	TOX145-RTS-9R03234M, Day1, control, -ext10/19/99						
21 102799033	TOX145-RBS10229-250ppb-1, RWV	248.00	7.593	4004	239.94 bb		-3
22 102799034	MeOH Blank						
23 102799035	TOX145-RTS-9R03235M, Day4, control, -ext10/19/99						
24 102799036	TOX145-RTS-9R03236M, Day4, control, -ext10/19/99						
25 102799037	TOX145-RTS-9R03237M, Day4, control, -ext10/19/99						
26 102799038	TOX145-RTS-9R03238M, Day4, control, -ext10/19/99						
27 102799039	TOX145-RTS-9R03239M, Day4, control, -ext10/19/99						
28 102799040	TOX145-RBS10229-250ppb-1, RWV	248.00	7.588	4224	253.16 bb		2
29 102799041	MeOH Blank						
30 102799042	TOX145-RTS-9R03245M, Day1, 5mg/kg, ext10/19/99		7.584	2501	149.67 bb		
31 102799043	TOX145-RTS-9R03246M, Day1, 5mg/kg, ext10/19/99		7.595	2969	177.80 bb		
32 102799044	TOX145-RTS-9R03247M, Day1, 5mg/kg, ext10/19/99		7.599	3580	214.47 bb		
33 102799045	TOX145-RTS-9R03248M, Day1, 5mg/kg, ext10/19/99		7.590	2742	164.16 bb		
34 102799046	TOX145-RTS-9R03249M, Day1, 5mg/kg, ext10/19/99		7.583	3343	200.26 bb		
35 102799047	TOX145-RBS10229-250ppb-1, RWV	248.00	7.590	4039	242.02 bb		-2
36 102799048	MeOH Blank						
37 102799049	TOX145-RTS-9R03250M, Day4, 5mg/kg, ext10/19/99		7.580	1770	105.83 bb		
38 102799050	TOX145-RTS-9R03251M, Day4, 5mg/kg, ext10/19/99		7.592	1098	65.43 bb		
39 102799051	TOX145-RTS-9R03252M, Day4, 5mg/kg, ext10/19/99		7.594	1444	86.25 bb		
40 102799052	TOX145-RTS-9R03254-1M, Day4, 5mg/kg, ext 10/19/99		7.593	1337	79.81 bb		
41 102799053	TOX145-RTS-9R03254-2M, Day4, 5mg/kg, ext 10/19/99		7.589	1603	95.79 bb		
42 102799054	TOX145-RBS10229-250ppb-1, RWV	248.00	7.598	4020	240.87 bb		-3
43 102799055	MeOH Blank						
44 102799056	TOX145-RTL-9R03247-M, Day1, 5mg/kg, ext 10/13/99		7.594	983	58.56 bb		
45 102799057	TOX145-RTL-9R03253-M, Day4, 5mg/kg, ext 10/13/99		7.587	526	31.13 bb		
46 102799058	TOX145-RBS10229-250ppb-1, RWV	248.00	7.591	3719	222.84 bb		-10
47 102799059	MeOH Blank						
48 102799060	TOX145-RBS10229-H2O Blk-1, RWV						
49 102799061	TOX145-RBS10229-Serum Blk-1, RWV						
50 102799062	TOX145-RBS10229-1ppb-1, RWV	0.99	7.525	23	0.93 MCK		-5
51 102799063	TOX145-RBS10229-5ppb-1, RWV	4.93	7.590	100	5.53 bb		12
52 102799064	TOX145-RBS10229-10ppb-1, RWV	9.76	7.578	152	8.68 bb		-11
53 102799065	TOX145-RBS10229-25ppb-1, RWV	24.80	7.586	427	25.15 bb		1
54 102799066	TOX145-RBS10229-50ppb-1, RWV	49.30	7.597	818	48.64 bb		-1
55 102799067	TOX145-RBS10229-100ppb-1, RWV	97.60	7.599	1808	108.08 bb		11
56 102799068	TOX145-RBS10229-250ppb-1, RWV	248.00	7.592	4202	251.81 bb		2
57 102799069	TOX145-RBS10229-500ppb-1, RWV	493.00	7.599	6743	404.41 bbX		-18
58 102799070	TOX145-RBS10229-750ppb-1, RWV	735.00	7.586	9918	595.01 bbX		-19
59 102799071	TOX145-RBS10229-1000ppb-1, RWV	976.00	7.596	11346	680.77 bbX		-30

(*)

do not
report
values
k_{in} 10/28/99

(*)

(*) curves are not representative
of PFOSAB levels
k_{in} 10/28/99

Sample List: C:\MASSLYNK\Davey070799.FPO\SampleDB\102799b
Last modified: Thu Oct 28 09:14:03 1999
Method: C:\MASSLYNK\Davey070799.FPO\MethDB\102799b
Last modified: Thu Oct 28 08:57:32 1999
Job Code:

Printed: Thu Oct 28 09:27:15 1999

Compound 3: surr (fune 3)

# Name	Samplelist Text	Std Conc	RT	Area	Conc. (ppt)	Flags	QDev
1 102799013	MeOH Blank		6.833	20	0.00 bb		-100
2 102799014	MeOH Blank		6.805	31	0.00 bb		-100
3 102799015	TOX145-RBS10229-H2O Blk-1, RMW		6.815	178594	1.00 bb		-0
4 102799016	TOX145-RBS10229-Serum Blk-1, RMW		6.802	179068	1.00 bb		-0
5 102799017	TOX145-RBS10229-1ppb-1, RMW	1.00	6.795	34459	0.19 RM		-81
6 102799018	TOX145-RBS10229-5ppb-1, RMW	1.00	6.808	182303	1.02 bb		2
7 102799019	TOX145-RBS10229-10ppb-1, RMW	1.00	6.804	207191	1.16 bb		16
8 102799020	TOX145-RBS10229-25ppb-1, RMW	1.00	6.810	207507	1.16 bb		16
9 102799021	TOX145-RBS10229-50ppb-1, RMW	1.00	6.804	199555	1.11 bb		11
10 102799022	TOX145-RBS10229-100ppb-1, RMW	1.00	6.811	192001	1.07 bb		7
11 102799023	TOX145-RBS10229-250ppb-1, RMW	1.00	6.805	179742	1.00 bb		0
12 102799024	TOX145-RBS10229-500ppb-1, RMW	1.00	6.800	158632	0.89 bb		-11
13 102799025	TOX145-RBS10229-750ppb-1, RMW	1.00	6.801	159083	0.89 bb		-11
14 102799026	TOX145-RBS10229-1000ppb-1, RMW	1.00	6.804	155316	0.87 bb		-13
15 102799027	MeOH Blank		6.806	111	0.00 bb		-100
16 102799028	TOX145-RTS-9R03230M, Day1, control, -ext10/19/99		6.808	249174	1.39 bb		39
17 102799029	TOX145-RTS-9R03231M, Day1, control, -ext10/19/99		6.806	238809	1.33 bb		33
18 102799030	TOX145-RTS-9R03232M, Day1, control, -ext10/19/99		6.799	225733	1.26 bb		26
19 102799031	TOX145-RTS-9R03233M, Day1, control, -ext10/19/99		6.798	233026	1.30 bb		30
20 102799032	TOX145-RTS-9R03234M, Day1, control, -ext10/19/99		6.813	224963	1.26 bb		26
21 102799033	TOX145-RBS10229-250ppb-1, RMW	1.00	6.799	179678	1.00 bb		0
22 102799034	MeOH Blank		6.814	133	0.00 bb		-100
23 102799035	TOX145-RTS-9R03235M, Day4, control, -ext10/19/99		6.805	204590	1.14 bb		14
24 102799036	TOX145-RTS-9R03236M, Day4, control, -ext10/19/99		6.812	210526	1.18 bb		18
25 102799037	TOX145-RTS-9R03237M, Day4, control, -ext10/19/99		6.810	201676	1.13 bb		13
26 102799038	TOX145-RTS-9R03238M, Day4, control, -ext10/19/99		6.802	191867	1.07 bb		7
27 102799039	TOX145-RTS-9R03239M, Day4, control, -ext10/19/99		6.806	177017	0.99 bb		-1
28 102799040	TOX145-RBS10229-250ppb-1, RMW	1.00	6.808	175534	0.98 bb		-2
29 102799041	MeOH Blank		6.827	194	0.00 bb		-100
30 102799042	TOX145-RTS-9R03245M, Day1, 5mg/kg, ext10/19/99		6.806	220905	1.23 bb		23
31 102799043	TOX145-RTS-9R03246M, Day1, 5mg/kg, ext10/19/99		6.801	205626	1.15 bb		15
32 102799044	TOX145-RTS-9R03247M, Day1, 5mg/kg, ext10/19/99		6.804	197683	1.10 bb		10
33 102799045	TOX145-RTS-9R03248M, Day1, 5mg/kg, ext10/19/99		6.816	174315	0.97 bb		-3
34 102799046	TOX145-RTS-9R03249M, Day1, 5mg/kg, ext10/19/99		6.804	175461	0.98 bb		-2
35 102799047	TOX145-RBS10229-250ppb-1, RMW	1.00	6.812	185134	1.03 bb		3
36 102799048	MeOH Blank		6.800	231	0.00 bb		-100
37 102799049	TOX145-RTS-9R03250M, Day4, 5mg/kg, ext10/19/99		6.805	164011	0.92 bb		-8
38 102799050	TOX145-RTS-9R03251M, Day4, 5mg/kg, ext10/19/99		6.814	165230	0.92 bb		-8
39 102799051	TOX145-RTS-9R03252M, Day4, 5mg/kg, ext10/19/99		6.804	199874	1.12 bb		12
40 102799052	TOX145-RTS-9R03254-1M, Day4, 5mg/kg, ext 10/19/99		6.798	197858	1.10 bb		10
41 102799053	TOX145-RTS-9R03254-2M, Day4, 5mg/kg, ext 10/19/99		6.809	182382	1.02 bb		2
42 102799054	TOX145-RBS10229-250ppb-1, RMW	1.00	6.805	181776	1.02 bb		2
43 102799055	MeOH Blank		6.820	131	0.00 bb		-100
44 102799056	TOX145-RTL-9R03247-M, Day1, 5mg/kg, ext 10/13/99		6.802	259082	1.45 bb		45
45 102799057	TOX145-RTL-9R03253-M, Day4, 5mg/kg, ext 10/13/99		6.791	299726	1.67 bb		67
46 102799058	TOX145-RBS10229-250ppb-1, RMW	1.00	6.786	208571	1.16 bb		16
47 102799059	MeOH Blank		6.800	152	0.00 bd		-100
48 102799060	TOX145-RBS10229-H2O Blk-1, RMW		6.801	203027	1.13 bb		13
49 102799061	TOX145-RBS10229-Serum Blk-1, RMW		6.808	205089	1.15 bb		15
50 102799062	TOX145-RBS10229-1ppb-1, RMW	1.00	6.785	40376	0.23 bb		-77
51 102799063	TOX145-RBS10229-5ppb-1, RMW	1.00	6.802	201154	1.12 bb		12
52 102799064	TOX145-RBS10229-10ppb-1, RMW	1.00	6.805	222858	1.24 bb		24
53 102799065	TOX145-RBS10229-25ppb-1, RMW	1.00	6.808	231660	1.29 bb		29
54 102799066	TOX145-RBS10229-50ppb-1, RMW	1.00	6.810	230939	1.29 bb		29
55 102799067	TOX145-RBS10229-100ppb-1, RMW	1.00	6.809	228963	1.28 bb		28
56 102799068	TOX145-RBS10229-250ppb-1, RMW	1.00	6.801	204091	1.14 bb		14
57 102799069	TOX145-RBS10229-500ppb-1, RMW	1.00	6.804	185891	1.04 bb		4
58 102799070	TOX145-RBS10229-750ppb-1, RMW	1.00	6.807	185314	1.03 bb		3
59 102799071	TOX145-RBS10229-1000ppb-1, RMW	1.00	6.807	174614	0.98 bb		-2

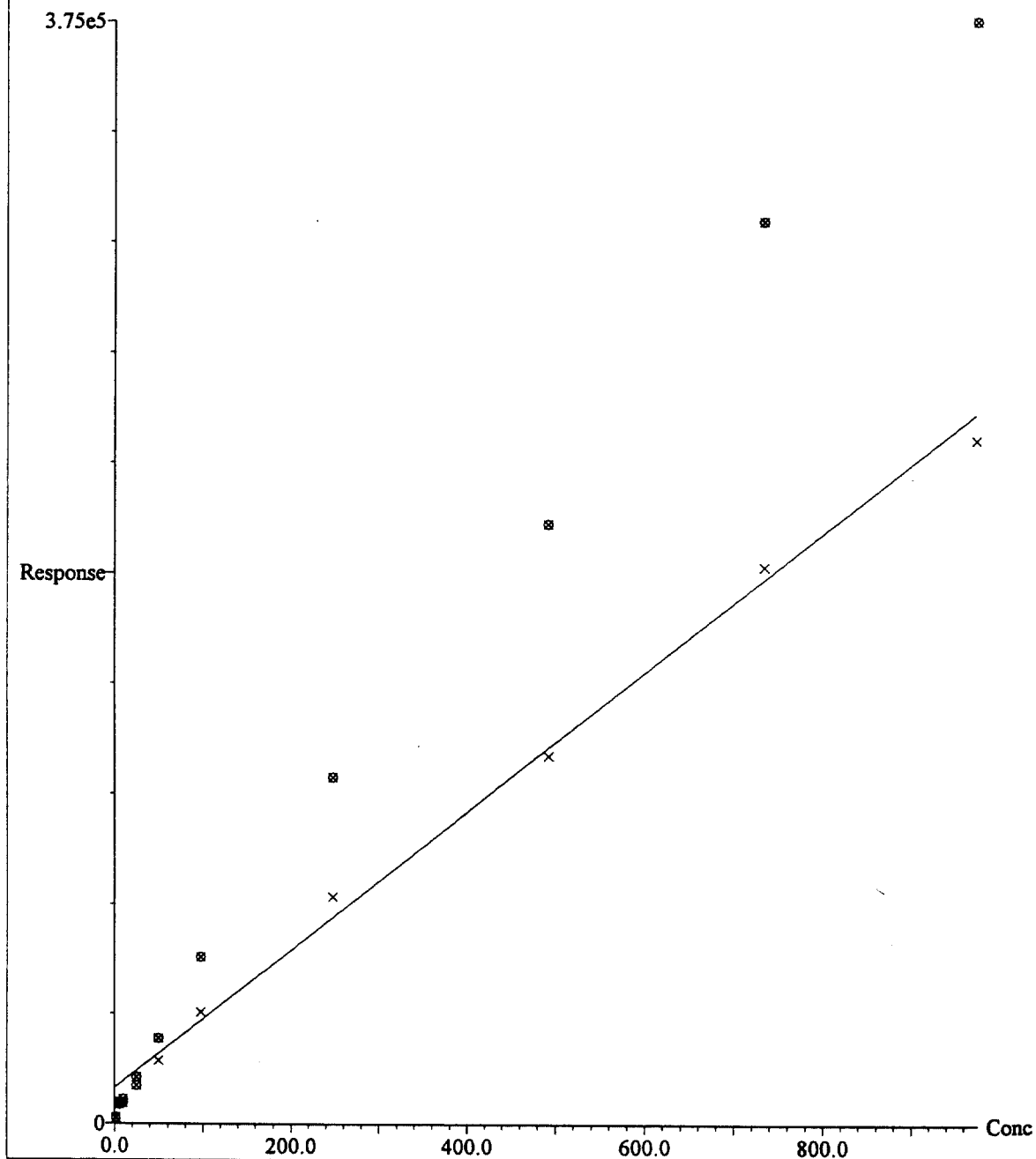
Not a target analyte - data not used.

UC 10/29/99

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10-28-99
P13
Page 1

Quantify Calibration Report
Tox 145: Rat Sera and Liver, Day 1 & 4

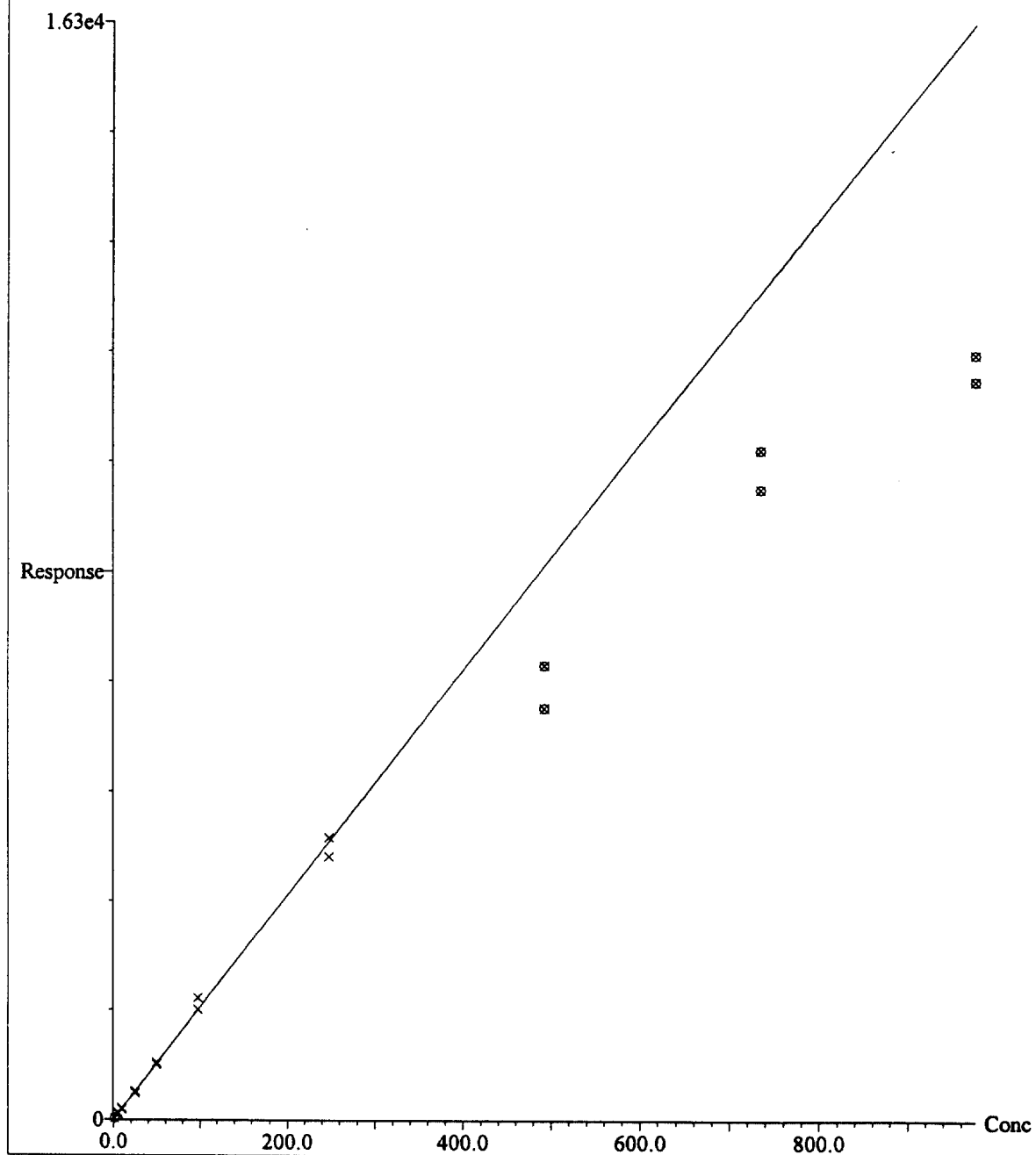
Compound 1 name: POAA (func 1)
Coefficient of Determination: 0.995226
Calibration curve: $234.909 * x + 12236.1$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



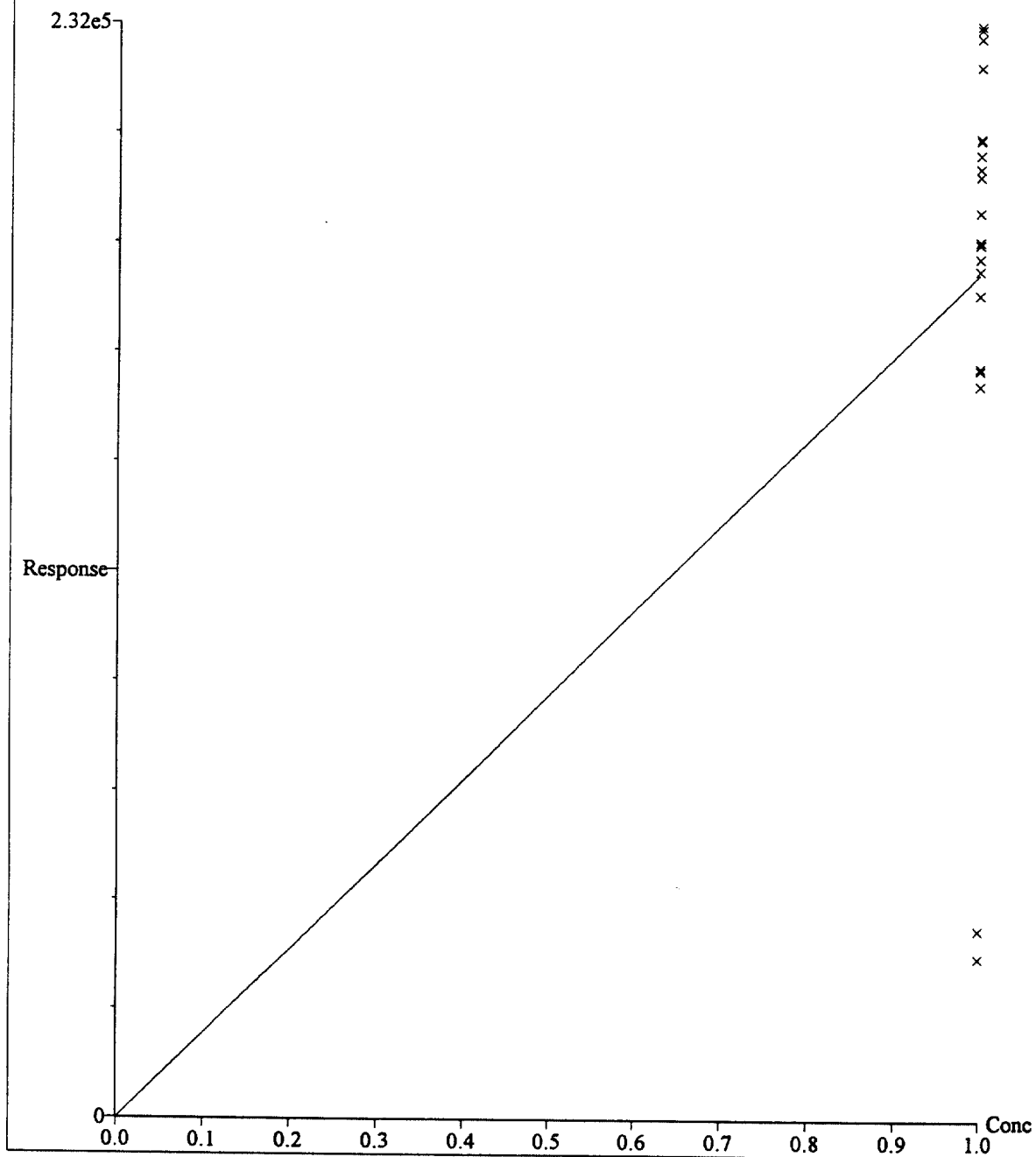
Davey 070799: quantitated and printed by mmh10/28/99

000640

Compound 2 name: 980 (func 2)
Coefficient of Determination: 0.996332
Calibration curve: $16.6556 * x + 7.74822$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound 3 name: surr (func 3)
Response Factor: 179082
Response type: External Std, Area
Curve type: RF



Davey 070799: quantitated and printed by mmh10/28/99

000642

10-28-99
P15
Page 1

Method Report

Method file: C:\MASSLYNK\Davey070799.PRO\MethDB\102799b.mdb
Last modified: Thu Oct 28 08:57:32 1999

Compound: 1: POAA (func 1)

Printed: Thu Oct 28 12:41:03 1999

Compound Name: POAA (func 1)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. A
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 6.842, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.414

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 492

Smothing: Disabled
Window size (scans): 1
Number of smooths: 1
Smothing method: Savitzky Golay

Join valley threshold: 5.00
Reduce peak tailing: 50.00
Raise baseline maximum: 10.00

Draw vertical threshold: 25.00
Detect shoulders: Disabled
Shoulder threshold: 5.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Enabled
Absolute area: 0.00, Disabled

000643

Method Report

Page 2

Method file: C:\MASSLYNX\Davey070799.PRO\MethDB\102799b.mdb
Last modified: Thu Oct 28 08:57:32 1999

Compound: 2: 980 (func 2)

Printed: Thu Oct 28 12:41:03 1999

Compound Name: 980 (func 2)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. A
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.620, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.359

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 444

Smoothing: Disabled
Window size (scans): 1
Number of smooths: 1
Smoothing method: Savitzky Golay

Join valley threshold: 5.00
Reduce peak tailing: 50.00
Raise baseline maximum: 10.00

Draw vertical threshold: 25.00
Detect shoulders: Disabled
Shoulder threshold: 5.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Enabled
Absolute area: 0.00, Disabled

000644

Method Report

Page 3

Method file: C:\MASSLYNX\Davey070799.PRO\MethDB\102799b.mdb
Last modified: Thu Oct 28 08:57:32 1999

Compound: 3: surr (func 3)

Printed: Thu Oct 28 12:41:03 1999

Compound Name: surr (func 3)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Fixed
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 6.820, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.265

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 629

Smoothing: Disabled
Window size (scans): 1
Number of smooths: 1
Smoothing method: Savitzky Golay

Join valley threshold: 5.00
Reduce peak tailing: 50.00
Raise baseline maximum: 10.00

Draw vertical threshold: 25.00
Detect shoulders: Disabled
Shoulder threshold: 5.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Enabled
Absolute area: 0.00, Disabled

000645

mmh
10-27-99

DAVEY 070799: FACT Equipment Log 99077
Quattro Ultima: #9261QB

PAGE _____

DATE: 10/27/99

ANALYST: mmh

STUDY NUMBER: Tox145: Matrix: Rat Sera& Liver DESCRIPTION: POAA& 980

FACT Method: ETS 8-5.1 & 8-7.0

Injection volume: 5 uL Cycle time: 10 min Electrode: Z-Spray

Mobile Phase: (A) 2.0 mM NH₄CH₃CO₂ (B) MeOH

TN-A- or stdrd number (A) 99085-11-3 (B) tn-a-2945

Prepared (date/by): (A) 10/23/99 sah (B) 10/23/99 sah

Expires: (A) 04/00 (B) 04/00

Store at room temp? ☒ Y or N Other: _____

Guard column: None

Part number: na Lot number: na

Analytical column: Keystone Betasil

Size: 2 x 50 mm Type: C18 Sphere size: 5 um

Part number: 055-701-2 Lot number: P9E11 Serial number: 0591825Z

Analytical parameters:

Drying gas (L/hr): 487 ESI nebulizing gas (L/hr) 10 Sheath gas (L/hr) 0

LC Flow rate: 300 uL/min Back pressure: 82 bar

HPLC solvent gradient: 10min-10-95

Time (min.)	%B
1) <u>0.00</u>	<u>10.0</u>
2) <u>1.00</u>	<u>10.0</u>
3) <u>5.50</u>	<u>95.0</u>
4) <u>7.50</u>	<u>95.0</u>
5) <u>8.00</u>	<u>10.0</u>

000646

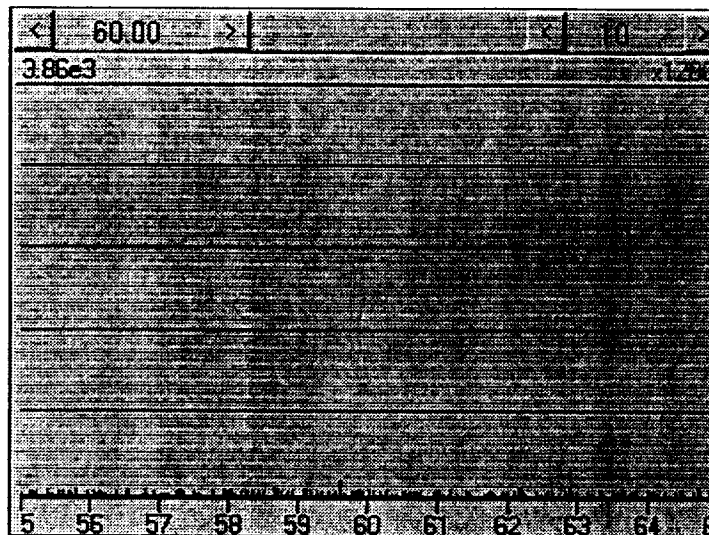
17284
10-27-99

Tuning Method Report

Page 1

Method: C:\MASSLYNX\DAVEY BACKUP AUG99\DAVEY-RATS.PRO\ACQUDB\PFOS-4

Printed: Wed Oct 27 14:31:21 1999



SOURCE (ESP-)	Set	Rdbk	Analyser	Set	Rdbk
Capillary(kV)	3.15	-3.07	LM Res 1	14.5	
Cone	10	-11	HM Res 1	14.5	
Hexapole 1	0.0		IEnergy 1	1.0	
Aperture 1	0.0		Entrance	-2	-3
Hexapole 2	0.0		Collision	2	2
Source Block Temp.	150	148	Exit	1	0
Desolvation Temp.	250	249	LM Res 2	12.0	
			HM Res 2	12.0	
			IEnergy 2	1.0	
			Multiplier	650	-646
Pressures		Rdbk	Gas Flows		Rdbk
Analyser Vacuum	OFF		Cone Gas	57.8	
Gas Cell	4.3e-3		Desolvation	484.8	

000647

7287X
102799

Scanning Method Report

Page 1

Method: C:\MASSLYNK\DAVEY070799.PRO\ACQUIS\10MIN-POAA-980-IS
Last Modified: Wed Oct 27 14:18:01 1999
Printed: Wed Oct 27 14:32:05 1999

Solvent Delay (mins) : 0.00

Function : 1 MRM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03
Span (Daltons) : 0.00
Start Time (Mins) : 0.00
End Time (Mins) : 10.00

Repeats : 1

	Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1		413.00	169.00	0.30	30	15

Function : 2 MRM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03
Span (Daltons) : 0.00
Start Time (Mins) : 0.00
End Time (Mins) : 10.00

Repeats : 1

	Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1		980.00	78.00	0.30	60	60

Function : 3 MRM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03
Span (Daltons) : 0.00
Start Time (Mins) : 0.00
End Time (Mins) : 10.00

Repeats : 1

	Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1		427.00	80.00	0.30	40	60

000648

Method Report

Page 1

Method File:
Last Modified:

C:\MASSLYNX\DAVEY070799.PRO\ACQUDB\10min
Wednesday, October 27, 1999 14:25:05

Printed:

Wednesday, October 27, 1999 14:25:31

HP1100 LC Pump Initial Conditions

Solvents

A%	90.0
B%	10.0
C%	0.0
D%	0.0

Flow (ml/min)	0.300
Stop Time (mins)	10.0
Min Pressure (bar)	0
Max Pressure (bar)	400
Oven Temperature Left (°C)	30.0
Oven Temperature Right (°C)	30.0

HP1100 LC Pump Gradient Timetable

The gradient Timetable contains 5 entries which are :

Time	A%	B%	C%	D%	Flow
0.00	90.0	10.0	0.0	0.0	0.300
1.00	90.0	10.0	0.0	0.0	0.300
5.50	5.0	95.0	0.0	0.0	0.300
7.50	5.0	95.0	0.0	0.0	0.300
8.00	90.0	10.0	0.0	0.0	0.300

HP1100 LC Pump External Event Timetable

The Timetable contains 3 entries which are :

Time	Column Switch	Contact1	Contact2	Contact3	Contact4
Initial	Off	Off	Off	Off	Off
0.00	Off	On	On	Off	On
0.10	Off	Off	Off	Off	Off

HP1100 Autosampler Initial Conditions

Draw Speed	200.0
Eject Speed (ul/min)	200
Draw Position (mm)	0.00
Stop Time (mins)	9.00
Injection Volume (ul)	10.0
Vial Number	12

000649

102799
102799

Masslynx - Sample List

Page 1

Sample List: C:\MASSLYNX\Davey070799.PRO\SampleDB\102799b.SPL
Printed: Wed Oct 27 14:34:05 1999

Page Position: (1, 1)

	File Name	File Text
1	102799013	MeOH Blank
2	102799014	MeOH Blank
3	102799015	TOX145-RBS10229-H2O Blk-1, RWW
4	102799016	TOX145-RBS10229-Serum Blk-1, RWW
5	102799017	TOX145-RBS10229-1ppb-1, RWW
6	102799018	TOX145-RBS10229-5ppb-1, RWW
7	102799019	TOX145-RBS10229-10ppb-1, RWW
8	102799020	TOX145-RBS10229-25ppb-1, RWW
9	102799021	TOX145-RBS10229-50ppb-1, RWW
10	102799022	TOX145-RBS10229-100ppb-1, RWW
11	102799023	TOX145-RBS10229-250ppb-1, RWW
12	102799024	TOX145-RBS10229-500ppb-1, RWW
13	102799025	TOX145-RBS10229-750ppb-1, RWW
14	102799026	TOX145-RBS10229-1000ppb-1, RWW
15	102799027	MeOH Blank
16	102799028	TOX145-RTS-9R03230M, Day1, control, -ext10/19/99
17	102799029	TOX145-RTS-9R03231M, Day1, control, -ext10/19/99
18	102799030	TOX145-RTS-9R03232M, Day1, control, -ext10/19/99
19	102799031	TOX145-RTS-9R03233M, Day1, control, -ext10/19/99
20	102799032	TOX145-RTS-9R03234M, Day1, control, -ext10/19/99
21	102799033	TOX145-RBS10229-250ppb-1, RWW
22	102799034	MeOH Blank
23	102799035	TOX145-RTS-9R03235M, Day4, control, -ext10/19/99
24	102799036	TOX145-RTS-9R03236M, Day4, control, -ext10/19/99
25	102799037	TOX145-RTS-9R03237M, Day4, control, -ext10/19/99
26	102799038	TOX145-RTS-9R03238M, Day4, control, -ext10/19/99
27	102799039	TOX145-RTS-9R03239M, Day4, control, -ext10/19/99
28	102799040	TOX145-RBS10229-250ppb-1, RWW
29	102799041	MeOH Blank
30	102799042	TOX145-RTS-9R03245M, Day1, 5mg/kg, D50-ext10/19/99
31	102799043	TOX145-RTS-9R03246M, Day1, 5mg/kg, D50-ext10/19/99
32	102799044	TOX145-RTS-9R03247M, Day1, 5mg/kg, D50-ext10/19/99
33	102799045	TOX145-RTS-9R03248M, Day1, 5mg/kg, D50-ext10/19/99
34	102799046	TOX145-RTS-9R03249M, Day1, 5mg/kg, D50-ext10/19/99
35	102799047	TOX145-RBS10229-250ppb-1, RWW
36	102799048	MeOH Blank
37	102799049	TOX145-RTS-9R03250M, Day4, 5mg/kg, ext10/19/99
38	102799050	TOX145-RTS-9R03251M, Day4, 5mg/kg, ext10/19/99
39	102799051	TOX145-RTS-9R03252M, Day4, 5mg/kg, ext10/19/99
40	102799052	TOX145-RTS-9R03254-1M, Day4, 5mg/kg, ext 10/19/99
41	102799053	TOX145-RTS-9R03254-2M, Day4, 5mg/kg, ext 10/19/99
42	102799054	TOX145-RBS10229-250ppb-1, RWW
43	102799055	MeOH Blank
44	102799056	TOX145-RTL-9R03247-M, Day1, 5mg/kg, ext 10/13/99
45	102799057	TOX145-RTL-9R03253-M, Day4, 5mg/kg, ext 10/13/99
46	102799058	TOX145-RBS10229-250ppb-1, RWW
47	102799059	MeOH Blank
48	102799060	TOX145-RBS10229-H2O Blk-1, RWW
49	102799061	TOX145-RBS10229-Serum Blk-1, RWW
50	102799062	TOX145-RBS10229-1ppb-1, RWW
51	102799063	TOX145-RBS10229-5ppb-1, RWW
52	102799064	TOX145-RBS10229-10ppb-1, RWW
53	102799065	TOX145-RBS10229-25ppb-1, RWW
54	102799066	TOX145-RBS10229-50ppb-1, RWW
55	102799067	TOX145-RBS10229-100ppb-1, RWW
56	102799068	TOX145-RBS10229-250ppb-1, RWW
57	102799069	TOX145-RBS10229-500ppb-1, RWW
58	102799070	TOX145-RBS10229-750ppb-1, RWW

000650

Handwritten: 102759

Masslynx - Sample List

Page 2

Sample List: C:\MASSLYNX\Davey070799.PRO\SampleDB\102799b.SPL
Printed: Wed Oct 27 14:34:05 1999

Page Position: (2, 1)

	MS File	Inlet File	Bottle	Inject Volume	Sample Type	Conc A
1	10MIN-POAA-980-IS	10min-10-95f	91	10	---	---
2	10MIN-POAA-980-IS	10min-10-95f	91	10	---	---
3	10MIN-POAA-980-IS	10min-10-95f	1	10	---	---
4	10MIN-POAA-980-IS	10min-10-95f	2	10	---	---
5	10MIN-POAA-980-IS	10min-10-95f	3	2	Standard	---
6	10MIN-POAA-980-IS	10min-10-95f	3	10	Standard	---
7	10MIN-POAA-980-IS	10min-10-95f	4	10	Standard	---
8	10MIN-POAA-980-IS	10min-10-95f	5	10	Standard	---
9	10MIN-POAA-980-IS	10min-10-95f	6	10	Standard	---
10	10MIN-POAA-980-IS	10min-10-95f	7	10	Standard	---
11	10MIN-POAA-980-IS	10min-10-95f	8	10	Standard	---
12	10MIN-POAA-980-IS	10min-10-95f	9	10	Standard	---
13	10MIN-POAA-980-IS	10min-10-95f	10	10	Standard	---
14	10MIN-POAA-980-IS	10min-10-95f	11	10	Standard	---
15	10MIN-POAA-980-IS	10min-10-95f	92	10	---	---
16	10MIN-POAA-980-IS	10min-10-95f	12	10	---	---
17	10MIN-POAA-980-IS	10min-10-95f	13	10	---	---
18	10MIN-POAA-980-IS	10min-10-95f	14	10	---	---
19	10MIN-POAA-980-IS	10min-10-95f	15	10	---	---
20	10MIN-POAA-980-IS	10min-10-95f	16	10	---	---
21	10MIN-POAA-980-IS	10min-10-95f	8	10	---	---
22	10MIN-POAA-980-IS	10min-10-95f	92	10	---	---
23	10MIN-POAA-980-IS	10min-10-95f	17	10	---	---
24	10MIN-POAA-980-IS	10min-10-95f	18	10	---	---
25	10MIN-POAA-980-IS	10min-10-95f	19	10	---	---
26	10MIN-POAA-980-IS	10min-10-95f	20	10	---	---
27	10MIN-POAA-980-IS	10min-10-95f	21	10	---	---
28	10MIN-POAA-980-IS	10min-10-95f	8	10	---	---
29	10MIN-POAA-980-IS	10min-10-95f	93	10	---	---
30	10MIN-POAA-980-IS	10min-10-95f	22	10	---	---
31	10MIN-POAA-980-IS	10min-10-95f	23	10	---	---
32	10MIN-POAA-980-IS	10min-10-95f	24	10	---	---
33	10MIN-POAA-980-IS	10min-10-95f	25	10	---	---
34	10MIN-POAA-980-IS	10min-10-95f	26	10	---	---
35	10MIN-POAA-980-IS	10min-10-95f	8	10	QC	---
36	10MIN-POAA-980-IS	10min-10-95f	93	10	---	---
37	10MIN-POAA-980-IS	10min-10-95f	27	10	---	---
38	10MIN-POAA-980-IS	10min-10-95f	28	10	---	---
39	10MIN-POAA-980-IS	10min-10-95f	29	10	---	---
40	10MIN-POAA-980-IS	10min-10-95f	30	10	---	---
41	10MIN-POAA-980-IS	10min-10-95f	31	10	---	---
42	10MIN-POAA-980-IS	10min-10-95f	8	10	QC	---
43	10MIN-POAA-980-IS	10min-10-95f	94	10	---	---
44	10MIN-POAA-980-IS	10min-10-95f	41	10	---	---
45	10MIN-POAA-980-IS	10min-10-95f	42	10	---	---
46	10MIN-POAA-980-IS	10min-10-95f	8	10	QC	---
47	10MIN-POAA-980-IS	10min-10-95f	94	10	---	---
48	10MIN-POAA-980-IS	10min-10-95f	1	10	---	---
49	10MIN-POAA-980-IS	10min-10-95f	2	10	---	---
50	10MIN-POAA-980-IS	10min-10-95f	3	2	Standard	---
51	10MIN-POAA-980-IS	10min-10-95f	3	10	Standard	---
52	10MIN-POAA-980-IS	10min-10-95f	4	10	Standard	---
53	10MIN-POAA-980-IS	10min-10-95f	5	10	Standard	---
54	10MIN-POAA-980-IS	10min-10-95f	6	10	Standard	---
55	10MIN-POAA-980-IS	10min-10-95f	7	10	Standard	---
56	10MIN-POAA-980-IS	10min-10-95f	8	10	Standard	---
57	10MIN-POAA-980-IS	10min-10-95f	9	10	Standard	---
58	10MIN-POAA-980-IS	10min-10-95f	10	10	Standard	---

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1700h
102799

Masslynx - Sample List

Page 4

Sample List: C:\MASSLYNX\Davey070799.PRO\SampleDB\102799b.SPL
Printed: Wed Oct 27 14:34:05 1999

Page Position: (1, 2)

	File Name	File Text
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59	102799071	TOX145-RBS10229-1000ppb-1, RWW
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000652

12/24/99
10-27-99

Masslynx - Sample List

Page 5

Sample List: C:\MASSLYNX\Davey070799.PRO\SampleDB\102799b.SPL
Printed: Wed Oct 27 14:34:05 1999

Page Position: (2, 2)

	MS File	Inlet File	Bottle	Inject Volume	Sample Type	Conc A
59	10MIN-POAA-980-IS	10min-10-95f	11	10	Standard	—

000653

FACT: DATA REVIEW SUMMARY FORM

page 1-2
LAC
11/05/99

Date: 11/05/99
Data Analyst: JAS
FACT Study #: Tox 145
Matrix/Timepoints: Pat/Se.a - Day 29 close and control - ext 11/3/99 MCH
Sample List: 110799a
Set-up Analysis Date/Analyst: JAS 11/04/99
Instrument: Simp 020199

Data Review Sign-Off

Analyst Review
Name / Date: JAS 11/05/99

Technical Review
Name / Date: kin Nov-5-99

Quality Assurance Review
Name / Date: _____

Data Entry: LAC 11/05/99

Comments / Justifications: _____

Analyte: POAA (CH1) ①
Acceptable Linear Range: NA
LOQ: NA Extraction Blank: NA
R²: NA Matrix Blank: NA
Calibration Checks: NA
MS/MSD: ① NA enter to comments LAC 11/05/99
Data Status: NA

Analyte: PFOS (CH2) ②
Acceptable Linear Range: 9.76 ppb → 735 ppb
LOQ: 9.76 ppb Extracted Blank: less than LOQ
R²: 0.980777 Matrix Blank: less than LOQ
Calibration Checks: OK - Range from -7% → -12%
MS/MSD: OK - one value at 69% still reported > LAC 11/05/99
Data Status: closed samples need 1:10 dilutions.

Analyte: PFOSA (CH3)
Acceptable Linear Range: 4.93 ppb → 977 ppb
LOQ: 4.93 ppb Extracted Blank: less than LOQ
R²: 0.997747 Matrix Blank: less than LOQ
Calibration Checks: OK - Range from -5% → -13%
MS/MSD: OK > LAC 11/05/99
Data Status: Entered

① A calibration curve was not run for POAA. Data is useful for area counts and retention times only. - JAS 11/05/99

② Low level point, 4.93 ppb, was not 2x higher than the extraction blank; this point was excluded from the curve to disqualify a data range that may have been significantly affected by background levels of the analyte. - JAS 11/05/99
High point 976 ppb was excluded to provide a better fit over the linear range appropriate to the data. - JAS 11/05/99.

Some cross talk between 499 → 498 observed.

kin Nov-5-99

JAS 11/05/99 p. 1 of 2

000654

page 2-2
UAC
11/05/99

Comments / Justifications:

Analyte: _____

Acceptable Linear Range: _____

LOQ: _____ **Extracted Blank:** _____

R²: _____ **Matrix Blank:** _____

Calibration Checks: _____

MS/MSD: _____

Data Status: _____

Ins 4/5/99 p. 2 of 2

000655

ias 11/05/99 p. 1 of 4

Quantify Compound Summary Report

TOX145-rat sera, day 29 dose and control- ext. 11/3/99 MCH

Sample List: C:\MASSLYMX\comp020199.pro\SampleDB\110499a

Last modified: Fri Nov 05 06:30:18 1999

Method: C:\MASSLYMX\comp020199.pro\MethDB\110499a

Last modified: Fri Nov 05 06:29:44 1999

Job Code:

Printed: Fri Nov 05 06:52:31 1999

Compound 1: POAA (Ch1)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1 110499001	MeOH Blank							
2 110499002	MeOH Blank							
3 110499003	MeOH Blank							
4 110499004	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH			7.067	102	bb	1.49	49
5 110499005	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.063	115	bb	1.67	67
6 110499006	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.102	94	bb	1.37	37
7 110499007	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.128	85	bb	1.36	36
8 110499008	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.128	85	bb	1.24	24
9 110499009	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.128	83	bb	1.20	20
10 110499010	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.111	73	bb	1.07	7
11 110499011	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.112	73	bb	1.06	6
12 110499012	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.151	65	bb	0.95	-5
13 110499013	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.127	82	bb	1.19	19
14 110499014	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.113	88	bb	1.28	28
15 110499015	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.090	103	bb	1.50	50
16 110499016	MeOH Blank							
17 110499017	TOX145-RBS11039-250ppb-M89, ext. 11/3/99 MCH			7.111	56	bb	0.82	-18
18 110499018	TOX145-RBS11039-250ppb-M8D10, ext. 11/3/99 MCH			7.083	44	bb	0.64	-36
19 110499019	TOX145-RBS11039-250ppb-M8D10, ext. 11/3/99 MCH			7.131	71	bb	1.04	4
20 110499020	TOX145-RBS11039-250ppb-M8D10, ext. 11/3/99 MCH			7.129	57	bb	0.82	-18
21 110499021	TOX145-RBS11039-250ppb-M8D10, ext. 11/3/99 MCH			7.148	55	bb	0.80	-20
22 110499022	TOX145-RBS11039-250ppb-M8D10, ext. 11/3/99 MCH	QC	1.00	7.132	43	bb	0.63	-37
23 110499023	MeOH Blank							
24 110499024	TOX145-RBS11039-250ppb-M8D10, ext. 11/3/99 MCH			7.150	54	bb	0.78	-22
25 110499025	TOX145-RBS11039-250ppb-M8D10, ext. 11/3/99 MCH			7.134	58	bb	0.85	-15
26 110499026	TOX145-RBS11039-250ppb-M8D10, ext. 11/3/99 MCH			7.151	56	bb	0.82	-18
27 110499027	TOX145-RBS11039-250ppb-M8D10, ext. 11/3/99 MCH			7.135	113	bb	1.65	65
28 110499028	TOX145-RBS11039-250ppb-M8D10, ext. 11/3/99 MCH			7.127	93	bb	1.36	36
29 110499029	TOX145-RBS11039-250ppb-M8D10, ext. 11/3/99 MCH	QC	1.00	7.086	61	bb	0.89	-11
30 110499030	MeOH Blank							
31 110499031	TOX145-RBS11039-250ppb-M8D10, ext. 11/3/99 MCH			7.108	112	bb	1.63	63
32 110499032	TOX145-RBS11039-250ppb-M8D10, ext. 11/3/99 MCH			7.089	132	bb	1.92	92
33 110499033	TOX145-RBS11039-250ppb-M8D10, ext. 11/3/99 MCH	QC	1.00	7.106	48	bb	0.69	-31
34 110499034	MeOH Blank							
35 110499035	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH			7.127	90	bb	1.31	31
36 110499036	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.159	52	bb	0.76	-24
37 110499037	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.197	46	bb	0.68	-32
38 110499038	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.131	36	bb	0.52	-48
39 110499039	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.107	39	bb	0.57	-43
40 110499040	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.150	45	bb	0.65	-35
41 110499041	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.085	47	bb	0.69	-31
42 110499042	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.105	50	bb	0.73	-27
43 110499043	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.111	52	bb	0.75	-25
44 110499044	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.131	59	bb	0.86	-14
45 110499045	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.110	70	bb	1.03	3
46 110499046	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.136	92	bb	1.34	34

Quantify Compound Summary Report
TOX145-rat sera, day 29 dose and control- ext. 11/3/99 MCH

Page 2

Sample List: C:\MASSLYNK\scup020199.pro\SampleDB\110499a
Last modified: Fri Nov 05 06:30:18 1999
Method: C:\MASSLYNK\scup020199.pro\MethDB\110499a
Last modified: Fri Nov 05 06:29:44 1999
Job Code:

Printed: Fri Nov 05 06:52:31 1999

Compound 2: PFOS (Ch2)

#	Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1	110499001	MeOH Blank			7.220	53	bb	0.00	
2	110499002	MeOH Blank			7.230	41	MM	0.00	
3	110499003	MeOH Blank			7.291	26	bb	0.00	
4	110499004	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH			7.206	108	bb	0.00	
5	110499005	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.180	72	bb	0.00	
6	110499006	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.242	84	MM	0.00	
7	110499007	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	Standard	4.93	7.267	208	bbX	4.89	-1
8	110499008	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	Standard	9.76	7.267	293	bb	9.02	-8
9	110499009	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	Standard	24.80	7.245	527	bb	20.47	-17
10	110499010	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	Standard	49.30	7.250	995	MM	43.28	-12
11	110499011	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	Standard	97.60	7.251	1921	bb	88.51	-9
12	110499012	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	Standard	248.00	7.291	4504	bd	214.64	-13
13	110499013	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	Standard	493.00	7.244	8983	MM	433.34	-12
14	110499014	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	Standard	735.00	7.252	13842	MM	670.62	-9
15	110499015	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	976.00	7.228	17240	bdX	836.53	-14
16	110499016	MeOH Blank			7.253	193	bb	4.12	
17	110499017	TOX145-RBS11039-250ppb-MS9, ext. 11/3/99 MCH			7.250	3926	bd	186.40	
18	110499018	TOX145-RBS11039-250ppb-MSD10, ext. 11/3/99 MCH			7.222	3634	bb	172.14	
19	110499019	TOX145-RBS11039-250ppb-MSD10, ext. 11/3/99 MCH			7.270	57	bb	0.00	
20	110499020	TOX145-RBS11039-250ppb-MSD10, ext. 11/3/99 MCH			7.268	54	MM	0.00	
21	110499021	TOX145-RBS11039-250ppb-MSD10, ext. 11/3/99 MCH			7.288	51	bb	0.00	
22	110499022	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH	QC	248.00	7.248	4344	MM	206.81	-17
23	110499023	MeOH Blank			7.289	81	bb	0.00	
24	110499024	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH			7.291	39	bb	0.00	
25	110499025	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH			7.251	40	bb	0.00	
26	110499026	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH			7.268	52663	bb	2566.17	
27	110499027	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH			7.275	58663	bb	2859.14	
28	110499028	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH			7.267	61721	bb	3008.48	
29	110499029	TOX145-RBS11039-250ppb-CCV10, ext. 11/3/99 MCH	QC	248.00	7.225	4581	bb	218.39	-12
30	110499030	MeOH Blank			7.271	133	MM	1.19	
31	110499031	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH			7.246	56708	bb	2763.69	
32	110499032	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH			7.251	98061	bb	4782.90	
33	110499033	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH	QC	248.00	7.224	4843	bd	231.17	-7
34	110499034	MeOH Blank			7.308	182	bb	3.59	
35	110499035	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH			7.266	87	bb	0.00	
36	110499036	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.298	61	bb	0.00	
37	110499037	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.314	107	bb	0.00	
38	110499038	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	Standard	4.93	7.248	183	MMX	3.65	-26
39	110499039	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	Standard	9.76	7.247	326	bb	10.65	9
40	110499040	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	Standard	24.80	7.289	656	bb	26.74	8
41	110499041	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	Standard	49.30	7.224	1268	bb	56.64	15
42	110499042	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	Standard	97.60	7.244	2489	bb	116.27	19
43	110499043	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	Standard	248.00	7.271	5714	bd	273.70	10
44	110499044	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	Standard	493.00	7.269	11073	bd	535.41	9
45	110499045	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	Standard	735.00	7.250	16812	bb	815.64	11
46	110499046	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	976.00	7.277	22935	bdX	1114.59	14

Entered UC 11/05/99
need 10x dilution
k'n 11/5/99

Quantify Compound Summary Report
TOX145-rat sera, day 29 dose and control- ext. 11/3/99 MCH

Page 3

Sample List: C:\MASSLYNK\scup020199.pro\SampleDB\110499a
Last modified: Fri Nov 05 06:30:16 1999
Method: C:\MASSLYNK\scup020199.pro\MathDB\110499a
Last modified: Fri Nov 05 06:29:44 1999
Job Code:

Printed: Fri Nov 05 06:52:31 1999

Compound 3: PFOSA (Ch3)

#	Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	hDev
1	110499001	MeOH Blank			7.805	236	MM	0.00	
2	110499002	MeOH Blank			7.815	840	MM	0.80	
3	110499003	MeOH Blank			7.809	522	MM	0.00	
4	110499004	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH			7.769	363	MM	0.00	
5	110499005	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.805	409	MM	0.00	
6	110499006	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.871	544	bb	0.00	
7	110499007	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	Standard	4.93	7.850	1849	db	4.91	-0
8	110499008	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	Standard	9.77	7.828	3250	db	10.61	9
9	110499009	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	Standard	24.80	7.875	7052	db	26.09	5
10	110499010	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	Standard	49.30	7.857	13252	db	51.32	4
11	110499011	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	Standard	97.70	7.816	28632	MM	113.91	17
12	110499012	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	Standard	248.00	7.853	66768	MM	269.12	9
13	110499013	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	Standard	493.00	7.875	125323	MM	507.43	3
14	110499014	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	Standard	736.00	7.837	187764	MM	761.56	3
15	110499015	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	977.00	7.835	241229	MM	979.16	0
16	110499016	MeOH Blank			7.860	1516	MM	3.55	
17	110499017	TOX145-RBS11039-250ppb-M89, ext. 11/3/99 MCH			7.832	61127	MM	246.16	
18	110499018	TOX145-RBS11039-250ppb-M810, ext. 11/3/99 MCH			7.808	60889	MM	245.19	
19	110499019	TOX145-RBS11039-250ppb-M810, ext. 11/3/99 MCH			7.855	674	MM	0.13	
20	110499020	TOX145-RBS11039-250ppb-M810, ext. 11/3/99 MCH			7.853	600	MM	0.00	
21	110499021	TOX145-RBS11039-250ppb-M810, ext. 11/3/99 MCH			7.847	417	MM	0.00	
22	110499022	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH	QC	248.00	7.811	58654	MM	236.10	-5
23	110499023	MeOH Blank			7.872	541	MM	0.00	
24	110499024	TOX145-RBS11039-250ppb-M810, ext. 11/3/99 MCH			7.874	370	MM	0.00	
25	110499025	TOX145-RBS11039-250ppb-M810, ext. 11/3/99 MCH			7.859	323	MM	0.00	
26	110499026	TOX145-RBS11039-250ppb-M810, ext. 11/3/99 MCH			7.875	677	db	0.14	
27	110499027	TOX145-RBS11039-250ppb-M810, ext. 11/3/99 MCH			7.926	939	MM	1.20	
28	110499028	TOX145-RBS11039-250ppb-M810, ext. 11/3/99 MCH			7.874	911	db	1.09	
29	110499029	TOX145-RBS11039-250ppb-CCV10, ext. 11/3/99 MCH	QC	248.00	7.831	55323	MM	222.54	-10
30	110499030	MeOH Blank			7.878	286	MM	0.00	
31	110499031	TOX145-RBS11039-250ppb-M810, ext. 11/3/99 MCH			7.831	929	MM	1.16	
32	110499032	TOX145-RBS11039-250ppb-M810, ext. 11/3/99 MCH			7.856	1875	db	5.01	
33	110499033	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH	QC	248.00	7.832	53678	MM	215.85	-13
34	110499034	MeOH Blank			7.938	160	MM	0.00	
35	110499035	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH			7.873	229	MM	0.00	
36	110499036	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.948	240	MM	0.00	
37	110499037	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.876	283	db	0.00	
38	110499038	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	Standard	4.93	7.810	1629	MM	4.01	-19
39	110499039	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	Standard	9.77	7.808	2912	MM	9.23	-5
40	110499040	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	Standard	24.80	7.918	6272	MM	22.91	-8
41	110499041	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	Standard	49.30	7.876	11757	MM	45.23	-8
42	110499042	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	Standard	97.70	7.872	24931	MM	98.85	1
43	110499043	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	Standard	248.00	7.845	60206	MM	242.42	-2
44	110499044	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	Standard	493.00	7.809	122823	MM	497.26	1
45	110499045	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	Standard	736.00	7.879	172738	MM	700.41	-5
46	110499046	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	977.00	7.861	230763	MM	936.56	-4

Entered LAC
11/05/99

Quantify Compound Summary Report

TOX145-rat sera, day 29 dose and control- ext. 11/3/99 MCH

Page 4

Sample List: C:\MASSLYNK\acup020199.pro\SampleDB\110499a
 Last modified: Fri Nov 05 06:30:18 1999
 Method: C:\MASSLYNK\acup020199.pro\MethDB\110499a
 Last modified: Fri Nov 05 06:29:44 1999
 Job Code:

Printed: Fri Nov 05 06:52:31 1999

Compound 4: Surr (Ch4)

#	Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1	110499001	MeOH Blank			7.075		14 NM	0.01	-99
2	110499002	MeOH Blank							
3	110499003	MeOH Blank							
4	110499004	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH			7.085	2657	bb	1.23	23
5	110499005	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.058	2772	bb	1.29	29
6	110499006	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.097	2201	bb	1.02	2
7	110499007	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.122	2219	bb	1.03	3
8	110499008	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.123	2541	bd	1.18	18
9	110499009	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.123	2030	bb	1.12	12
10	110499010	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.128	2372	bb	0.94	-6
11	110499011	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.105	2030	bb	1.10	10
12	110499012	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.168	2178	bb	1.01	1
13	110499013	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.121	2352	bb	1.09	9
14	110499014	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.130	2306	bb	1.07	7
15	110499015	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.106	2191	bb	1.02	2
16	110499016	MeOH Blank			7.131	10	bb	0.00	-100
17	110499017	TOX145-RBS11039-250ppb-MS9, ext. 11/3/99 MCH			7.106	1907	bb	0.89	-11
18	110499018	TOX145-RBS11039-250ppb-MSD10, ext. 11/3/99 MCH			7.077	1730	bb	0.80	-20
19	110499019	TOX145-RBS-9R03240M-0-Day 29, ext. 11/3/99 MCH			7.125	1490	bb	0.69	-31
20	110499020	TOX145-RBS-9R03241M-0-Day 29, ext. 11/3/99 MCH			7.146	2231	bb	1.04	4
21	110499021	TOX145-RBS-9R03242M-0-Day 29, ext. 11/3/99 MCH			7.143	2377	bb	1.10	10
22	110499022	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH	QC	1.00	7.126	2260	bb	1.05	5
23	110499023	MeOH Blank			7.167	11	MM	0.01	-99
24	110499024	TOX145-RBS-9R03243M-0-Day 29, ext. 11/3/99 MCH			7.167	2212	bb	1.03	3
25	110499025	TOX145-RBS-9R03243M-0-Day 29, ext. 11/3/99 MCH			7.128	2451	bd	1.14	14
26	110499026	TOX145-RBS-9R03255M-5mg/kg-Day29, ext. 11/3/99 MCH			7.124	2005	bb	0.93	-7
27	110499027	TOX145-RBS-9R03256M-5mg/kg-Day29, ext. 11/3/99 MCH			7.129	2380	bb	1.11	11
28	110499028	TOX145-RBS-9R03257M-5mg/kg-Day29, ext. 11/3/99 MCH			7.122	2507	bb	1.16	16
29	110499029	TOX145-RBS11039-250ppb-CCV10, ext. 11/3/99 MCH	QC	1.00	7.080	1986	bb	0.92	-8
30	110499030	MeOH Blank							
31	110499031	TOX145-RBS-9R03258M-5mg/kg-Day29, ext. 11/3/99 MCH			7.102	2213	bb	1.03	3
32	110499032	TOX145-RBS-9R03259M-5mg/kg-Day29, ext. 11/3/99 MCH			7.105	2002	bb	0.93	-7
33	110499033	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH	QC	1.00	7.100	2025	bb	0.94	-6
34	110499034	MeOH Blank			7.163	9	bb	0.00	-100
35	110499035	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH			7.122	1877	MM	0.87	-13
36	110499036	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.154	2008	bb	0.93	-7
37	110499037	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.191	1671	bb	0.78	-22
38	110499038	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.124	1925	bb	0.89	-11
39	110499039	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.102	2102	bd	0.98	-2
40	110499040	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.144	2068	bb	0.96	-4
41	110499041	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.080	1741	bb	0.81	-19
42	110499042	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.099	2120	bb	0.98	-2
43	110499043	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.127	2076	bd	0.96	-4
44	110499044	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.126	2239	bb	1.04	4
45	110499045	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.105	2002	MM	0.93	-7
46	110499046	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.130	1894	bb	0.88	-12

Soup 020199: Quantitated and printed by ias on 11/05/99

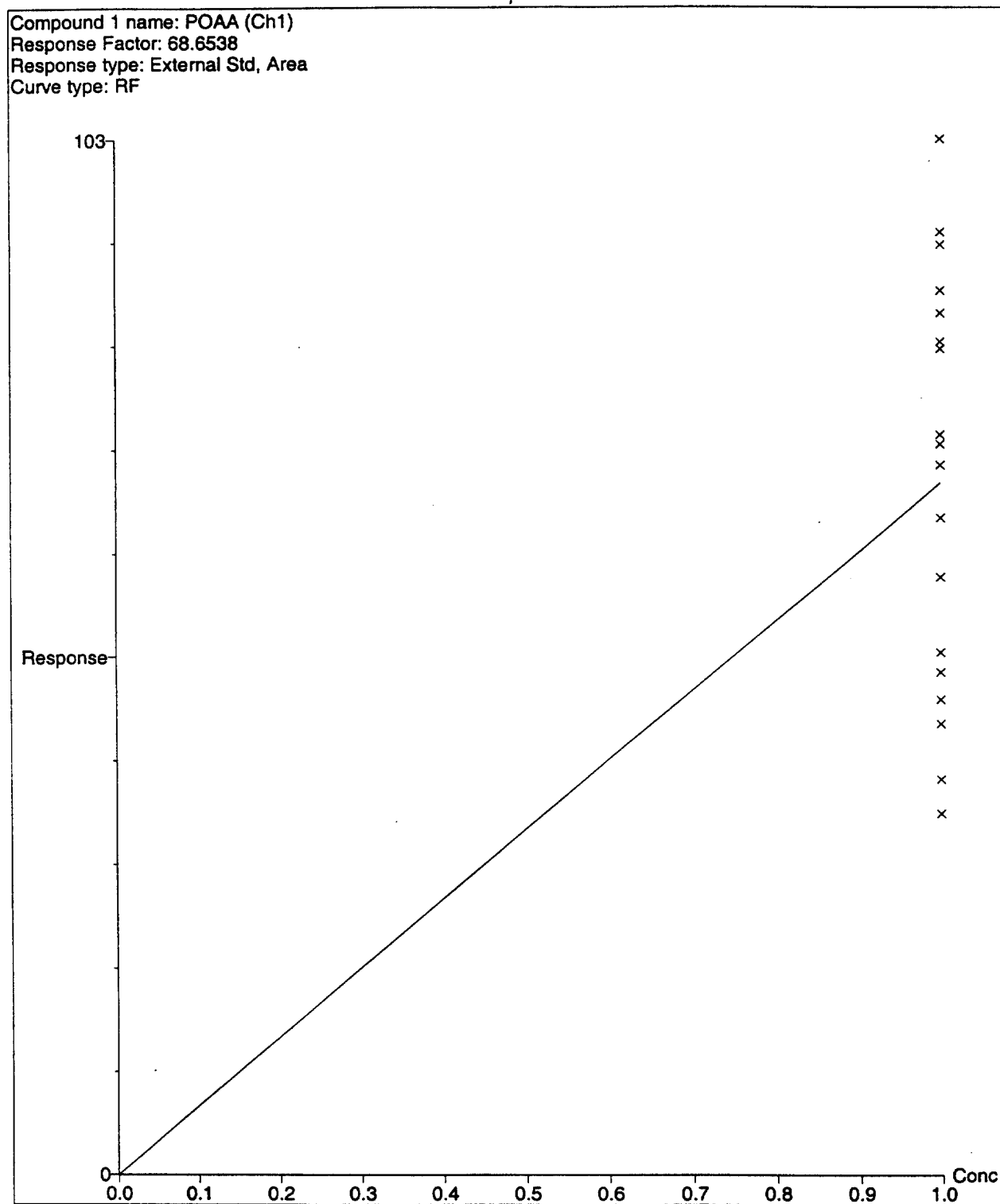
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IAS 11/05/99 p 1 of 4

Quantify Calibration Report
TOK145-rat sera, day 29 dose and control- ext. 11/3/99 MCH

Page 1

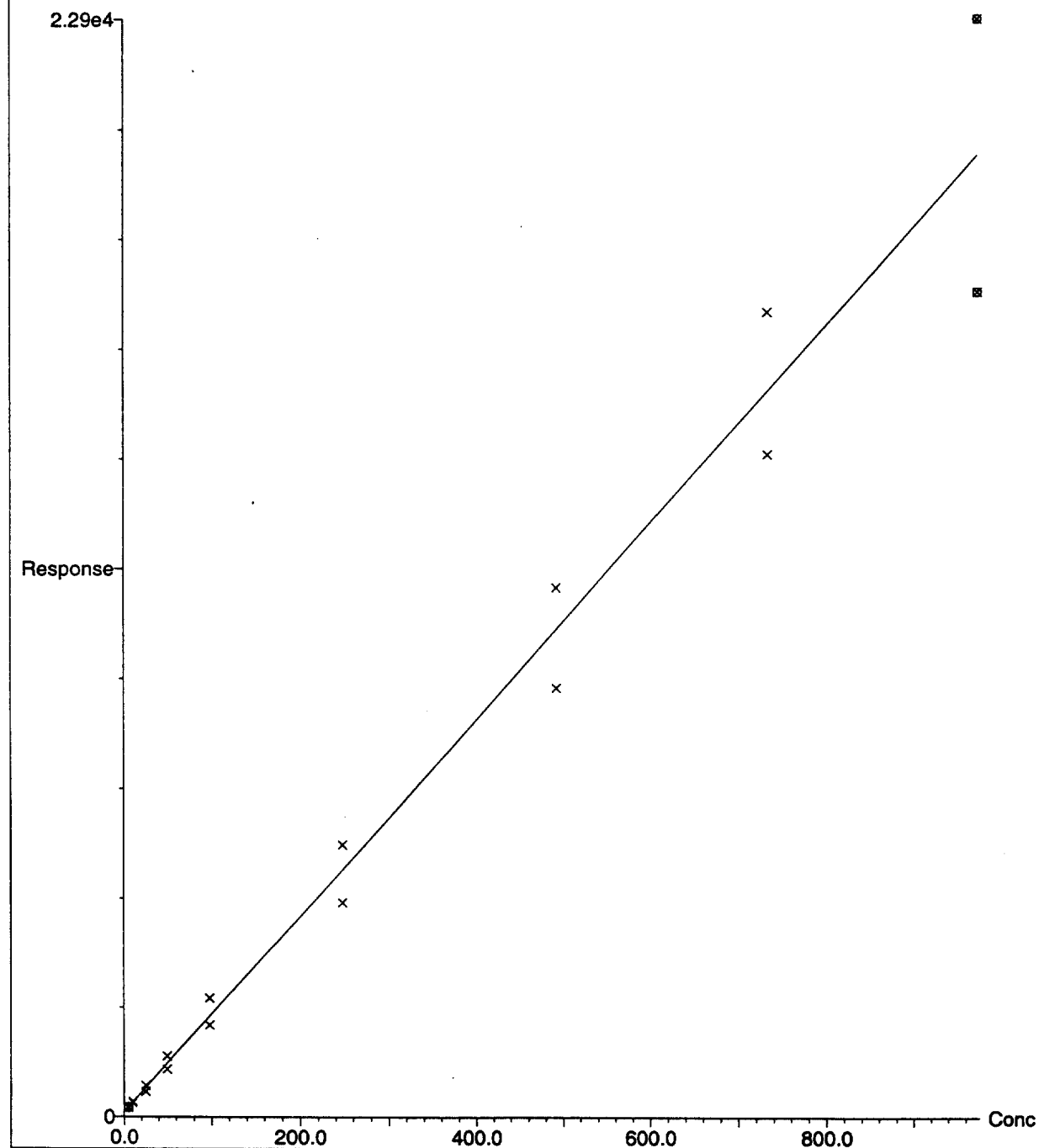
Compound 1 name: POAA (Ch1)
Response Factor: 68.6538
Response type: External Std, Area
Curve type: RF



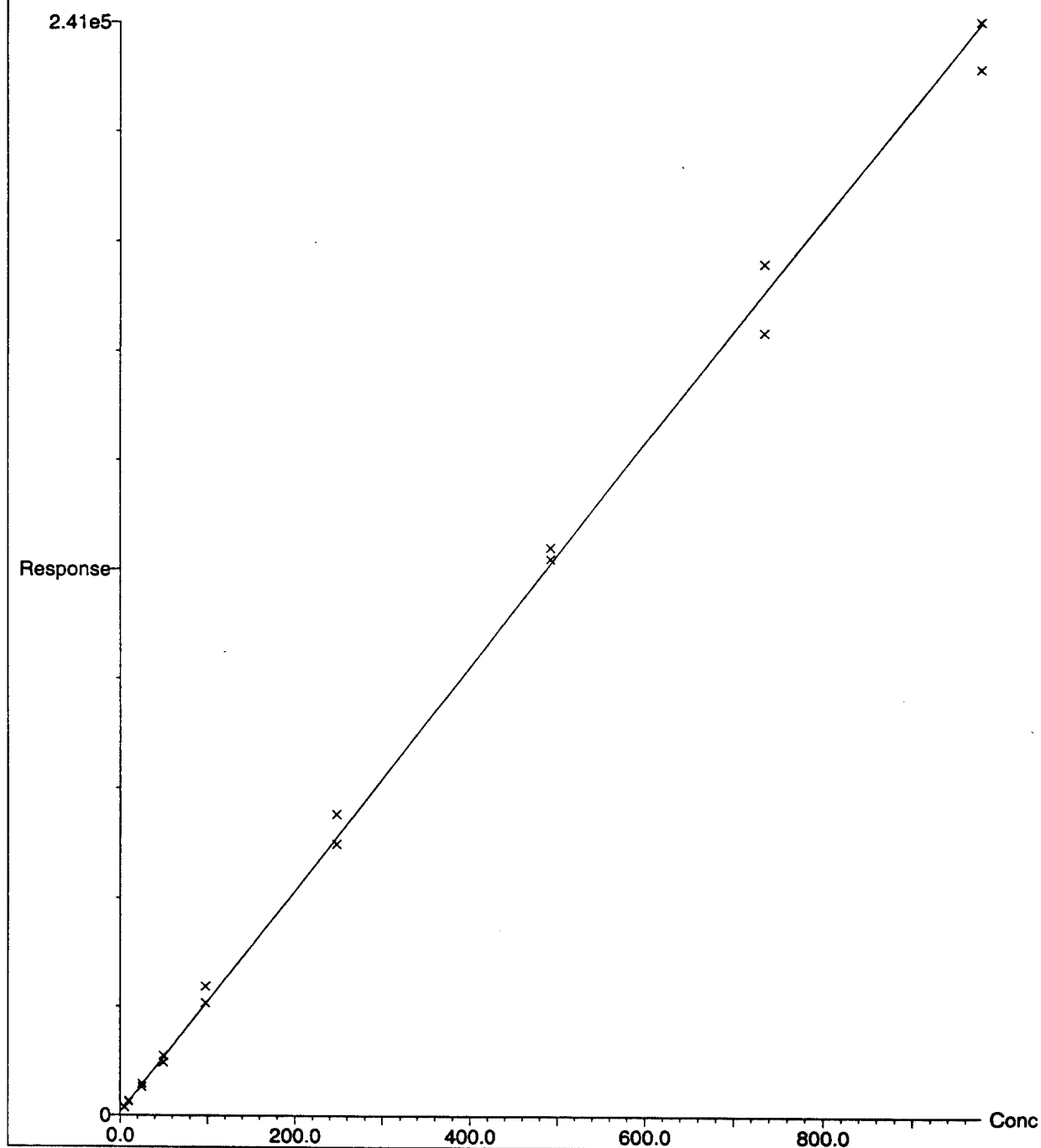
Scup 020199: Quantitated and printed by ias on 11/05/99

000660

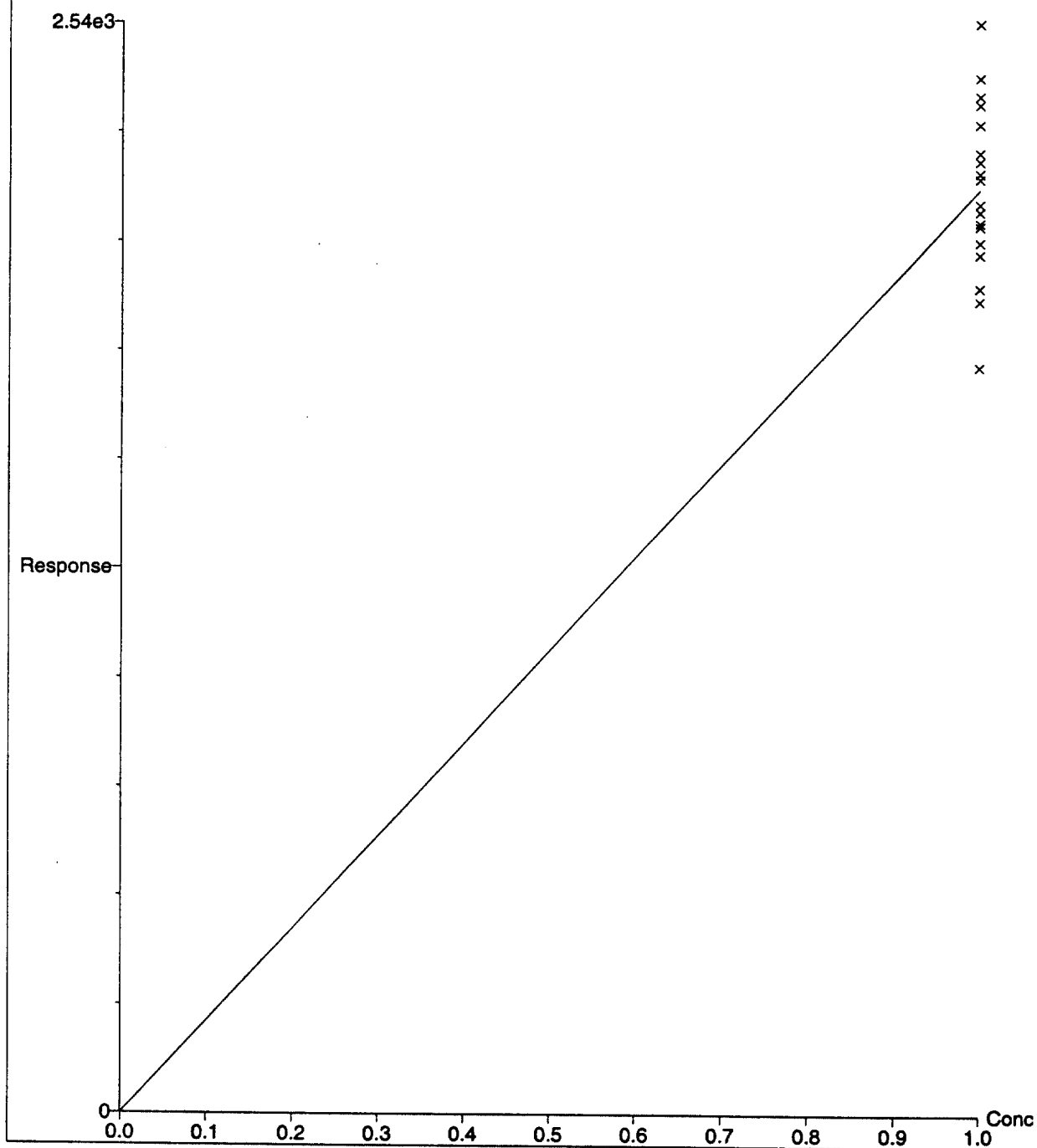
Compound 2 name: PFOS (Ch2)
Coefficient of Determination: 0.980777
Calibration curve: $20.4798 * x + 108.210$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound 3 name: PFOSA (Ch3)
Coefficient of Determination: 0.997747
Calibration curve: $245.706 * x + 643.062$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound 4 name: Surr (Ch4)
Response Factor: 2153.97
Response type: External Std, Area
Curve type: RF



Jas #105/69 p 1 of 4

Page 1

Method Report

Method file: C:\MASSLYNX\scup020199.pro\MethDB\110499a.mdb
Last modified: Fri Nov 05 06:29:44 1999

Compound: 1: POAA (Ch1)

Printed: Fri Nov 05 06:56:56 1999

Compound Name: POAA (Ch1)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Fixed
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.167, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.345

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 81

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 30.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000664

Method Report

Page 2

Method file: C:\MASSLYNX\scup020199.pro\MethDB\110499a.mdb
Last modified: Fri Nov 05 06:29:44 1999

Compound: 2: PFOS (Ch2)

Printed: Fri Nov 05 06:56:56 1999

Compound Name: PFOS (Ch2)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. A
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.429, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.467

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 81

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000665

Method Report

Page 3

Method file: C:\MASSLYNX\scup020199.pro\MethDB\110499a.mdb
Last modified: Fri Nov 05 06:29:44 1999

Compound: 3: PFOSA (Ch3)

Printed: Fri Nov 05 06:56:56 1999

Compound Name: PFOSA (Ch3)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. B
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 8.147, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.556

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 81

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000666

Method Report

Page 4

Method file: C:\MASSLYNX\scup020199.pro\MethDB\110499a.mdb
Last modified: Fri Nov 05 06:29:44 1999
Compound: 4: Surr (Ch4)
Printed: Fri Nov 05 06:56:56 1999

Compound Name: Surr (Ch4)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Fixed
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.218, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.379

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 81

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000667

Jas 11/05/99

SOUP 020199: FACT Equipment
Log 99092 PAGE 1/1

Quattro II, #6414E

DATE: 11-04-99

ANALYST: jas

STUDY NUMBER: TOX145 MATRIX: rat sera DESCRIPTION: day 29- dose & control

FACT Method: ETS-8-5.1

Injection volume: 10 uL Cycle time: 10 min Electrode: Z-Spray

Mobile Phase: (A) 2.0 mM NH₄CH₃CO₂ (B) MeOH

TN-A- or stdrd number (A) 99085-11-15 (B) TN-A-2997

Prepared (date/by): (A) 10/30/99 sah (B) 10/30/99 sah

Expires: (A) 04/00 (B) 2010

Store at room temp? ☒ Y or N Other: _____

Guard column: None

Part number: na Lot number: na

Analytical column: Keystone Betasil

Size: 2 x 50 mm Type: C18 Sphere size: 5 um

Part number: 055-701-2 Lot number: P9E11 Serial number: 0591804Z

Analytical parameters:

Drying gas (L/hr): 350 ESI nebulizing gas (L/hr) 13 Sheath gas (L/hr) 0

LC Flow rate: 300 uL/min Back pressure: 65 bar

HPLC solvent gradient: 10min-10-95

Time (min.)	%B
1) <u>0.00</u>	<u>10.0</u>
2) <u>1.00</u>	<u>10.0</u>
3) <u>5.50</u>	<u>95.0</u>
4) <u>7.50</u>	<u>95.0</u>
5) <u>8.00</u>	<u>10.0</u>

000668

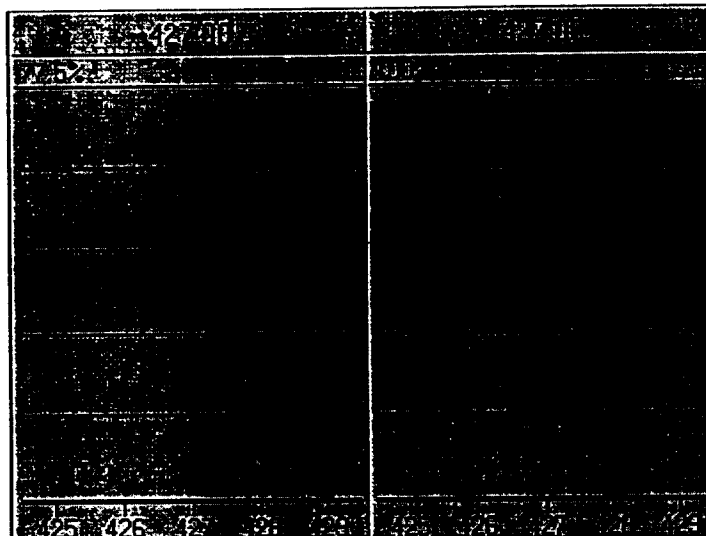
BEST COPY AVAILABLE

Tuning Method Report

Page 1

Method: C:\MASSLYNX\SOUP020199.PRO\ACQUDB\PFOS3

Printed: Thu Nov 04 14:02:27 1999



MS

MS2

SOURCE (ESP-)

Capillary
Cone
Extractor
RF Lens
Source Block Temp.
Desolvation Temp.

Set	Rdbk
1.60	1.62
40	-41
5	-7
0.2	
150	150
250	250

MS1

LM Resolution
HM Resolution
Ion Energy
Ion Energy Ramp
Lens 6
Multiplier

Set	Rdbk
14.0	
14.0	
0.9	
0.0	
4	3
650	

Pressures

Analyser Vacuum
Gas Cell

Rdbk
5.1e-5
4.0e-3

MS2

LM Resolution
HM Resolution
Collision
Ion Energy
Ion Energy Ramp
Lens 8
Lens 9
Multiplier

Set	Rdbk
14.0	
14.0	
10	7
2.0	
0.0	
40	36
0	1
650	

000669

Scanning Method Report

Page 1

Method: C:\MSASLYNK\SCUP020199.FPO\ACQDS\10MIN-PP0S-FOAA-PP0SA-IS
Last Modified: Fri Oct 15 10:35:03 1999
Printed: Thu Nov 04 13:18:28 1999

Solvent Delay (mins) : 0.00

Function : 1 MSN of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03
Span (Daltons) : 0.00
Start Time (Mins) : 0.00
End Time (Mins) : 10.00
Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	413.00	169.00	0.30	30	15

Function : 2 MSN of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03
Span (Daltons) : 0.00
Start Time (Mins) : 0.00
End Time (Mins) : 10.00
Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	499.00	99.00	0.30	45	60

Function : 3 MSN of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03
Span (Daltons) : 0.00
Start Time (Mins) : 0.00
End Time (Mins) : 10.00
Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	498.00	78.00	0.30	45	60

Function : 4 MSN of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03
Span (Daltons) : 0.00
Start Time (Mins) : 0.00
End Time (Mins) : 10.00
Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	427.00	80.00	0.30	40	60

000670

Method Report

Page 1

Method File: C:\MASSLYNX\SCUP020199.PRO\ACQUDE\10min-10-95
 Last Modified: Thursday, November 04, 1999 11:22:29
 Printed: Thursday, November 04, 1999 13:18:38

HP1100 LC Pump Initial Conditions

Solvents
 A% 90.0
 B% 10.0
 C% 0.0
 D% 0.0
 Flow (ml/min) 0.300
 Stop Time (mins) 10.0
 Min Pressure (bar) 0
 Max Pressure (bar) 400
 Oven Temperature Left(°C) 30.0
 Oven Temperature Right(°C) 30.0

HP1100 LC Pump Gradient Timetable

The gradient Timetable contains 5 entries which are :

Time	A%	B%	C%	D%	Flow	Pressure
0.00	90.0	10.0	0.0	0.0	0.300	400
1.00	90.0	10.0	0.0	0.0	0.300	400
5.50	5.0	95.0	0.0	0.0	0.300	400
7.50	5.0	95.0	0.0	0.0	0.300	400
8.00	90.0	10.0	0.0	0.0	0.300	400

HP1100 LC Pump External Event Timetable

The Timetable contains 3 entries which are :

Time	Column Switch	Contact1	Contact2	Contact3	Contact4
Initial	Off	Off	Off	Off	Off
0.00	Off	On	On	Off	On
0.10	Off	Off	Off	Off	Off

HP1100 Autosampler Initial Conditions

Draw Speed 200.0
 Eject Speed (µl/min) 200
 Draw Position (mm) 0.00
 Stop Time (mins) 9.00
 Injection Volume (µl) 10.0
 Vial Number 51

000671

Sample List: C:\MASSLYNX\soup020199.pro\SampleDB\110499a.SPL
Printed: Thu Nov 04 13:18:22 1999

Page Position: (1, 1)

	File Name	File Text	MS File
1	110499001	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
2	110499002	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
3	110499003	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
4	110499004	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
5	110499005	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
6	110499006	TOX145-RTS11039-Sera Blk-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
7	110499007	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
8	110499008	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
9	110499009	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
10	110499010	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
11	110499011	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
12	110499012	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
13	110499013	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
14	110499014	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
15	110499015	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
16	110499016	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
17	110499017	TOX145-RTS11039-250ppb-MS9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
18	110499018	TOX145-RTS11039-250ppb-MSD10, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
19	110499019	TOX145-RTS-9R03240M-0-Day 29, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
20	110499020	TOX145-RTS-9R03241M-0-Day 29, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
21	110499021	TOX145-RTS-9R03242M-0-Day 29, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
22	110499022	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
23	110499023	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
24	110499024	TOX145-RTS-9R03243M-0-Day 29, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
25	110499025	TOX145-RTS-9R03243M-0-Day 29, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
26	110499026	TOX145-RTS-9R03255M-5mg/kg-Day29, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
27	110499027	TOX145-RTS-9R03256M-5mg/kg-Day29, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
28	110499028	TOX145-RTS-9R03257M-5mg/kg-Day29, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
29	110499029	TOX145-RBS11039-250ppb-CCV10, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
30	110499030	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
31	110499031	TOX145-RTS-9R03258M-5mg/kg-Day29, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
32	110499032	TOX145-RTS-9R03259M-5mg/kg-Day29, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
33	110499033	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
34	110499034	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
35	110499035	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
36	110499036	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
37	110499037	TOX145-RTS11039-Sera Blk-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
38	110499038	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
39	110499039	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
40	110499040	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
41	110499041	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
42	110499042	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
43	110499043	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
44	110499044	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
45	110499045	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS
46	110499046	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS

Masslynx - Sample List

Page 2

Sample List: C:\MASSLYNX\soup020199.pro\SampleDB\110499a.SPL
Printed: Thu Nov 04 13:18:22 1999

Page Position: (2, 1)

	Inlet File	Bottle	Inject Volume	Sample Type	Conc A	Conc B	Conc C
1	10min-10-95	91	10	--	--	--	--
2	10min-10-95	91	10	--	--	--	--
3	10min-10-95	91	10	--	--	--	--
4	10min-10-95	1	10	--	--	--	--
5	10min-10-95	2	10	--	--	--	--
6	10min-10-95	3	10	--	--	--	--
7	10min-10-95	4	10	--	--	--	--
8	10min-10-95	5	10	--	--	--	--
9	10min-10-95	6	10	--	--	--	--
10	10min-10-95	7	10	--	--	--	--
11	10min-10-95	8	10	--	--	--	--
12	10min-10-95	9	10	--	--	--	--
13	10min-10-95	10	10	--	--	--	--
14	10min-10-95	11	10	--	--	--	--
15	10min-10-95	12	10	--	--	--	--
16	10min-10-95	92	10	--	--	--	--
17	10min-10-95	13	10	--	--	--	--
18	10min-10-95	14	10	--	--	--	--
19	10min-10-95	15	10	--	--	--	--
20	10min-10-95	16	10	--	--	--	--
21	10min-10-95	17	10	--	--	--	--
22	10min-10-95	25	10	--	--	--	--
23	10min-10-95	92	10	--	--	--	--
24	10min-10-95	18	10	--	--	--	--
25	10min-10-95	19	10	--	--	--	--
26	10min-10-95	20	10	--	--	--	--
27	10min-10-95	21	10	--	--	--	--
28	10min-10-95	22	10	--	--	--	--
29	10min-10-95	26	10	--	--	--	--
30	10min-10-95	93	10	--	--	--	--
31	10min-10-95	23	10	--	--	--	--
32	10min-10-95	24	10	--	--	--	--
33	10min-10-95	25	10	--	--	--	--
34	10min-10-95	93	10	--	--	--	--
35	10min-10-95	1	10	--	--	--	--
36	10min-10-95	2	10	--	--	--	--
37	10min-10-95	3	10	--	--	--	--
38	10min-10-95	4	10	--	--	--	--
39	10min-10-95	5	10	--	--	--	--
40	10min-10-95	6	10	--	--	--	--
41	10min-10-95	7	10	--	--	--	--
42	10min-10-95	8	10	--	--	--	--
43	10min-10-95	9	10	--	--	--	--
44	10min-10-95	10	10	--	--	--	--
45	10min-10-95	11	10	--	--	--	--
46	10min-10-95	12	10	--	--	--	--

000673

FACT: DATA REVIEW SUMMARY FORM

Date: 11/08/99
 Data Analyst: JAS
 FACT Study #: Tox 145
 Matrix/Timepoints: Ref Sam - day 29 control / dose - oct 11/3/99
 Sample List: 1105996
 Set-up Analysis
 Date/Analyst: MMH 11/05/99
 Instrument: Samp 020199

Data Review Sign-Off

Analyst Review
 Name / Date: JAS 11/05/99

Technical Review
 Name / Date: MMH 11/8/99 / kln NOV-8-99

Quality Assurance Review
 Name / Date: _____

Data Entry: GML 11/8/99

Comments / Justifications: _____

Analyte: POAA (CH1) ①
 Acceptable Linear Range: NA
 LOQ: NA Extraction Blank: NA
 R²: NA Matrix Blank: NA
 Calibration Checks: NA
 MS/MSD: N/A
 Data Status: DATA NOT reported 3 GML 11/8/99

Analyte: PFOS (CH2) ②
 Acceptable Linear Range: 24.80 ppb → 976 ppb
 LOQ: 24.80 ppb Extracted Blank: less than LOQ
 R²: 0.999549 Matrix Blank: less than LOQ
 Calibration Checks: OK - (-6%)
 MS/MSD: N/A
 Data Status: Reported Data for Gp. 6, Du. 110 GML 11/8/99

Analyte: PFOSA (CH3)
 Acceptable Linear Range: 4.93 ppb → 977 ppb
 LOQ: 4.93 ppb Extracted Blank: less than LOQ
 R²: 0.999708 Matrix Blank: less than LOQ
 Calibration Checks: OK → -5%
 MS/MSD: N/A
 Data Status: Data not reported 3 GML 11/10/99

① A calibration curve was not run for POAA. Data is useful for area counts and retention times only. - JAS 11/08/99.

② Low curve points 7.93 ppb and 9.76 ppb were excluded as they were not within the ± 30% accuracy requirements of the method when the curves were evaluated over a linear range appropriate to the data. - JAS 11/08/99

③ Internal std area counts on samples were low due to the dilution factor. - JAS 11/08/99.

GML
11/08/99

JAS 11/08/99 p. 1 of 2

000674

FACT: DATA REVIEW SUMMARY FORM

Data Review Sign-Off

Date: 11/08/99
 Data Analyst: IAS
 FACT Study #: Tox MS
 Matrix/Timepoints: Ref Sera - day 29 dose & control - ext 11/3/99
 Sample List: 1105996
 Set-up Analysis
 Date/Analyst: MMH 11/05/99
 Instrument: Sep 020199

Analyst Review
 Name / Date: IAS 11/08/99

Technical Review
 Name / Date: Refer to page one LAC 11/9/99

Quality Assurance Review
 Name / Date: _____

Data Entry: Refer to page one LAC 11/9/99

Comments / Justifications: _____

Analyte: Sarr (CH4) ③ P. 141
 Acceptable Linear Range: NA
 LOQ: NA Extraction Blank: NA
 R²: NA Matrix Blank: NA
 Calibration Checks: NA
 MS/MSD: _____
 Data Status: _____

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: IAS 11/08/99
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: _____

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: _____

Gal
11/08/99

IAS 11/08/99 p. 2 of 2

000675

IAS 4/08/99 p. 1 of 4

Quantify Compound Summary Report
TOX145-day29 control and dose- ext. 11/3/99

Page 1

Sample List: C:\MASSELYNK\soup020199.pro\SampleDB\110599b
Last modified: Fri Nov 05 15:26:11 1999
Method: C:\MASSELYNK\soup020199.pro\MethDB\110599b
Last modified: Mon Nov 08 07:09:58 1999
Job Code:

Printed: Mon Nov 08 07:28:42 1999

Compound 1: POAA (Ch1)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1 110599028	MeOH Blank			7.172	38	bb	0.49	-51
2 110599029	MeOH Blank			7.174	20	MM	0.35	-65
3 110599030	MeOH Blank			7.192	17	bb	0.29	-71
4 110599031	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH			7.177	69	bb	1.20	20
5 110599032	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.153	54	bb	0.93	-7
6 110599033	TOX145-RTS11039-Sera Blk-9, ext. 11/3/99 MCH			7.172	45	bb	0.78	-22
7 110599034	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.147	48	bb	0.84	-16
8 110599035	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.117	42	bb	0.74	-26
9 110599036	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.155	39	MM	0.67	-33
10 110599037	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.174	53	MM	0.92	-8
11 110599038	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.131	61	MM	1.06	6
12 110599039	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.106	63	MM	1.09	9
13 110599040	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.172	57	MM	1.00	-0
14 110599041	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.151	85	MM	1.48	48
15 110599042	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.152	87	bb	1.51	51
16 110599043	MeOH Blank							
17 110599044	TOX145-RTS-9R03255M-5mg/kg-Day29, D:10			7.170	10	MM	0.18	-82
18 110599045	TOX145-RTS-9R03256M-5mg/kg-Day29, D:10			7.195	16	bb	0.27	-73
19 110599046	TOX145-RTS-9R03257M-5mg/kg-Day29, D:10			7.173	21	MM	0.36	-64
20 110599047	TOX145-RTS-9R03258M-5mg/kg-Day29, D:10			7.175	32	MM	0.56	-44
21 110599048	TOX145-RTS-9R03259M-5mg/kg-Day29, D:10			7.192	17	MM	0.29	-71
22 110599049	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH	QC	1.00	7.196	45	bb	0.78	-22
23 110599050	MeOH Blank							
24 110599051	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH			7.133	67	bb	1.17	17
25 110599052	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.198	61	MM	1.05	5
26 110599053	TOX145-RTS11039-Sera Blk-9, ext. 11/3/99 MCH			7.133	43	MM	0.75	-25
27 110599054	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.172	41	bb	0.71	-29
28 110599055	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.147	44	MM	0.76	-24
29 110599056	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.141	45	MM	0.78	-22
30 110599057	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.171	38	bb	0.67	-33
31 110599058	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.146	51	MM	0.89	-11
32 110599059	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.152	58	MM	1.00	0
33 110599060	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.153	63	bb	1.10	10
34 110599061	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.174	77	MM	1.34	34
35 110599062	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.131	83	bb	1.44	44

Soup 020199: Quantitated and printed by ias on 11/08/99

000676

Quantify Compound Summary Report
TOX145-day29 control and dose- ext. 11/3/99

Page 2

Sample List: C:\MSASLYNK\scup020199.pro\SampleDB\110599b
Last modified: Fri Nov 05 15:26:11 1999
Method: C:\MSASLYNK\scup020199.pro\MethDB\110599b
Last modified: Mon Nov 08 07:09:58 1999
Job Code:

Printed: Mon Nov 08 07:28:42 1999

Compound 2: PFOS (Ch2)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc. uDev
1 110599028	MeOH Blank			7.288	452	bb	8.88
2 110599029	MeOH Blank			7.290	394	bb	6.89
3 110599030	MeOH Blank			7.309	386	bb	6.62
4 110599031	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH			7.316	159	bb	0.00
5 110599032	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.270	219	bb	0.96
6 110599033	TOX145-RTS11039-Sera Blk-9, ext. 11/3/99 MCH			7.288	255	bb	2.17
7 110599034	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	Standard	4.93	7.264	318	MMX	4.30 -13
8 110599035	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	Standard	9.76	7.255	641	MMX	15.30 57
9 110599036	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	Standard	24.80	7.294	927	bb	25.05 1
10 110599037	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	Standard	49.30	7.292	1581	bb	47.30 -4
11 110599038	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	Standard	97.60	7.271	3003	bb	95.70 -2
12 110599039	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	Standard	248.00	7.246	7723	bb	256.37 3
13 110599040	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	Standard	493.00	7.289	14580	bb	489.79 -1
14 110599041	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	Standard	735.00	7.290	21377	bb	721.17 -2
15 110599042	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	976.00	7.290	29202	bb	967.51 1
16 110599043	MeOH Blank			7.245	300	bb	1.70
17 110599044	TOX145-RTS-9R03255M-5mg/kg-Day29, D:10			7.310	10749	bb	359.37
18 110599045	TOX145-RTS-9R03256M-5mg/kg-Day29, D:10			7.335	11879	bb	397.84
19 110599046	TOX145-RTS-9R03257M-5mg/kg-Day29, D:10			7.311	12061	bb	404.03
20 110599047	TOX145-RTS-9R03258M-5mg/kg-Day29, D:10			7.314	12313	bb	412.64
21 110599048	TOX145-RTS-9R03259M-5mg/kg-Day29, D:10			7.331	21806	bb	735.75
22 110599049	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH	QC	248.00	7.313	7016	bb	232.31 -6
23 110599050	MeOH Blank			7.265	179	MM	0.00
24 110599051	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH			7.273	155	bb	0.00
25 110599052	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.337	169	bb	0.00
26 110599053	TOX145-RTS11039-Sera Blk-9, ext. 11/3/99 MCH			7.272	168	bb	0.00
27 110599054	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	Standard	4.93	7.290	298	MMX	1.62 -27
28 110599055	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	Standard	9.76	7.286	552	MMX	12.29 26
29 110599056	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	Standard	24.80	7.279	879	bb	23.42 -6
30 110599057	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	Standard	49.30	7.286	1703	bb	51.46 4
31 110599058	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	Standard	97.60	7.286	3319	bb	106.45 9
32 110599059	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	Standard	248.00	7.269	7091	bd	234.86 -5
33 110599060	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	Standard	493.00	7.293	14578	bb	489.73 -1
34 110599061	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	Standard	735.00	7.290	22027	bb	743.27 1
35 110599062	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	976.00	7.271	28844	bd	975.32 -0

Data Entered
GML 11/8/99

Quantify Compound Summary Report
TOX145-day29 control and dose- ext. 11/3/99

Page 3

Sample List: C:\MSAS\LYNK\comp020199.pro\SampleDB\110599b
Last modified: Fri Nov 05 15:26:11 1999
Method: C:\MSAS\LYNK\comp020199.pro\MethDB\110599b
Last modified: Mon Nov 08 07:09:58 1999
Job Code:

Printed: Mon Nov 08 07:28:42 1999

Compound 3: PFOA (Ch3)

#	Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1	110599028	MeOH Blank			7.852	2750	db	8.08	
2	110599029	MeOH Blank			7.852	1436	db	2.63	
3	110599030	MeOH Blank			7.894	748	db	0.00	
4	110599031	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH			7.878	723	db	0.00	
5	110599032	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.832	508	MM	0.00	
6	110599033	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.851	568	db	0.00	
7	110599034	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	Standard	4.93	7.870	1898	MM	4.55	-8
8	110599035	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	Standard	9.77	7.885	3162	MM	9.78	0
9	110599036	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	Standard	24.80	7.878	6543	MM	23.79	-4
10	110599037	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	Standard	49.30	7.831	12951	MM	50.34	2
11	110599038	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	Standard	97.70	7.878	25648	MM	103.03	5
12	110599039	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	Standard	248.00	7.897	58946	MM	239.25	-4
13	110599040	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	Standard	493.00	7.829	119341	MM	491.13	-0
14	110599041	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	Standard	736.00	7.830	177760	MM	733.17	-0
15	110599042	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	977.00	7.832	232968	MM	961.90	-2
16	110599043	MeOH Blank			7.829	1621	MM	3.40	
17	110599044	TOX145-RTS-9R03255M-5mg/kg-Day29, D:10			7.871	676	MM	0.00	
18	110599045	TOX145-RTS-9R03256M-5mg/kg-Day29, D:10			7.895	541	MM	0.00	
19	110599046	TOX145-RTS-9R03257M-5mg/kg-Day29, D:10			7.874	412	MM	0.00	
20	110599047	TOX145-RTS-9R03258M-5mg/kg-Day29, D:10			7.854	429	MM	0.00	
21	110599048	TOX145-RTS-9R03259M-5mg/kg-Day29, D:10			7.872	534	MM	0.00	
22	110599049	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH	QC	248.00	7.828	57924	MM	236.67	-5
23	110599050	MeOH Blank			7.828	518	db	0.00	
24	110599051	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH			7.968	459	MM	0.00	
25	110599052	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.921	517	MM	0.00	
26	110599053	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.833	457	MM	0.00	
27	110599054	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	Standard	4.93	7.874	1777	MM	4.04	-18
28	110599055	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	Standard	9.77	7.848	3350	MM	10.56	8
29	110599056	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	Standard	24.80	7.816	6639	MM	24.19	-2
30	110599057	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	Standard	49.30	7.826	13374	MM	52.09	6
31	110599058	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	Standard	97.70	7.826	27595	MM	111.01	14
32	110599059	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	Standard	248.00	7.810	62165	MM	254.24	3
33	110599060	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	Standard	493.00	7.852	120742	MM	496.93	1
34	110599061	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	Standard	736.00	7.831	177761	MM	733.17	-0
35	110599062	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	977.00	7.808	236809	MM	977.81	0

Original
data points
will be
used.
4/11 Nov-8-99

Quantify Compound Summary Report
TOX145-day29 control and dose- ext. 11/3/99

Page 4

Sample List: C:\MASSLYNK\soup020199.pro\SampleDB\110599b
Last modified: Fri Nov 05 15:26:11 1999
Method: C:\MASSLYNK\soup020199.pro\MethDB\110599b
Last modified: Mon Nov 08 07:09:58 1999
Job Code:

Printed: Mon Nov 08 07:28:42 1999

Compound 4: Butt (CH4)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc. %Dev
1 110599028	MeOH Blank			7.189	15	bb	0.01 -99
2 110599029	MeOH Blank						
3 110599030	MeOH Blank						
4 110599031	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH			7.171	1783	bb	0.83 -17
5 110599032	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.148	2056	bd	0.95 -5
6 110599033	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.167	1850	bd	0.86 -14
7 110599034	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.141	2048	bb	0.95 -5
8 110599035	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.109	2266	bd	1.05 5
9 110599036	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.149	2181	bb	1.01 1
10 110599037	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.169	1840	bb	0.85 -15
11 110599038	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.126	2250	bb	1.04 4
12 110599039	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.100	2303	bb	1.07 7
13 110599040	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.167	2399	bb	1.11 11
14 110599041	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.168	2114	bb	0.98 -2
15 110599042	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.146	2021	bb	0.94 -6
16 110599043	MeOH Blank			7.124	10	MM	0.00 -100
17 110599044	TOX145-RTS-9R03255M-5mg/kg-Day29, D:10			7.165	244	bb	0.11 -89
18 110599045	TOX145-RTS-9R03256M-5mg/kg-Day29, D:10			7.189	264	bb	0.12 -88
19 110599046	TOX145-RTS-9R03257M-5mg/kg-Day29, D:10			7.190	269	bb	0.12 -88
20 110599047	TOX145-RTS-9R03258M-5mg/kg-Day29, D:10			7.192	253	bb	0.12 -88
21 110599048	TOX145-RTS-9R03259M-5mg/kg-Day29, D:10			7.187	234	bb	0.11 -89
22 110599049	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH	QC	1.00	7.191	2199	bb	1.02 2
23 110599050	MeOH Blank						
24 110599051	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH			7.150	2009	bb	0.93 -7
25 110599052	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.193	2498	bb	1.16 16
26 110599053	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH			7.127	1956	bb	0.91 -9
27 110599054	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.167	2085	bb	0.97 -3
28 110599055	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.142	2249	bb	1.04 4
29 110599056	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.136	2233	bb	1.04 4
30 110599057	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.164	1846	bb	0.86 -14
31 110599058	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.141	2432	bb	1.13 13
32 110599059	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.147	2167	bb	1.00 0
33 110599060	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.147	2280	bb	1.06 6
34 110599061	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.168	2135	bd	0.99 -1
35 110599062	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	1.00	7.125	1983	bb	0.92 -8

Soup 020199: Quantitated and printed by las on 11/08/99

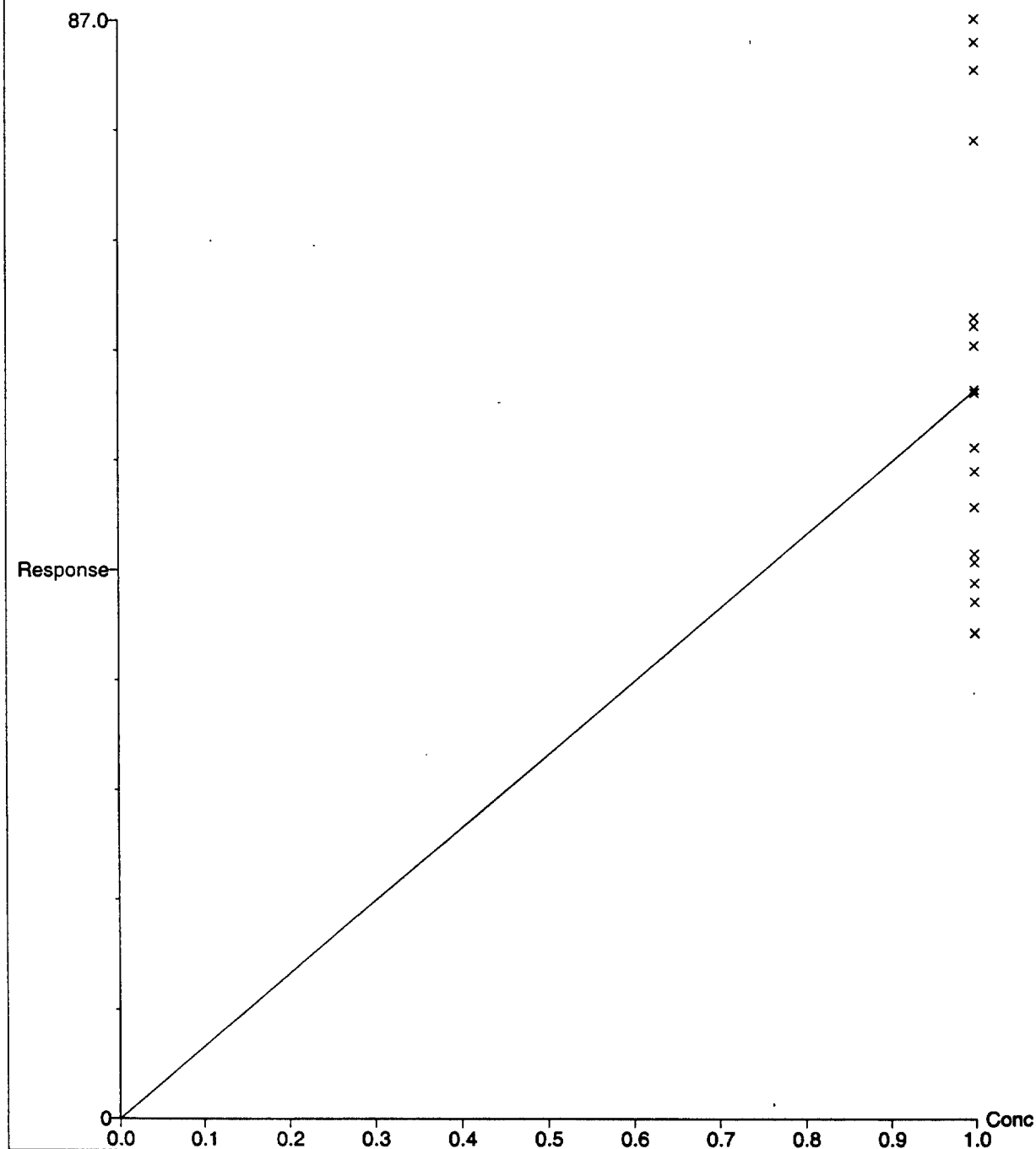
000679

Gas 4/08/99 p. 1 of 4

Quantify Calibration Report
TOX145-day29 control and dose- ext. 11/3/99

Page 1

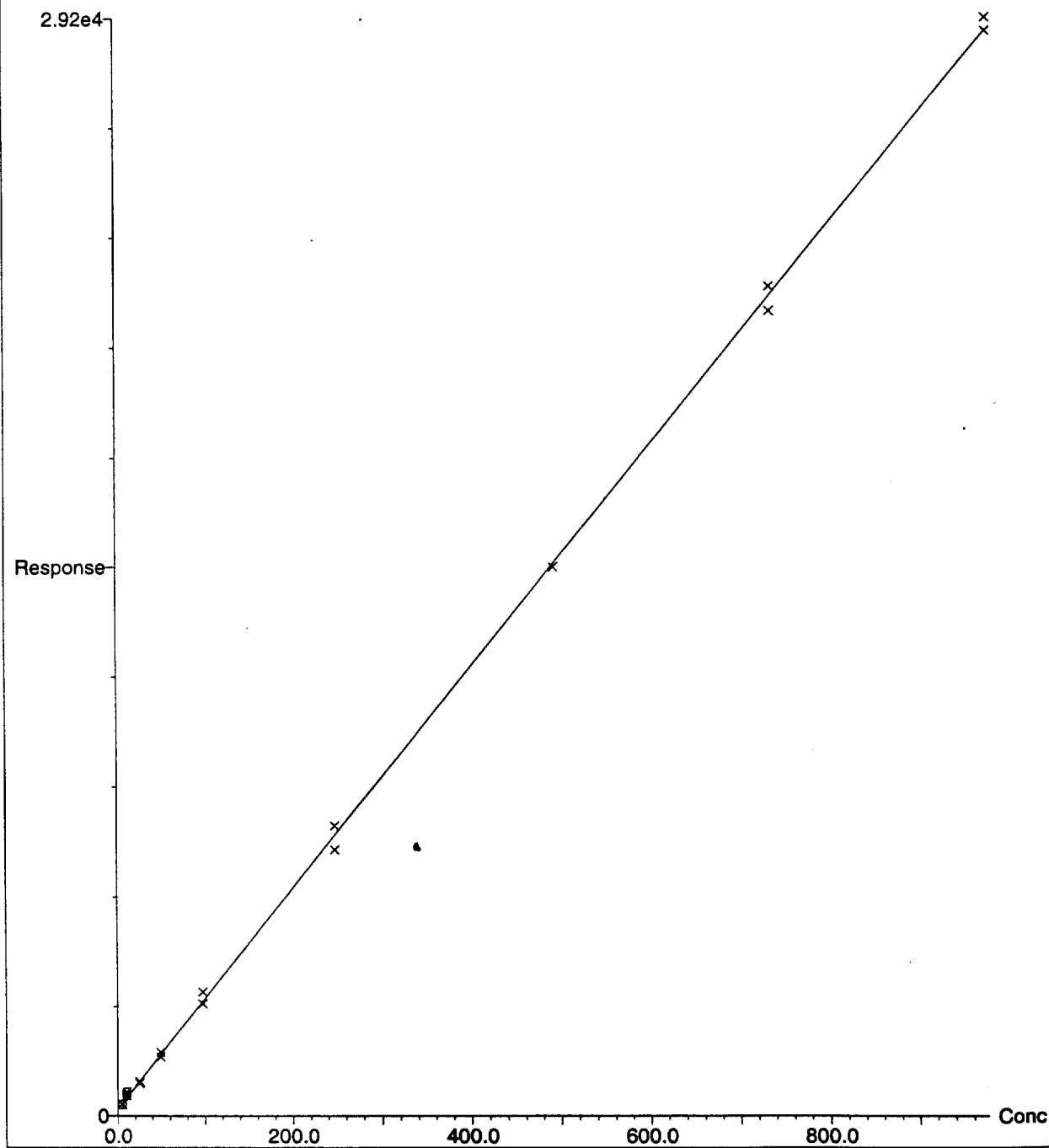
Compound 1 name: POAA (Ch1)
Response Factor: 57.5599
Response type: External Std, Area
Curve type: RF



Soup 020199: Quantitated and printed by las on 11/08/99

000680

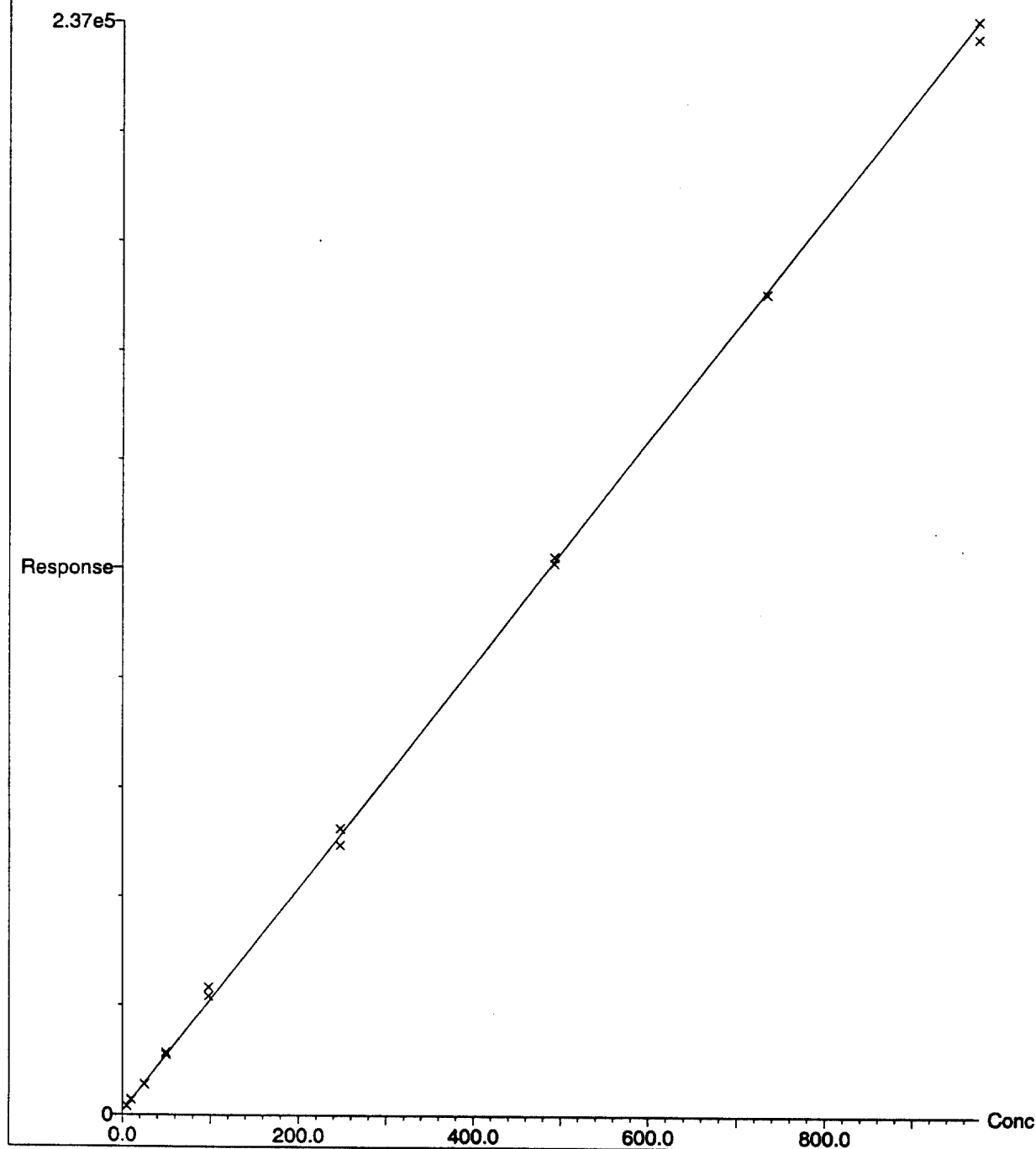
Compound 2 name: PFOS (Ch2)
Coefficient of Determination: 0.999549
Calibration curve: $29.3773 * x + 191.305$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



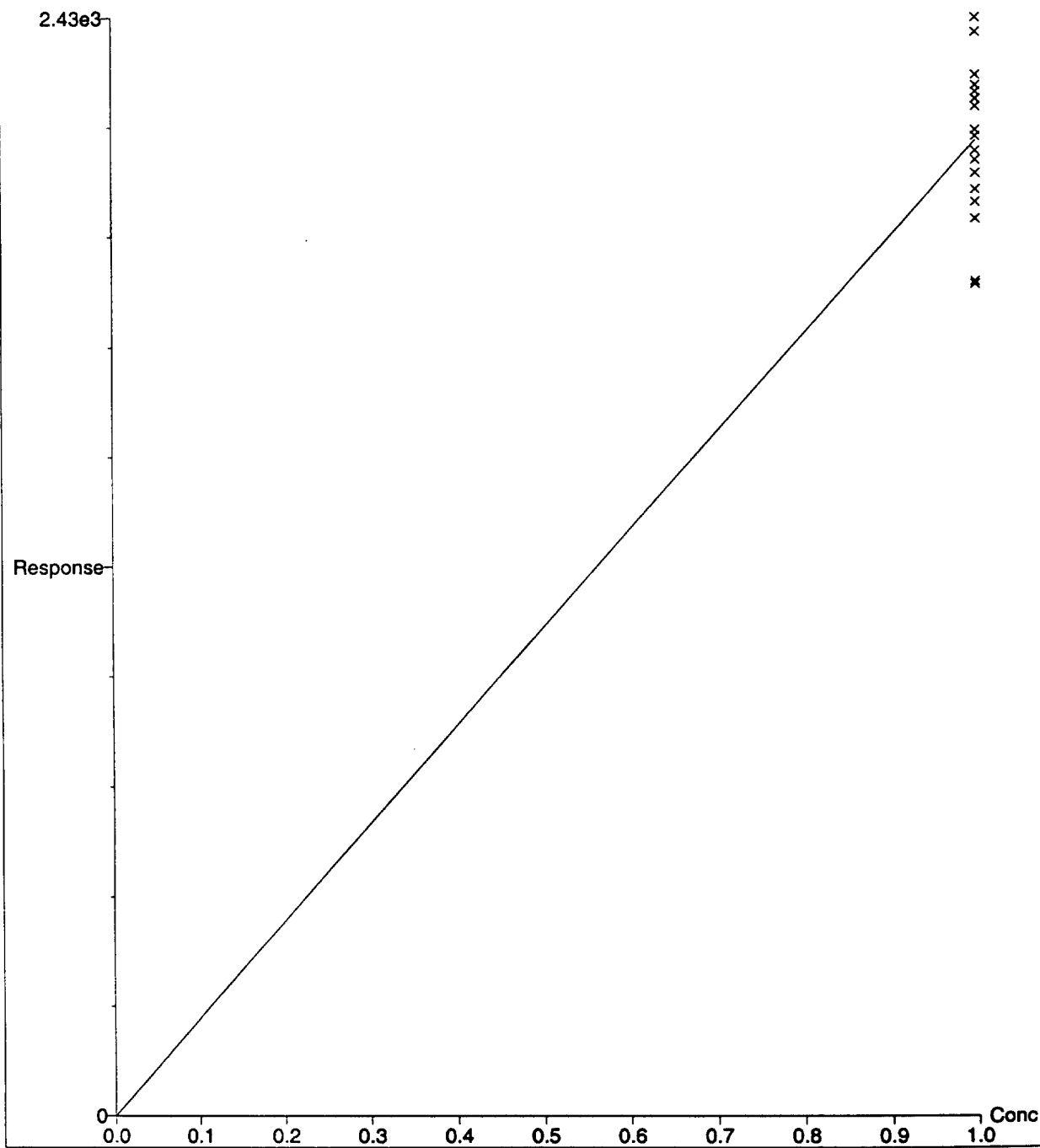
Soup 020199: Quantitated and printed by ias on 11/08/99

000681

Compound 3 name: PFOSA (Ch3)
Coefficient of Determination: 0.999708
Calibration curve: $241.363 * x + 800.807$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound 4 name: Surr (Ch4)
Response Factor: 2157.29
Response type: External Std, Area
Curve type: RF



Soup 020199: Quantitated and printed by ias on 11/08/99

000683

Method Report

Method file: C:\MSDCHEM\scup\00199.pro\MethDB\110599b.mdb
 Last modified: Mon Nov 03 07:09:38 1999
 Compound: 1: PCNA (Ch1)
 Printed: Mon Nov 03 07:29:54 1999

Compound Name: PCNA (Ch1)
 Internal Ref.: (None)
 Quantify Trace: TIC
 Conc. of Standards: Fixed
 User RF Value: 1.000000, Disabled
 User Peak Factor: 1.000100
 Integrate Window Factor: 2.000
 Chromatogram mass window: 1.000000

Retention Time (mins): 7.170, Enabled
 Relative Retention Time: 0.000, Disabled
 RT Reference: (None)
 Time Window (mins): 0.200

Peak Selection: Nearest

Response Type: External
 Response: Area

Polynomial Type: Linear
 Point of Origin: Exclude
 Fit Weighting: 1/X
 Axis Transformation: None

Units:

Peak-to-peak amplitude: 100

Smoothing: Enabled
 Window size (scans): 3
 Number of smooths: 1
 Smoothing method:

Join valley threshold: 50.00
 Reduce peak tailing: 200.00
 Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
 Detect shoulders: Disabled
 Shoulder threshold: 30.00

Relative height: 1.50, Disabled
 Absolute height: 10.00, Disabled
 Relative area: 0.10, Disabled
 Absolute area: 1.00, Disabled

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000684

Method Report

Method file: C:\MASSLYNX\source\20199.pro\MethDB\110599b.mdb
Last modified: Mon Nov 08 07:09:53 1999
Compound: 2: PPS (CH2)
Printed: Mon Nov 08 07:29:54 1999

Compound Name: PPS (Ch2)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. A
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 1.0000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.421, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.321

Peak Selection: Nearest

Response Type: External
Response: Area

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 100

Smoothing: Enabled
Window size (scans): 3
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 50.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.00, Disabled
Absolute height: 2.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 2.00, Disabled

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000685

Method Report

Method file: C:\MASSLYNK\setup020199.pro\MethDB\110599b.mdb
Last modified: Mon Nov 08 07:09:58 1999
Compound: 3: PFDSA (CH3)
Printed: Mon Nov 08 07:29:54 1999

Compound Name: PFDSA (CH3)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. B
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.00000Da

Retention Time (mins): 3.078, Enabled
Relative Retention Time: 3.078, Disabled
RT Reference: [None]
Time Window (mins): 0.468

Peak Selection: Nearest

Response Type: External
Response: Area

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 100

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 10.00
Reduce peak calling: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 93.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.00, Disabled
Absolute height: 1.00, Disabled
Relative area: 1.00, Disabled
Absolute area: 1.00, Disabled

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Method Report

Method file: C:\MASSLYNX\src\20199.pro\MethDB\110599b.mdb
Last modified: Mon Nov 08 07:22:58 1999
Compound: 4: Surr (Ch4)
Printed: Mon Nov 08 07:22:54 1999

Compound Name: Surr (Ch4)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Fixed
User RF Value: 1.00000, Disabled
User Peak Factor: 1.00000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.267, Enabled
Relative Retention Time: [None], Disabled
RT Reference: [None]
Time Window (mins): 0.267

Peak Selection: Nearest

Response Type: External
Response: Area

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 70

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 10.00
Reduce peak tailing: 10.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 50.00

Relative height: 1.00, Disabled
Absolute height: 0.00, Disabled
Relative area: 1.00, Disabled
Absolute area: 0.00, Disabled

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mmh
11-3-99

SOUP 020199: FACT Equipment
Log 99092 PAGE 1/1

Quattro II, #6414E

DATE: 11-05-99

ANALYST: mmh

STUDY NUMBER: TOX145 MATRIX: rat sera DESCRIPTION: day 29- dil

FACT Method: ETS-8-5.1

Injection volume: 10 uL Cycle time: 10 min Electrode: Z-Spray

Mobile Phase: (A) 2.0 mM NH₄CH₃CO₂ (B) MeOH

TN-A- or stdrd number (A) 99085-11-15 (B) TN-A-2997

Prepared (date/by): (A) 10/30/99 sah (B) 10/30/99 sah

Expires: (A) 04/00 (B) 2010

Store at room temp? ☒ or N Other: _____

Guard column: None

Part number: na Lot number: na

Analytical column: Keystone Betasil

Size: 2 x 50 mm Type: C18 Sphere size: 5 um

Part number: 055-701-2 Lot number: P9E11 Serial number: 0591804Z

Analytical parameters:

Drying gas (L/hr): 350 ESI nebulizing gas (L/hr) 13 Sheath gas (L/hr) 0

LC Flow rate: 300 uL/min Back pressure: 65 bar

HPLC solvent gradient: 10min-10-95

Time (min.)	%B
1) <u>0.00</u>	<u>10.0</u>
2) <u>1.00</u>	<u>10.0</u>
3) <u>5.50</u>	<u>95.0</u>
4) <u>7.50</u>	<u>95.0</u>
5) <u>8.00</u>	<u>10.0</u>

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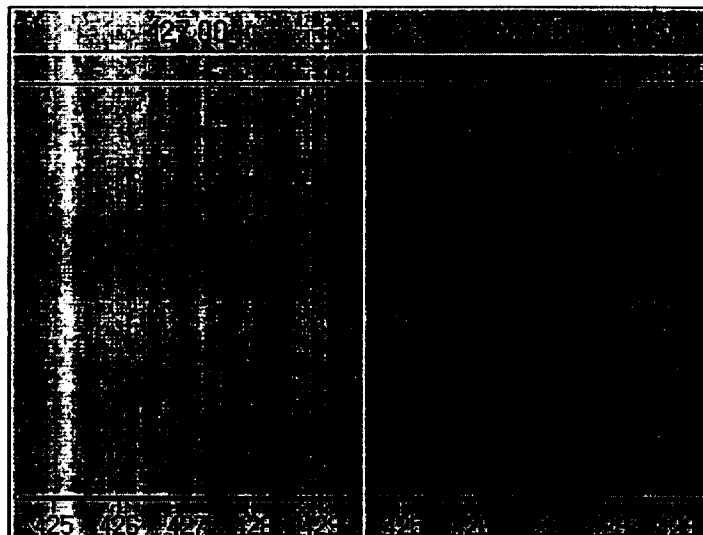
11-589

Tuning Method Report

Page 1

Method: C:\MASSLYNK\SOUP020199.PRO\ACQUDE\PFOS3

Printed: Fri Nov 05 15:32:05 1999



MS

MS2

SOURCE (ESP-)	Set	Rdbk	MS1	Set	Rdbk
Capillary	1.60	1.60	LM Resolution	14.0	
Cone	40	-60	HM Resolution	14.0	
Extractor	5	-6	Ion Energy	0.9	
RF Lens	0.2		Ion Energy Ramp	0.0	
Source Block Temp.	150	149	Lens 6	4	3
Desolvation Temp.	250	249	Multiplier	650	

Pressures	Rdbk	MS2	Set	Rdbk
Analysar Vacuum	5.1e-5	LM Resolution	14.0	
		HM Resolution	14.0	
		Collision	10	42
Gas Cell	4.1e-3	Ion Energy	2.0	
		Ion Energy Ramp	0.0	
		Lens 8	40	40
		Lens 9	0	45
		Multiplier	650	

000689

11551

Scanning Method Report

Page 1

Method: C:\MASSLYNK\SCUP020199.FRO\ACQDS\10KIN-PP06-POAA-PP06A-18
Last Modified: Fri Oct 15 10:38:03 1999
Printed: Fri Nov 05 15:31:38 1999

Solvent Delay (mins) : 0.00

Function : 1 NRM of 1 Mass Pair (EEP-)

Inter Channel Delay (Secs) : 0.03
Span (Daltons) : 0.00
Start Time (Mins) : 0.00
End Time (Mins) : 10.00
Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	413.00	169.00	0.30	30	15

Function : 2 NRM of 1 Mass Pair (EEP-)

Inter Channel Delay (Secs) : 0.03
Span (Daltons) : 0.00
Start Time (Mins) : 0.00
End Time (Mins) : 10.00
Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	499.00	99.00	0.30	45	60

Function : 3 NRM of 1 Mass Pair (EEP-)

Inter Channel Delay (Secs) : 0.03
Span (Daltons) : 0.00
Start Time (Mins) : 0.00
End Time (Mins) : 10.00
Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	498.00	78.00	0.30	45	60

Function : 4 NRM of 1 Mass Pair (EEP-)

Inter Channel Delay (Secs) : 0.03
Span (Daltons) : 0.00
Start Time (Mins) : 0.00
End Time (Mins) : 10.00
Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	427.00	80.00	0.30	40	60

000690

11-59

Masslynx - Sample List

Page 1

Sample List: C:\MASSLYNX\soup020199.pro\SampleDB\110599b.SPL
Printed: Fri Nov 05 15:26:20 1999

Page Position: (1, 1)

	File Name	File Text	MS File	Inlet File
1	110599028	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
2	110599029	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
3	110599030	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
4	110599031	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
5	110599032	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
6	110599033	TOX145-RTS11039-Sera Blk-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
7	110599034	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
8	110599035	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
9	110599036	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
10	110599037	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
11	110599038	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
12	110599039	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
13	110599040	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
14	110599041	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
15	110599042	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
16	110599043	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
17	110599044	TOX145-RTS-9R03255M-5mg/kg-Day29, D:10	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
18	110599045	TOX145-RTS-9R03256M-5mg/kg-Day29, D:10	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
19	110599046	TOX145-RTS-9R03257M-5mg/kg-Day29, D:10	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
20	110599047	TOX145-RTS-9R03258M-5mg/kg-Day29, D:10	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
21	110599048	TOX145-RTS-9R03259M-5mg/kg-Day29, D:10	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
22	110599049	TOX145-RBS11039-250ppb-CCV9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
23	110599050	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
24	110599051	TOX145-RBS11039-H2O Blk-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
25	110599052	TOX145-RBS11039-Sera Blk-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
26	110599053	TOX145-RTS11039-Sera Blk-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
27	110599054	TOX145-RBS11039-5ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
28	110599055	TOX145-RBS11039-10ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
29	110599056	TOX145-RBS11039-25ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
30	110599057	TOX145-RBS11039-50ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
31	110599058	TOX145-RBS11039-100ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
32	110599059	TOX145-RBS11039-250ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
33	110599060	TOX145-RBS11039-500ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
34	110599061	TOX145-RBS11039-750ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95
35	110599062	TOX145-RBS11039-1000ppb-9, ext. 11/3/99 MCH	10MIN-PFOS-POAA-PFOSA-IS	10min-10-95

000691

FACT: DATA REVIEW SUMMARY FORM

FAS 10/18/99 p. 1 of 2

Data Review Sign-Off

Date: 10/18/99
 Data Analyst: JAS
 FACT Study #: TOXINS
 Matrix/Timepoints: Day 1 & 4, Rat Liver, Control & dose, ext. 10/18/99
 Sample List: 101599a
 Set-up Analysis Date/Analyst: 10/15/99 JAS (5)
 Instrument: Solup 020199

Analyst Review
 Name / Date: JAS 10/18/99

Technical Review
 Name / Date: MEE 10/18/99 / h 10/18/99

Quality Assurance Review
 Name / Date: _____

Data Entry: UAC 10/21/99

Comments / Justifications: _____

Analyte: POAA (CH1) (1) (8)
 Acceptable Linear Range: No curve available - see comments
 LOQ: NA Extraction Blank: NA
 R²: NA Matrix Blank: NA
 Calibration Checks: NA
 MS/MSD: OK (5) UAC 10/21/99
 Data Status: ET

Analyte: PFOS (CH2) (2) (9)
 Acceptable Linear Range: JAS 10/18/99 30-70 → 920 ppb
 LOQ: 30-70 ppb Extracted Blank: less than LOQ
 R²: 0.983941 Matrix Blank: less than LOQ
 Calibration Checks: OK - Range from 16% → 23%
 MS/MSD: OK UAC 10/21/99
 Data Status: Entered + 10 Dilutions needed

Analyte: PFOSA (CH3) (3) (9)
 Acceptable Linear Range: 12.40 ppb → 614 ppb
 LOQ: 12.40 ppb Extracted Blank: less than LOQ
 R²: 0.999594 Matrix Blank: less than LOQ
 Calibration Checks: OK - Range from -30% → (-19%)
 MS/MSD: OK
 Data Status: Entered UAC 10/21/99

① POAA was not present in the calibration curves & calibration checks. Area counts are the only useful data for POAA.

Samples should be rerun for useable POAA data. - JAS 10/18/99

② The 6.13 ppb and 1227 ppb points were dropped from both curves because they did not fit into the linear range of the curve. The 12.30 ppb point was dropped so that the H₂O and matrix blank were less than the LOQ. Some samples need dilutions. One of the control samples was really high. The first methanol blank (file # 101599015) shows some carryover. - JAS 10/18/99

③ The 5 ppb (6.14 ppb), 921 ppb, and 1227 ppb curve points were dropped because they didn't fit into the linear range of the curve. Some samples need

⑧ No POAA detected in this screen; do not re-run.

⑨ Re-run sample in 101599043; suspect carry-over in results. h 10/18/99

000692

FACT: DATA REVIEW SUMMARY FORM

IAS 10/10/99 p. 2 of 2

Data Review Sign-Off

Date: 10/17/99
 Data Analyst: IAS
 FACT Study #: Tox 145
 Matrix/Timepoints: Day 1 & 4, Red Liver, Control & dose, ext 10/13/99
 Sample List: 10K592
 Set-up Analysis
 Date/Analyst: 10/18/99 IAS
 Instrument: Simp 020199

Analyst Review
 Name / Date: IAS 10/18/99

Technical Review
 Name / Date: Refer to page 1 of 2 LAC 10/21/99

Quality Assurance Review
 Name / Date: _____

Data Entry: LAC 10/21/99

Comments / Justifications: _____

Analyte: IS (CH4)①
 Acceptable Linear Range: NA
 LOQ: NA Extraction Blank: NA
 R²: NA Matrix Blank: NA
 Calibration Checks: NA
 MS/MSD: _____
 Data Status: _____

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____ IAS 10/18/99
 MS/MSD: _____
 Data Status: _____

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: _____

dilutions. One of the control samples was high (file # 101599025). A small peak that elutes just before the PDSA peak was noticed for most samples. The identity of the peak was not discovered. - IAS 10/18/99

④ Area counts for the ISTD were good. All samples had deviations under 30% except the H₂O blank which was at 42%. - IAS 10/18/99

⑤ A second cal check separated by a MWH blank was used in the sample run in anticipation of carryover. - IAS 10/18/99.

⑥ PDAA AREA COUNTS ARE VERY LOW. QUESTION NAME OF REPEATING ANALYSIS. MEE 10/18/99
 ⑦ SECOND PEAK IN PDSA CHROMATOGRAM IS NOT A TARGET ANALYTE AND DOES NOT NEED TO BE QUANTIFIED. MEE 10/18/99

000693

Ins 10/18/99 p. 1 of 4

Page 1

Quantify Compound Summary Report

TOX145- rat liver, day 1 & 4, control and dose- ext. 10/13/99

Sample List: C:\MASSLYNK\soup-rats.pro\SampleDB\101599a

Last modified: Mon Oct 18 06:42:08 1999

Method: C:\MASSLYNK\soup-rats.pro\MethDB\101599a

Last modified: Mon Oct 18 06:41:21 1999

Job Code:

Printed: Mon Oct 18 09:20:02 1999

Compound 1: POAA (Chl)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1 101599001	MeOH Blank							
2 101599002	MeOH Blank							
3 101599003	MeOH Blank							
4 101599004	TOX145, RBL10139-H2O Blank-11, SEE			7.072	41	MM	950.81	
5 101599005	TOX145, RBL10139-Liver Blank-11, SEE			7.086	12	bb	0.00	
6 101599006	TOX145, RBL10139-5ppb-11, SEE	Standard	5.00	7.088	12	bb	0.00	-100
7 101599007	TOX145, RBL10139-10ppb-11, SEE	Standard	10.00	7.083	17	MM	51.45	414
8 101599008	TOX145, RBL10139-25ppb-11, SEE	Standard	25.00	7.081	34	MM	709.73	2739
9 101599009	TOX145, RBL10139-50ppb-11, SEE	Standard	50.00	7.091	25	MM	363.70	627
10 101599010	TOX145, RBL10139-100ppb-11, SEE	Standard	100.00	7.088	29	MM	500.43	400
11 101599011	TOX145, RBL10139-250ppb-11, SEE	Standard	250.00	7.091	25	MM	343.34	37
12 101599012	TOX145, RBL10139-500ppb-11, SEE	Standard	500.00	7.105	12	MM	617.92	24
13 101599013	TOX145, RBL10139-750ppb-11, SEE	Standard	750.00	7.089	14	MM	0.00	-100
14 101599014	TOX145, RBL10139-1000ppb-11, SEE	Standard	1000.00	7.085	14	bb	690.12	-31
15 101599015	MeOH Blank			7.057	14	MM	0.00	
16 101599016	TOX145, RTL-9R03230M, Dy1, Control- SEE 10/13/99			7.084	45	MM	1090.29	
17 101599017	TOX145, RTL-9R03231M, Dy1, Control- SEE 10/13/99			7.088	33	bb	665.68	
18 101599018	TOX145, RTL-9R03232M, Dy1, Control- SEE 10/13/99			7.087	12	bb	0.00	
19 101599019	TOX145, RTL-9R03233M, Dy1, Control- SEE 10/13/99			7.088	29	MM	514.82	
20 101599020	TOX145, RTL-9R03234M, Dy1, Control- SEE 10/13/99			7.134	9	MM	0.00	
21 101599021	TOX145, RBL10139-250ppb-CCV-11-1, SEE	QC	250.00	7.112	22	bb	240.31	-4
22 101599022	MeOH Blank							
23 101599023	TOX145, RTL-9R03235M, Dy4, Control- SEE 10/13/99			7.079	34	MM	676.86	
24 101599024	TOX145, RTL-9R03236M, Dy4, Control- SEE 10/13/99			7.087	24	MM	302.04	
25 101599025	TOX145, RTL-9R03237M, Dy4, Control- SEE 10/13/99			7.083	54	MM	1464.13	
26 101599026	TOX145, RTL-9R03238M, Dy4, Control- SEE 10/13/99			7.111	13	MM	0.00	
27 101599027	TOX145, RTL-9R03239M, Dy4, Control- SEE 10/13/99			7.087	14	bb	0.00	
28 101599028	TOX145, RBL10139-250ppb-CCV-11-2, SEE	QC	250.00	7.085	17	MM	38.49	-85
29 101599029	MeOH Blank							
30 101599030	TOX145, 9R03230M, Dy1, Control-250pb-MS-11			7.090	25	MM	344.55	
31 101599031	TOX145, 9R03230M, Dy1, Control-250pb-MSD-11			7.110	22	bb	248.47	
32 101599032	TOX145, 9R03235M, Dy4, Control-250pb-MS-12			7.068	23	bb	291.47	
33 101599033	TOX145, 9R03235M, Dy4, Control-250pb-MSD-12			7.088	19	bb	134.30	
34 101599034	TOX145, RTL-9R03245M, Dy1, 5mg/kg- SEE 10/13/99			7.106	56	MM	1520.80	
35 101599035	TOX145, RBL10139-250ppb-CCV-11-3, SEE	QC	250.00	7.091	24	bb	298.95	20
36 101599036	MeOH Blank							
37 101599037	TOX145, RBL10139-250ppb-CCV-11-3, SEE	QC	250.00	7.084	24	bb	330.57	32
38 101599038	MeOH Blank							
39 101599039	TOX145, RTL-9R03246M, Dy1, 5mg/kg- SEE 10/13/99			7.105	62	MM	1734.25	
40 101599040	TOX145, RTL-9R03247M, Dy1, 5mg/kg- SEE 10/13/99			7.104	52	MM	1370.02	
41 101599041	TOX145, RTL-9R03248M, Dy1, 5mg/kg- SEE 10/13/99			7.067	59	MM	1638.25	
42 101599042	TOX145, RTL-9R03249M, Dy1, 5mg/kg- SEE 10/13/99			7.090	53	MM	1422.83	
43 101599043	TOX145, RTL-9R03250M, Dy4, 5mg/kg- SEE 10/13/99			7.089	20	MM	163.54	
44 101599044	TOX145, RBL10139-250ppb-CCV-11-4, SEE	QC	250.00	7.069	20	MM	179.22	-28
45 101599045	MeOH Blank							
46 101599046	TOX145, RBL10139-250ppb-CCV-11-4, SEE	QC	250.00	7.086	14	MM	0.00	-100
47 101599047	MeOH Blank							
48 101599048	TOX145, RTL-9R03251M, Dy4, 5mg/kg- SEE 10/13/99			7.091	40	bb	909.32	
49 101599049	TOX145, RTL-9R03252M, Dy4, 5mg/kg- SEE 10/13/99			7.083	18	bb	837.01	
50 101599050	TOX145, RTL-9R03253M, Dy4, 5mg/kg- SEE 10/13/99			7.088	39	MM	867.46	
51 101599051	TOX145, RTL-9R03254M, Dy4, 5mg/kg- SEE 10/13/99			7.089	50	MM	1283.05	
52 101599052	TOX145, RBL10139-250ppb-CCV-11-4, SEE	QC	250.00	7.058	15	MM	0.00	-100
53 101599053	MeOH Blank							
54 101599054	TOX145, RBL10139-250ppb-CCV-11-4, SEE	QC	250.00	7.064	19	MM	115.94	-54
55 101599055	MeOH Blank							
56 101599056	TOX145, RBL10139-5ppb-11, SEE	Standard	5.00	7.113	12	bb	0.00	-100
57 101599057	TOX145, RBL10139-10ppb-11, SEE	Standard	10.00	7.090	16	MM	27.68	177
58 101599058	TOX145, RBL10139-25ppb-11, SEE	Standard	25.00	7.112	18	MM	106.27	325
59 101599059	TOX145, RBL10139-50ppb-11, SEE	Standard	50.00	7.110	31	MM	566.58	1033
60 101599060	TOX145, RBL10139-100ppb-11, SEE	Standard	100.00	7.091	25	bb	355.31	255
61 101599061	TOX145, RBL10139-250ppb-11, SEE	Standard	250.00	7.113	31	MM	582.45	133
62 101599062	TOX145, RBL10139-500ppb-11, SEE	Standard	500.00	7.067	23	bb	283.57	-43
63 101599063	TOX145, RBL10139-750ppb-11, SEE	Standard	750.00	7.088	16	bb	28.33	-96
64 101599064	TOX145, RBL10139-1000ppb-11, SEE	Standard	1000.00	7.086	29	MM	496.73	-50

Not a target analyte. Data not used

LAC 10/18/99

Sample List: C:\MSBLLYME\soup-rats-pro\SampleDB\101599a
Last modified: Mon Oct 18 06:42:08 1999
Method: C:\MSBLLYME\soup-rats-pro\MethDB\101599a
Last modified: Mon Oct 18 06:41:21 1999
Job Code:

Printed: Mon Oct 18 09:20:02 1999

Compound 2: PFOS (Ch2)

#	Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1	101599001	MeOH Blank			7.315	27	bb	0.00	
2	101599002	MeOH Blank			7.242	14	bb	0.00	
3	101599003	MeOH Blank			7.255	31	MM	0.00	
4	101599004	TOX145, RBL10139-MQO Blank-11, SEE			7.233	59	MM	0.00	
5	101599005	TOX145, RBL10139-Liver Blank-11, SEE			7.247	55	MM	0.00	
6	101599006	TOX145, RBL10139-5ppb-11, SEE	Standard	6.13	7.250	75	MMX	0.00	-100
7	101599007	TOX145, RBL10139-10ppb-11, SEE	Standard	12.30	7.243	95	MMX	2.58	-79
8	101599008	TOX145, RBL10139-25ppb-11, SEE	Standard	30.70	7.243	155	MM	26.34	-14
9	101599009	TOX145, RBL10139-50ppb-11, SEE	Standard	61.30	7.231	254	MM	66.51	9
10	101599010	TOX145, RBL10139-100ppb-11, SEE	Standard	123.00	7.249	408	bb	128.32	4
11	101599011	TOX145, RBL10139-250ppb-11, SEE	Standard	307.00	7.252	934	MM	340.19	11
12	101599012	TOX145, RBL10139-500ppb-11, SEE	Standard	613.00	7.245	1815	MM	694.33	13
13	101599013	TOX145, RBL10139-750ppb-11, SEE	Standard	920.00	7.250	2347	bb	908.67	-1
14	101599014	TOX145, RBL10139-1000ppb-11, SEE	Standard	1227.00	7.246	2856	bbX	1113.21	-9
15	101599015	MeOH Blank			7.242	175	bb	34.56	
16	101599016	TOX145, RTL-9R03230M, Dy1, Control- SEE 10/13/99			7.245	37	bb	0.00	
17	101599017	TOX145, RTL-9R03231M, Dy1, Control- SEE 10/13/99			7.272	37	bb	0.00	
18	101599018	TOX145, RTL-9R03232M, Dy1, Control- SEE 10/13/99			7.272	58	bb	0.00	
19	101599019	TOX145, RTL-9R03233M, Dy1, Control- SEE 10/13/99			7.250	38	MM	0.00	
20	101599020	TOX145, RTL-9R03234M, Dy1, Control- SEE 10/13/99			7.273	59	MM	0.00	
21	101599021	TOX145, RBL10139-250ppb-CCV-11-1, SEE	QC	307.00	7.251	746	bb	264.24	-14
22	101599022	MeOH Blank			7.245	146	bb	22.73	
23	101599023	TOX145, RTL-9R03235M, Dy4, Control- SEE 10/13/99			7.240	77	MM	0.00	
24	101599024	TOX145, RTL-9R03236M, Dy4, Control- SEE 10/13/99			7.226	58	MM	0.00	
25	101599025	TOX145, RTL-9R03237M, Dy4, Control- SEE 10/13/99			7.245	71286	bb	28648.20	*
26	101599026	TOX145, RTL-9R03238M, Dy4, Control- SEE 10/13/99			7.294	143	bb	21.75	
27	101599027	TOX145, RTL-9R03239M, Dy4, Control- SEE 10/13/99			7.271	60	bb	0.00	
28	101599028	TOX145, RBL10139-250ppb-CCV-11-2, SEE	QC	307.00	7.247	731	bb	258.48	-16
29	101599029	MeOH Blank			7.271	49	MM	0.00	
30	101599030	TOX145, 9R03230M, Dy1, Control-250pb-MS-11			7.250	773	bb	275.14	
31	101599031	TOX145, 9R03230M, Dy1, Control-250pb-MSD-11			7.249	742	bb	262.88	
32	101599032	TOX145, 9R03235M, Dy4, Control-250pb-MS-12			7.230	652	bb	226.68	
33	101599033	TOX145, 9R03235M, Dy4, Control-250pb-MSD-12			7.227	627	bb	216.37	
34	101599034	TOX145, RTL-9R03245M, Dy1, 5mg/kg- SEE 10/13/99			7.245	43197	bb	17345.69	-
35	101599035	TOX145, RBL10139-250ppb-CCV-11-3, SEE	QC	307.00	7.229	803	bb	287.13	-6
36	101599036	MeOH Blank			7.273	68	MM	0.00	
37	101599037	TOX145, RBL10139-250ppb-CCV-11-3, SEE	QC	307.00	7.245	732	bd	258.80	-16
38	101599038	MeOH Blank			7.295	32	bb	0.00	
39	101599039	TOX145, RTL-9R03246M, Dy1, 5mg/kg- SEE 10/13/99			7.244	58246	bb	23401.31	
40	101599040	TOX145, RTL-9R03247M, Dy1, 5mg/kg- SEE 10/13/99			7.244	62508	bb	25116.09	
41	101599041	TOX145, RTL-9R03248M, Dy1, 5mg/kg- SEE 10/13/99			7.250	41365	bb	16608.81	
42	101599042	TOX145, RTL-9R03249M, Dy1, 5mg/kg- SEE 10/13/99			7.229	51215	bb	20572.04	
43	101599043	TOX145, RTL-9R03250M, Dy4, 5mg/kg- SEE 10/13/99			7.273	163	bb	29.63	*
44	101599044	TOX145, RBL10139-250ppb-CCV-11-4, SEE	QC	307.00	7.231	692	bb	242.59	-21
45	101599045	MeOH Blank			7.297	70	bb	0.00	
46	101599046	TOX145, RBL10139-250ppb-CCV-11-4, SEE	QC	307.00	7.247	673	bb	234.98	-23
47	101599047	MeOH Blank			7.250	48	MM	0.00	
48	101599048	TOX145, RTL-9R03251M, Dy4, 5mg/kg- SEE 10/13/99			7.231	60338	bb	24242.92	
49	101599049	TOX145, RTL-9R03252M, Dy4, 5mg/kg- SEE 10/13/99			7.244	73238	bb	29433.70	
50	101599050	TOX145, RTL-9R03253M, Dy4, 5mg/kg- SEE 10/13/99			7.250	68971	bb	27716.59	
51	101599051	TOX145, RTL-9R03254M, Dy4, 5mg/kg- SEE 10/13/99			7.228	65760	bb	26424.89	
52	101599052	TOX145, RBL10139-250ppb-CCV-11-4, SEE	QC	307.00	7.242	978	MM	357.59	16
53	101599053	MeOH Blank			7.294	175	bb	34.45	
54	101599054	TOX145, RBL10139-250ppb-CCV-11-4, SEE	QC	307.00	7.248	696	bb	244.39	-20
55	101599055	MeOH Blank			7.271	105	MM	6.44	
56	101599056	TOX145, RBL10139-5ppb-11, SEE	Standard	6.13	7.275	84	bbX	0.00	-100
57	101599057	TOX145, RBL10139-10ppb-11, SEE	Standard	12.30	7.251	96	MMX	2.99	-76
58	101599058	TOX145, RBL10139-25ppb-11, SEE	Standard	30.70	7.250	169	MM	31.97	4
59	101599059	TOX145, RBL10139-50ppb-11, SEE	Standard	61.30	7.227	221	bb	53.13	-13
60	101599060	TOX145, RBL10139-100ppb-11, SEE	Standard	123.00	7.229	394	MM	122.60	-0
61	101599061	TOX145, RBL10139-250ppb-11, SEE	Standard	307.00	7.251	836	bd	300.55	-2
62	101599062	TOX145, RBL10139-500ppb-11, SEE	Standard	613.00	7.229	1637	bb	622.81	2
63	101599063	TOX145, RBL10139-750ppb-11, SEE	Standard	920.00	7.227	2113	bd	814.57	-11
64	101599064	TOX145, RBL10139-1000ppb-11, SEE	Standard	1227.00	7.226	2575	MMX	1000.46	-18

Entered all
except those
needing a dilution.
UC
10/24/99

1:100
Need a 1:150 dilution
UC
10/18/99

1:100
Need a 1:150 dilution
UC

1:100
Need a 1:150 dilution
UC

1:100
Need a 1:150 dilution
UC

1:100
Need a 1:150 dilution
UC
10/18/99

* Could these samples be mislabeled/switched? by 10/18/99

0 1:100 dilution will be sufficient. UC 10/18/99

Sample List: C:\MASSLYTK\soup-rats-pro\SampleDB\101599a
Last modified: Mon Oct 18 06:42:08 1999
Method: C:\MASSLYTK\soup-rats-pro\MethDB\101599a
Last modified: Mon Oct 18 06:41:21 1999
Job Code:

Printed: Mon Oct 18 09:20:02 1999

Compound 3: PFOA (Ch3)

#	Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1	101599001	MeOH Blank			7.810	177	bd	0.00	
2	101599002	MeOH Blank			7.826	339	bb	4.25	
3	101599003	MeOH Blank			7.839	178	MM	0.00	
4	101599004	TOX145, RBL10139-H2O Blank-11, SEE			7.796	145	MM	0.00	
5	101599005	TOX145, RBL10139-Liver Blank-11, SEE			7.807	159	MM	0.00	
6	101599006	TOX145, RBL10139-5ppb-11, SEE	Standard	6.14	7.766	321	MMX	3.70	-40
7	101599007	TOX145, RBL10139-10ppb-11, SEE	Standard	12.40	7.783	522	MM	9.78	-21
8	101599008	TOX145, RBL10139-25ppb-11, SEE	Standard	31.00	7.805	1469	MM	38.38	24
9	101599009	TOX145, RBL10139-50ppb-11, SEE	Standard	62.00	7.792	2173	MM	59.64	-4
10	101599010	TOX145, RBL10139-100ppb-11, SEE	Standard	124.00	7.808	4263	MM	122.79	-1
11	101599011	TOX145, RBL10139-250ppb-11, SEE	Standard	307.00	7.814	10398	MM	308.16	0
12	101599012	TOX145, RBL10139-500ppb-11, SEE	Standard	614.00	7.807	20565	MM	615.30	0
13	101599013	TOX145, RBL10139-750ppb-11, SEE	Standard	921.00	7.813	26902	MMX	806.77	-12
14	101599014	TOX145, RBL10139-1000ppb-11, SEE	Standard	1228.00	7.808	35703	MMX	1072.65	-13
15	101599015	MeOH Blank			7.848	166	MM	0.00	
16	101599016	TOX145, RTL-9R03230M, Dy1, Control- SEE 10/13/99			7.852	147	MM	0.00	
17	101599017	TOX145, RTL-9R03231M, Dy1, Control- SEE 10/13/99			7.833	115	MM	0.00	
18	101599018	TOX145, RTL-9R03232M, Dy1, Control- SEE 10/13/99			7.835	127	MM	0.00	
19	101599019	TOX145, RTL-9R03233M, Dy1, Control- SEE 10/13/99			7.813	140	MM	0.00	
20	101599020	TOX145, RTL-9R03234M, Dy1, Control- SEE 10/13/99			7.813	127	MM	0.00	
21	101599021	TOX145, RBL10139-250ppb-CCV-11-1, SEE	QC	307.00	7.813	8203	MM	241.81	-21
22	101599022	MeOH Blank			7.874	105	MM	0.00	
23	101599023	TOX145, RTL-9R03235M, Dy4, Control- SEE 10/13/99			7.824	277	MM	2.35	
24	101599024	TOX145, RTL-9R03236M, Dy4, Control- SEE 10/13/99			7.833	147	MM	0.00	
25	101599025	TOX145, RTL-9R03237M, Dy4, Control- SEE 10/13/99			7.807	77671	db	2340.62	* - need a 1:100 dilution
26	101599026	TOX145, RTL-9R03238M, Dy4, Control- SEE 10/13/99			7.857	352	MM	4.62	
27	101599027	TOX145, RTL-9R03239M, Dy4, Control- SEE 10/13/99			7.833	183	MM	0.00	
28	101599028	TOX145, RTL-9R03239M, Dy4, Control- SEE 10/13/99	QC	307.00	7.809	8271	MM	243.87	-21
29	101599029	MeOH Blank			7.855	123	MM	0.00	
30	101599030	TOX145, 9R03230M, Dy1, Control-250pb-MS-11			7.813	10526	MM	312.02	
31	101599031	TOX145, 9R03230M, Dy1, Control-250pb-MSD-11			7.813	9524	MM	281.72	
32	101599032	TOX145, 9R03235M, Dy4, Control-250pb-MS-12			7.814	7882	MM	232.13	
33	101599033	TOX145, 9R03235M, Dy4, Control-250pb-MSD-12			7.811	7389	MM	217.22	
34	101599034	TOX145, RTL-9R03245M, Dy1, 5mg/kg- SEE 10/13/99			7.807	147648	db	4454.76	- Need a 1:100 dilution
35	101599035	TOX145, RBL10139-250ppb-CCV-11-3, SEE	QC	307.00	7.813	8382	MM	247.24	-19
36	101599036	MeOH Blank			7.858	158	MM	0.00	
37	101599037	TOX145, RBL10139-250ppb-CCV-11-3, SEE	QC	307.00	7.807	7941	MM	233.92	-24
38	101599038	MeOH Blank			7.857	138	MM	0.00	
39	101599039	TOX145, RTL-9R03246M, Dy1, 5mg/kg- SEE 10/13/99			7.806	176413	db	5323.83	
40	101599040	TOX145, RTL-9R03247M, Dy1, 5mg/kg- SEE 10/13/99			7.806	153273	db	4624.70	
41	101599041	TOX145, RTL-9R03248M, Dy1, 5mg/kg- SEE 10/13/99			7.813	156909	bb	4734.57	} Need a 1:100 dilution
42	101599042	TOX145, RTL-9R03249M, Dy1, 5mg/kg- SEE 10/13/99			7.814	160875	db	4854.39	
43	101599043	TOX145, RTL-9R03250M, Dy4, 5mg/kg- SEE 10/13/99			7.857	932	MM	22.16	*
44	101599044	TOX145, RBL10139-250ppb-CCV-11-4, SEE	QC	307.00	7.792	7589	MM	223.26	-27
45	101599045	MeOH Blank			7.881	202	MM	0.11	
46	101599046	TOX145, RBL10139-250ppb-CCV-11-4, SEE	QC	307.00	7.810	7441	MM	218.79	-29
47	101599047	MeOH Blank			7.879	172	MM	0.00	
48	101599048	TOX145, RTL-9R03251M, Dy4, 5mg/kg- SEE 10/13/99			7.816	66310	bb	1997.35	
49	101599049	TOX145, RTL-9R03252M, Dy4, 5mg/kg- SEE 10/13/99			7.806	73496	dd	2214.46	
50	101599050	TOX145, RTL-9R03253M, Dy4, 5mg/kg- SEE 10/13/99			7.812	68288	dd	2057.11	
51	101599051	TOX145, RTL-9R03254M, Dy4, 5mg/kg- SEE 10/13/99			7.790	79669	bb	2400.98	
52	101599052	TOX145, RBL10139-250ppb-CCV-11-4, SEE	QC	307.00	7.804	7292	MM	214.29	-30
53	101599053	MeOH Blank			7.880	211	MM	0.38	
54	101599054	TOX145, RBL10139-250ppb-CCV-11-4, SEE	QC	307.00	7.810	7480	MM	219.99	-28
55	101599055	MeOH Blank			7.879	148	MM	0.00	
56	101599056	TOX145, RBL10139-5ppb-11, SEE	Standard	6.14	7.836	367	MMX	5.07	-17
57	101599057	TOX145, RBL10139-10ppb-11, SEE	Standard	12.40	7.791	551	MM	10.64	-14
58	101599058	TOX145, RBL10139-25ppb-11, SEE	Standard	31.00	7.790	1467	MM	38.33	24
59	101599059	TOX145, RBL10139-50ppb-11, SEE	Standard	62.00	7.789	2205	MM	60.61	-2
60	101599060	TOX145, RBL10139-100ppb-11, SEE	Standard	124.00	7.813	4136	MM	118.95	-4
61	101599061	TOX145, RBL10139-250ppb-11, SEE	Standard	307.00	7.813	10131	MM	300.07	-2
62	101599062	TOX145, RBL10139-500ppb-11, SEE	Standard	614.00	7.792	20659	MM	618.16	1
63	101599063	TOX145, RBL10139-750ppb-11, SEE	Standard	921.00	7.812	23247	MMX	756.78	-18
64	101599064	TOX145, RBL10139-1000ppb-11, SEE	Standard	1228.00	7.810	35385	MMX	1063.05	-13

Entered all
except those
needing a
dilution.

uc
10/21/99

* - need a 1:100 dilution

- Need a 1:100 dilution

} Need a 1:100 dilution

} Need a 1:100 dilution

uc
10/18/99

* Could these samples be switched/mislabeled?

h 10/17/99

Quantify Compound Summary Report

Page 4

TOX145- rat liver, day 1 & 4, control and dose- ext. 10/13/99

Sample List: C:\MASSLYNK\scup-rats.pro\sampleDB\101599a

Last modified: Mon Oct 18 06:42:08 1999

Method: C:\MASSLYNK\scup-rats.pro\Method\101599a

Last modified: Mon Oct 18 06:41:21 1999

Job Code:

Printed: Mon Oct 18 09:20:02 1999

Compound 4: IS (Ch4)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1 101599001	MeOH Blank							
2 101599002	MeOH Blank							
3 101599003	MeOH Blank							
4 101599004	TOX145, RBL10139-H2O Blank-11, SEE			7.067	887	bb	0.58	-42
5 101599005	TOX145, RBL10139-Liver Blank-11, SEE			7.081	1255	bb	0.81	-19
6 101599006	TOX145, RBL10139-5ppb-11, SEE	Standard	1.00	7.083	1358	bb	0.88	-12
7 101599007	TOX145, RBL10139-10ppb-11, SEE	Standard	1.00	7.099	1473	bb	0.96	-4
8 101599008	TOX145, RBL10139-25ppb-11, SEE	Standard	1.00	7.098	1675	bb	1.09	9
9 101599009	TOX145, RBL10139-50ppb-11, SEE	Standard	1.00	7.086	1670	bb	1.08	8
10 101599010	TOX145, RBL10139-100ppb-11, SEE	Standard	1.00	7.083	1608	bb	1.04	4
11 101599011	TOX145, RBL10139-250ppb-11, SEE	Standard	1.00	7.085	1640	bb	1.06	6
12 101599012	TOX145, RBL10139-500ppb-11, SEE	Standard	1.00	7.078	1651	bb	1.07	7
13 101599013	TOX145, RBL10139-750ppb-11, SEE	Standard	1.00	7.084	1551	bb	1.01	1
14 101599014	TOX145, RBL10139-1000ppb-11, SEE	Standard	1.00	7.079	1487	bb	0.96	-4
15 101599015	MeOH Blank							
16 101599016	TOX145, RTL-9R03230M, Dy1, Control- SEE 10/13/99			7.078	1609	bb	1.04	4
17 101599017	TOX145, RTL-9R03231M, Dy1, Control- SEE 10/13/99			7.082	1873	bb	1.02	2
18 101599018	TOX145, RTL-9R03232M, Dy1, Control- SEE 10/13/99			7.082	1757	bb	1.14	14
19 101599019	TOX145, RTL-9R03233M, Dy1, Control- SEE 10/13/99			7.083	1612	bb	1.04	4
20 101599020	TOX145, RTL-9R03234M, Dy1, Control- SEE 10/13/99			7.083	1698	bb	1.10	10
21 101599021	TOX145, RBL10139-250ppb-CCV-11-1, SEE	QC	1.00	7.084	1396	bb	0.90	-10
22 101599022	MeOH Blank							
23 101599023	TOX145, RTL-9R03235M, Dy4, Control- SEE 10/13/99			7.095	1583	bb	1.03	3
24 101599024	TOX145, RTL-9R03236M, Dy4, Control- SEE 10/13/99			7.082	1519	bb	0.99	-1
25 101599025	TOX145, RTL-9R03237M, Dy4, Control- SEE 10/13/99			7.078	1435	bb	0.93	-7
26 101599026	TOX145, RTL-9R03238M, Dy4, Control- SEE 10/13/99			7.082	1439	bb	0.93	-7
27 101599027	TOX145, RTL-9R03239M, Dy4, Control- SEE 10/13/99			7.082	1466	bb	0.95	-5
28 101599028	TOX145, RBL10139-250ppb-CCV-11-2, SEE	QC	1.00	7.079	1417	bb	0.92	-8
29 101599029	MeOH Blank							
30 101599030	TOX145, 9R03230M, Dy1, Control-250pb-MS-11			7.083	1494	bb	0.97	-3
31 101599031	TOX145, 9R03230M, Dy1, Control-250pb-MSD-11			7.082	1426	bb	0.92	-8
32 101599032	TOX145, 9R03235M, Dy4, Control-250pb-MS-12			7.086	1224	bb	0.79	-21
33 101599033	TOX145, 9R03235M, Dy4, Control-250pb-MSD-12			7.082	1187	bb	0.77	-23
34 101599034	TOX145, RTL-9R03245M, Dy1, 5mg/kg- SEE 10/13/99			7.078	1484	bb	0.96	-4
35 101599035	TOX145, RBL10139-250ppb-CCV-11-3, SEE	QC	1.00	7.084	1379	bb	0.89	-11
36 101599036	MeOH Blank							
37 101599037	TOX145, RBL10139-250ppb-CCV-11-3, SEE	QC	1.00	7.100	1313	bb	0.85	-15
38 101599038	MeOH Blank							
39 101599039	TOX145, RTL-9R03246M, Dy1, 5mg/kg- SEE 10/13/99			7.100	1390	bb	0.90	-10
40 101599040	TOX145, RTL-9R03247M, Dy1, 5mg/kg- SEE 10/13/99			7.099	1409	bb	0.91	-9
41 101599041	TOX145, RTL-9R03248M, Dy1, 5mg/kg- SEE 10/13/99			7.083	1305	bb	0.85	-15
42 101599042	TOX145, RTL-9R03249M, Dy1, 5mg/kg- SEE 10/13/99			7.084	1413	bb	0.92	-8
43 101599043	TOX145, RTL-9R03250M, Dy4, 5mg/kg- SEE 10/13/99			7.084	1386	bb	0.90	-10
44 101599044	TOX145, RBL10139-250ppb-CCV-11-4, SEE	QC	1.00	7.086	1235	bb	0.80	-20
45 101599045	MeOH Blank							
46 101599046	TOX145, RBL10139-250ppb-CCV-11-4, SEE	QC	1.00	7.080	1239	bb	0.80	-20
47 101599047	MeOH Blank							
48 101599048	TOX145, RTL-9R03251M, Dy4, 5mg/kg- SEE 10/13/99			7.106	1	bb	0.00	-100
49 101599049	TOX145, RTL-9R03252M, Dy4, 5mg/kg- SEE 10/13/99			7.086	1289	bb	0.84	-16
50 101599050	TOX145, RTL-9R03253M, Dy4, 5mg/kg- SEE 10/13/99			7.077	1382	bb	0.90	-10
51 101599051	TOX145, RTL-9R03254M, Dy4, 5mg/kg- SEE 10/13/99			7.083	1460	bb	0.95	-5
52 101599052	TOX145, RBL10139-250ppb-CCV-11-4, SEE	QC	1.00	7.083	1341	bb	0.87	-13
53 101599053	MeOH Blank							
54 101599054	TOX145, RBL10139-250ppb-CCV-11-4, SEE	QC	1.00	7.081	1253	bb	0.81	-19
55 101599055	MeOH Blank							
56 101599056	TOX145, RBL10139-5ppb-11, SEE	Standard	1.00	7.084	1495	bb	0.97	-3
57 101599057	TOX145, RBL10139-10ppb-11, SEE	Standard	1.00	7.084	1498	bb	0.97	-3
58 101599058	TOX145, RBL10139-25ppb-11, SEE	Standard	1.00	7.084	1574	bb	1.02	2
59 101599059	TOX145, RBL10139-50ppb-11, SEE	Standard	1.00	7.082	1572	bb	1.02	2
60 101599060	TOX145, RBL10139-100ppb-11, SEE	Standard	1.00	7.084	1585	bb	1.03	3
61 101599061	TOX145, RBL10139-250ppb-11, SEE	Standard	1.00	7.084	1607	bb	1.04	4
62 101599062	TOX145, RBL10139-500ppb-11, SEE	Standard	1.00	7.084	1527	bb	0.99	-1
63 101599063	TOX145, RBL10139-750ppb-11, SEE	Standard	1.00	7.082	1400	bb	0.91	-9
64 101599064	TOX145, RBL10139-1000ppb-11, SEE	Standard	1.00	7.081	1392	bb	0.90	-10

Not a target analyte + not used in any calculations.

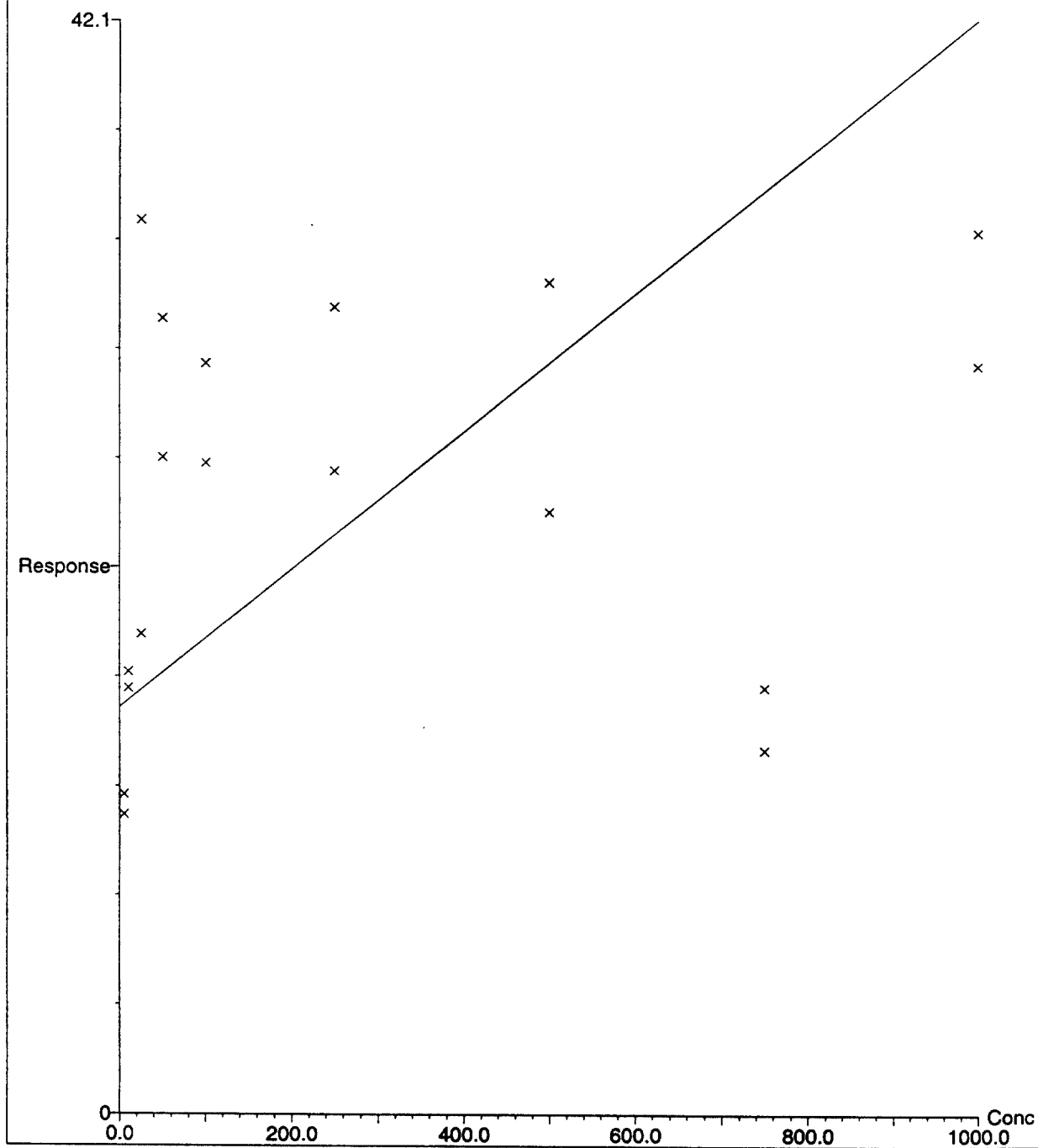
UC 10/22/99

IAS 10/18/99 p. 1 of 4

Quantify Calibration Report
TOX145- rat liver, day 1 & 4, control and dose- ext. 10/13/99

Page 1

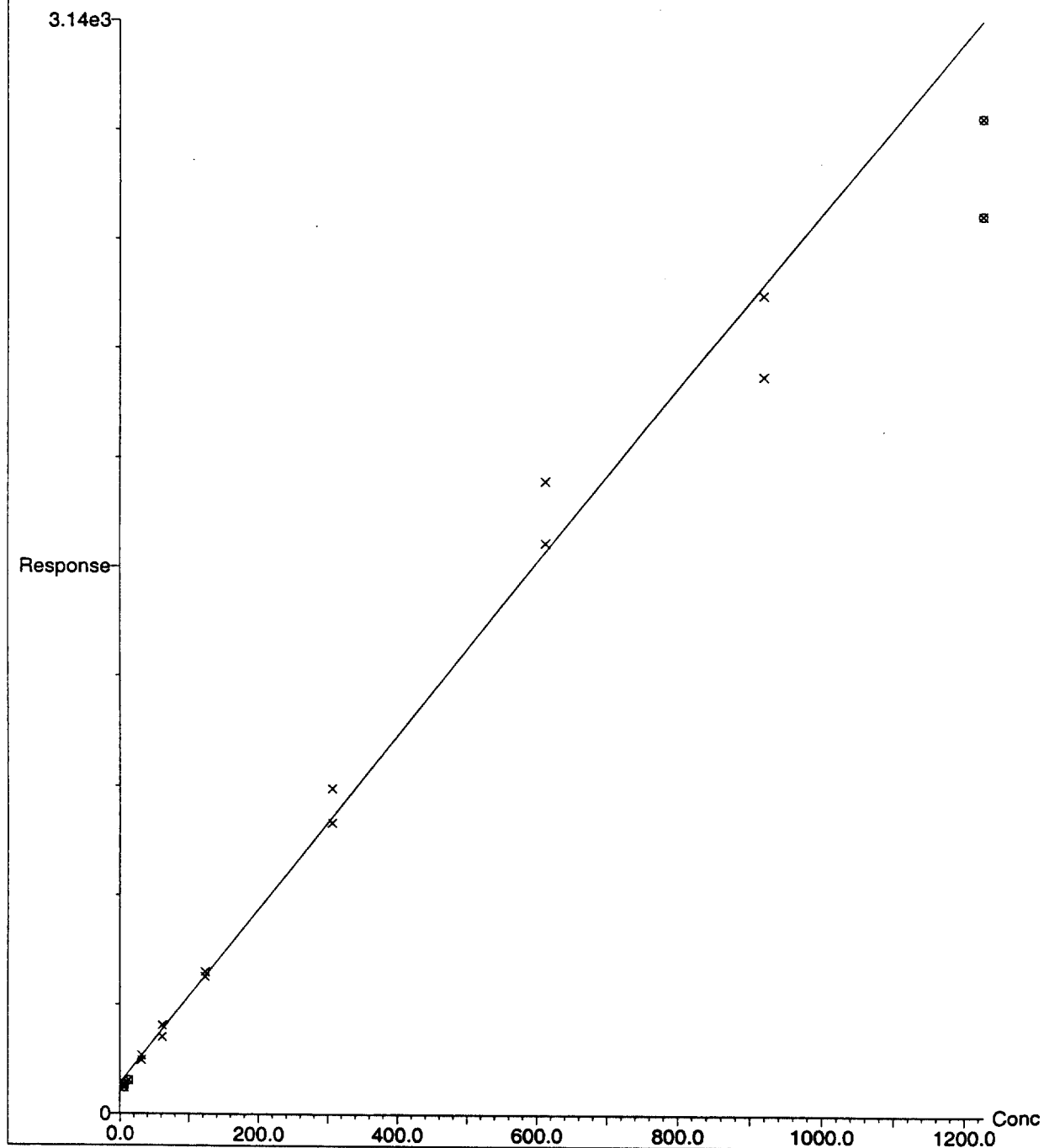
Compound 1 name: POAA (Ch1)
Coefficient of Determination: 89334.567901
Calibration curve: $0.0264689 * x + 15.6723$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Soup 020199: Quantified and printed by ias 10/18/99

000698

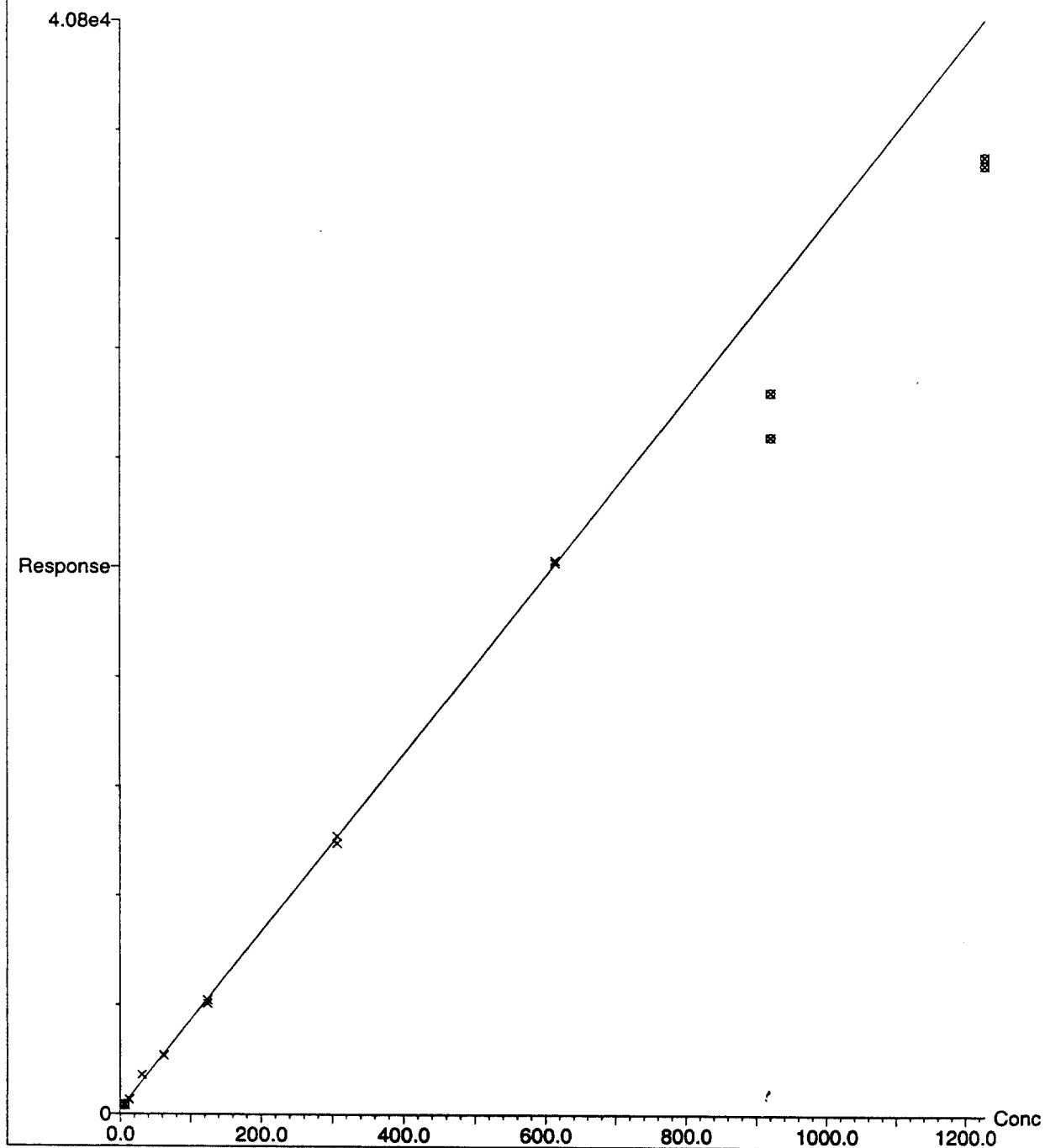
Compound 2 name: PFOS (Ch2)
Coefficient of Determination: 0.983941
Calibration curve: $2.48521 * x + 89.0578$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Soup 020199: Quantified and printed by las 10/18/99

000699

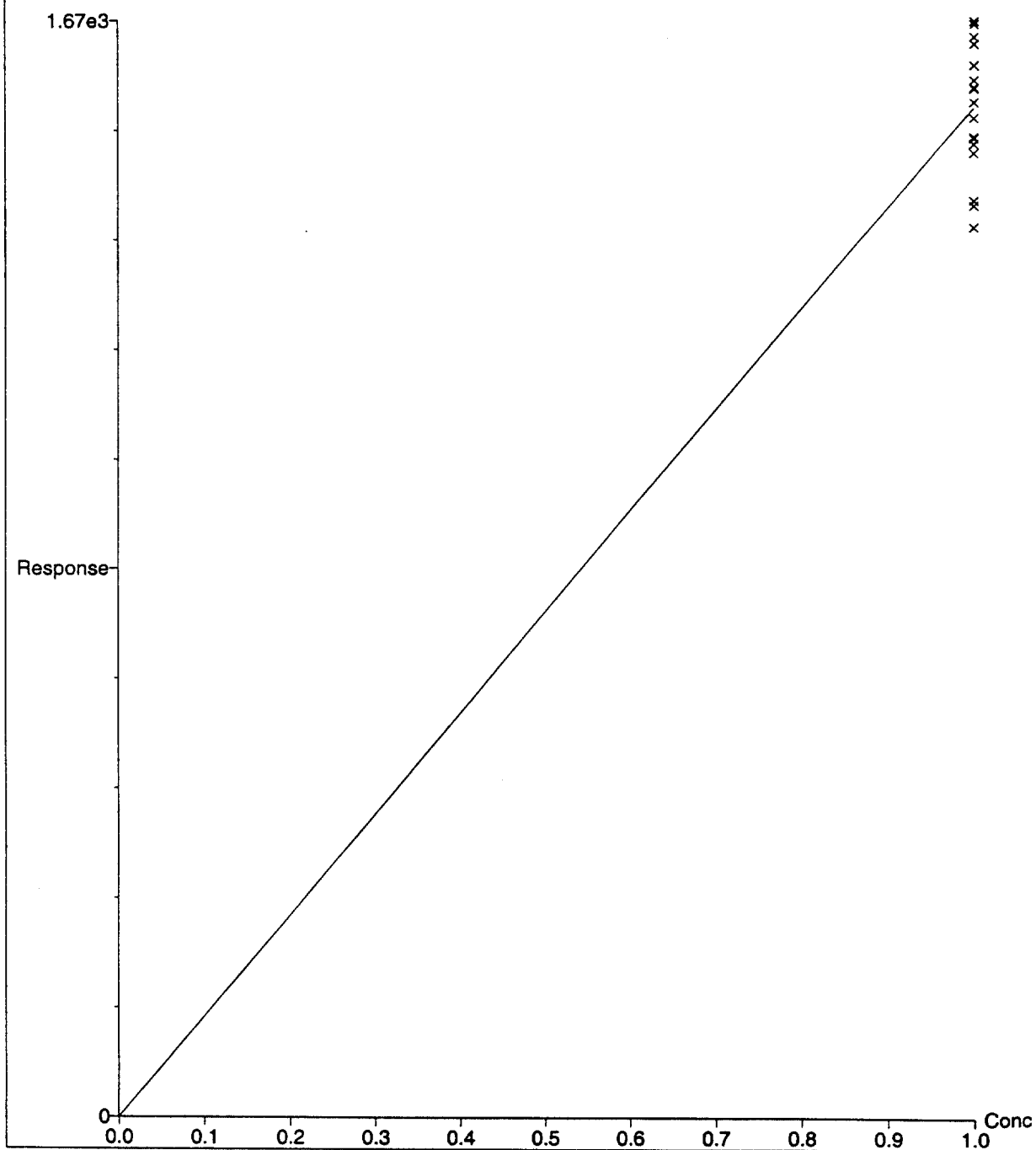
Compound 3 name: PFOSA (Ch3)
Coefficient of Determination: 0.999594
Calibration curve: $33.0992 * x + 198.731$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Soup 020199: Quantified and printed by ias 10/19/99

000700

Compound 4 name: IS (Ch4)
Response Factor: 1542.30
Response type: External Std, Area
Curve type: RF



Soup 020199: Quantified and printed by las 10/18/99

000701

Method Report

Method file: C:\MASSLYNK\asoup-rats.pro\MethDB\101599a.mdb
 Last modified: Mon Oct 18 06:41:21 1999

Compound: 1: POAA (Ch1)

Printed: Mon Oct 18 07:46:51 1999

Soup 020199 IAS 10/18/99

Compound Name: POAA (Ch1)
 Internal Ref.: [None]
 Quantify Trace: TIC
 Conc. of Standards: Conc. A
 User RF Value: 1.000000, Disabled
 User Peak Factor: 1.000000
 Integrate Window Factor: 2.000
 Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.129, Enabled
 Relative Retention Time: 0.000, Disabled
 RT Reference: [None]
 Time Window (mins): 0.356

Peak Selection: Nearest

Response Type: External
 Response: Areas

Polynomial Type: Linear
 Point of Origin: Exclude
 Fit Weighting: 1/X
 Axis Transformation: None

Units:

Peak-to-peak amplitude: 66

Smoothing: Enabled
 Window size (scans): 2
 Number of smooths: 1
 Smoothing method:

Join valley threshold: 30.00
 Reduce peak tailing: 200.00
 Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
 Detect shoulders: Disabled
 Shoulder threshold: 30.00

Relative height: 1.50, Disabled
 Absolute height: 0.00, Disabled
 Relative area: 2.00, Disabled
 Absolute area: 0.00, Disabled

Method Report

Page 2

Method file: C:\MASSLYNX\scup-rats.pro\MethDB\101599a.mdb
Last modified: Mon Oct 18 06:41:21 1999

Compound: 2: PFOS (Ch2)

Printed: Mon Oct 18 07:46:51 1999

Scup 020199 FAS 10/18/99

Compound Name: PFOS (Ch2)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. B
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.313, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.400

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 66

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000703

Method Report

Page 3

Method file: C:\MASSLYNX\soup-rats.pro\MethDB\101599a.mdb
Last modified: Mon Oct 18 06:41:21 1999

Compound: 3: PFOSA (Ch3)

Printed: Mon Oct 18 07:46:51 1999

Soup 020179 FMS 10/18/99

Compound Name: PFOSA (Ch3)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. C
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.797, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.679

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 80

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000704

Method Report

Page 4

Method file: C:\MASSLYNX\soup-rats.pro\MethDB\101599a.mdb
Last modified: Mon Oct 18 06:41:21 1999

Compound: 4: IS (Ch4)

Printed: Mon Oct 18 07:46:51 1999

Soup 22-199 Ins 10/18/99

Compound Name: IS (Ch4)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Fixed
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.157, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.411

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 67

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000705

Jas 12/15/99

SOUP 020199: FACT Equipment
Log 99092 PAGE 1/1

Quattro II, #6414E

DATE: 10/15/99

ANALYST: jas

STUDY NUMBER: TOX145 MATRIX: rat liver DESCRIPTION: days 1&4

FACT Method: ETS-8-7.0

Injection volume: 10 uL Cycle time: 10 min. Electrode: Z-Spray

Mobile Phase: (A) 2.0 mM NH₄CH₃CO₂ (B) MeOH

TN-A- or stdrd number (A) 99085-10-5 (B) TN-A-2878

Prepared (date/by): (A) 10/10/99 sah (B) 10/10/99 sah

Expires: (A) 04/00 (B) 2010

Store at room temp? ☒ Y or N Other: _____

Guard column: None

Part number: na Lot number: na

Analytical column: Keystone Betasil

Size: 2 x 50 mm Type: C18 Sphere size: 5 um

Part number: 055-701-2 Lot number: P9E11 Serial number: 0591804Z

Analytical parameters:

Drying gas (L/hr): 350 ESI nebulizing gas (L/hr) 13 Sheath gas (L/hr) 0

LC Flow rate: 300 uL/min. Back pressure: 65 bar

HPLC solvent gradient: 10min-10-95

Time (min.)	%B
1) 0.00	10.0
2) 1.00	10.0
3) 5.50	95.0
4) 7.50	95.0
5) 8.00	10.0

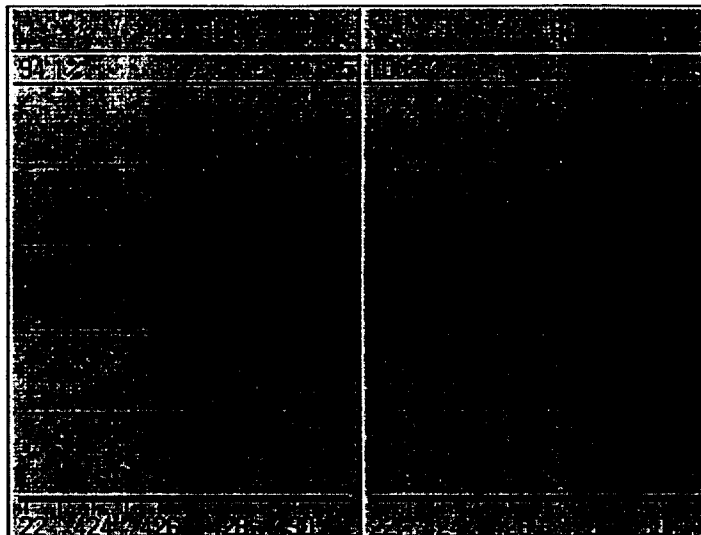
000706

Tuning Method Report

Page 1

Method: C:\MASSLYNX\SOUP-RATS.PRO\ACQUDE\PF083

Printed: Fri Oct 15 11:42:32 1999



BEST COPY AVAILABLE

MS

MS2

SOURCE (ESP-)	Set	Rdbk	MS1	Set	Rdbk
Capillary	3.00	3.00	LM Resolution	14.0	
Cone	40	-41	HM Resolution	14.0	
Extractor	5	-6	Ion Energy	0.9	
RF Lens	0.2		Ion Energy Ramp	0.0	
Source Block Temp.	150	153	Lens 6	4	3
Desolvation Temp.	250	250	Multiplier	650	

Pressures	Rdbk	MS2	Set	Rdbk
Analysar Vacuum	6.6e-5	LM Resolution	14.0	
		HM Resolution	14.0	
		Collision	10	7
		Ion Energy	2.0	
		Ion Energy Ramp	0.0	
		Lens 8	40	38
		Lens 9	0	-0
		Multiplier	650	

000707

Scanning Method Report

Page 1

Method: C:\MSSELYNK\SOUP-RATS.PRO\ACQUIS\10MIN-PP08-POAA-PP08A-IS

Last Modified: Fri Oct 15 11:35:03 1999

Printed: Fri Oct 15 11:42:48 1999

Solvent Delay (mins) : 0.00

Function : 1 MSN of 1 Mass Pair (MSP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	413.00	169.00	0.30	30	15

Function : 2 MSN of 1 Mass Pair (MSP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	499.00	99.00	0.30	45	60

Function : 3 MSN of 1 Mass Pair (MSP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	498.00	78.00	0.30	45	60

Function : 4 MSN of 1 Mass Pair (MSP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	427.00	80.00	0.30	40	60

000708

Method Report

Page 1

Method File: C:\MASSLYNX\SOUP-RATS.PRO\ACQUDE\10min-10-95
Last Modified: Friday, October 15, 1999 11:43:04
Printed: Friday, October 15, 1999 11:43:13

HP1100 LC Pump Initial Conditions

Solvents
A% 90.0
B% 10.0
C% 0.0
D% 0.0
Flow (ml/min) 0.300
Stop Time (mins) 10.0
Min Pressure (bar) 0
Max Pressure (bar) 400
Oven Temperature Left(°C) 30.0
Oven Temperature Right(°C) 30.0

HP1100 LC Pump Gradient Timetable

The gradient Timetable contains 5 entries which are :

Time	A%	B%	C%	D%	Flow	Pressure
0.00	90.0	10.0	0.0	0.0	0.300	400
1.00	90.0	10.0	0.0	0.0	0.300	400
5.50	5.0	95.0	0.0	0.0	0.300	400
7.50	5.0	95.0	0.0	0.0	0.300	400
8.00	90.0	10.0	0.0	0.0	0.300	400

HP1100 LC Pump External Event Timetable

The Timetable contains 3 entries which are :

Time	Column Switch	Contact1	Contact2	Contact3	Contact4
Initial	Off	Off	Off	Off	Off
0.00	Off	On	On	Off	On
0.10	Off	Off	Off	Off	Off

HP1100 Autosampler Initial Conditions

Draw Speed 200.0
Eject Speed (µl/min) 200
Draw Position (mm) 0.00
Stop Time (mins) 9.00
Injection Volume(µl) 5.0
Vial Number 91

000709

Sample List: C:\MASSLYNX\soup-rats.pro\SampleDB\101599a.SPL
Printed: Fri Oct 15 11:56:25 1999

Page Position: (1, 1)

	File Name	File Text	MS File
1	101599001	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
2	101599002	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
3	101599003	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
4	101599004	TOX145, RBL10139-H2O Blank-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
5	101599005	TOX145, RBL10139-Liver Blank-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
6	101599006	TOX145, RBL10139-5ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
7	101599007	TOX145, RBL10139-10ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
8	101599008	TOX145, RBL10139-25ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
9	101599009	TOX145, RBL10139-50ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
10	101599010	TOX145, RBL10139-100ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
11	101599011	TOX145, RBL10139-250ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
12	101599012	TOX145, RBL10139-500ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
13	101599013	TOX145, RBL10139-750ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
14	101599014	TOX145, RBL10139-1000ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
15	101599015	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
16	101599016	TOX145, RTL-9R03230M, Dy1, Control- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
17	101599017	TOX145, RTL-9R03231M, Dy1, Control- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
18	101599018	TOX145, RTL-9R03232M, Dy1, Control- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
19	101599019	TOX145, RTL-9R03233M, Dy1, Control- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
20	101599020	TOX145, RTL-9R03234M, Dy1, Control- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
21	101599021	TOX145, RBL10139-250ppb-CCV-11-1, SEE	10MIN-PFOS-POAA-PFOSA-IS
22	101599022	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
23	101599023	TOX145, RTL-9R03235M, Dy4, Control- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
24	101599024	TOX145, RTL-9R03236M, Dy4, Control- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
25	101599025	TOX145, RTL-9R03237M, Dy4, Control- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
26	101599026	TOX145, RTL-9R03238M, Dy4, Control- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
27	101599027	TOX145, RTL-9R03239M, Dy4, Control- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
28	101599028	TOX145, RBL10139-250ppb-CCV-11-2, SEE	10MIN-PFOS-POAA-PFOSA-IS
29	101599029	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
30	101599030	TOX145, 9R03230M, Dy1, Control-250pb-MS-11	10MIN-PFOS-POAA-PFOSA-IS
31	101599031	TOX145, 9R03230M, Dy1, Control-250pb-MSD-11	10MIN-PFOS-POAA-PFOSA-IS
32	101599032	TOX145, 9R03235M, Dy4, Control-250pb-MS-12	10MIN-PFOS-POAA-PFOSA-IS
33	101599033	TOX145, 9R03235M, Dy4, Control-250pb-MSD-12	10MIN-PFOS-POAA-PFOSA-IS
34	101599034	TOX145, RTL-9R03245M, Dy1, 5mg/kg- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
35	101599035	TOX145, RBL10139-250ppb-CCV-11-3, SEE	10MIN-PFOS-POAA-PFOSA-IS
36	101599036	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
37	101599037	TOX145, RBL10139-250ppb-CCV-11-3, SEE	10MIN-PFOS-POAA-PFOSA-IS
38	101599038	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
39	101599039	TOX145, RTL-9R03246M, Dy1, 5mg/kg- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
40	101599040	TOX145, RTL-9R03247M, Dy1, 5mg/kg- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
41	101599041	TOX145, RTL-9R03248M, Dy1, 5mg/kg- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
42	101599042	TOX145, RTL-9R03249M, Dy1, 5mg/kg- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
43	101599043	TOX145, RTL-9R03250M, Dy4, 5mg/kg- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
44	101599044	TOX145, RBL10139-250ppb-CCV-11-4, SEE	10MIN-PFOS-POAA-PFOSA-IS
45	101599045	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
46	101599046	TOX145, RBL10139-250ppb-CCV-11-4, SEE	10MIN-PFOS-POAA-PFOSA-IS
47	101599047	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
48	101599048	TOX145, RTL-9R03251M, Dy4, 5mg/kg- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
49	101599049	TOX145, RTL-9R03252M, Dy4, 5mg/kg- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
50	101599050	TOX145, RTL-9R03253M, Dy4, 5mg/kg- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
51	101599051	TOX145, RTL-9R03254M, Dy4, 5mg/kg- SEE 10/13/99	10MIN-PFOS-POAA-PFOSA-IS
52	101599052	TOX145, RBL10139-250ppb-CCV-11-4, SEE	10MIN-PFOS-POAA-PFOSA-IS
53	101599053	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
54	101599054	TOX145, RBL10139-250ppb-CCV-11-4, SEE	10MIN-PFOS-POAA-PFOSA-IS
55	101599055	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
56	101599056	TOX145, RBL10139-5ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
57	101599057	TOX145, RBL10139-10ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
58	101599058	TOX145, RBL10139-25ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
59	101599059	TOX145, RBL10139-50ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS

Masslynx - Sample List

Page 2

Sample List: C:\MASSLYNX\soup-rats.pro\SampleDB\101599a.SPL
Printed: Fri Oct 15 11:56:25 1999

Page Position: (2, 1)

	Inlet File	Bottle	Inject Volume	Sample Type	Conc A
1	10min-10-95	91	10.000	---	---
2	10min-10-95	91	10.000	---	---
3	10min-10-95	91	10.000	---	---
4	10min-10-95	1	10.000	---	---
5	10min-10-95	2	10.000	---	---
6	10min-10-95	3	10.000	---	---
7	10min-10-95	4	10.000	---	---
8	10min-10-95	5	10.000	---	---
9	10min-10-95	6	10.000	---	---
10	10min-10-95	7	10.000	---	---
11	10min-10-95	8	10.000	---	---
12	10min-10-95	9	10.000	---	---
13	10min-10-95	10	10.000	---	---
14	10min-10-95	11	10.000	---	---
15	10min-10-95	92	10.000	---	---
16	10min-10-95	12	10.000	---	---
17	10min-10-95	13	10.000	---	---
18	10min-10-95	14	10.000	---	---
19	10min-10-95	15	10.000	---	---
20	10min-10-95	16	10.000	---	---
21	10min-10-95	36	10.000	---	---
22	10min-10-95	92	10.000	---	---
23	10min-10-95	17	10.000	---	---
24	10min-10-95	18	10.000	---	---
25	10min-10-95	19	10.000	---	---
26	10min-10-95	20	10.000	---	---
27	10min-10-95	21	10.000	---	---
28	10min-10-95	37	10.000	---	---
29	10min-10-95	93	10.000	---	---
30	10min-10-95	22	10.000	---	---
31	10min-10-95	23	10.000	---	---
32	10min-10-95	24	10.000	---	---
33	10min-10-95	25	10.000	---	---
34	10min-10-95	26	10.000	---	---
35	10min-10-95	38	10.000	---	---
36	10min-10-95	93	10.000	---	---
37	10min-10-95	38	10.000	---	---
38	10min-10-95	93	10.000	---	---
39	10min-10-95	27	10.000	---	---
40	10min-10-95	28	10.000	---	---
41	10min-10-95	29	10.000	---	---
42	10min-10-95	30	10.000	---	---
43	10min-10-95	31	10.000	---	---
44	10min-10-95	39	10.000	---	---
45	10min-10-95	94	10.000	---	---
46	10min-10-95	39	10.000	---	---
47	10min-10-95	94	10.000	---	---
48	10min-10-95	32	10.000	---	---
49	10min-10-95	33	10.000	---	---
50	10min-10-95	34	10.000	---	---
51	10min-10-95	35	10.000	---	---
52	10min-10-95	39	10.000	---	---
53	10min-10-95	95	10.000	---	---
54	10min-10-95	39	10.000	---	---
55	10min-10-95	95	10.000	---	---
56	10min-10-95	3	10.000	---	---
57	10min-10-95	4	10.000	---	---
58	10min-10-95	5	10.000	---	---
59	10min-10-95	6	10.000	---	---

000711

Masslynx - Sample List**Page 3****Sample List:** C:\MASSLYNX\soup-rats.pro\SampleDB\101599a.SPL
Printed: Fri Oct 15 11:56:26 1999**Page Position:** (1, 2)

	File Name	File Text	MS File
60	101599060	TOX145, RBL10139-100ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
61	101599061	TOX145, RBL10139-250ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
62	101599062	TOX145, RBL10139-500ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
63	101599063	TOX145, RBL10139-750ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS
64	101599064	TOX145, RBL10139-1000ppb-11, SEE	10MIN-PFOS-POAA-PFOSA-IS

000712

Masslynx - Sample List

Page 4

Sample List: C:\MASSLYNX\soup-rats.pro\SampleDB\101599a.SPL
Printed: Fri Oct 15 11:56:26 1999

Page Position: (2, 2)

	Inlet File	Bottle	Inject Volume	Sample Type	Conc A
60	10min-10-95	7	10.000	--	--
61	10min-10-95	8	10.000	--	--
62	10min-10-95	9	10.000	--	--
63	10min-10-95	10	10.000	--	--
64	10min-10-95	11	10.000	--	--

000713

FACT: DATA REVIEW SUMMARY FORM

P/2

Date: 10-20-99
 Data Analyst: MMX
 FACT Study #: Tox 145 (2)
 Matrix/Timepoints: But for ^{live} day 1, 4 Dilutions
 Sample List: 10/9990
 Set-up Analysis Date/Analyst: MMX 10-19-99
 Instrument: Amelia DMS-95

Data Review Sign-Off

Analyst Review Name / Date: MMX 10-20-99

Technical Review Name / Date: MEE 10/20/99 / kh 10/20/99

Quality Assurance Review Name / Date: _____

Data Entry: LAC 10/22/99

Comments / Justifications: _____

Analyte: PFOS
 Acceptable Linear Range: 24.80 - 976.0 ppb
 LOQ: 24.80 Extraction Blank: < 1/2 LOQ
 R²: 0.9898 Matrix Blank: < 1/2 LOQ
 Calibration Checks: OK
 MS/MSD: OK
 Data Status: (1) Entered LAC 10/22/99

Analyte: PFOSA
 Acceptable Linear Range: 4.93 - 493 ppb
 LOQ: 4.93 ppb Extracted Blank: < 1/2 LOQ
 R²: 0.9979 Matrix Blank: < 1/2 LOQ
 Calibration Checks: OK
 MS/MSD: OK
 Data Status: (2) Entered LAC 10/22/99

Analyte: POPA (3)
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: used to monitor levels of POPA not for quantitation.

- ① excluded lowest two points MMX 10-20-99
to meet 30% deviation criteria
- ② excluded highest two points MMX 10-20-99
to meet R² criteria.
- ③ POAA WAS NOT INCLUDED IN
THE CALIBRATION CURVE BECAUSE POAA
WAS NOT CONSIDERED TO BE A PRIMARY
TARGET ANALYTE. MEE 10/20/99.
- ④ SAMPLE DILUENT DID NOT
CONTAIN INTERNAL STANDARD.
SAMPLES WERE QUANTITATED VS AN
EXTERNAL STANDARD CURVE. MEE 10/20/99.

LAC 10/22/99

000714

FACT: DATA REVIEW SUMMARY FORM

P2/2

Date: 10-20-99
 Data Analyst: MMN
 FACT Study #: Tox 145
 Matrix/Timepoints: But Liver Day 1, 4 D1
 Sample List: 101999a
 Set-up Analysis Date/Analyst: 101999 MMN
 Instrument: Amelia R23498

Data Review Sign-Off

Analyst Review Name / Date: MMN 10-20-99

Technical Review Name / Date: Refer to page 1 of 2 LAC 10/22/99

Quality Assurance Review Name / Date: _____

Data Entry: LAC 10/22/99

Comments / Justifications: _____

Analyte: Sur (4)
 Acceptable Linear Range: _____
 LOQ: _____ Extraction Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: _____

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: LAC 10/21/99
 MS/MSD: _____
 Data Status: _____

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: LAC 10/21/99
 MS/MSD: _____
 Data Status: _____

mmh
10/20/99
P1/4
Page 1

Quantify Compound Summary Report
Tox 145: Rat ~~Sees~~ Dilutions, Day 1,4
Sample List: C:\MSL\TOK\amelia-rats.FPO\Samples\101999a
Last modified: Wed Oct 20 08:31:47 1999
Method: C:\MSL\TOK\amelia-rats.FPO\Methods\101999a
Last modified: Wed Oct 20 08:16:18 1999
Job Code:

Printed: Wed Oct 20 09:00:41 1999

Compound 1: PFO8

#	Name	Samplelist Text	Type	Std Conc	RT	Area	Conc.	Flags	%Dev
1	101999001	MeOH			7.159	33	0.00	bb	
2	101999002	MeOH			7.171	29	0.00	bb	
3	101999003	Tox 145: RBL10139-H2O blk-11, Ext SEE 10-13-99			7.167	109	0.00	bb	
4	101999004	Tox 145: RBL10139-Liver blk-11, Ext SEE 10-13-99			7.140	127	0.00	bb	
5	101999005	Tox 145: RBL10139-5 ppb-11, Ext SEE 10-13-99	Standard	4.93	7.123	266	0.00	MMX	-100
6	101999006	Tox 145: RBL10139-10 ppb-11, Ext SEE 10-13-99	Standard	9.76	7.120	326	0.00	MMX	-100
7	101999007	Tox 145: RBL10139-25 ppb-11, Ext SEE 10-13-99	Standard	24.80	7.120	563	25.44	MM	3
8	101999008	Tox 145: RBL10139-50 ppb-11, Ext SEE 10-13-99	Standard	49.30	7.137	778	51.46	MM	4
9	101999009	Tox 145: RBL10139-100 ppb-11, Ext SEE 10-13-99	Standard	97.60	7.120	1265	110.57	MM	13
10	101999010	Tox 145: RBL10139-250 ppb-11, Ext SEE 10-13-99	Standard	248.00	7.141	2818	298.68	MM	20
11	101999011	Tox 145: RBL10139-500 ppb-11, Ext SEE 10-13-99	Standard	493.00	7.118	4996	562.73	MM	14
12	101999012	Tox 145: RBL10139-750 ppb-11, Ext SEE 10-13-99	Standard	735.00	7.120	6355	727.43	MM	-1
13	101999013	Tox 145: RBL10139-1000 ppb-11, Ext SEE 10-13-99	Standard	976.00	7.119	8033	930.73	MM	-5
14	101999014	MeOH			7.149	41	0.00	bb	
15	101999015	Tox 145: 9R03237M, Day 4, Control, D:100			7.119	3396	368.79	bd	+
16	101999016	Tox 145: 9R03245M, Day 1, 5 mg/kg, D:100			7.138	2339	240.65	bb	
17	101999017	Tox 145: 9R03246M, Day 1, 5 mg/kg, D:100			7.141	3037	325.31	MM	
18	101999018	Tox 145: 9R03247M, Day 1, 5 mg/kg, D:100			7.139	3370	365.59	MM	
19	101999019	Tox 145: 9R03248M, Day 1, 5 mg/kg, D:100			7.139	2132	215.58	MM	
20	101999020	Tox 145: RBL10139-250 ppb-11-2, Ext SEE 10-13-99	QC	248.00	7.144	2043	204.85	MM	-17
21	101999021	MeOH			7.163	25	0.00	bb	
22	101999022	Tox 145: 9R03249M, Day 1, 5 mg/kg, D:100			7.146	2630	276.01	MM	
23	101999023	Tox 145: 9R03251M, Day 4, 5 mg/kg, D:100			7.145	3050	326.91	MM	
24	101999024	Tox 145: 9R03252M, Day 4, 5 mg/kg, D:100			7.143	3897	429.51	bb	
25	101999025	Tox 145: 9R03253M, Day 4, 5 mg/kg, D:100			7.145	3395	368.64	MM	
26	101999026	Tox 145: 9R03254M, Day 4, 5 mg/kg, D:100			7.168	3570	389.84	bb	
27	101999027	Tox 145: RBL10139-250 ppb-11-1, Ext SEE 10-13-99	QC	248.00	7.166	1805	176.02	MM	-29
28	101999028	MeOH			7.165	35	0.00	bb	
29	101999029	Tox 145: RBL10139-H2O blk-11, Ext SEE 10-13-99			7.191	98	0.00	bb	
30	101999030	Tox 145: RBL10139-Liver blk-11, Ext SEE 10-13-99			7.189	118	0.00	MM	
31	101999031	Tox 145: RBL10139-5 ppb-11, Ext SEE 10-13-99	Standard	4.93	7.165	270	0.00	MMX	-100
32	101999032	Tox 145: RBL10139-10 ppb-11, Ext SEE 10-13-99	Standard	9.76	7.140	289	0.00	MMX	-100
33	101999033	Tox 145: RBL10139-25 ppb-11, Ext SEE 10-13-99	Standard	24.80	7.149	498	17.53	bb	-29
34	101999034	Tox 145: RBL10139-50 ppb-11, Ext SEE 10-13-99	Standard	49.30	7.148	707	42.92	bb	-13
35	101999035	Tox 145: RBL10139-100 ppb-11, Ext SEE 10-13-99	Standard	97.60	7.120	1087	88.98	MM	-9
36	101999036	Tox 145: RBL10139-250 ppb-11, Ext SEE 10-13-99	Standard	248.00	7.145	2653	278.80	MM	12
37	101999037	Tox 145: RBL10139-500 ppb-11, Ext SEE 10-13-99	Standard	493.00	7.142	4435	494.75	MM	0
38	101999038	Tox 145: RBL10139-750 ppb-11, Ext SEE 10-13-99	Standard	735.00	7.123	6109	697.60	MM	-5
39	101999039	Tox 145: RBL10139-1000 ppb-11, Ext SEE 10-13-99	Standard	976.00	7.141	7943	919.78	MM	-6

+ This animal number may have been switched with animal # 9R0250M, Day 4, 5 mg/kg.

000716

Quantify Compound Summary Report
Tox 145: Rat ~~Surv~~ Dilutions, Day 1, 4

Page 2

Sample List: C:\MASSL\TOK\amelia-rats.FPO\SampleDB\1019999
Last modified: Wed Oct 20 08:31:47 1999
Method: C:\MASSL\TOK\amelia-rats.FPO\MethDB\1019999
Last modified: Wed Oct 20 08:16:18 1999
Job Code:

Printed: Wed Oct 20 09:00:41 1999

Compound 2: PFOCA

# Name	Samplelist Text	Type	Std Conc	RT	Area	Conc.	Flags	tDev
1 101999001	MeOH			7.841	24	0.00	bb	
2 101999002	MeOH			7.875	19	0.00	bb	
3 101999003	Tox 145: RBL10139-M20 blk-11, Ext SEE 10-13-99			7.848	29	0.21	bb	
4 101999004	Tox 145: RBL10139-Liver blk-11, Ext SEE 10-13-99			7.821	37	0.51	MM	
5 101999005	Tox 145: RBL10139-5 ppb-11, Ext SEE 10-13-99	Standard	4.93	7.803	146	4.61	MM	-7
6 101999006	Tox 145: RBL10139-10 ppb-11, Ext SEE 10-13-99	Standard	9.77	7.800	247	8.41	MM	-14
7 101999007	Tox 145: RBL10139-25 ppb-11, Ext SEE 10-13-99	Standard	24.80	7.801	810	29.60	bb	19
8 101999008	Tox 145: RBL10139-50 ppb-11, Ext SEE 10-13-99	Standard	49.30	7.817	1304	48.21	MM	-2
9 101999009	Tox 145: RBL10139-100 ppb-11, Ext SEE 10-13-99	Standard	97.70	7.800	2496	93.11	bb	-5
10 101999010	Tox 145: RBL10139-250 ppb-11, Ext SEE 10-13-99	Standard	248.00	7.798	6404	240.28	bd	-3
11 101999011	Tox 145: RBL10139-500 ppb-11, Ext SEE 10-13-99	Standard	493.00	7.799	12860	483.44	MM	-2
12 101999012	Tox 145: RBL10139-750 ppb-11, Ext SEE 10-13-99	Standard	736.00	7.800	14433	542.66	MM	-26
13 101999013	Tox 145: RBL10139-1000 ppb-11, Ext SEE 10-13-99	Standard	977.00	7.800	20057	754.50	MM	-23
14 101999014	MeOH			7.852	30	0.23	bb	
15 101999015	Tox 145: 9R03237M, Day 4, Control, D:100			7.822	961	35.31	MM	+
16 101999016	Tox 145: 9R03245M, Day 1, 5 mg/kg, D:100			7.819	1650	61.24	MM	
17 101999017	Tox 145: 9R03246M, Day 1, 5 mg/kg, D:100			7.822	1902	70.74	MM	
18 101999018	Tox 145: 9R03247M, Day 1, 5 mg/kg, D:100			7.844	1698	63.05	MM	
19 101999019	Tox 145: 9R03248M, Day 1, 5 mg/kg, D:100			7.820	1821	67.70	MM	
20 101999020	Tox 145: RBL10139-250 ppb-11-2, Ext SEE 10-13-99	QC	248.00	7.799	6666	250.15	MM	
21 101999021	MeOH			7.820	6	0.00	bd	
22 101999022	Tox 145: 9R03249M, Day 1, 5 mg/kg, D:100			7.826	2344	87.39	MM	
23 101999023	Tox 145: 9R03251M, Day 4, 5 mg/kg, D:100			7.825	947	34.78	bb	
24 101999024	Tox 145: 9R03252M, Day 4, 5 mg/kg, D:100			7.826	1083	39.91	MM	
25 101999025	Tox 145: 9R03253M, Day 4, 5 mg/kg, D:100			7.826	939	34.46	bb	
26 101999026	Tox 145: 9R03254M, Day 4, 5 mg/kg, D:100			7.849	1134	41.82	bb	
27 101999027	Tox 145: RBL10139-250 ppb-11-1, Ext SEE 10-13-99	QC	248.00	7.822	7149	268.36	MM	
28 101999028	MeOH			7.870	32	0.32	bb	
29 101999029	Tox 145: RBL10139-M20 blk-11, Ext SEE 10-13-99			7.871	60	1.38	bb	
30 101999030	Tox 145: RBL10139-Liver blk-11, Ext SEE 10-13-99			7.845	53	1.11	bb	
31 101999031	Tox 145: RBL10139-5 ppb-11, Ext SEE 10-13-99	Standard	4.93	7.821	147	4.65	bb	-6
32 101999032	Tox 145: RBL10139-10 ppb-11, Ext SEE 10-13-99	Standard	9.77	7.820	271	9.32	bb	-5
33 101999033	Tox 145: RBL10139-25 ppb-11, Ext SEE 10-13-99	Standard	24.80	7.829	864	31.65	bb	28
34 101999034	Tox 145: RBL10139-50 ppb-11, Ext SEE 10-13-99	Standard	49.30	7.806	1312	48.51	bb	-2
35 101999035	Tox 145: RBL10139-100 ppb-11, Ext SEE 10-13-99	Standard	97.70	7.798	2454	91.53	bb	-6
36 101999036	Tox 145: RBL10139-250 ppb-11, Ext SEE 10-13-99	Standard	248.00	7.826	6538	245.33	bb	-1
37 101999037	Tox 145: RBL10139-500 ppb-11, Ext SEE 10-13-99	Standard	493.00	7.823	13734	516.36	MM	5
38 101999038	Tox 145: RBL10139-750 ppb-11, Ext SEE 10-13-99	Standard	736.00	7.806	15935	599.24	MM	-19
39 101999039	Tox 145: RBL10139-1000 ppb-11, Ext SEE 10-13-99	Standard	977.00	7.821	20765	781.16	MM	-20

Entered
LAC
10/22/99

+ This animal number may have been switched with animal # 9R03250M, Day 4, 5 mg/kg

LAC
10/22/99

Quantify Compound Summary Report
Tox 145: Rat Liver, Dilutions, Day 1,4
Sample List: C:\MSL\YOK\amelia-rats.FRO\SampleDB\101990a
Last modified: Wed Oct 20 08:31:47 1999
Method: C:\MSL\YOK\amelia-rats.FRO\MethDB\101999a
Last modified: Wed Oct 20 08:16:18 1999
Job Code:
Printed: Wed Oct 20 09:00:41 1999

Page 3

Compound 3: POAA

# Name	Samplelist Text	Type	Std Conc	RT	Area	Conc.	Flags	tDev
1 101999001	MeOH							
2 101999002	MeOH							
3 101999003	Tox 145: RBL10139-H2O blk-11, Ext SEE 10-13-99			6.971	87	2.61	bb	161
4 101999004	Tox 145: RBL10139-Liver blk-11, Ext SEE 10-13-99			6.968	31	0.92	bb	-8
5 101999005	Tox 145: RBL10139-5 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.974	29	0.86	bb	-14
6 101999006	Tox 145: RBL10139-10 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.947	29	0.88	bb	-12
7 101999007	Tox 145: RBL10139-25 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.971	54	1.63	MM	63
8 101999008	Tox 145: RBL10139-50 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.964	34	1.02	MM	2
9 101999009	Tox 145: RBL10139-100 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.972	36	1.07	bb	7
10 101999010	Tox 145: RBL10139-250 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.969	38	1.15	bb	15
11 101999011	Tox 145: RBL10139-500 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.946	31	0.92	bb	-8
12 101999012	Tox 145: RBL10139-750 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.971	19	0.56	bb	-44
13 101999013	Tox 145: RBL10139-1000 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.946	41	1.23	bb	23
14 101999014	MeOH							
15 101999015	Tox 145: 9R03237M, Day 4, Control, D:100			6.970	17	0.51	bb	-49
16 101999016	Tox 145: 9R03245M, Day 1, 5 mg/kg, D:100			6.965	16	0.48	bb	-52
17 101999017	Tox 145: 9R03246M, Day 1, 5 mg/kg, D:100			6.993	18	0.53	bb	-47
18 101999018	Tox 145: 9R03247M, Day 1, 5 mg/kg, D:100			6.967	19	0.56	bb	-44
19 101999019	Tox 145: 9R03248M, Day 1, 5 mg/kg, D:100			6.968	18	0.54	bb	-46
20 101999020	Tox 145: RBL10139-250 ppb-11-2, Ext SEE 10-13-99	QC	1.00	6.997	65	1.94	bb	94
21 101999021	MeOH							
22 101999022	Tox 145: 9R03249M, Day 1, 5 mg/kg, D:100			6.973	24	0.72	bb	-28
23 101999023	Tox 145: 9R03251M, Day 4, 5 mg/kg, D:100			6.974	29	0.87	bb	-13
24 101999024	Tox 145: 9R03252M, Day 4, 5 mg/kg, D:100			6.972	27	0.82	bb	-18
25 101999025	Tox 145: 9R03253M, Day 4, 5 mg/kg, D:100			6.948	19	0.56	bb	-44
26 101999026	Tox 145: 9R03254M, Day 4, 5 mg/kg, D:100			6.996	20	0.59	MM	-41
27 101999027	Tox 145: RBL10139-250 ppb-11-1, Ext SEE 10-13-99	QC	1.00	6.994	45	1.34	bb	34
28 101999028	MeOH							
29 101999029	Tox 145: RBL10139-H2O blk-11, Ext SEE 10-13-99			6.994	109	3.27	bb	227
30 101999030	Tox 145: RBL10139-Liver blk-11, Ext SEE 10-13-99			7.016	35	1.06	bb	6
31 101999031	Tox 145: RBL10139-5 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.992	19	0.57	bb	-43
32 101999032	Tox 145: RBL10139-10 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.969	27	0.82	bb	-18
33 101999033	Tox 145: RBL10139-25 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.976	41	1.23	bb	23
34 101999034	Tox 145: RBL10139-50 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.975	29	0.88	bb	-12
35 101999035	Tox 145: RBL10139-100 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.948	38	1.14	bb	14
36 101999036	Tox 145: RBL10139-250 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.971	38	1.13	bb	13
37 101999037	Tox 145: RBL10139-500 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.970	35	1.07	bb	7
38 101999038	Tox 145: RBL10139-750 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.974	22	0.65	bb	-35
39 101999039	Tox 145: RBL10139-1000 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.968	40	1.20	bb	20

Data not used, not a target analyte for this study

UC 10/22/99

Quantify Compound Summary Report
Tox 145: Rat 6666-Dilutions, Day 1,4

Page 4

Sample List: C:\MSL\TOK\Amelia-rats.PRO\SampleDB\101999a
Last modified: Wed Oct 20 08:31:47 1999
Method: C:\MSL\TOK\Amelia-rats.PRO\MethDB\101999a
Last modified: Wed Oct 20 08:16:18 1999
Job Code:

Printed: Wed Oct 20 09:00:41 1999

Compound 4: 18 (Ch2)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Conc.	Flags	Dev
1 101999001	MeOH							
2 101999002	MeOH							
3 101999003	Tox 145: RBL10139-H2O blk-11, Ext SEE 10-13-99			6.982	2946	0.74	bb	-26
4 101999004	Tox 145: RBL10139-Liver blk-11, Ext SEE 10-13-99			6.955	3227	0.81	bb	-19
5 101999005	Tox 145: RBL10139-5 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.961	4774	1.20	bb	20
6 101999006	Tox 145: RBL10139-10 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.958	4858	1.22	bb	22
7 101999007	Tox 145: RBL10139-25 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.958	4227	1.06	bb	6
8 101999008	Tox 145: RBL10139-50 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.951	4612	1.16	bb	16
9 101999009	Tox 145: RBL10139-100 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.960	3958	1.00	bb	-0
10 101999010	Tox 145: RBL10139-250 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.956	4194	1.05	bb	-5
11 101999011	Tox 145: RBL10139-500 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.957	3767	0.95	bb	-5
12 101999012	Tox 145: RBL10139-750 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.958	3827	0.96	bb	-4
13 101999013	Tox 145: RBL10139-1000 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.934	3281	0.83	bb	-17
14 101999014	MeOH							
15 101999015	Tox 145: 9R03237M, Day 4, Control, D:100			6.957	30	0.01	bb	-99
16 101999016	Tox 145: 9R03245M, Day 1, 5 mg/kg, D:100			6.976	36	0.01	bd	-99
17 101999017	Tox 145: 9R03246M, Day 1, 5 mg/kg, D:100			6.980	43	0.01	bd	-99
18 101999018	Tox 145: 9R03247M, Day 1, 5 mg/kg, D:100			6.978	34	0.01	bd	-99
19 101999019	Tox 145: 9R03248M, Day 1, 5 mg/kg, D:100			6.955	32	0.01	bb	-99
20 101999020	Tox 145: RBL10139-250 ppb-11-2, Ext SEE 10-13-99	QC	1.00	6.984	2585	0.65	bb	-35
21 101999021	MeOH							
22 101999022	Tox 145: 9R03249M, Day 1, 5 mg/kg, D:100			6.984	32	0.01	bb	-99
23 101999023	Tox 145: 9R03251M, Day 4, 5 mg/kg, D:100			6.961	38	0.01	bb	-99
24 101999024	Tox 145: 9R03252M, Day 4, 5 mg/kg, D:100			6.982	28	0.01	bd	-99
25 101999025	Tox 145: 9R03253M, Day 4, 5 mg/kg, D:100			6.960	27	0.01	bd	-99
26 101999026	Tox 145: 9R03254M, Day 4, 5 mg/kg, D:100			6.983	35	0.01	bd	-99
27 101999027	Tox 145: RBL10139-250 ppb-11-1, Ext SEE 10-13-99	QC	1.00	6.980	2759	0.69	bb	-31
28 101999028	MeOH							
29 101999029	Tox 145: RBL10139-H2O blk-11, Ext SEE 10-13-99			6.980	8	0.00	bb	-100
30 101999030	Tox 145: RBL10139-Liver blk-11, Ext SEE 10-13-99			7.007	2979	0.75	bb	-25
31 101999031	Tox 145: RBL10139-5 ppb-11, Ext SEE 10-13-99	Standard	1.00	7.003	3288	0.83	bb	-17
32 101999032	Tox 145: RBL10139-10 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.979	3941	0.99	bb	-1
33 101999033	Tox 145: RBL10139-25 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.956	4388	1.10	bb	10
34 101999034	Tox 145: RBL10139-50 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.987	4134	1.04	bb	4
35 101999035	Tox 145: RBL10139-100 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.963	4085	1.03	bd	3
36 101999036	Tox 145: RBL10139-250 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.959	3571	0.90	bb	-10
37 101999037	Tox 145: RBL10139-500 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.958	3937	0.99	bd	-1
38 101999038	Tox 145: RBL10139-750 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.957	3460	0.87	bb	-13
39 101999039	Tox 145: RBL10139-1000 ppb-11, Ext SEE 10-13-99	Standard	1.00	6.961	3218	0.81	bb	-19

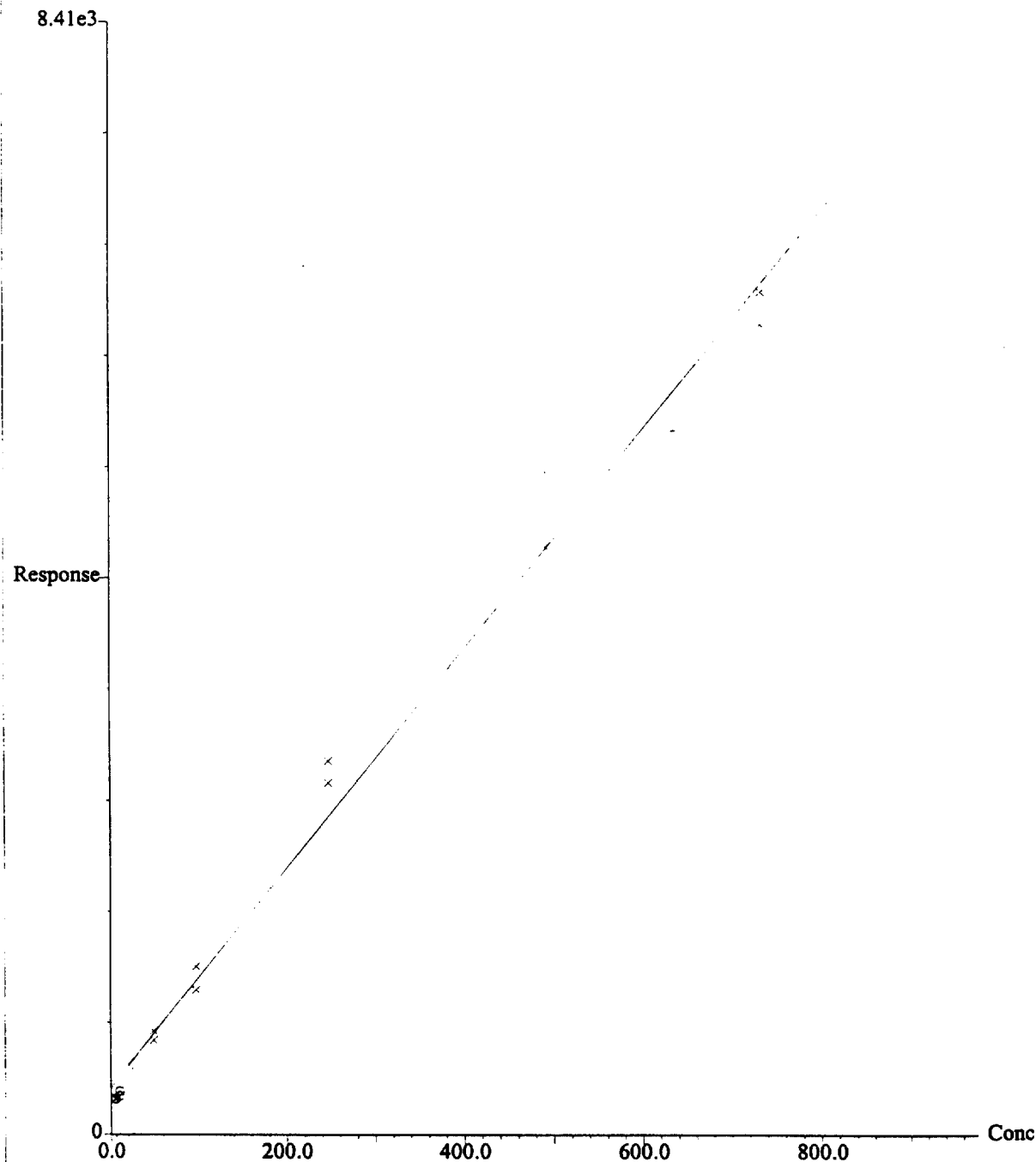
① mmh 10/21/99 (RC)

Data not used.

LAC 10/22/99

mmh
10-20-99
p14
Page 1Quantify Calibration Report
Tox 145: Rat ~~perf~~ Dilutions, Day 1,4

Compound 1 name: PFOS
Coefficient of Determination: 0.989844
Calibration curve: $8.25151 * x + 352.987$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Amelia 062498: Quantitated and Printed by mmh 10/20/99

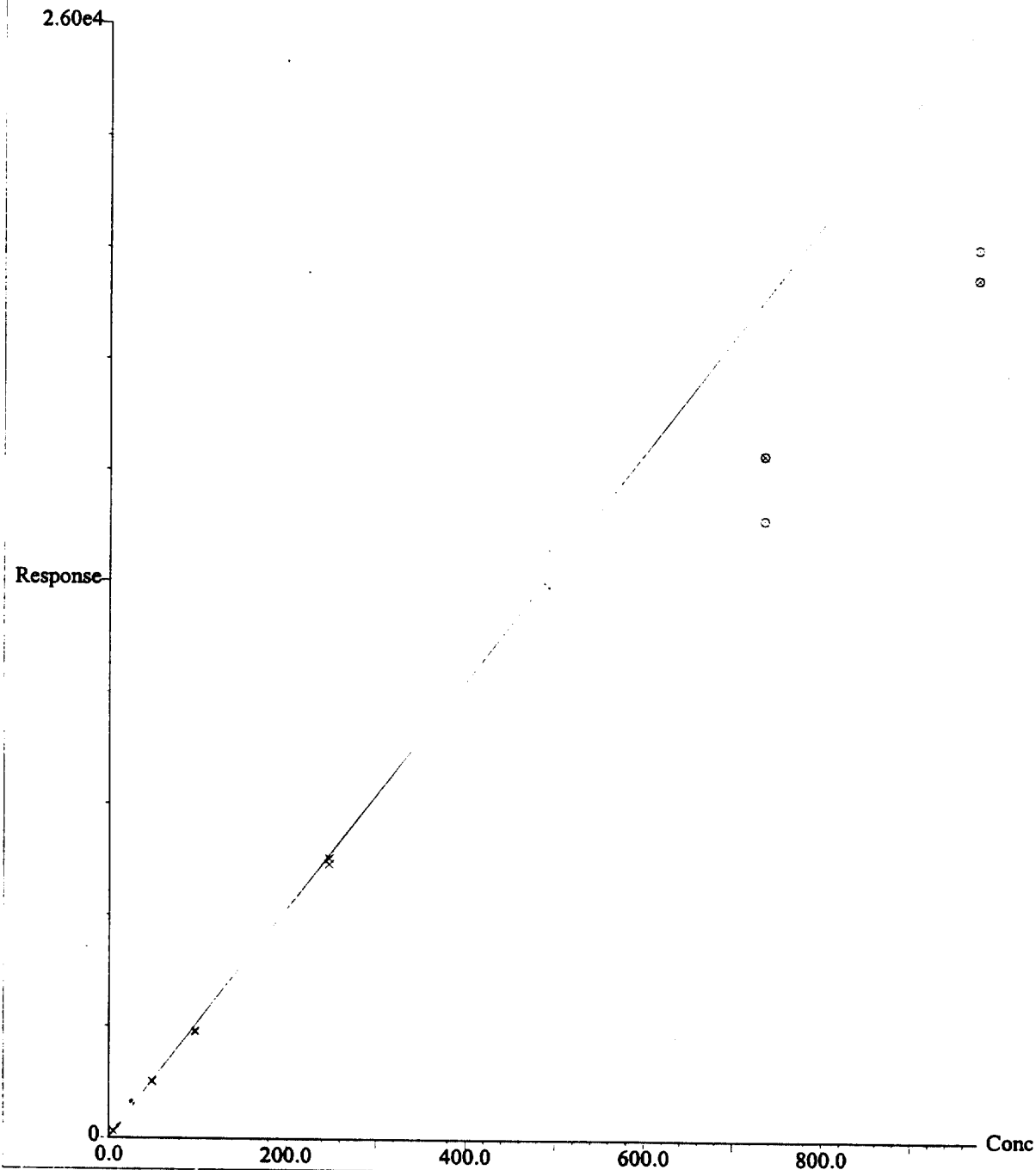
① RE mmh 10-21-99

000720

Quantify Calibration Report
Tox 145: Rat ~~Serum~~ Dilutions, Day 1,4

Page 2

Compound 2 name: PFOSA
Coefficient of Determination: 0.997888
Calibration curve: $26.5524 * x + 23.6545$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None

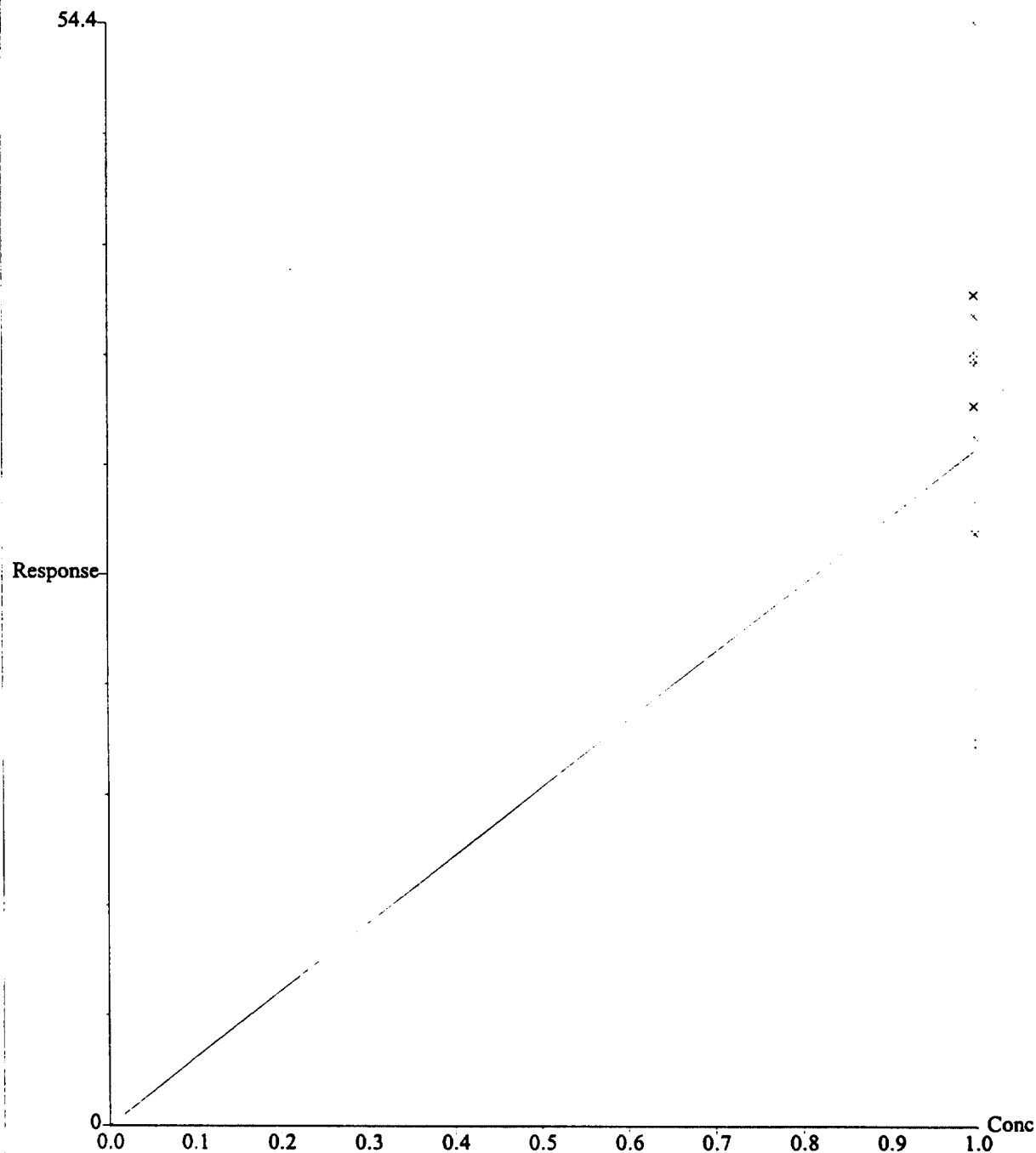


Amelia 062498: Quantitated and Printed by wnh 10/20/99

① RE mm. 102139

000721

Compound 3 name: POAA
Response Factor: 33.2811
Response type: External Std, Area
Curve type: RF

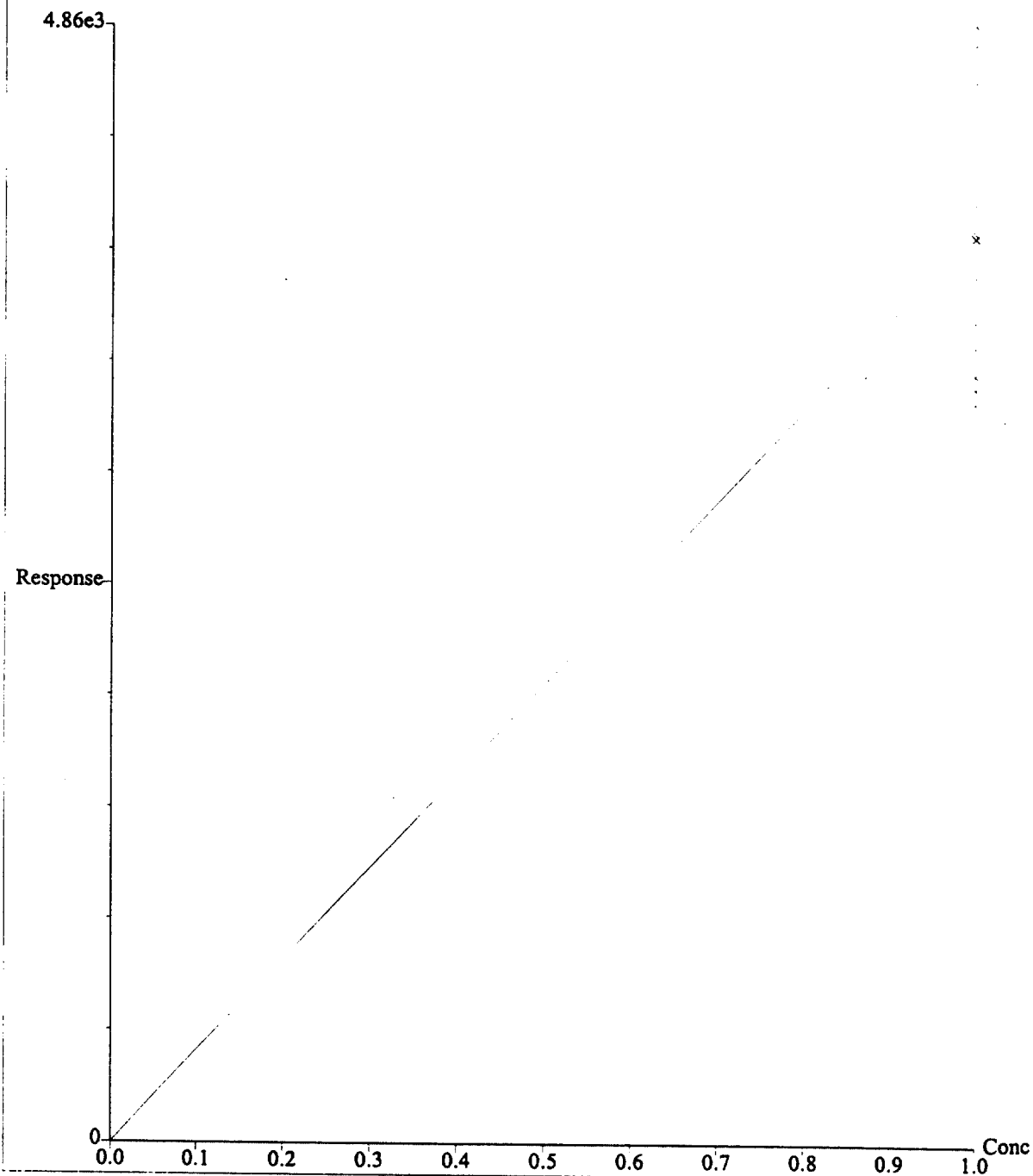


Amelia 062498: Quantitated and Printed by mmh 10/20/99

① RE mmh 10-21-99

000722

Compound 4 name: IS (Ch2)
Response Factor: 3976.31
Response type: External Std, Area
Curve type: RF



Amelia 062498: Quantitated and Printed by mmh 10/20/99

① RE MIN. 10-24-99

000723

Amelia 062498 10-21-99

Method Report

Page 1

Method file: N:\amelia-rats.PRO\MethDB\101999a.mdb
Last modified: Wed Oct 20 08:16:18 1999
Compound: 1: PFOS
Printed: Thu Oct 21 15:13:17 1999

Compound Name: PFOS
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. A
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da
Peak Location
Retention Time (mins): 7.167, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.358
Peak Selection: Nearest
Response
Response Type: External
Response: Areas
Calibration Curves
Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None
Concentration
Units:
Noise
Peak-to-peak amplitude: 100
Smooth
Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:
Baselines
Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00
Peak Separation
Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00
Response Threshold
Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000724

Amelia 062498 MMX 10-21-99

Method Report

Page 2

Method file: M:\amelia-rats.PRO\MethDB\101999a.mdb
Last modified: Wed Oct 20 08:16:18 1999
Compound: 2: PFOSA
Printed: Thu Oct 21 15:13:18 1999

Compound Name: PFOSA
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. B

User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da
Peak Location

Retention Time (mins): 7.624, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.262

Peak Selection: Nearest
Response

Response Type: External
Response: Areas
Calibration Curves

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None
Concentration

Units:
Noise

Peak-to-peak amplitude: 94
Smooth

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:
Baselines

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00
Peak Separation

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00
Response Threshold

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000725

Amelia 02/24/98 10-21-99

Method Report

Page 3

Method file: N:\amelia-rats.PRO\MethDB\101999a.mdb
Last modified: Wed Oct 20 08:16:18 1999

Compound: 3: POAA

Printed: Thu Oct 21 15:13:18 1999

Compound Name: POAA
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Fixed

User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da
Peak Location

Retention Time (mins): 6.947, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.238

Peak Selection: Nearest
Response

Response Type: External
Response: Areas
Calibration Curves

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None
Concentration

Units:
Noise

Peak-to-peak amplitude: 94
Smooth

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:
Baselines

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00
Peak Separation

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00
Response Threshold

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000726

Amelia 002498 10/21/99

Method Report

Page 4

Method file: N:\amelia-rats.PRO\MethDB\101999a.mdb
Last modified: Wed Oct 20 08:16:18 1999
Compound: 4: IS (Ch2)
Printed: Thu Oct 21 15:13:18 1999

Compound Name: IS (Ch2)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Fixed

User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Peak Location

Retention Time (mins): 6.910, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.406

Peak Selection: Nearest
Response

Response Type: External
Response: Areas
Calibration Curves

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None
Concentration

Units:
Noise

Peak-to-peak amplitude: 94
Smooth

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:
Baselines

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00
Peak Separation

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00
Response Threshold

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000727

mmh
10-19-99

AMELIA 062498: FACT Equipment Log ,99114
Quattro II, #6448E

PAGE _____

DATE: 10/19/99

ANALYST: mmh

STUDY NUMBER: TOX145 MATRIX: rat liver DESCRIPTION: dil

FACT Method: ETS-8-7.0

Injection volume: 10 uL Cycle time: 10 min. Electrode: Z-Spray

Mobile Phase: (A) 2.0 mM NH₄CH₃CO₂ (B) MeOH

TN-A- or stdrd number (A) 99085-10-14 (B) TN-A-2997

Prepared (date/by): (A) 10/16/99 sah (B) 10/16/99 sah

Expires: (A) 04/00 (B) 04/00

Store at room temp? ☒ Y or N Other: _____

Guard column: None

Part number: na Lot number: na

Analytical column: Keystone Betasil

Size: 2 x 50 mm Type: C18 Sphere size: 5 um

Part number: 055-701-2 Lot number: P9E11 Serial number: 0591816Z

Analytical parameters:

Drying gas (L/hr): 300 ESI nebulizing gas (L/hr) 10 Sheath gas (L/hr) 0

LC Flow rate: 300 uL/min. Back pressure: 62 bar

HPLC solvent gradient: 10min-10-95

Time (min.)	%B
1) <u>0.00</u>	<u>10.0</u>
2) <u>1.00</u>	<u>10.0</u>
3) <u>5.50</u>	<u>95.0</u>
4) <u>7.50</u>	<u>95.0</u>
5) <u>8.00</u>	<u>10.0</u>

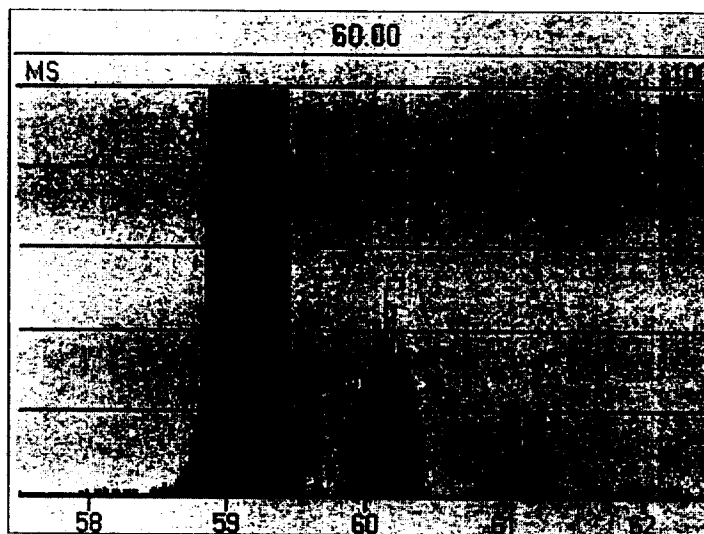
000728

Tuning Method Report

Page 1

Method: C:\MASSLYNX\AMELIA-RATS.PRO\ACQUDE\PFOS1-6KV

Printed: Tue Oct 19 10:25:49 1999



MS

SOURCE (ESP-)	Set	Rdbk	MS1	Set	Rdbk
Capillary	1.60	1.59	LM Resolution	15.0	
Cone	40	-40	HM Resolution	15.0	
Extractor	5	-5	Ion Energy	2.0	
RF Lens	0.2		Ion Energy Ramp	0.0	
Source Block Temp.	150	153	Lens 6	4	8
Desolvation Temp.	250	248	Multiplier	650	

Pressures	Rdbk	MS2	Set	Rdbk
Analyser Vacuum	1.2e-4	LM Resolution	14.0	
		HM Resolution	14.0	
		Collision	10	11
Gas Cell	4.6e-3	Ion Energy	2.0	
		Ion Energy Ramp	0.0	
		Lens 8	40	39
		Lens 9	0	12
		Multiplier	650	

000729

177N
10-A-99

Scanning Method Report

Page 1

Method: C:\MSSELYNK\AMELIA-RATS.PRO\ACQUIS\IONIN-PTOS-FOAA-IS-PTOSA
Last Modified: Mon Oct 11 08:22:17 1999

Printed: Tue Oct 19 10:22:46 1999

Solvent Delay (mins) : 0.00

Function : 1 MS/MS of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	413.00	169.00	0.30	30	15

Function : 2 MS/MS of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	499.00	99.00	0.30	45	60

Function : 3 MS/MS of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	427.00	80.00	0.30	40	60

Function : 4 MS/MS of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	498.00	78.00	0.40	45	60

000730

MMX
10-19-99

Masslynx - Sample List

Page 1

Sample List: C:\MASSLYNX\amelia-rats.PRO\SampleDB\101999a.SPL
Printed: Tue Oct 19 14:10:27 1999

Page Position: (1, 1)

	File Name	File Text	MS File
1	101999001	MeOH	10MIN-PFOS-POAA-IS-PFOSA
2	101999002	MeOH	10MIN-PFOS-POAA-IS-PFOSA
3	101999003	Tox 145: RBL10139-H2O blk-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
4	101999004	Tox 145: RBL10139-Liver blk-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
5	101999005	Tox 145: RBL10139-5 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
6	101999006	Tox 145: RBL10139-10 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
7	101999007	Tox 145: RBL10139-25 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
8	101999008	Tox 145: RBL10139-50 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
9	101999009	Tox 145: RBL10139-100 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
10	101999010	Tox 145: RBL10139-250 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
11	101999011	Tox 145: RBL10139-500 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
12	101999012	Tox 145: RBL10139-750 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
13	101999013	Tox 145: RBL10139-1000 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
14	101999014	MeOH	10MIN-PFOS-POAA-IS-PFOSA
15	101999015	Tox 145: 9R03237M, Day 4, Control, D:100	10MIN-PFOS-POAA-IS-PFOSA
16	101999016	Tox 145: 9R03245M, Day 1, 5 mg/kg, D:100	10MIN-PFOS-POAA-IS-PFOSA
17	101999017	Tox 145: 9R03246M, Day 1, 5 mg/kg, D:100	10MIN-PFOS-POAA-IS-PFOSA
18	101999018	Tox 145: 9R03247M, Day 1, 5 mg/kg, D:100	10MIN-PFOS-POAA-IS-PFOSA
19	101999019	Tox 145: 9R03248M, Day 1, 5 mg/kg, D:100	10MIN-PFOS-POAA-IS-PFOSA
20	101999020	Tox 145: RBL10139-250 ppb-11-2, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
21	101999021	MeOH	10MIN-PFOS-POAA-IS-PFOSA
22	101999022	Tox 145: 9R03249M, Day 1, 5 mg/kg, D:100	10MIN-PFOS-POAA-IS-PFOSA
23	101999023	Tox 145: 9R03251M, Day 4, 5 mg/kg, D:100	10MIN-PFOS-POAA-IS-PFOSA
24	101999024	Tox 145: 9R03252M, Day 4, 5 mg/kg, D:100	10MIN-PFOS-POAA-IS-PFOSA
25	101999025	Tox 145: 9R03253M, Day 4, 5 mg/kg, D:100	10MIN-PFOS-POAA-IS-PFOSA
26	101999026	Tox 145: 9R03254M, Day 4, 5 mg/kg, D:100	10MIN-PFOS-POAA-IS-PFOSA
27	101999027	Tox 145: RBL10139-250 ppb-11-1, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
28	101999028	MeOH	10MIN-PFOS-POAA-IS-PFOSA
29	101999029	Tox 145: RBL10139-H2O blk-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
30	101999030	Tox 145: RBL10139-Liver blk-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
31	101999031	Tox 145: RBL10139-5 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
32	101999032	Tox 145: RBL10139-10 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
33	101999033	Tox 145: RBL10139-25 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
34	101999034	Tox 145: RBL10139-50 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
35	101999035	Tox 145: RBL10139-100 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
36	101999036	Tox 145: RBL10139-250 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
37	101999037	Tox 145: RBL10139-500 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
38	101999038	Tox 145: RBL10139-750 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA
39	101999039	Tox 145: RBL10139-1000 ppb-11, Ext SEE 10-13-99	10MIN-PFOS-POAA-IS-PFOSA

000731

MDX
101999

Masslynx - Sample List

Page 2

Sample List: C:\MASSLYNX\amelia-rats.PRO\SampleDB\101999a.SPL
Printed: Tue Oct 19 14:10:27 1999

Page Position: (2, 1)

	Inlet File	Bottle	Inject Volume	Sample Type	Conc A	Conc B	Conc C	Conc D	Conc E	Conc F
1	10min-10-95	91	10	---	---	---	---	---	---	---
2	10min-10-95	91	10	---	---	---	---	---	---	---
3	10min-10-95	1	10	---	---	---	---	---	---	---
4	10min-10-95	2	10	---	---	---	---	---	---	---
5	10min-10-95	3	10	---	---	---	---	---	---	---
6	10min-10-95	4	10	---	---	---	---	---	---	---
7	10min-10-95	5	10	---	---	---	---	---	---	---
8	10min-10-95	6	10	---	---	---	---	---	---	---
9	10min-10-95	7	10	---	---	---	---	---	---	---
10	10min-10-95	8	10	---	---	---	---	---	---	---
11	10min-10-95	9	10	---	---	---	---	---	---	---
12	10min-10-95	10	10	---	---	---	---	---	---	---
13	10min-10-95	11	10	---	---	---	---	---	---	---
14	10min-10-95	91	10	---	---	---	---	---	---	---
15	10min-10-95	12	10	---	---	---	---	---	---	---
16	10min-10-95	13	10	---	---	---	---	---	---	---
17	10min-10-95	14	10	---	---	---	---	---	---	---
18	10min-10-95	15	10	---	---	---	---	---	---	---
19	10min-10-95	16	10	---	---	---	---	---	---	---
20	10min-10-95	81	10	---	---	---	---	---	---	---
21	10min-10-95	92	10	---	---	---	---	---	---	---
22	10min-10-95	17	10	---	---	---	---	---	---	---
23	10min-10-95	18	10	---	---	---	---	---	---	---
24	10min-10-95	19	10	---	---	---	---	---	---	---
25	10min-10-95	20	10	---	---	---	---	---	---	---
26	10min-10-95	21	10	---	---	---	---	---	---	---
27	10min-10-95	82	10	---	---	---	---	---	---	---
28	10min-10-95	92	10	---	---	---	---	---	---	---
29	10min-10-95	1	10	---	---	---	---	---	---	---
30	10min-10-95	2	10	---	---	---	---	---	---	---
31	10min-10-95	3	10	---	---	---	---	---	---	---
32	10min-10-95	4	10	---	---	---	---	---	---	---
33	10min-10-95	5	10	---	---	---	---	---	---	---
34	10min-10-95	6	10	---	---	---	---	---	---	---
35	10min-10-95	7	10	---	---	---	---	---	---	---
36	10min-10-95	8	10	---	---	---	---	---	---	---
37	10min-10-95	9	10	---	---	---	---	---	---	---
38	10min-10-95	10	10	---	---	---	---	---	---	---
39	10min-10-95	11	10	---	---	---	---	---	---	---

000732

FACT: DATA REVIEW SUMMARY FORM

Page 1/2

Date: 10/26/99
 Data Analyst: IAS
 FACT Study #: Tox 145 - MD/V
 Matrix/Timepoints: Rabbit Liver Curve - est. 10/22/99 RWJ
 Sample List: 102599C
 Set-up Analysis Date/Analyst: 10/25/99 IAS
 Instrument: Analisa 062498

Data Review Sign-Off

Analyst Review
 Name / Date: IAS 10/26/99

Technical Review
 Name / Date: MEE 10/27/99

Quality Assurance Review
 Name / Date: _____

Data Entry: No entry LAC 10/29/99

Comments / Justifications: _____

Analyte: POAA (CH1)^①
 Acceptable Linear Range: 61.30 ppb → 1227 ppb
 LOQ: 61.30 ppb Extraction Blank: greater than LOQ
 R²: 0.996986 Matrix Blank: greater than LOQ
 Calibration Checks: N/A
 MS/MSD: N/A
 Data Status: N/A

Analyte: TFOS (CH2)^②
 Acceptable Linear Range: 12.30 ppb → 307 ppb
 LOQ: 12.30 ppb Extracted Blank: less than LOQ
 R²: 0.972922 Matrix Blank: less than LOQ
 Calibration Checks: N/A
 MS/MSD: N/A
 Data Status: N/A

Analyte: IS (CH3)^③
 Acceptable Linear Range: NA
 LOQ: NA Extracted Blank: NA
 R²: NA Matrix Blank: NA
 Calibration Checks: NA
 MS/MSD: N/A 7 LAC 10/29/99
 Data Status: N/A

- ① The 6.13 ppb, 12.30 ppb, and 30.70 ppb points were dropped from the curve due to their large deviations from the linear range. The 307 ppb point had to be dropped in order to get a r² value of more than 0.98. This curve point was outside of the linear range of the points around it. - IAS 10/26/99 Area counts for POAA were low. - IAS 10/26/99
- ② The 6.14 ppb point was dropped from the curve so that the Liver BK would be less than the LOQ. The 614 ppb, 921 ppb, and 1228 ppb points were excluded in order to provide a good linear range with curve point deviations less than ± 30%. - IAS 10/26/99
- ③ Area counts of IS looked consistent. The H₂O BK had a deviation of 56%. It is not known why the deviation of the H₂O BK is so high. - IAS 10/26/99

000733

FACT: DATA REVIEW SUMMARY FORM

Page 2/2

Date: 10/26/99
 Data Analyst: IAS
 FACT Study #: Tox 145 - MD/V
 Matrix/Timepoints: Robert Liver/urine - cut. 10/22/99 PNH
 Sample List: 102599C
 Set-up Analysis
 Date/Analyst: 10/25/99 IAS
 Instrument: Amelia 062998

Data Review Sign-Off

Analyst Review
 Name / Date: IAS 10/26/99

Technical Review
 Name / Date: Refer to page 1/2 UAC 10/29/99

Quality Assurance Review
 Name / Date: _____

Data Entry: No entry IAC 10/29/99

Comments / Justifications: _____

Analyte: PFOSA (CH4) ④
 Acceptable Linear Range: 30.70 ppb → 1227 ppb
 LOQ: 30.70 ppb Extraction Blank: less than LOQ
 R²: 0.996087 Matrix Blank: less than LOQ
 Calibration Checks: NA
 MS/MSD: N/A
 Data Status: N/A

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: IAS 10/26/99
 Data Status: _____

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: _____

④ The 6.13 and 12.80 ppb points were dropped so that the H₂O & Matrix blanks would be less than the LOQ. Methanol blks for PFOSA were fairly high - IAS 10/26/99.
 ⑤ POAA EXTRACTION EFFICIENCY APPEARS TO BE LOW BASED ON TIC AREA COUNTS. MEE 10/27/99.

IAC 10/29/99

000734

inc 10/26/99 p. 1 of 4
Page 1

Quantify Compound Summary Report
TQX145-rabbit liver curve- ext. 10/22/99 RWW

Sample List: C:\MSL\TQX\Amalia062498.PRO\SampleDB\102599c
Last modified: Tue Oct 26 09:34:39 1999
Method: C:\MSL\TQX\Amalia062498.PRO\MethDB\102599c
Last modified: Tue Oct 26 09:34:42 1999
Job Code:

Printed: Tue Oct 26 09:43:32 1999

Compound 1: FOAA (Ch1)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Conc. Flags	%Dev
1 102599035	MeOH Blank						
2 102599036	MeOH Blank						
3 102599037	TQX145-RBL10229-H2O Blank-1, 10/22/99 RWW			6.976	100	253.08 bb	
4 102599038	TQX145-RBL10229-Liver Blank-1, 10/22/99 RWW			6.972	48	45.88 bb	
5 102599039	TQX145-RBL10229-5ppb-1, 10/22/99 RWW	Standard	6.13	6.968	51	59.89 bbX	877
6 102599040	TQX145-RBL10229-12ppb-1, 10/22/99 RWW	Standard	12.30	6.973	42	21.77 bbX	77
7 102599041	TQX145-RBL10229-30ppb-1, 10/22/99 RWW	Standard	30.70	6.941	36	0.00 HMX	-100
8 102599042	TQX145-RBL10229-60ppb-1, 10/22/99 RWW	Standard	61.30	6.947	56	76.64 bb	25
9 102599043	TQX145-RBL10229-120ppb-1, 10/22/99 RWW	Standard	123.00	6.923	59	87.83 HMX	-29
10 102599044	TQX145-RBL10229-300ppb-1, 10/22/99 RWW	Standard	307.00	6.920	151	454.03 bbX	48
11 102599045	TQX145-RBL10229-600ppb-1, 10/22/99 RWW	Standard	613.00	6.921	200	644.57 bb	5
12 102599046	TQX145-RBL10229-900ppb-1, 10/22/99 RWW	Standard	920.00	6.918	263	897.16 bb	-2
13 102599047	TQX145-RBL10229-1200ppb-1, 10/22/99 RWW	Standard	1227.00	6.924	350	1238.10 bb	1

Amalia 062498: Quantitated and printed by inc 10/26/99

000735

Quantify Compound Summary Report
 TOX145-rabbit liver curve- ext. 10/22/99 RWW

Page 2

Sample List: C:\MASSLYNK\Amalia\062498.PRO\SampleDB\102599c
 Last modified: Tue Oct 26 09:34:39 1999
 Method: C:\MASSLYNK\Amalia\062498.PRO\MethDB\102599c
 Last modified: Tue Oct 26 09:34:42 1999
 Job Code:

Printed: Tue Oct 26 09:43:52 1999

Compound 2: PFO6 (Ch2)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Conc. Flag	%Dev
1 102599035	MeOH Blank			7.148	49	0.00 bb	
2 102599036	MeOH Blank			7.144	33	0.00 bb	
3 102599037	TOX145-RBL10229-H2O Blank-1, 10/22/99 RWW			7.126	74	0.00 bb	
4 102599038	TOX145-RBL10229-Liver Blank-1, 10/22/99 RWW			7.144	94	0.00 bb	
5 102599039	TOX145-RBL10229-5ppb-1, 10/22/99 RWW	Standard	6.14	7.117	168	2.95 bbX	-52
6 102599040	TOX145-RBL10229-12ppb-1, 10/22/99 RWW	Standard	12.30	7.121	226	12.07 MM	-2
7 102599041	TOX145-RBL10229-30ppb-1, 10/22/99 RWW	Standard	30.70	7.116	352	31.89 bb	4
8 102599042	TOX145-RBL10229-60ppb-1, 10/22/99 RWW	Standard	61.40	7.119	571	66.44 bb	8
9 102599043	TOX145-RBL10229-120ppb-1, 10/22/99 RWW	Standard	123.00	7.119	823	106.12 bb	-14
10 102599044	TOX145-RBL10229-300ppb-1, 10/22/99 RWW	Standard	307.00	7.116	2167	317.88 bb	4
11 102599045	TOX145-RBL10229-600ppb-1, 10/22/99 RWW	Standard	614.00	7.096	3114	467.13 bbX	-24
12 102599046	TOX145-RBL10229-900ppb-1, 10/22/99 RWW	Standard	921.00	7.115	4458	678.84 bbX	-26
13 102599047	TOX145-RBL10229-1200ppb-1, 10/22/99 RWW	Standard	1228.00	7.097	6026	925.79 bbX	-25

Quantify Compound Summary Report
TOX145-rabbit liver curve- ext. 10/22/99 RMW

Page 3

Sample List: C:\MSL\TOK\Amelia062498.PRO\SampleDB\102599c
Last modified: Tue Oct 26 09:34:39 1999
Method: C:\MSL\TOK\Amelia062498.PRO\MethDB\102599c
Last modified: Tue Oct 26 09:34:42 1999
Job Code:

Printed: Tue Oct 26 09:43:52 1999

Compound 3: IS (Ch3)

#	Name	Sample List Text	Type	Std Conc	RT	Area	Conc.	Flags	%Dev
1	102599035	MeOH Blank							
2	102599036	MeOH Blank							
3	102599037	TOX145-RBL10229-H2O Blank-1, 10/22/99 RMW			6.964	3718	1.56	bb	56
4	102599038	TOX145-RBL10229-Liver Blank-1, 10/22/99 RMW			6.959	2208	0.93	bb	-7
5	102599039	TOX145-RBL10229-5ppb-1, 10/22/99 RMW	Standard	1.00	6.956	2692	1.13	bb	13
6	102599040	TOX145-RBL10229-12ppb-1, 10/22/99 RMW	Standard	1.00	6.960	2725	1.15	bb	15
7	102599041	TOX145-RBL10229-30ppb-1, 10/22/99 RMW	Standard	1.00	6.929	2635	1.11	bb	11
8	102599042	TOX145-RBL10229-60ppb-1, 10/22/99 RMW	Standard	1.00	6.958	2559	1.08	bb	8
9	102599043	TOX145-RBL10229-120ppb-1, 10/22/99 RMW	Standard	1.00	6.934	2230	0.94	bb	-6
10	102599044	TOX145-RBL10229-300ppb-1, 10/22/99 RMW	Standard	1.00	6.931	2409	1.01	bb	1
11	102599045	TOX145-RBL10229-600ppb-1, 10/22/99 RMW	Standard	1.00	6.932	2079	0.87	bb	-13
12	102599046	TOX145-RBL10229-900ppb-1, 10/22/99 RMW	Standard	1.00	6.929	2041	0.86	bb	-14
13	102599047	TOX145-RBL10229-1200ppb-1, 10/22/99 RMW	Standard	1.00	6.912	2040	0.86	bb	-14

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000737

Quantify Compound Summary Report
 TOX145-rabbit liver curve- ext. 10/22/99 RWW

Page 4

Sample List: C:\MSL\TQC\Amelia062498.PRO\SampleDB\102599c
 Last modified: Tue Oct 26 09:34:39 1999
 Method: C:\MSL\TQC\Amelia062498.PRO\MethDB\102599c
 Last modified: Tue Oct 26 09:34:42 1999
 Job Code:

Printed: Tue Oct 26 09:43:52 1999

Compound 4: PFOSA (Ch4)

#	Name	Samplelist Text	Type	Std Conc	RT	Area	Conc.	Flags	StdDev
1	102599035	MeOH Blank			7.781	677	34.09	MM	
2	102599036	MeOH Blank			7.774	350	15.55	bb	
3	102599037	TOX145-RBL10229-H2O Blank-1, 10/22/99 RWW			7.783	268	10.88	bb	
4	102599038	TOX145-RBL10229-Liver Blank-1, 10/22/99 RWW			7.777	208	7.44	bb	
5	102599039	TOX145-RBL10229-5ppb-1, 10/22/99 RWW	Standard	6.13	7.774	242	9.40	MMX	53
6	102599040	TOX145-RBL10229-12ppb-1, 10/22/99 RWW	Standard	12.30	7.777	336	14.72	bbX	20
7	102599041	TOX145-RBL10229-30ppb-1, 10/22/99 RWW	Standard	30.70	7.772	644	32.22	bb	5
8	102599042	TOX145-RBL10229-60ppb-1, 10/22/99 RWW	Standard	61.30	7.775	1233	65.73	bb	7
9	102599043	TOX145-RBL10229-120ppb-1, 10/22/99 RWW	Standard	123.00	7.777	1939	105.80	bb	-14
10	102599044	TOX145-RBL10229-300ppb-1, 10/22/99 RWW	Standard	307.00	7.773	5751	322.47	bb	5
11	102599045	TOX145-RBL10229-600ppb-1, 10/22/99 RWW	Standard	613.00	7.778	10450	589.48	bb	-4
12	102599046	TOX145-RBL10229-900ppb-1, 10/22/99 RWW	Standard	920.00	7.775	15649	884.92	bb	-4
13	102599047	TOX145-RBL10229-1200ppb-1, 10/22/99 RWW	Standard	1227.00	7.778	22627	1281.39	bb	4

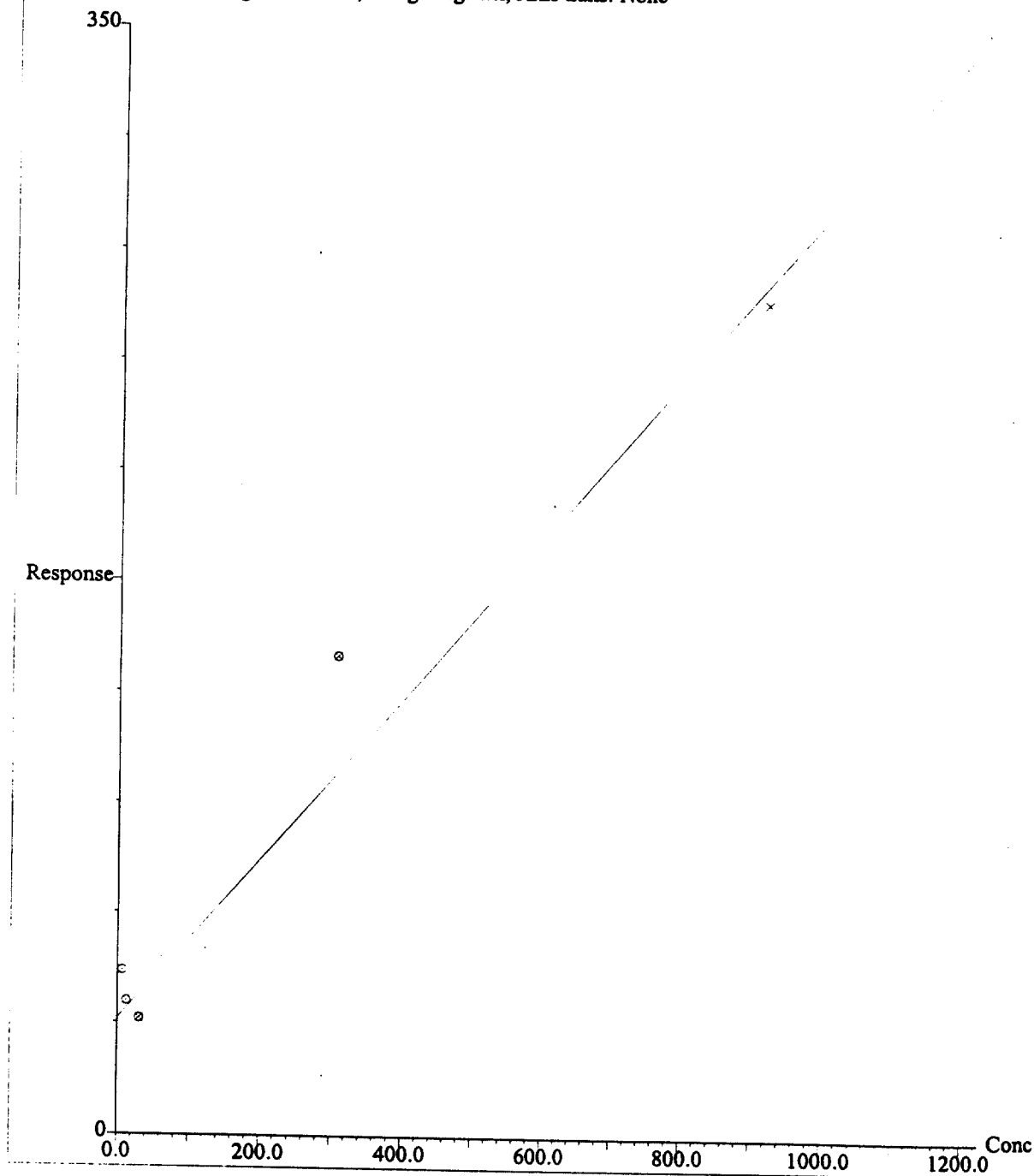
BEST COPY AVAILABLE

IAS 10/26/99 7. 1.44

Quantify Calibration Report
TOX145-rabbit liver curve- ext. 10/22/99 JMW

Page 1

Compound I name: POAA (Ch1)
Coefficient of Determination: 0.996986
Calibration curve: $0.253166 * x + 36.3287$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



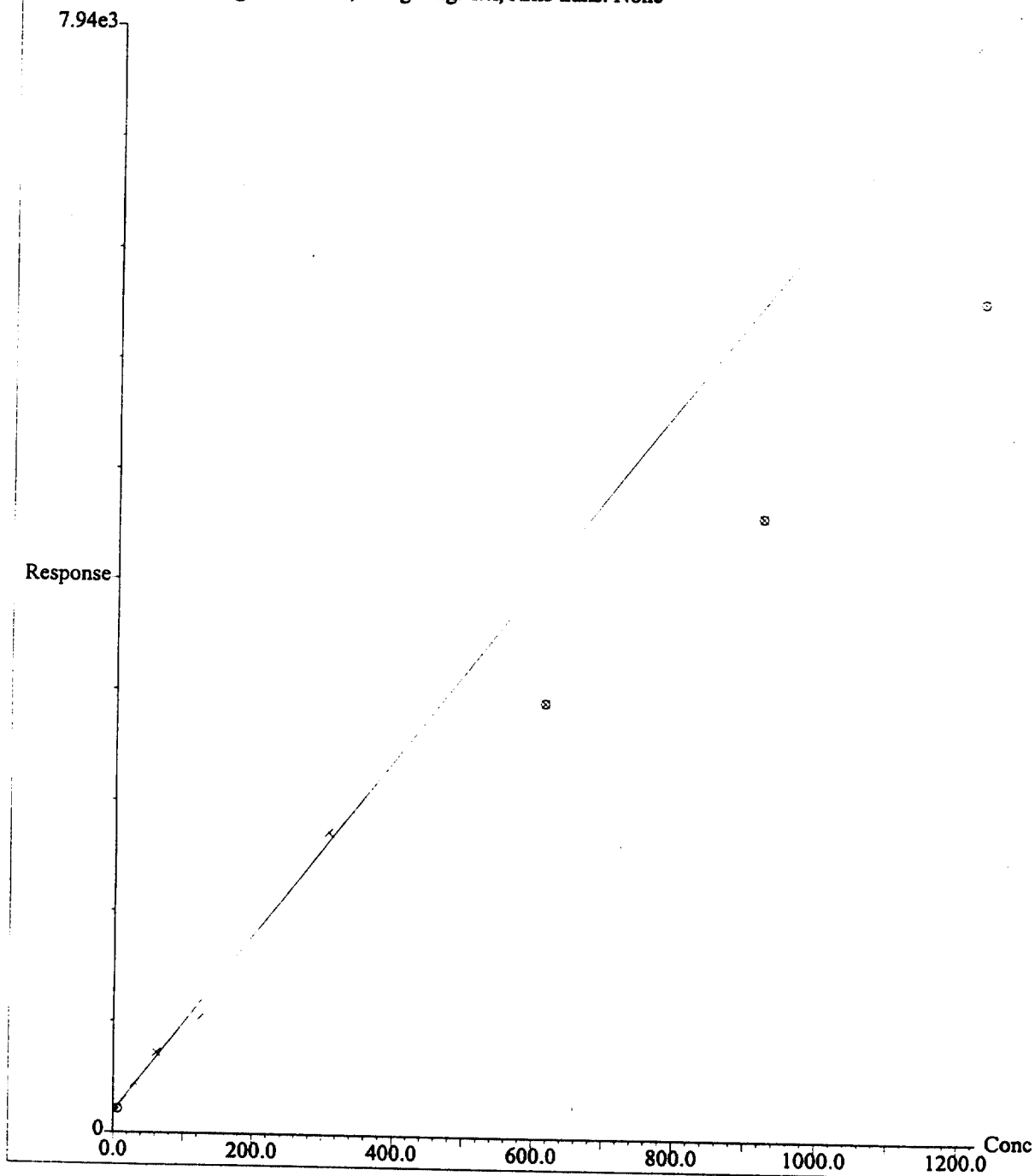
Amelia 062498: Quantitated and printed by ias 10/26/99

000739

Quantify Calibration Report
TOX145-rabbit liver curve- ext. 10/22/99 NW

Page 2

Compound 2 name: PFOS (Ch2)
Coefficient of Determination: 0.992922
Calibration curve: $6.34685 * x + 149.692$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



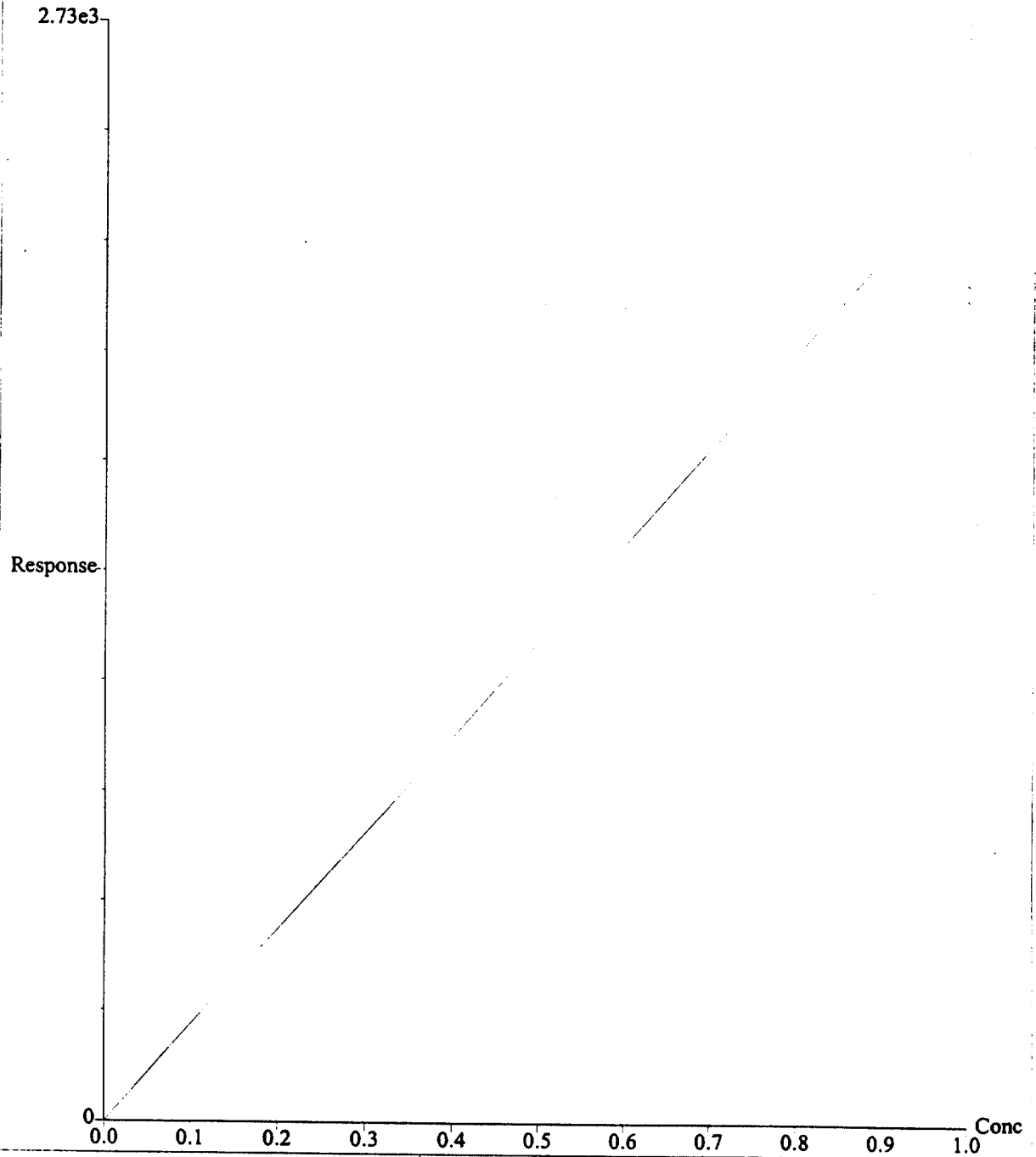
Amelia 062498: Quantitated and printed by las 10/26/99

000740

Quantify Calibration Report
TOX145-rabbit liver curve- ext. 10/22/99 ROW

Page 3

Compound 3 name: IS (Ch3)
Response Factor: 2378.96
Response type: External Std, Area
Curve type: RF



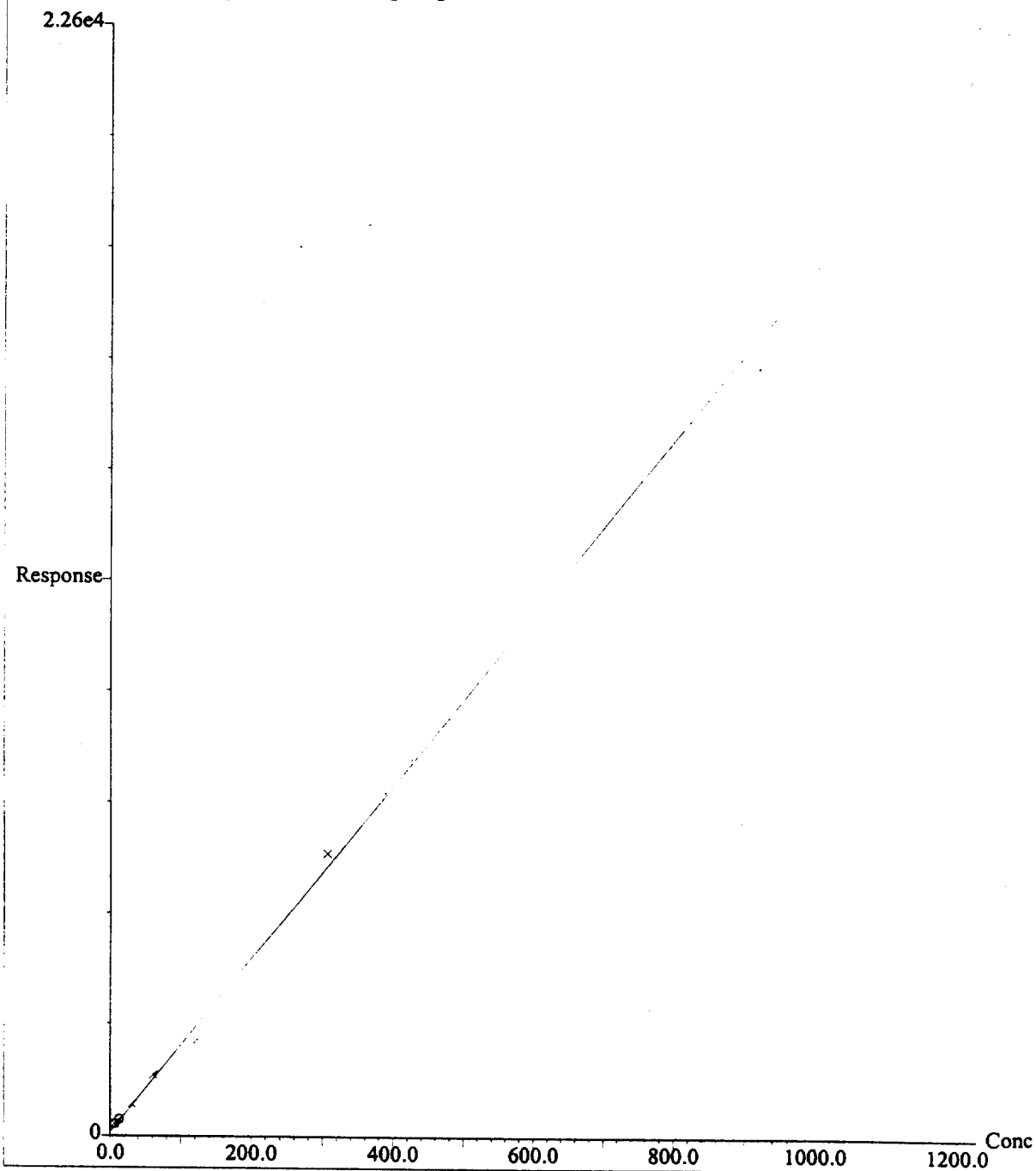
Amelia 062490: Quantitated and printed by ias 10/26/99

000741

Quantify Calibration Report
TOX145-rabbit liver curve- ext. 10/22/99 RMW

Page 4

Compound 4 name: PFOSA (Ch4)
Coefficient of Determination: 0.996087
Calibration curve: $17.5980 * x + 76.6733$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Amelia 062498: Quantitated and printed by ias 10/26/99

000742

Ins 10/26/99 p. 1st 4

Page 1

Method Report

Method file: C:\MASSLYNX\amelia062498.PRO\MethDB\102599c.mdb
Last modified: Tue Oct 26 09:34:42 1999C
Compound: 1: POAA (Ch1)
Printed: Tue Oct 26 09:44:34 1999

Compound Name: POAA (Ch1)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. B
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da
Peak Location

Retention Time (mins): 6.889, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.252

Peak Selection: Nearest
Response

Response Type: External
Response: Areas
Calibration Curves

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None
Concentration

Units:
Noise

Peak-to-peak amplitude: 91
Smooth

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:
Baselines

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00
Peak Separation

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00
Response Threshold

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000743

Method Report

Page 2

Method file: C:\MASSLYNX\amelia062498.PRO\MethDB\102599c.mdb
Last modified: Tue Oct 26 09:34:42 1999C
Compound: 2: PFOS (Ch2)
Printed: Tue Oct 26 09:44:34 1999

Compound Name: PFOS (Ch2)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. A

User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da
Peak Location

Retention Time (mins): 7.109, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.227

Peak Selection: Nearest
Response

Response Type: External
Response: Areas
Calibration Curves

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None
Concentration

Units:
Noise

Peak-to-peak amplitude: 96
Smooth

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:
Baselines

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00
Peak Separation

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00
Response Threshold

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000744

Method Report

Page 3

Method file: C:\MASSLYNK\amelia062498.PRO\MethDB\102599c.mdb
Last modified: Tue Oct 26 09:34:42 1999

Compound: 3: IS (Ch3)

Printed: Tue Oct 26 09:44:34 1999

Compound Name: IS (Ch3)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Fixed

User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da
Peak Location

Retention Time (mins): 6.924, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.252

Peak Selection: Nearest
Response

Response Type: External
Response: Areas
Calibration Curves

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None
Concentration

Units:
Noise

Peak-to-peak amplitude: 74
Smooth

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:
Baselines

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00
Peak Separation

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00
Response Threshold

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000745

Method Report

Page 4

Method file: C:\MASSLYNK\amelia062498.PRO\MethDB\102599c.mdb
Last modified: Tue Oct 26 09:34:42 1999C

Compound: 4: PFOSA (Ch4)

Printed: Tue Oct 26 09:44:34 1999

Compound Name: PFOSA (Ch4)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. B

User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da
Peak Location

Retention Time (mins): 7.851, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.311

Peak Selection: Nearest
Response

Response Type: External
Response: Area
Calibration Curves

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None
Concentration

Units:
Noise

Peak-to-peak amplitude: 318
Smooth

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:
Baselines

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00
Peak Separation

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00
Response Threshold

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000746

IAS 10/25/99

AMELIA 062498: FACT Equipment Log 99114
Quattro II, #6448E

PAGE _____

DATE: 10/25/99

ANALYST: ias

STUDY NUMBER: TO145 MATRIX: Rabbit Liver DESCRIPTION: Curve

FACT Method: ETS-8-7.0

Injection volume: 10 uL Cycle time: 10 min Electrode: Z-Spray

Mobile Phase: (A) 2.0 mM NH₄CH₃CO₂ (B) MeOH

TN-A- or stdrd number (A) 99085-11-3 (B) TN-A-2945

Prepared (date/by): (A) 10/21/99 sah (B) 10/20/99 ias

Expires: (A) 04/00 (B) 04/00

Store at room temp? ☒ Y or N Other: _____

Guard column: None

Part number: na Lot number: na

Analytical column: Keystone Betasil

Size: 2 x 50 mm Type: C18 Sphere size: 5 um

Part number: 055-701-2 Lot number: P9E11 Serial number: 0591816Z

Analytical parameters:

Drying gas (L/hr): 300 ESI nebulizing gas (L/hr) 10 Sheath gas (L/hr) 0

LC Flow rate: 300 uL/min Back pressure: 62 bar

HPLC solvent gradient: 10min-10-95

Time (min.)	%B
1) <u>0.00</u>	<u>10.0</u>
2) <u>1.00</u>	<u>10.0</u>
3) <u>5.50</u>	<u>95.0</u>
4) <u>7.50</u>	<u>95.0</u>
5) <u>8.00</u>	<u>10.0</u>

000747

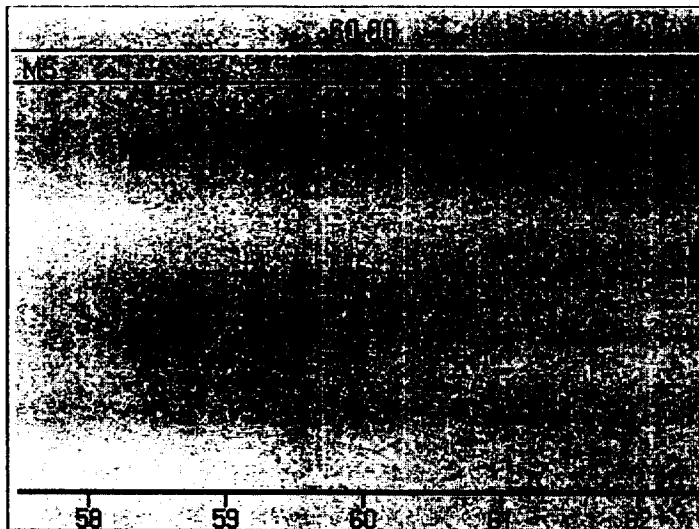
BEST COPY AVAILABLE

Tuning Method Report

Page 1

Method: C:\MASSLYNX\AMELIA-RATS.PRO\ACQUDB\PFOS1-6KV

Printed: Mon Oct 25 10:13:58 1999



MS

SOURCE (ESP-)	Set	Rdbk	MS1	Set	Rdbk
Capillary	1.60	1.60	LM Resolution	15.0	
Cone	40	-60	HM Resolution	15.0	
Extractor	5	-6	Ion Energy	2.0	
RF Lens	0.2		Ion Energy Ramp	0.0	
Source Block Temp.	150	151	Lens 6	4	7
Desolvation Temp.	250	250	Multiplier	650	

Pressures	Rdbk	MS2	Set	Rdbk
Analyser Vacuum	1.2e-4	LM Resolution	14.0	
		HM Resolution	14.0	
		Collision	10	46
Gas Cell	4.5e-3	Ion Energy	2.0	
		Ion Energy Ramp	0.0	
		Lens 8	40	39
		Lens 9	0	48
		Multiplier	650	

000748

Scanning Method Report

Page 1

Method: C:\MSSELYOK\AMELIA-RATS.PRO\ACQUIS\IONIN-PP06-POAA-IS-PP06A
Last Modified: Mon Oct 11 08:22:17 1999

Printed: Mon Oct 25 10:13:30 1999

Solvent Delay (mins) : 0.00

Function : 1 MM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	413.00	169.00	0.30	30	15

Function : 2 MM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	499.00	99.00	0.30	45	60

Function : 3 MM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	427.00	80.00	0.30	40	60

Function : 4 MM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	498.00	78.00	0.40	45	60

000749

Method Report

Page 1

Method File: c:\masslynx\amelia-rats.pro\acqddb\10min-10-95
Last Modified: Monday, October 25, 1999 10:12:15
Printed: Monday, October 25, 1999 10:13:44

HP1100 LC Pump Initial Conditions

Solvents
A% 90.0
B% 10.0
C% 0.0
D% 0.0
Flow (ml/min) 0.300
Stop Time (mins) 10.0
Min Pressure (bar) 0
Max Pressure (bar) 400
Oven Temperature Left(°C) 30.0
Oven Temperature Right(°C) 30.0

HP1100 LC Pump Gradient Timetable

The gradient Timetable contains 5 entries which are :

Time	A%	B%	C%	D%	Flow	Pressure
0.00	90.0	10.0	0.0	0.0	0.300	400
1.00	90.0	10.0	0.0	0.0	0.300	400
5.50	5.0	95.0	0.0	0.0	0.300	400
7.50	5.0	95.0	0.0	0.0	0.300	400
8.00	90.0	10.0	0.0	0.0	0.300	400

HP1100 LC Pump External Event Timetable

The Timetable contains 3 entries which are :

Time	Column Switch	Contact1	Contact2	Contact3	Contact4
Initial	Off	Off	Off	Off	Off
0.00	Off	On	On	Off	On
0.10	Off	Off	Off	Off	Off

HP1100 Autosampler Initial Conditions

Draw Speed 200.0
Eject Speed (µl/min) 200
Draw Position (mm) 0.00
Stop Time (mins) 9.00
Injection Volume(µl) 10.0
Vial Number 13

000750

Masslynx - Sample List

Page 1

Sample List: C:\MASSLYNX\amelia-rats.PRO\SampleDB\102599c.SPL
Printed: Mon Oct 25 10:13:12 1999

Page Position: (1, 1)

	File Name	File Text	MS File	Inlet File
1	102599035	MeOH Blank	10MIN-PFOS-POAA-IS-PFOSA	10min-10-95
2	102599036	MeOH Blank	10MIN-PFOS-POAA-IS-PFOSA	10min-10-95
3	102599037	TOX145-RBL10229-H2O Blank-1, 10/22/99 RWW	10MIN-PFOS-POAA-IS-PFOSA	10min-10-95
4	102599038	TOX145-RBL10229-Liver Blank-1, 10/22/99 RWW	10MIN-PFOS-POAA-IS-PFOSA	10min-10-95
5	102599039	TOX145-RBL10229-5ppb-1, 10/22/99 RWW	10MIN-PFOS-POAA-IS-PFOSA	10min-10-95
6	102599040	TOX145-RBL10229-12ppb-1, 10/22/99 RWW	10MIN-PFOS-POAA-IS-PFOSA	10min-10-95
7	102599041	TOX145-RBL10229-30ppb-1, 10/22/99 RWW	10MIN-PFOS-POAA-IS-PFOSA	10min-10-95
8	102599042	TOX145-RBL10229-60ppb-1, 10/22/99 RWW	10MIN-PFOS-POAA-IS-PFOSA	10min-10-95
9	102599043	TOX145-RBL10229-120ppb-1, 10/22/99 RWW	10MIN-PFOS-POAA-IS-PFOSA	10min-10-95
10	102599044	TOX145-RBL10229-300ppb-1, 10/22/99 RWW	10MIN-PFOS-POAA-IS-PFOSA	10min-10-95
11	102599045	TOX145-RBL10229-600ppb-1, 10/22/99 RWW	10MIN-PFOS-POAA-IS-PFOSA	10min-10-95
12	102599046	TOX145-RBL10229-900ppb-1, 10/22/99 RWW	10MIN-PFOS-POAA-IS-PFOSA	10min-10-95
13	102599047	TOX145-RBL10229-1200ppb-1, 10/22/99 RWW	10MIN-PFOS-POAA-IS-PFOSA	10min-10-95

000751

Masslynx - Sample List

Page 2

Sample List: C:\MASSLYNX\amelia-rats.PRO\SampleDB\102599c.SPL
Printed: Mon Oct 25 10:13:12 1999

Page Position: (2, 1)

	Bottle	Inject Volume
1	92	10
2	92	10
3	12	10
4	13	10
5	14	10
6	15	10
7	16	10
8	17	10
9	18	10
10	19	10
11	20	10
12	21	10
13	22	10

000752

FACT: DATA REVIEW SUMMARY FORM

page 1 of 2

UC
11/05/99

Data Review Sign-Off

Date: 10/29/99
 Data Analyst: JAS
 FACT Study #: T2X145
 Matrix/Timepoints: Twice 80-dose Confirmation - Oct 10/29/99
 Sample List: 102899a
 Set-up Analysis
 Date/Analyst: 10/29/99 NEE/EAS
 Instrument: Sup 020199

Analyst Review

Name / Date: JAS 10/29/99

Technical Review

Name / Date: NEE 10/29/99 / kh Nov-4-99

Quality Assurance Review

Name / Date: _____

Data Entry: DATA NOT Reported (E) GML 11/9/99

Comments / Justifications:

Analyte: FOAA (CH1) (3)(5)
 Acceptable Linear Range: 1.00 ppb → 251 ppb
 LOQ: 1.00 ppb Extraction Blank: NA
 R²: 0.996738 Matrix Blank: NA
 Calibration Checks: OK - Range from 13% → -4%
 MS/MSD: (*) DATA NOT Reported GML 11/9/99
 Data Status: _____

Analyte: PFOS (CH2) (3)(5)
 Acceptable Linear Range: 1.01 ppb → 252 ppb
 LOQ: 1.01 ppb Extracted Blank: NA
 R²: 0.999345 Matrix Blank: NA
 Calibration Checks: OK - Range from 6 → 4%
 MS/MSD: (*) Data NOT Reported GML 11/9/99
 Data Status: _____

Analyte: PFOSA (CH3) (3)(5)
 Acceptable Linear Range: 25.10 ppb → 754 ppb
 LOQ: 25.10 ppb Extracted Blank: NA
 R²: _____ Matrix Blank: NA
 Calibration Checks: _____
 MS/MSD: (*) Data NOT Reported 11/9/99
 Data Status: _____

① The 502 ppb and 752 ppb curve points were dropped in order to provide a good linear fit to the curve and to keep deviations in the lower half of the curve to less than $\pm 30\%$. There were no blanks to extract and run with the curve. - JAS 10/29/99

② The 503 ppb and 755 ppb points were dropped from the curve in order to provide a good linear fit and to keep deviations less than $\pm 30\%$ in the lower half of the curve. There were no blanks to extract and run with the sample set. - JAS 10/29/99

③ The 1.01 ppb, 5.03 ppb, and 10.10 ppb points were dropped because they had deviations from their expected conc. of more than $\pm 30\%$. Samples were too high due to a miscalculation and should be re-extracted - JAS 10/29/99

④ DATA WILL NOT BE USED for this Analysis
 Due to errors in spiking calculations. Further analysis will occur after re-extraction of the samples. GML 11/9/99

000753

FACT: DATA REVIEW SUMMARY FORM

page 2 of 2
UC
11/05/99

Data Review Sign-Off

Date: 10/29/99
Data Analyst: IAS
FACT Study #: Tox145
Matrix/Timepoints: Tissue 80 - dose confirmation - ext 10/28/99
Sample List: 102899a
Set-up Analysis
Date/Analyst: 10/28/99 MEE/IAS
Instrument: Sony D20199

Analyst Review

Name / Date: _____

Technical Review

Name / Date: _____

Quality Assurance Review

Name / Date: _____

Data Entry:

Comments / Justifications: _____

Analyte: To (C4H4)
Acceptable Linear Range: NA
LOQ: NA Extraction Blank: NA
R²: NA Matrix Blank: NA
Calibration Checks: NA
MS/MSD: _____
Data Status: _____

Analyte: _____
Acceptable Linear Range: _____
LOQ: _____ Extracted Blank: _____
R²: _____ Matrix Blank: _____
Calibration Checks: _____
MS/MSD: _____
Data Status: _____

Analyte: _____
Acceptable Linear Range: _____
LOQ: _____ Extracted Blank: _____
R²: _____ Matrix Blank: _____
Calibration Checks: _____
MS/MSD: _____
Data Status: _____

① Is area counts looked fairly consistent among the samples. The curves had area counts which were high in the lower side and low in the upper part of the curve. Area counts for the second dropped slightly compared to the initial curve - IAS 10/28/99
② A miscalculation was made when preparing the sample. Samples are much higher in concentration than they should be. Samples will be reextracted and rerun. Data should not be used - IAS 10/29/99

000754

Quantify Compound Summary Report
TOX145-tween 80 dose confirmation- PPOS/PFOSA/POAA ext. 10/28/99 ias

Sample List: C:\MSL\YHX\scup020199.pro\SampleDB\102899a
Last modified: Fri Oct 29 07:02:56 1999
Method: C:\MSL\YHX\scup020199.pro\MethDB\102899a
Last modified: Fri Oct 29 06:53:35 1999
Job Code:

Printed: Fri Oct 29 07:37:25 1999

Compound 1: POAA (Chl)

#	Name	Samplelist Test	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1	102899001	MeOH Blk							
2	102899002	MeOH Blk							
3	102899003	FCMlx-99086-43-1, 1ppb- 9/10/99 RMW	Standard	1.00	7.043	17	MM	0.85	-15
4	102899004	FCMlx-99086-43-2, 5ppb- 9/10/99 RMW	Standard	5.02	7.107	38	bb	3.69	-26
5	102899005	FCMlx-99086-43-3, 10ppb- 9/10/99 RMW	Standard	10.00	7.085	87	MM	10.39	4
6	102899006	FCMlx-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.10	7.087	193	bb	24.79	-1
7	102899007	FCMlx-99086-43-5, 50ppb- 9/10/99 RMW	Standard	50.20	7.092	364	bb	48.04	-4
8	102899008	FCMlx-99086-43-6, 100ppb- 9/10/99 RMW	Standard	100.00	7.085	743	bb	99.65	-0
9	102899009	FCMlx-99086-43-7, 250ppb- 9/10/99 RMW	Standard	251.00	7.110	1780	bb	240.63	-4
10	102899010	FCMlx-99086-43-8, 500ppb- 9/10/99 RMW	Standard	502.00	7.102	3005	bbX	407.36	-19
11	102899011	FCMlx-99086-43-9, 750ppb- 9/10/99 RMW	Standard	752.00	7.131	3991	bbX	541.42	-28
12	102899012	MeOH Blk				9	bb	0.00	
13	102899013	TOX145-PPOS/POAA in Tween-dose-2ppb- 10/28/99 IAS			7.106				
14	102899014	TOX145-PPOS/POAA in Tween-ma-4ppb- 10/28/99 IAS			7.130	447	bb	59.13	
15	102899015	FCMlx-99086-43-4, 25ppb- 9/10/99 RMW	QC	25.10	7.125	415	bb	55.07	
16	102899016	MeOH Blk			7.129	219	MM	28.36	13
17	102899017	TOX145-PFOSA in Tween-dose-200ppb- 10/28/99 IAS			7.111	418	bb	55.45	
18	102899018	TOX145-PFOSA in Tween-ma-300ppb- 10/28/99 IAS			7.106	434	MM	57.55	
19	102899019	FCMlx-99086-43-7, 250ppb- 9/10/99 RMW	QC	251.00	7.132	1775	bb	240.01	-4
20	102899020	MeOH Blk							
21	102899021	FCMlx-99086-43-1, 1ppb- 9/10/99 RMW	Standard	1.00	7.128	19	MM	1.10	10
22	102899022	FCMlx-99086-43-2, 5ppb- 9/10/99 RMW	Standard	5.02	7.126	46	bb	4.76	-5
23	102899023	FCMlx-99086-43-3, 10ppb- 9/10/99 RMW	Standard	10.00	7.130	95	MM	11.48	15
24	102899024	FCMlx-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.10	7.157	212	bb	27.42	9
25	102899025	FCMlx-99086-43-5, 50ppb- 9/10/99 RMW	Standard	50.20	7.125	422	bb	56.00	12
26	102899026	FCMlx-99086-43-6, 100ppb- 9/10/99 RMW	Standard	100.00	7.130	819	bb	109.94	10
27	102899027	FCMlx-99086-43-7, 250ppb- 9/10/99 RMW	Standard	251.00	7.133	1818	bb	245.90	-2
28	102899028	FCMlx-99086-43-8, 500ppb- 9/10/99 RMW	Standard	502.00	7.128	3158	bbX	428.05	-15
29	102899029	FCMlx-99086-43-9, 750ppb- 9/10/99 RMW	Standard	752.00	7.129	4081	bbX	553.60	-26

Sample List: C:\MASSLYNX\scup020199.pro\SampleDB\102899a
 Last modified: Fri Oct 29 07:02:56 1999
 Method: C:\MASSLYNX\scup020199.pro\MethDB\102899a
 Last modified: Fri Oct 29 06:53:35 1999
 Job Code:

Printed: Fri Oct 29 07:37:25 1999

Compound 2: PFOS (Ch3)

# Name	Sample/Std Test	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1 102899001	MeOH Blk			7.248	95	MM	0.63	
2 102899002	MeOH Blk			7.265	53	MM	0.14	
3 102899003	FCMIX-99086-43-1, 1ppb- 9/10/99 RMW	Standard	1.01	7.226	109	bb	0.79	-21
4 102899004	FCMIX-99086-43-2, 5ppb- 9/10/99 RMW	Standard	5.03	7.268	442	bb	4.67	-7
5 102899005	FCMIX-99086-43-3, 10ppb- 9/10/99 RMW	Standard	10.00	7.247	905	bb	10.07	1
6 102899006	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.20	7.248	2086	bb	23.83	-5
7 102899007	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	50.30	7.254	4177	bb	48.19	-4
8 102899008	FCMIX-99086-43-6, 100ppb- 9/10/99 RMW	Standard	101.00	7.247	8256	bb	95.72	-5
9 102899009	FCMIX-99086-43-7, 250ppb- 9/10/99 RMW	Standard	252.00	7.272	21703	bb	252.39	0
10 102899010	FCMIX-99086-43-8, 500ppb- 9/10/99 RMW	Standard	503.00	7.287	39235	bbX	456.65	-9
11 102899011	FCMIX-99086-43-9, 750ppb- 9/10/99 RMW	Standard	755.00	7.290	54526	bbX	634.82	-16
12 102899012	MeOH Blk			7.289	167	bb	1.47	
13 102899013	TOX145-PFOS/POAA in Tween-dose-2ppb- 10/28/99 IAS			7.291	29376	bb	341.79	
14 102899014	TOX145-PFOS/POAA in Tween-ma-4ppb- 10/28/99 IAS			7.287	30229	bb	351.73	
15 102899015	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	QC	25.20	7.291	2326	bb	26.62	6
16 102899016	MeOH Blk			7.294	55	MM	0.16	
17 102899017	TOX145-PFOA in Tween-dose-200ppb- 10/28/99 IAS			7.273	30004	bb	349.11	
18 102899018	TOX145-PFOA in Tween-ma-300ppb- 10/28/99 IAS			7.267	29852	bb	347.34	
19 102899019	FCMIX-99086-43-7, 250ppb- 9/10/99 RMW	QC	252.00	7.294	22582	bb	262.63	4
20 102899020	MeOH Blk			7.288	88	bb	0.54	
21 102899021	FCMIX-99086-43-1, 1ppb- 9/10/99 RMW	Standard	1.01	7.288	141	bb	1.17	16
22 102899022	FCMIX-99086-43-2, 5ppb- 9/10/99 RMW	Standard	5.03	7.287	514	bb	5.51	10
23 102899023	FCMIX-99086-43-3, 10ppb- 9/10/99 RMW	Standard	10.00	7.268	987	bb	11.02	10
24 102899024	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	2248	bb	25.71	2
25 102899025	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	50.30	7.286	4459	bd	51.47	2
26 102899026	FCMIX-99086-43-6, 100ppb- 9/10/99 RMW	Standard	101.00	7.291	8735	bb	101.29	0
27 102899027	FCMIX-99086-43-7, 250ppb- 9/10/99 RMW	Standard	252.00	7.295	22119	bd	257.24	2
28 102899028	FCMIX-99086-43-8, 500ppb- 9/10/99 RMW	Standard	503.00	7.289	38876	bbX	452.47	-10
29 102899029	FCMIX-99086-43-9, 750ppb- 9/10/99 RMW	Standard	755.00	7.266	53247	bbX	619.91	-18

Quantify Compound Summary Report
 TOX145-tween 80 dose confirmation- PFOS/PFOA/FOAA ext. 10/28/99 ias

Page 3

Sample List: C:\MASSLYNK\soup020199.pro\SampleDB\102899a
 Last modified: Fri Oct 29 07:02:56 1999
 Method: C:\MASSLYNK\soup020199.pro\MethodDB\102899a
 Last modified: Fri Oct 29 06:53:35 1999
 Job Code:

Printed: Fri Oct 29 07:37:25 1999

Compound 3: PFOA (Ch3)

#	Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1	102899001	MeOH Blk			7.811	559	db	0.00	
2	102899002	MeOH Blk			7.802	204	MM	0.00	
3	102899003	FCMIX-99086-43-1, 1ppb- 9/10/99 RWW	Standard	1.01	7.766	762	MMX	0.00	-100
4	102899004	FCMIX-99086-43-2, 5ppb- 9/10/99 RWW	Standard	5.03	7.786	2539	bbX	0.00	-100
5	102899005	FCMIX-99086-43-3, 10ppb- 9/10/99 RWW	Standard	10.10	7.764	4612	MMX	2.11	-79
6	102899006	FCMIX-99086-43-4, 25ppb- 9/10/99 RWW	Standard	25.10	7.765	10800	MM	17.23	-31
7	102899007	FCMIX-99086-43-5, 50ppb- 9/10/99 RWW	Standard	50.10	7.771	19708	bb	39.00	-22
8	102899008	FCMIX-99086-43-6, 100ppb- 9/10/99 RWW	Standard	101.00	7.765	43375	bb	96.84	-4
9	102899009	FCMIX-99086-43-7, 250ppb- 9/10/99 RWW	Standard	251.00	7.811	105704	bb	249.16	-1
10	102899010	FCMIX-99086-43-8, 500ppb- 9/10/99 RWW	Standard	503.00	7.826	205809	bb	493.80	-2
11	102899011	FCMIX-99086-43-9, 750ppb- 9/10/99 RWW	Standard	754.00	7.831	301294	bb	727.16	-4
12	102899012	MeOH Blk			7.829	2511	MM	0.00	
13	102899013	TOX145-PFOS/FOAA in Tween-dose-2ppb- 10/28/99 IAS			7.853	4283752	bb	10459.75	
14	102899014	TOX145-PFOS/FOAA in Tween-ma-4ppb- 10/28/99 IAS			7.849	4315922	bb	10538.37	
15	102899015	FCMIX-99086-43-4, 25ppb- 9/10/99 RWW	QC	25.10	7.831	63186	MM	145.25	479
16	102899016	MeOH Blk			7.856	12460	MM	21.29	
17	102899017	TOX145-PFOA in Tween-dose-200ppb- 10/28/99 IAS			7.835	4302369	bb	10505.25	
18	102899018	TOX145-PFOA in Tween-ma-300ppb- 10/28/99 IAS			7.852	4271995	bb	10431.02	
19	102899019	FCMIX-99086-43-7, 250ppb- 9/10/99 RWW	QC	251.00	7.833	159170	MM	379.83	51
20	102899020	MeOH Blk			7.829	14836	MM	27.09	
21	102899021	FCMIX-99086-43-1, 1ppb- 9/10/99 RWW	Standard	1.01	7.851	8217	MMX	10.92	981
22	102899022	FCMIX-99086-43-2, 5ppb- 9/10/99 RWW	Standard	5.03	7.828	8161	MMX	10.78	114
23	102899023	FCMIX-99086-43-3, 10ppb- 9/10/99 RWW	Standard	10.10	7.829	9260	MMX	13.46	33
24	102899024	FCMIX-99086-43-4, 25ppb- 9/10/99 RWW	Standard	25.10	7.836	16554	bb	31.29	25
25	102899025	FCMIX-99086-43-5, 50ppb- 9/10/99 RWW	Standard	50.10	7.823	25954	bb	54.26	8
26	102899026	FCMIX-99086-43-6, 100ppb- 9/10/99 RWW	Standard	101.00	7.831	51871	bb	117.60	16
27	102899027	FCMIX-99086-43-7, 250ppb- 9/10/99 RWW	Standard	251.00	7.830	120810	bb	286.08	14
28	102899028	FCMIX-99086-43-8, 500ppb- 9/10/99 RWW	Standard	503.00	7.830	217005	bb	521.17	4
29	102899029	FCMIX-99086-43-9, 750ppb- 9/10/99 RWW	Standard	754.00	7.808	304590	bb	735.21	-2

Soup 020199: Quantitated and printed by ias on 10/29/99

000757

Sample List: C:\NASSELYNK\scup020199.pro\SampleDS\102899a
 Last modified: Fri Oct 29 07:02:56 1999
 Method: C:\NASSELYNK\scup020199.pro\MethodS\102899a
 Last modified: Fri Oct 29 06:53:35 1999
 Job Code:

Printed: Fri Oct 29 07:37:25 1999

Compound 4: IS (Ch4)

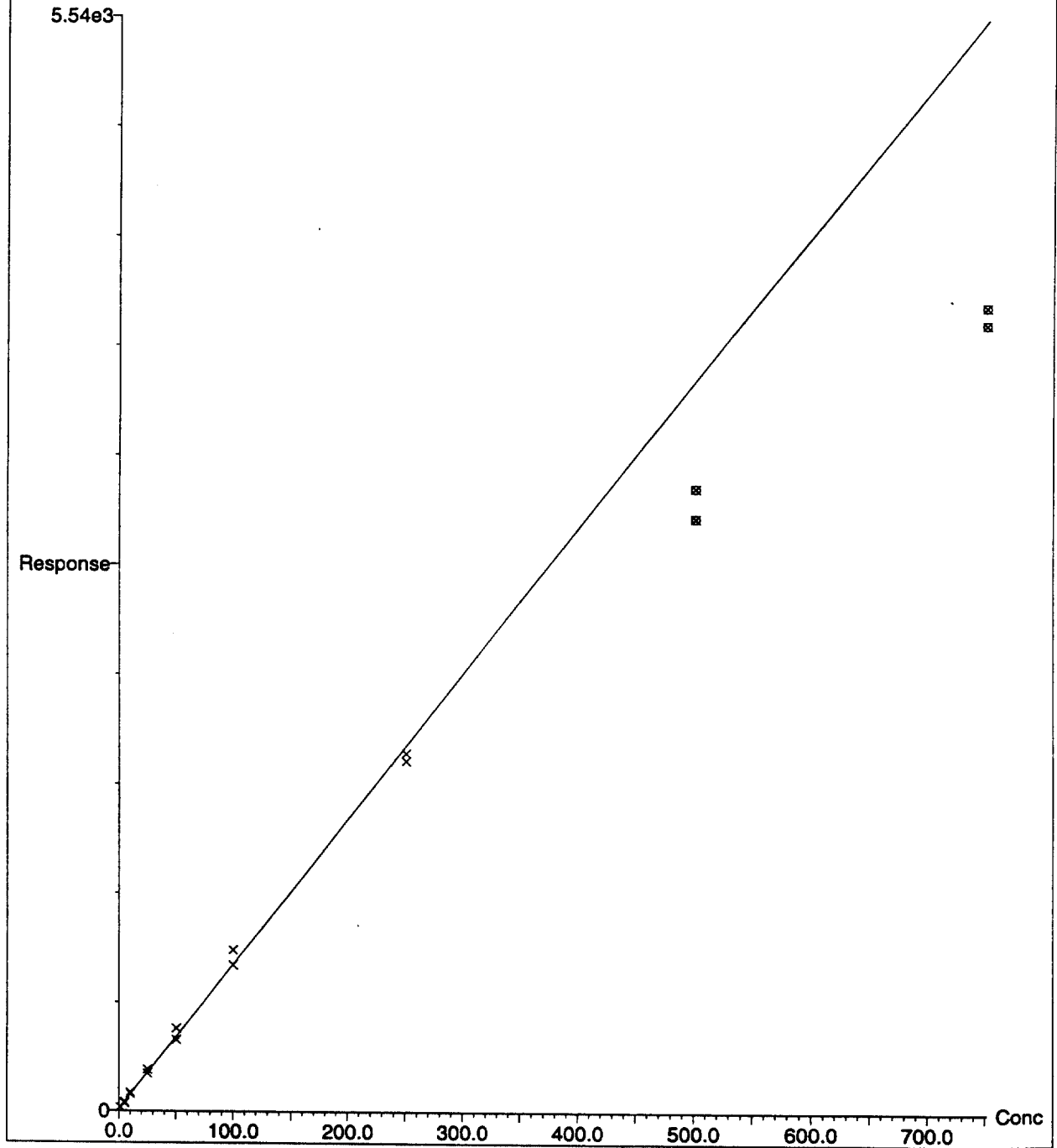
# Name	Samplelist Test	Type	Std Conc	RT	Area	Flags	Conc. %Dev
1 102899001	MeOH Blk			7.081	16	bb	0.00 -100
2 102899002	MeOH Blk						
3 102899003	FCMIX-99086-43-1, 1ppb- 9/10/99 RMW	Standard	1.00	7.083	4165	bb	1.26 26
4 102899004	FCMIX-99086-43-2, 5ppb- 9/10/99 RMW	Standard	1.00	7.102	4646	bd	1.40 40
5 102899005	FCMIX-99086-43-3, 10ppb- 9/10/99 RMW	Standard	1.00	7.101	4643	bb	1.40 40
6 102899006	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	1.00	7.103	4814	bb	1.45 45
7 102899007	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	1.00	7.109	4546	bb	1.37 37
8 102899008	FCMIX-99086-43-6, 100ppb- 9/10/99 RMW	Standard	1.00	7.102	4172	bb	1.26 26
9 102899009	FCMIX-99086-43-7, 250ppb- 9/10/99 RMW	Standard	1.00	7.105	2878	bd	0.87 -13
10 102899010	FCMIX-99086-43-8, 500ppb- 9/10/99 RMW	Standard	1.00	7.120	2479	bb	0.75 -25
11 102899011	FCMIX-99086-43-9, 750ppb- 9/10/99 RMW	Standard	1.00	7.124	2203	bb	0.67 -33
12 102899012	MeOH Blk						
13 102899013	TOX145-PFOS/POAA in Tween-dose-2ppb- 10/28/99 IAS			7.124	3463	bb	1.05 5
14 102899014	TOX145-PFOS/POAA in Tween-ma-4ppb- 10/28/99 IAS			7.120	3528	bb	1.07 7
15 102899015	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	QC	1.00	7.124	3185	bb	0.96 -4
16 102899016	MeOH Blk						
17 102899017	TOX145-PFOSA in Tween-dose-200ppb- 10/28/99 IAS			7.128	3679	bb	1.11 11
18 102899018	TOX145-PFOSA in Tween-ma-300ppb- 10/28/99 IAS			7.124	3545	bb	1.07 7
19 102899019	FCMIX-99086-43-7, 250ppb- 9/10/99 RMW	QC	1.00	7.127	2681	bb	0.81 -19
20 102899020	MeOH Blk						
21 102899021	FCMIX-99086-43-1, 1ppb- 9/10/99 RMW	Standard	1.00	7.145	3119	bb	0.94 -6
22 102899022	FCMIX-99086-43-2, 5ppb- 9/10/99 RMW	Standard	1.00	7.143	3041	bb	0.92 -8
23 102899023	FCMIX-99086-43-3, 10ppb- 9/10/99 RMW	Standard	1.00	7.125	2979	bb	0.90 -10
24 102899024	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	1.00	7.151	2937	bb	0.89 -11
25 102899025	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	1.00	7.141	2907	bb	0.88 -12
26 102899026	FCMIX-99086-43-6, 100ppb- 9/10/99 RMW	Standard	1.00	7.147	2907	bb	0.88 -12
27 102899027	FCMIX-99086-43-7, 250ppb- 9/10/99 RMW	Standard	1.00	7.149	2595	bb	0.78 -22
28 102899028	FCMIX-99086-43-8, 500ppb- 9/10/99 RMW	Standard	1.00	7.145	2441	bb	0.74 -26
29 102899029	FCMIX-99086-43-9, 750ppb- 9/10/99 RMW	Standard	1.00	7.121	2112	bb	0.64 -36

Inc 10/29/99 7-104

Quantify Calibration Report
TOX145-tween 80 dose confirmation- PFOS/PFOA/POAA ext. 10/28/99 ias

Page 1

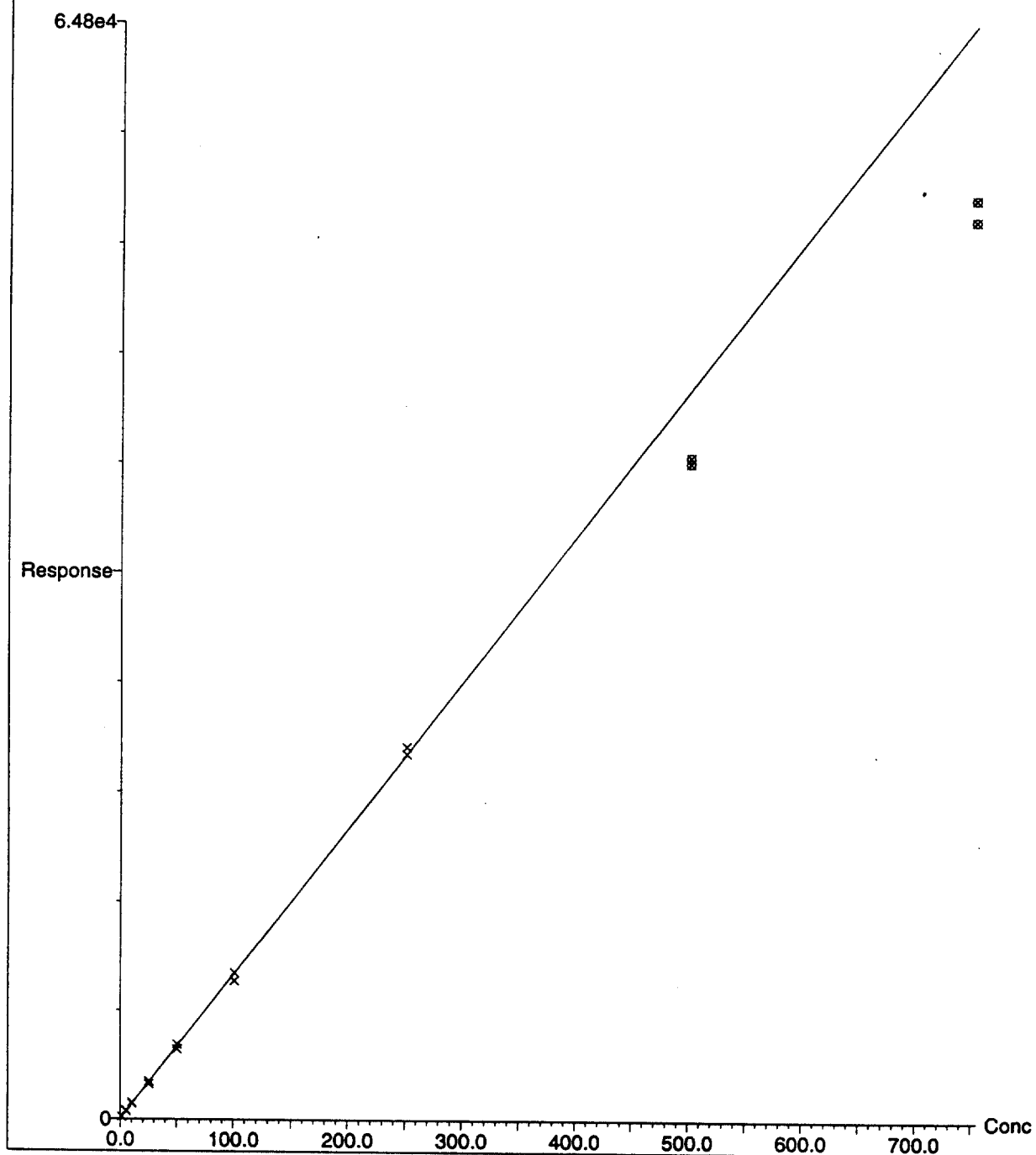
Compound 1 name: POAA (Ch1)
Coefficient of Determination: 0.996938
Calibration curve: $7.35186 \cdot x + 10.5814$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



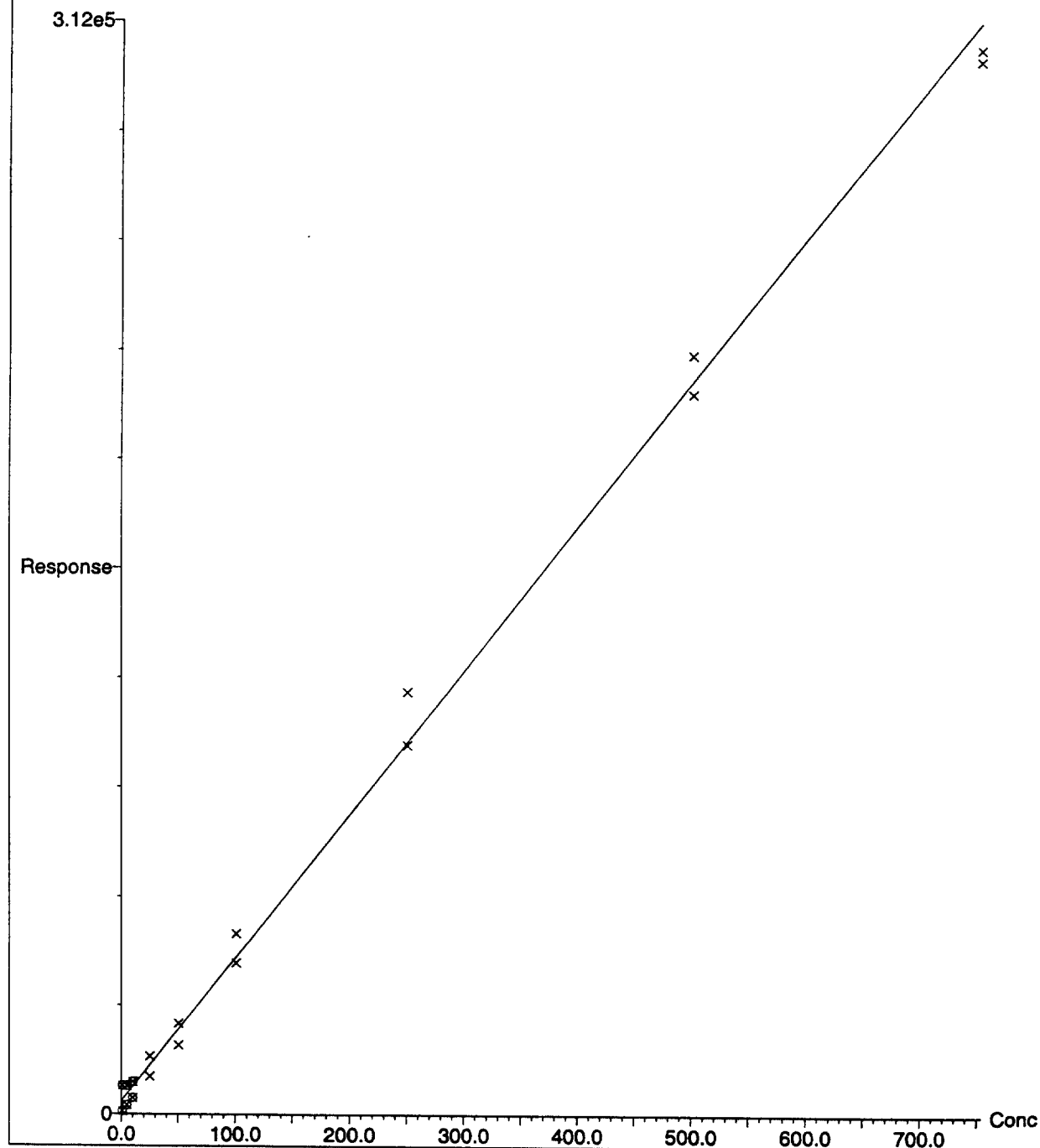
Soup 020199: Quantitated and printed by ias on 10/29/99

000759

Compound 2 name: PFOS (Ch2)
Coefficient of Determination: 0.999345
Calibration curve: $85.8285 * x + 40.9705$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



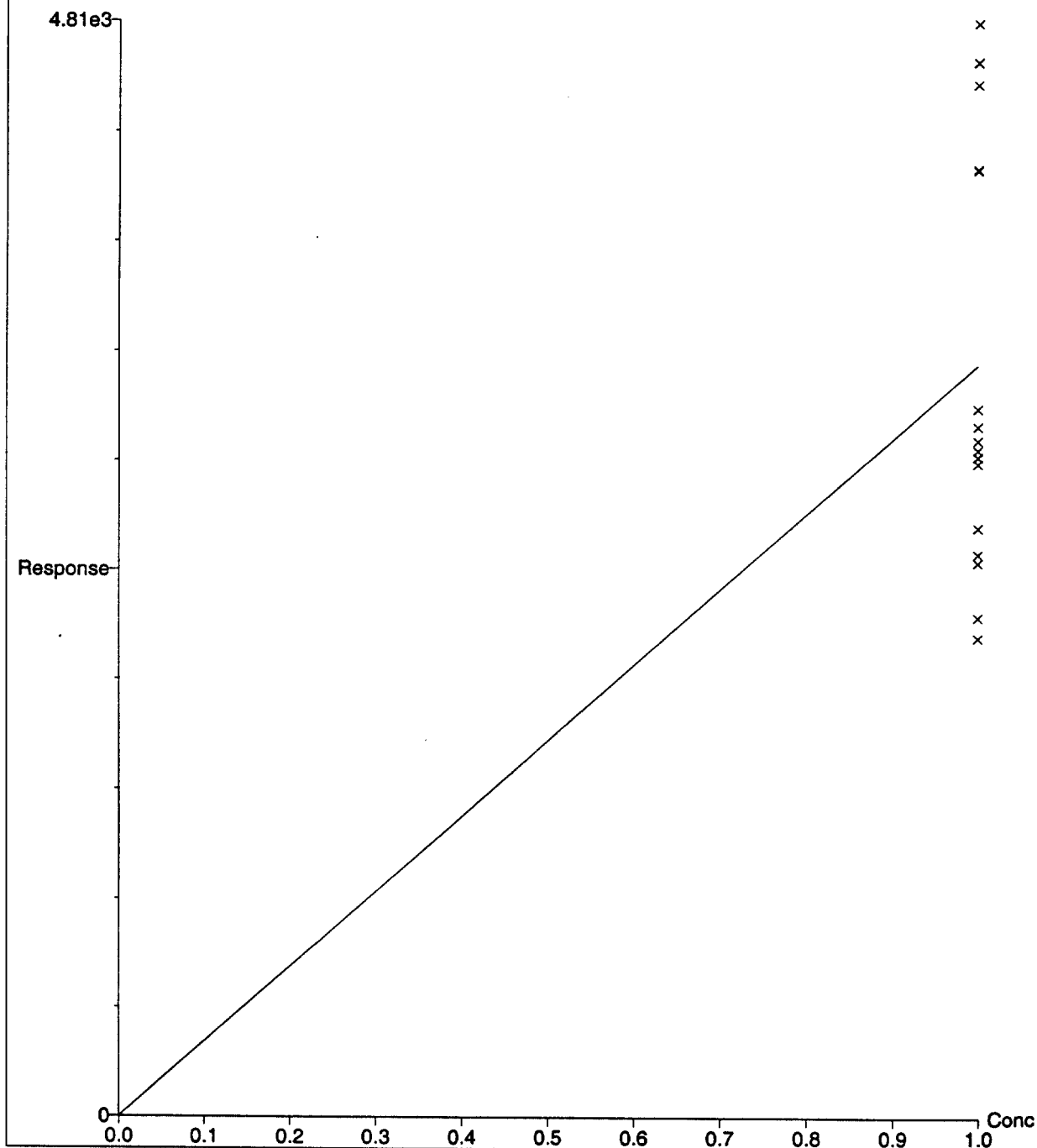
Compound 3 name: PFOSA (Ch3)
Coefficient of Determination: 0.995986
Calibration curve: $409.188 * x + 3750.14$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Soup 020199: Quantitated and printed by las on 10/29/99

000761

Compound 4 name: IS (Ch4)
Response Factor: 3310.11
Response type: External Std, Area
Curve type: RF



Las 10/29/99 7 10/4

Method Report

Method file: C:\MASSLYNX\scup020199.pro\MethDB\102899a.mdb
Last modified: Fri Oct 29 06:53:35 1999

Compound: 1: POAA (Ch1)

Printed: Fri Oct 29 07:38:25 1999

Compound Name: POAA (Ch1)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. A
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.152, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.356

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 63

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

Method Report

Page 2

Method file: C:\MASSLYNK\scup020199.pro\MethDB\102899a.mdb
Last modified: Fri Oct 29 06:53:35 1999

Compound: 2: PFOS (Ch2)

Printed: Fri Oct 29 07:38:25 1999

Compound Name: PFOS (Ch2)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. 8
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.335, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.334

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 88

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000764

Method Report

Page 3

Method file: C:\MASSLYNK\scup020199.pro\MethDB\102899a.mdb
Last modified: Fri Oct 29 06:53:35 1999

Compound: 3: PFOSA (Ch3)

Printed: Fri Oct 29 07:38:25 1999

Compound Name: PFOSA (Ch3)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. C
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 8.053, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.445

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 147

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000765

Method Report

Page 4

Method file: C:\MASSLYNX\scup020199.pro\MethDB\102899a.mdb
Last modified: Fri Oct 29 06:53:35 1999

Compound: 4: IS (Ch4)

Printed: Fri Oct 29 07:38:25 1999

Compound Name: IS (Ch4)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Fixed
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.190, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.290

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 70

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000766

10/28/99
MCE

SOUP 020199: FACT Equipment
Log 99092 PAGE 1/1

Quattro II, #6414E

DATE: 10-28-99

ANALYST: ias

STUDY NUMBER: TOX145 MATRIX: Tween samples DESCRIPTION: dose confirmation

FACT Method:

Injection volume: 10 uL Cycle time: 10 min. Electrode: Z-Spray

Mobile Phase: (A) 2.0 mM NH₄CH₃CO₂ (B) MeOH

TN-A- or stdrd number (A) 99085-11-3 (B) TN-A-2997

Prepared (date/by): (A) 10/21/99 mmh (B) 10/16/99 sah

Expires: (A) 04/00 (B) 2010

Store at room temp? ☒ Y or N Other: _____

Guard column: None

Part number: na Lot number: na

Analytical column: Keystone Betasil

Size: 2 x 50 mm Type: C18 Sphere size: 5 um

Part number: 055-701-2 Lot number: P9E11 Serial number: 0591804Z

Analytical parameters:

Drying gas (L/hr): 350 ESI nebulizing gas (L/hr) 13 Sheath gas (L/hr) 0

LC Flow rate: 300 uL/min. Back pressure: 65 bar

HPLC solvent gradient: 10min-10-95

Time (min.)	%B
1) 0.00	10.0
2) 1.00	10.0
3) 5.50	95.0
4) 7.50	95.0
5) 8.00	10.0

000767

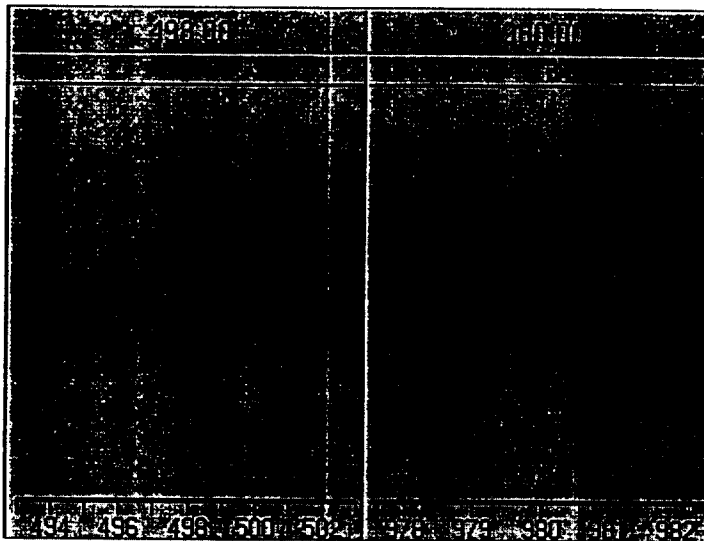
10/28/99
MEE

Tuning Method Report

Page 1

Method: C:\MASSLYNX\SOUP020199.PRO\ACQUD8\PFOS3

Printed: Thu Oct 28 15:21:34 1999



MS

MS

SOURCE (ESP-)

Set Rdbk

Capillary	1.60	1.59
Cone	60	-60
Extractor	5	-7
RF Lens	0.2	
Source Block Temp.	150	153
Desolvation Temp.	250	249

MS1

Set Rdbk

LM Resolution	14.0
HM Resolution	14.0
Ion Energy	0.9
Ion Energy Ramp	0.0
Lens 6	4
Multiplier	650

Pressures

Rdbk

Analyser Vacuum	6.1e-5
Gas Cell	4.1e-3

MS2

Set Rdbk

LM Resolution	14.0
HM Resolution	14.0
Collision	60
Ion Energy	2.0
Ion Energy Ramp	0.0
Lens 8	40
Lens 9	0
Multiplier	650

10/28/99 MEE

Method Report

Page 1

Method File:

C:\MASSLYNX\SOUP020199.PRO\ACQ008\10min-10-95

Last Modified:

Thursday, October 28, 1999 13:44:34

Printed:

Thursday, October 28, 1999 13:44:44

HP1100 LC Pump Initial Conditions

Solvents

A%	90.0
B%	10.0
C%	0.0
D%	0.0

Flow (ml/min)	0.300
Stop Time (mins)	10.0
Min Pressure (bar)	0
Max Pressure (bar)	400
Oven Temperature Left(°C)	30.0
Oven Temperature Right(°C)	30.0

HP1100 LC Pump Gradient Timetable

The gradient Timetable contains 5 entries which are :

Time	A%	B%	C%	D%	Flow	Pressure
0.00	90.0	10.0	0.0	0.0	0.300	400
1.00	90.0	10.0	0.0	0.0	0.300	400
5.50	5.0	95.0	0.0	0.0	0.300	400
7.50	5.0	95.0	0.0	0.0	0.300	400
8.00	90.0	10.0	0.0	0.0	0.300	400

HP1100 LC Pump External Event Timetable

The Timetable contains 3 entries which are :

Time	Column Switch	Contact1	Contact2	Contact3	Contact4
Initial	Off	Off	Off	Off	Off
0.00	Off	On	On	Off	On
0.10	Off	Off	Off	Off	Off

HP1100 Autosampler Initial Conditions

Draw Speed	200.0
Eject Speed (ul/min)	200
Draw Position (mm)	0.00
Stop Time (mins)	9.00
Injection Volume(ul)	10.0
Vial Number	41

000769

12/28/99
MEE

Scanning Method Report

Page 1

Method: C:\MASSLYNK\SCUP020199.FPO\ACQUDE\10MIN-PPOM-POAA-PPOMA-XS
Last Modified: Fri Oct 15 11:35:03 1999

Printed: Thu Oct 28 13:44:18 1999

Solvent Delay (mins) : 0.00

Function : 1 MMN of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	413.00	169.00	0.30	30	15

Function : 2 MMN of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	499.00	99.00	0.30	45	60

Function : 3 MMN of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	498.00	78.00	0.30	45	60

Function : 4 MMN of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	427.00	80.00	0.30	40	60

000770

10/28/99
MEE

Masslynx - Sample List

Page 1

Sample List: C:\MASSLYNX\soup020199.pro\SampleDB\102899a.SPL
Printed: Thu Oct 28 13:44:09 1999

Page Position: (1, 1)

	File Name	File Text	MS File
1	102899001	MeOH Blk	10MIN-PFOS-POAA-PFOSA-IS
2	102899002	MeOH Blk	10MIN-PFOS-POAA-PFOSA-IS
3	102899003	FCMix-99086-43-1, 1ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
4	102899004	FCMix-99086-43-2, 5ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
5	102899005	FCMix-99086-43-3, 10ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
6	102899006	FCMix-99086-43-4, 25ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
7	102899007	FCMix-99086-43-5, 50ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
8	102899008	FCMix-99086-43-6, 100ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
9	102899009	FCMix-99086-43-7, 250ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
10	102899010	FCMix-99086-43-8, 500ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
11	102899011	FCMix-99086-43-9, 750ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
12	102899012	MeOH Blk	10MIN-PFOS-POAA-PFOSA-IS
13	102899013	TOX145-PFOS/POAA in Tween-dose-2ppb- 10/28/99 IAS	10MIN-PFOS-POAA-PFOSA-IS
14	102899014	TOX145-PFOS/POAA in Tween-ms-4ppb- 10/28/99 IAS	10MIN-PFOS-POAA-PFOSA-IS
15	102899015	FCMix-99086-43-4, 25ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
16	102899016	MeOH Blk	10MIN-PFOS-POAA-PFOSA-IS
17	102899017	TOX145-PFOSA in Tween-dose-200ppb- 10/28/99 IAS	10MIN-PFOS-POAA-PFOSA-IS
18	102899018	TOX145-PFOSA in Tween-ms-300ppb- 10/28/99 IAS	10MIN-PFOS-POAA-PFOSA-IS
19	102899019	FCMix-99086-43-7, 250ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
20	102899020	MeOH Blk	10MIN-PFOS-POAA-PFOSA-IS
21	102899021	FCMix-99086-43-1, 1ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
22	102899022	FCMix-99086-43-2, 5ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
23	102899023	FCMix-99086-43-3, 10ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
24	102899024	FCMix-99086-43-4, 25ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
25	102899025	FCMix-99086-43-5, 50ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
26	102899026	FCMix-99086-43-6, 100ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
27	102899027	FCMix-99086-43-7, 250ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
28	102899028	FCMix-99086-43-8, 500ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS
29	102899029	FCMix-99086-43-9, 750ppb- 9/10/99 RWW	10MIN-PFOS-POAA-PFOSA-IS

000771

10/28/99
MEE

Masslynx - Sample List

Page 2

Sample List: C:\MASSLYNX\lsoup020199.pro\SampleDB\102899a.SPL
Printed: Thu Oct 28 13:44:09 1999

Page Position: (2, 1)

	Inlet File	Bottle	Inject Volume	Sample Type	Conc A	Conc B	Conc C
1	10min-10-95	91	10	---	---	---	---
2	10min-10-95	91	10	---	---	---	---
3	10min-10-95	1	10	---	---	---	---
4	10min-10-95	2	10	---	---	---	---
5	10min-10-95	3	10	---	---	---	---
6	10min-10-95	4	10	---	---	---	---
7	10min-10-95	5	10	---	---	---	---
8	10min-10-95	6	10	---	---	---	---
9	10min-10-95	7	10	---	---	---	---
10	10min-10-95	8	10	---	---	---	---
11	10min-10-95	9	10	---	---	---	---
12	10min-10-95	92	10	---	---	---	---
13	10min-10-95	10	10	---	---	---	---
14	10min-10-95	11	10	---	---	---	---
15	10min-10-95	4	10	---	---	---	---
16	10min-10-95	92	10	---	---	---	---
17	10min-10-95	12	10	---	---	---	---
18	10min-10-95	13	10	---	---	---	---
19	10min-10-95	7	10	---	---	---	---
20	10min-10-95	92	10	---	---	---	---
21	10min-10-95	1	10	---	---	---	---
22	10min-10-95	2	10	---	---	---	---
23	10min-10-95	3	10	---	---	---	---
24	10min-10-95	4	10	---	---	---	---
25	10min-10-95	5	10	---	---	---	---
26	10min-10-95	6	10	---	---	---	---
27	10min-10-95	7	10	---	---	---	---
28	10min-10-95	8	10	---	---	---	---
29	10min-10-95	9	10	---	---	---	---

000772

FACT: DATA REVIEW SUMMARY FORM

Date: 11/08/99
 Data Analyst: IAS
 FACT Study #: TOX145
 Matrix/Timepoints: Rat Liver - Control & dose day 29 - ext 11/4/99
 Sample List: 110599a
 Set-up Analysis Date/Analyst: IAS 11/05/99
 Instrument: Amulix 062499

Data Review Sign-Off

Analyst Review
 Name / Date: IAS 11/08/99
 Technical Review
 Name / Date: MEG 11/8/99 / Kih 11/18/99
 Quality Assurance Review
 Name / Date: _____
 Data Entry: Data Reported GML 11/8/99

Analyte: POAA (CH1) ①
 Acceptable Linear Range: 61.30ppb → 1227ppb
 LOQ: 61.30ppb Extraction Blank: less than LOQ
 R²: 0.987198 Matrix Blank: less than LOQ
 Calibration Checks: OK - Range from -5% → -10%
 MS/MSD: N/A
 Data Status: DATA NOT REPORTED GML 11/8/99

Analyte: PFOS (CH2) ② - Dilutions needed. IAS 11/08/99
 Acceptable Linear Range: 12.30ppb → 921ppb
 LOQ: 12.30ppb Extracted Blank: less than LOQ
 R²: 0.982749 Matrix Blank: greater than LOQ
 Calibration Checks: OK - Range from -27% → -13%
 MS/MSD: 87%, 78%
 Data Status: DATA for Control Gp 3 reported. DR 1:100 Gp 6 GML 11/8/99

Analyte: Surr (CH3) ③
 Acceptable Linear Range: NA
 LOQ: NA Extracted Blank: NA
 R²: NA Matrix Blank: NA
 Calibration Checks: NA
 MS/MSD: N/A
 Data Status: OK GML 11/8/99

Comments / Justifications:
 ① IAS 11/08/99
The low level points (6.13ppb, 12.30ppb, & 30.70ppb), were not 2x higher than the extraction blank; these points were excluded from the curve to disqualify a data range that may have been significantly affected by background levels of the analyte.
- IAS 11/08/99

② Low level point 6.14ppb was not 2x higher than the extraction blank; this point was excluded from the curve to disqualify a data range that may have been significantly affected by background levels of the analyte.
- IAS 11/08/99

High curve point 1228ppb was excluded to provide a better fit over the linear range appropriate to the data.
- IAS 11/08/99

③ Internal Std area counts in all control samples and the MS/MSD were low.
- IAS 11/8/99

IAS 11/08/99 p. 1 of 2

000773

FACT: DATA REVIEW SUMMARY FORM

Date: 11/08/99
 Data Analyst: JAS
 FACT Study #: Tox145
 Matrix/Timepoints: Rat Liver - control & dose day 29 - ext 11/1/99
 Sample List: 110599a
 Set-up Analysis Date/Analyst: JAS 11/05/99
 Instrument: Amulim 062498

Data Review Sign-Off

Analyst Review Name / Date: JAS 11/08/99

Technical Review Name / Date: Refer to page 1 11/09/99

Quality Assurance Review Name / Date: _____

Data Entry: refer to page 1 11/09/99

Comments / Justifications:

① Low level points 6.83 ppb and 12.30 ppb were not 2x higher than the extraction blank; these points were excluded from the curve to disqualify a data range that may have been significantly affected by background levels of the analyte.
 - JAS 11/08/99

Analyte: PFOSA (CH4)
 Acceptable Linear Range: 30.70 ppb → 1227 ppb
 LOQ: 30.70 ppb Extraction Blank: less than LOQ
 R²: 0.988845 Matrix Blank: less than LOQ
 Calibration Checks: OK - Range from 15% → 26%
 MS/MSD: 1466, 1370 (High)
 Data Status: OK - GML 11/8/99 Data Reported @ 3+6 OK

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: JAS 11/08/99
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: _____

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: _____

ORE GML 11/8/99

JAS 11/08/99 p. 2 of 2

000774

Quantify Compound Summary Report

TOX145-rat liver, Day29 dose and control- ext. 11/4/99 RWW

Sample List: C:\MASSLIT\Amelia062498.PRO\SampleDB\110599a
 Last modified: Mon Nov 08 08:54:18 1999
 Method: F:\Amelia backup sep99\Amelia-rats.pro\MethDB\110599a
 Last modified: Mon Nov 08 08:20:27 1999
 Job Code:

Printed: Mon Nov 08 09:18:10 1999

Compound 1: POAA (Ch1)

# Name	Sample List Text	Type	Std Conc	RT	Area	Conc.	Flags	%Dev	Mod	Comment
1 110599001	MeOH Blank									
2 110599002	MeOH Blank									
3 110599003	MeOH Blank									
4 110599004	TOX145-RBL11049-WBlk-1, RWW- Box 99-211			7.065	68	0.00	bb			
5 110599005	TOX145-RBL11049-RBL Blk-1, RWW- Box 99-211			7.041	60	0.00	bb			
6 110599006	TOX145-RBL11049-6ppb-1, RWW- Box 99-211	Standard	6.13	7.015	79	0.00	bbX	-100		
7 110599007	TOX145-RBL11049-12ppb-1, RWW- Box 99-211	Standard	12.30	7.009	100	0.00	MMX	-100		
8 110599008	TOX145-RBL11049-30ppb-1, RWW- Box 99-211	Standard	30.70	7.016	115	0.00	MMX	-100		
9 110599009	TOX145-RBL11049-60ppb-1, RWW- Box 99-211	Standard	61.30	7.021	171	52.87	bb	-14		
10 110599010	TOX145-RBL11049-120ppb-1, RWW- Box 99-211	Standard	123.00	7.013	234	117.58	bb	-4		
11 110599011	TOX145-RBL11049-300ppb-1, RWW- Box 99-211	Standard	307.00	6.998	442	330.89	bb	-8		
12 110599012	TOX145-RBL11049-600ppb-1, RWW- Box 99-211	Standard	613.00	6.996	786	684.65	bb	12		
13 110599013	TOX145-RBL11049-900ppb-1, RWW- Box 99-211	Standard	920.00	6.972	990	894.00	bb	-3		
14 110599014	TOX145-RBL11049-1200ppb-1, RWW- Box 99-211	Standard	1227.00	6.965	1228	1138.49	bb	-7		
15 110599015	MeOH Blank									
16 110599016	TOX145-RTL, 9R03240-300ppb-MS, 11/4/99- Box 99-211			6.993	378	265.61	bb			
17 110599017	TOX145-RTL, 9R03240-300ppb-MSD, 11/4/99 Box 99-211			6.976	379	265.72	bb			
18 110599018	TOX145-RTL, 9R03240/M/Day29/Control, 11/4/99			7.015	89	0.00	bb			
19 110599019	TOX145-RTL, 9R03241/M/Day29/Control, 11/4/99			7.018	94	0.00	bb			
20 110599020	TOX145-RTL, 9R03242/M/Day29/Control, 11/4/99			7.017	94	0.00	bb			
21 110599021	TOX145-RBL11049-300ppb-CC1-1, RWW- Box 99-211	QC	307.00	6.990	404	291.80	bb	-5		
22 110599022	MeOH Blank									
23 110599023	TOX145-RTL, 9R03243/M/Day29/Control, 11/4/99			7.015	93	0.00	bb			
24 110599024	TOX145-RTL, 9R03244/M/Day29/Control, 11/4/99			7.019	93	0.00	bb			
25 110599025	TOX145-RTL, 9R03255/M/Day29/5mg/kg, 11/4/99			7.010	91	0.00	bb			
26 110599026	TOX145-RTL, 9R03256/M/Day29/5mg/kg, 11/4/99			6.992	98	0.00	bb			
27 110599027	TOX145-RTL, 9R03257/M/Day29/5mg/kg, 11/4/99			6.999	91	0.00	bb			
28 110599028	TOX145-RBL11049-300ppb-CC1-2, RWW- Box 99-211	QC	307.00	6.994	390	277.30	bb	-10		
29 110599029	MeOH Blank									
30 110599030	TOX145-RTL, 9R03258/M/Day29/5mg/kg, 11/4/99			7.019	77	0.00	bb			
31 110599031	TOX145-RTL, 9R03259/M/Day29/5mg/kg, 11/4/99			6.995	96	0.00	bb			
32 110599032	TOX145-RBL11049-300ppb-CC1-1, RWW- Box 99-211	QC	307.00	6.975	402	289.39	bb	-6		
33 110599033	MeOH Blank									
34 110599034	TOX145-RBL11049-WBlk-1, RWW- Box 99-211			7.011	89	0.00	bb			
35 110599035	TOX145-RBL11049-RBL Blk-1, RWW- Box 99-211			7.021	76	0.00	MM			
36 110599036	TOX145-RBL11049-6ppb-1, RWW- Box 99-211	Standard	6.13	7.016	78	0.00	bbX	-100		
37 110599037	TOX145-RBL11049-12ppb-1, RWW- Box 99-211	Standard	12.30	6.994	104	0.00	MMX	-100		
38 110599038	TOX145-RBL11049-30ppb-1, RWW- Box 99-211	Standard	30.70	6.985	120	0.00	MMX	-100		
39 110599039	TOX145-RBL11049-60ppb-1, RWW- Box 99-211	Standard	61.30	6.987	173	54.77	bb	-11		
40 110599040	TOX145-RBL11049-120ppb-1, RWW- Box 99-211	Standard	123.00	6.997	240	123.52	bb	0		
41 110599041	TOX145-RBL11049-300ppb-1, RWW- Box 99-211	Standard	307.00	6.991	468	357.80	bb	17		
42 110599042	TOX145-RBL11049-600ppb-1, RWW- Box 99-211	Standard	613.00	6.965	760	657.12	bb	7		
43 110599043	TOX145-RBL11049-900ppb-1, RWW- Box 99-211	Standard	920.00	6.969	1007	911.72	bb	-1		
44 110599044	TOX145-RBL11049-1200ppb-1, RWW- Box 99-211	Standard	1227.00	6.968	1268	1179.19	bb	-4		

Amelia 062498: Quantitated and reviewed by ias on 11/8/99

000775

Quantify Compound Summary Report

TOX145-rat liver, Day29 dose and control- ext. 11/4/99 RWW

Page 2

Sample List: C:\MASSLYNX\Amelia062498.PRO\SampleDB\110599a
Last modified: Mon Nov 08 08:54:18 1999
Method: F:\Amelia backup sep99\Amelia-rats.pro\MethDB\110599a
Last modified: Mon Nov 08 08:20:27 1999
Job Code:

Printed: Mon Nov 08 09:18:10 1999

Compound 2: PFCs (Ch2)

#	Name	Samplelist Text	Type	Std Conc	RT	Area	Conc.	Flags	Dev	Mod	Comment
1	110599001	MeOH Blank			7.256	77	0.00	bb			
2	110599002	MeOH Blank			7.238	55	0.00	bb			
3	110599003	MeOH Blank			7.242	39	0.00	bb			
4	110599004	TOX145-RBL11049-WBlk-1, RWW- Box 99-211			7.214	93	0.00	bb			
5	110599005	TOX145-RBL11049-RBL Blk-1, RWW- Box 99-211			7.213	149	0.00	bb			
6	110599006	TOX145-RBL11049-6ppb-1, RWW- Box 99-211	Standard	6.14	7.163	245	0.00	bbX	-100		
7	110599007	TOX145-RBL11049-12ppb-1, RWW- Box 99-211	Standard	12.30	7.181	362	10.61	MM	-14		
8	110599008	TOX145-RBL11049-30ppb-1, RWW- Box 99-211	Standard	30.70	7.165	508	25.94	bb	-15		
9	110599009	TOX145-RBL11049-60ppb-1, RWW- Box 99-211	Standard	61.40	7.194	848	61.71	bb	1		
10	110599010	TOX145-RBL11049-120ppb-1, RWW- Box 99-211	Standard	123.00	7.163	1539	134.29	bb	9		
11	110599011	TOX145-RBL11049-300ppb-1, RWW- Box 99-211	Standard	307.00	7.171	3586	349.39	bb	14		
12	110599012	TOX145-RBL11049-600ppb-1, RWW- Box 99-211	Standard	614.00	7.168	6303	634.97	bb	3		
13	110599013	TOX145-RBL11049-900ppb-1, RWW- Box 99-211	Standard	921.00	7.144	8113	825.08	bb	-10		
14	110599014	TOX145-RBL11049-1200ppb-1, RWW- Box 99-211	Standard	1228.00	7.161	9947	1017.86	bbX	-17		
15	110599015	MeOH Blank			7.193	52	0.00	MM			
16	110599016	TOX145-RTL, 9R03240-300ppb-MS, 11/4/99- Box 99-211			7.166	2315	215.83	dd			
17	110599017	TOX145-RTL, 9R03240-300ppb-MSD, 11/4/99 Box 99-211			7.174	2113	194.65	bb			
18	110599018	TOX145-RTL, 9R03240/M/Day29/Control, 11/4/99			7.164	201	0.00	bb			
19	110599019	TOX145-RTL, 9R03241/M/Day29/Control, 11/4/99			7.166	196	0.00	bb			
20	110599020	TOX145-RTL, 9R03242/M/Day29/Control, 11/4/99			7.165	157	0.00	bb			
21	110599021	TOX145-RBL11049-300ppb-CC1-1, RWW- Box 99-211	QC	307.00	7.163	2388	223.50	MM			-27 Data Reported
22	110599022	MeOH Blank			7.191	69	0.00	MM			
23	110599023	TOX145-RTL, 9R03243/M/Day29/Control, 11/4/99			7.188	148	0.00	bb			
24	110599024	TOX145-RTL, 9R03244/M/Day29/Control, 11/4/99			7.168	145	0.00	bb			
25	110599025	TOX145-RTL, 9R03255/M/Day29/5mg/kg, 11/4/99			7.182	96493	10112.21	bb			
26	110599026	TOX145-RTL, 9R03256/M/Day29/5mg/kg, 11/4/99			7.165	114302	11983.63	bb			
27	110599027	TOX145-RTL, 9R03257/M/Day29/5mg/kg, 11/4/99			7.171	108784	11403.79	bb			
28	110599028	TOX145-RBL11049-300ppb-CC1-2, RWW- Box 99-211	QC	307.00	7.167	2676	253.78	bb			-17 Dilute 1:100
29	110599029	MeOH Blank			7.186	72	0.00	bb			
30	110599030	TOX145-RTL, 9R03258/M/Day29/5mg/kg, 11/4/99			7.167	107695	11289.34	bb			
31	110599031	TOX145-RTL, 9R03259/M/Day29/5mg/kg, 11/4/99			7.167	130162	13650.29	bb			
32	110599032	TOX145-RBL11049-300ppb-CC1-1, RWW- Box 99-211	QC	307.00	7.147	2816	268.48	bb			-13
33	110599033	MeOH Blank			7.172	98	0.00	bb			
34	110599034	TOX145-RBL11049-WBlk-1, RWW- Box 99-211			7.193	136	0.00	bb			
35	110599035	TOX145-RBL11049-RBL Blk-1, RWW- Box 99-211			7.170	191	0.00	bb			
36	110599036	TOX145-RBL11049-6ppb-1, RWW- Box 99-211	Standard	6.14	7.164	286	2.64	bbX	-57		
37	110599037	TOX145-RBL11049-12ppb-1, RWW- Box 99-211	Standard	12.30	7.166	355	9.93	bb	-19		
38	110599038	TOX145-RBL11049-30ppb-1, RWW- Box 99-211	Standard	30.70	7.157	545	29.07	bb	-3		
39	110599039	TOX145-RBL11049-60ppb-1, RWW- Box 99-211	Standard	61.40	7.160	878	64.86	bb	6		
40	110599040	TOX145-RBL11049-120ppb-1, RWW- Box 99-211	Standard	123.00	7.168	1608	141.52	bb	15		
41	110599041	TOX145-RBL11049-300ppb-1, RWW- Box 99-211	Standard	307.00	7.163	3667	357.88	bb	17		
42	110599042	TOX145-RBL11049-600ppb-1, RWW- Box 99-211	Standard	614.00	7.137	6459	651.32	bb	6		
43	110599043	TOX145-RBL11049-900ppb-1, RWW- Box 99-211	Standard	921.00	7.165	8268	841.43	bb	-9		
44	110599044	TOX145-RBL11049-1200ppb-1, RWW- Box 99-211	Standard	1228.00	7.142	10512	1077.24	bbX	-12		

Amelia 062498: Quantitated and reviewed by iac on 11/8/99

000776

Quantify Compound Summary Report

TOX145-rat liver, Day29 dose and control- ext. 11/4/99 RWW

Page 3

Sample List: C:\MSL\YOK\amelia062498.PRO\SampleDB\110599a
 Last modified: Mon Nov 08 08:54:18 1999
 Method: F:\amelia backup sep99\amelia-rats.pro\MethDB\110599a
 Last modified: Mon Nov 08 08:20:27 1999
 Job Code:

Printed: Mon Nov 08 09:18:10 1999

Compound 3: Surr (Ch3)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Conc.	Flags	Dev	Mod	Comment
1 110599001	MeOH Blank									
2 110599002	MeOH Blank									
3 110599003	MeOH Blank									
4 110599004	TOX145-RBL11049-WBLK-1, RWW- Box 99-211			7.052	4714	0.99 bb		-1		
5 110599005	TOX145-RBL11049-RBL B1K-1, RWW- Box 99-211			7.028	3503	0.73 bb		-27		
6 110599006	TOX145-RBL11049-6ppb-1, RWW- Box 99-211	Standard	1.00	7.026	4801	1.01 bb		1		
7 110599007	TOX145-RBL11049-12ppb-1, RWW- Box 99-211	Standard	1.00	7.020	4858	1.02 bb		2		
8 110599008	TOX145-RBL11049-30ppb-1, RWW- Box 99-211	Standard	1.00	7.003	4445	0.93 bb		-7		
9 110599009	TOX145-RBL11049-60ppb-1, RWW- Box 99-211	Standard	1.00	7.032	5132	1.08 bb		8		
10 110599010	TOX145-RBL11049-120ppb-1, RWW- Box 99-211	Standard	1.00	7.024	5052	1.06 bb		6		
11 110599011	TOX145-RBL11049-300ppb-1, RWW- Box 99-211	Standard	1.00	7.011	4750	1.00 bb		-0		
12 110599012	TOX145-RBL11049-600ppb-1, RWW- Box 99-211	Standard	1.00	7.007	4715	0.99 bb		-1		
13 110599013	TOX145-RBL11049-900ppb-1, RWW- Box 99-211	Standard	1.00	6.983	4022	0.84 bb		-16		
14 110599014	TOX145-RBL11049-1200ppb-1, RWW- Box 99-211	Standard	1.00	7.000	3891	0.82 bb		-18		
15 110599015	MeOH Blank			7.031	10	0.00 bb		-100		
16 110599016	TOX145-RTL, 9R03240-300ppb-MS, 11/4/99- Box 99-211			7.004	2638	0.55 bd		-45		
17 110599017	TOX145-RTL, 9R03240-300ppb-MSD, 11/4/99 Box 99-211			7.012	2387	0.50 bb		-50		
18 110599018	TOX145-RTL, 9R03240/M/Day29/Control, 11/4/99			7.002	2619	0.55 bb		-45		
19 110599019	TOX145-RTL, 9R03241/M/Day29/Control, 11/4/99			7.005	2695	0.56 bb		-44		
20 110599020	TOX145-RTL, 9R03242/M/Day29/Control, 11/4/99			7.005	2498	0.52 bb		-48		
21 110599021	TOX145-RBL11049-300ppb-CC1-1, RWW- Box 99-211	QC	1.00	7.001	2769	0.58 bb		-42		
22 110599022	MeOH Blank									
23 110599023	TOX145-RTL, 9R03243/M/Day29/Control, 11/4/99			7.003	2439	0.51 bb		-49		
24 110599024	TOX145-RTL, 9R03244/M/Day29/Control, 11/4/99			7.007	2179	0.46 bd		-54		
25 110599025	TOX145-RTL, 9R03255/M/Day29/5mg/kg, 11/4/99			7.021	3535	0.74 bb		-26		
26 110599026	TOX145-RTL, 9R03256/M/Day29/5mg/kg, 11/4/99			7.003	4856	1.02 bb		2		
27 110599027	TOX145-RTL, 9R03257/M/Day29/5mg/kg, 11/4/99			6.986	5394	1.13 bb		13		
28 110599028	TOX145-RBL11049-300ppb-CC1-2, RWW- Box 99-211	QC	1.00	7.005	4664	0.98 bb		-2		
29 110599029	MeOH Blank									
30 110599030	TOX145-RTL, 9R03258/M/Day29/5mg/kg, 11/4/99			7.006	3378	0.71 bb		-29		
31 110599031	TOX145-RTL, 9R03259/M/Day29/5mg/kg, 11/4/99			7.006	4967	1.04 bb		4		
32 110599032	TOX145-RBL11049-300ppb-CC1-1, RWW- Box 99-211	QC	1.00	6.986	4412	0.92 bb		-8		
33 110599033	MeOH Blank									
34 110599034	TOX145-RBL11049-WBLK-1, RWW- Box 99-211			7.022	3963	0.83 bb		-17		
35 110599035	TOX145-RBL11049-RBL B1K-1, RWW- Box 99-211			7.009	3436	0.72 bb		-28		
36 110599036	TOX145-RBL11049-6ppb-1, RWW- Box 99-211	Standard	1.00	7.003	5129	1.07 bb		7		
37 110599037	TOX145-RBL11049-12ppb-1, RWW- Box 99-211	Standard	1.00	7.005	5309	1.11 bb		11		
38 110599038	TOX145-RBL11049-30ppb-1, RWW- Box 99-211	Standard	1.00	6.996	4827	1.01 bb		1		
39 110599039	TOX145-RBL11049-60ppb-1, RWW- Box 99-211	Standard	1.00	6.998	5250	1.10 bb		10		
40 110599040	TOX145-RBL11049-120ppb-1, RWW- Box 99-211	Standard	1.00	7.008	5272	1.10 bb		10		
41 110599041	TOX145-RBL11049-300ppb-1, RWW- Box 99-211	Standard	1.00	7.002	4975	1.04 bb		4		
42 110599042	TOX145-RBL11049-600ppb-1, RWW- Box 99-211	Standard	1.00	6.976	5000	1.05 bb		5		
43 110599043	TOX145-RBL11049-900ppb-1, RWW- Box 99-211	Standard	1.00	7.004	4346	0.91 bb		-9		
44 110599044	TOX145-RBL11049-1200ppb-1, RWW- Box 99-211	Standard	1.00	7.004	4149	0.87 bb		-13		

Amelia 062498: Quantitated and reviewed by ias on 11/8/99

000777

Quantify Compound Summary Report
TOX145-rat liver, Day29 dose and control- ext. 11/4/99 RWW

Page 4

Sample List: C:\HAASLYN\Amelia062498.PRO\SampleDB\110599a
Last modified: Mon Nov 08 08:54:18 1999
Method: F:\Amelia backup sep99\Amelia-rats.pro\MethDB\110599a
Last modified: Mon Nov 08 08:20:27 1999
Job Code:

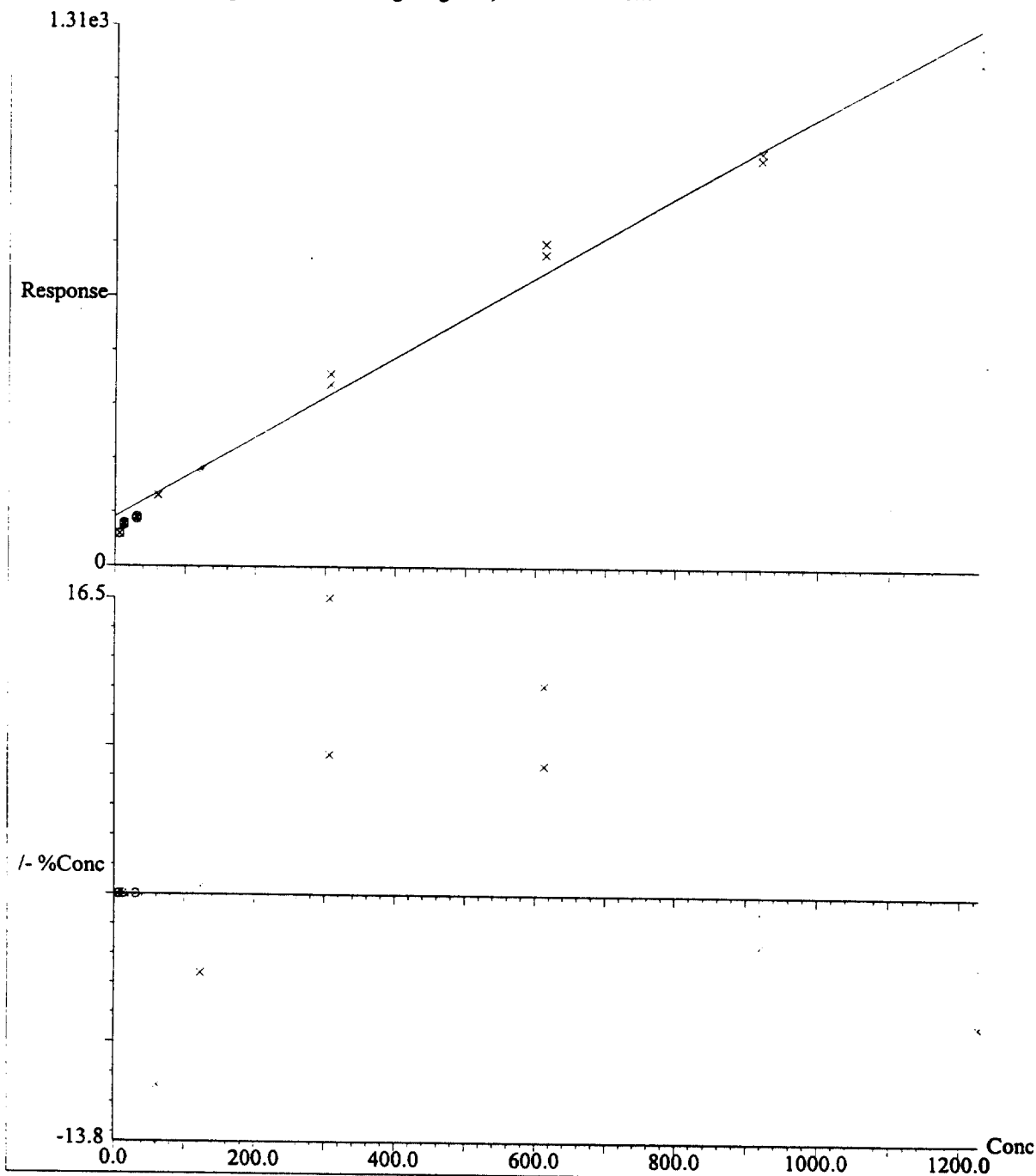
Printed: Mon Nov 08 09:18:10 1999

Compound 4: PFOSA (Ch4)

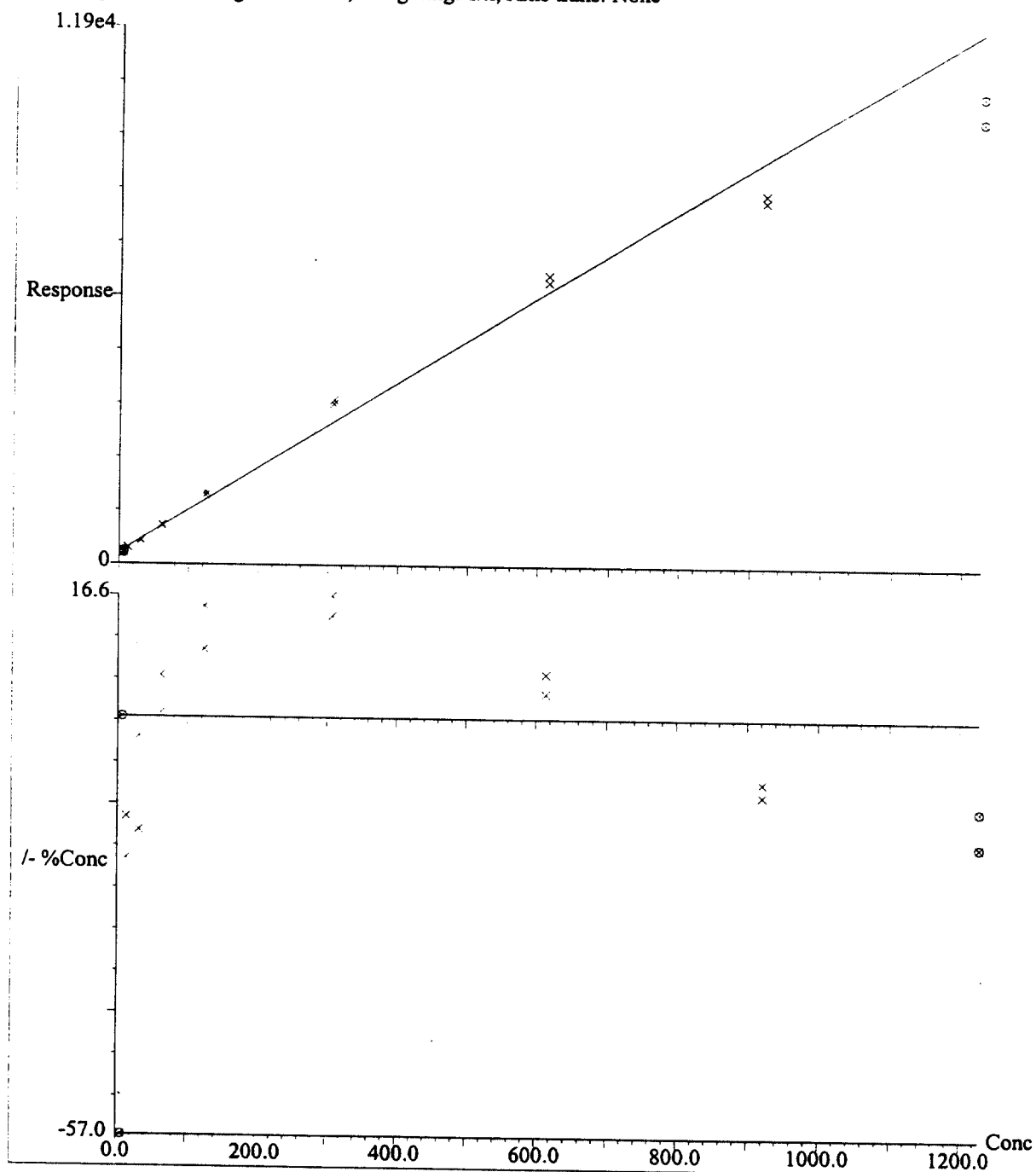
# Name	Samplelist Text	Type	Std Conc	RT	Area	Conc.	Flags	Dev	Mod	Comment
1 110599001	MeOH Blank			7.794	331	11.56	MM			
2 110599002	MeOH Blank			7.776	255	7.16	MM			
3 110599003	MeOH Blank			7.756	65	0.00	bb			
4 110599004	TOX145-RBL11049-WB1k-1, RWW- Box 99-211			7.774	158	1.54	MM			
5 110599005	TOX145-RBL11049-RBL Blk-1, RWW- Box 99-211			7.750	156	1.46	MM			
6 110599006	TOX145-RBL11049-6ppb-1, RWW- Box 99-211	Standard	6.13	7.726	196	3.79	MMX	-38		
7 110599007	TOX145-RBL11049-12ppb-1, RWW- Box 99-211	Standard	12.30	7.742	281	8.67	MMX	-30		
8 110599008	TOX145-RBL11049-30ppb-1, RWW- Box 99-211	Standard	30.70	7.747	571	25.50	MM	-17		
9 110599009	TOX145-RBL11049-60ppb-1, RWW- Box 99-211	Standard	61.30	7.755	1229	63.60	MM	4		
10 110599010	TOX145-RBL11049-120ppb-1, RWW- Box 99-211	Standard	123.00	7.748	2412	132.12	MM	7		
11 110599011	TOX145-RBL11049-300ppb-1, RWW- Box 99-211	Standard	307.00	7.757	6347	360.04	MM	17		
12 110599012	TOX145-RBL11049-600ppb-1, RWW- Box 99-211	Standard	613.00	7.778	12091	692.71	MM	13		
13 110599013	TOX145-RBL11049-900ppb-1, RWW- Box 99-211	Standard	920.00	7.753	15506	890.50	MM	-3		
14 110599014	TOX145-RBL11049-1200ppb-1, RWW- Box 99-211	Standard	1227.00	7.746	20418	1175.01	MM	-4		
15 110599015	MeOH Blank			7.778	47	0.00	MM			
16 110599016	TOX145-RTL, 9R03240-300ppb-MS, 11/4/99- Box 99-211			7.750	6382	362.04	MM			
17 110599017	TOX145-RTL, 9R03240-300ppb-MSD, 11/4/99 Box 99-211			7.758	5982	338.88	MM			
18 110599018	TOX145-RTL, 9R03240/M/Day29/Control, 11/4/99			7.773	176	2.62	MM			
19 110599019	TOX145-RTL, 9R03241/M/Day29/Control, 11/4/99			7.776	162	1.76	MM			
20 110599020	TOX145-RTL, 9R03242/M/Day29/Control, 11/4/99			7.774	144	0.74	MM			
21 110599021	TOX145-RBL11049-300ppb-CC1-1, RWW- Box 99-211	QC	307.00	7.748	6532	370.74	MM	21		
22 110599022	MeOH Blank			7.799	47	0.00	bb			
23 110599023	TOX145-RTL, 9R03243/M/Day29/Control, 11/4/99			7.774	134	0.18	MM			
24 110599024	TOX145-RTL, 9R03244/M/Day29/Control, 11/4/99			7.778	131	0.01	MM			
25 110599025	TOX145-RTL, 9R03255/M/Day29/5mg/kg, 11/4/99			7.768	1639	87.33	bb			
26 110599026	TOX145-RTL, 9R03256/M/Day29/5mg/kg, 11/4/99			7.774	3532	196.95	bb			
27 110599027	TOX145-RTL, 9R03257/M/Day29/5mg/kg, 11/4/99			7.781	1971	106.57	MM			
28 110599028	TOX145-RBL11049-300ppb-CC1-2, RWW- Box 99-211	QC	307.00	7.752	4071	228.17	MM	-26		
29 110599029	MeOH Blank			7.772	55	0.00	MM			
30 110599030	TOX145-RTL, 9R03258/M/Day29/5mg/kg, 11/4/99			7.776	2652	146.00	bb			
31 110599031	TOX145-RTL, 9R03259/M/Day29/5mg/kg, 11/4/99			7.777	5573	315.19	bb			
32 110599032	TOX145-RBL11049-300ppb-CC1-1, RWW- Box 99-211	QC	307.00	7.755	6243	354.01	MM	15		
33 110599033	MeOH Blank			7.781	51	0.00	MM			
34 110599034	TOX145-RBL11049-WB1k-1, RWW- Box 99-211			7.791	138	0.41	MM			
35 110599035	TOX145-RBL11049-RBL Blk-1, RWW- Box 99-211			7.780	141	0.57	MM			
36 110599036	TOX145-RBL11049-6ppb-1, RWW- Box 99-211	Standard	6.13	7.749	181	2.87	MMX	-53		
37 110599037	TOX145-RBL11049-12ppb-1, RWW- Box 99-211	Standard	12.30	7.752	293	9.37	MMX	-24		
38 110599038	TOX145-RBL11049-30ppb-1, RWW- Box 99-211	Standard	30.70	7.743	559	24.78	MM	-19		
39 110599039	TOX145-RBL11049-60ppb-1, RWW- Box 99-211	Standard	61.30	7.770	1172	60.28	MM	-2		
40 110599040	TOX145-RBL11049-120ppb-1, RWW- Box 99-211	Standard	123.00	7.754	2299	125.54	MM	2		
41 110599041	TOX145-RBL11049-300ppb-1, RWW- Box 99-211	Standard	307.00	7.772	5742	324.98	MM	6		
42 110599042	TOX145-RBL11049-600ppb-1, RWW- Box 99-211	Standard	613.00	7.747	11584	663.36	MM	8		
43 110599043	TOX145-RBL11049-900ppb-1, RWW- Box 99-211	Standard	920.00	7.751	14521	833.45	MM	-9		
44 110599044	TOX145-RBL11049-1200ppb-1, RWW- Box 99-211	Standard	1227.00	7.750	20714	1192.13	MM	-3		

*Data Reported
Dec
11/8/99*

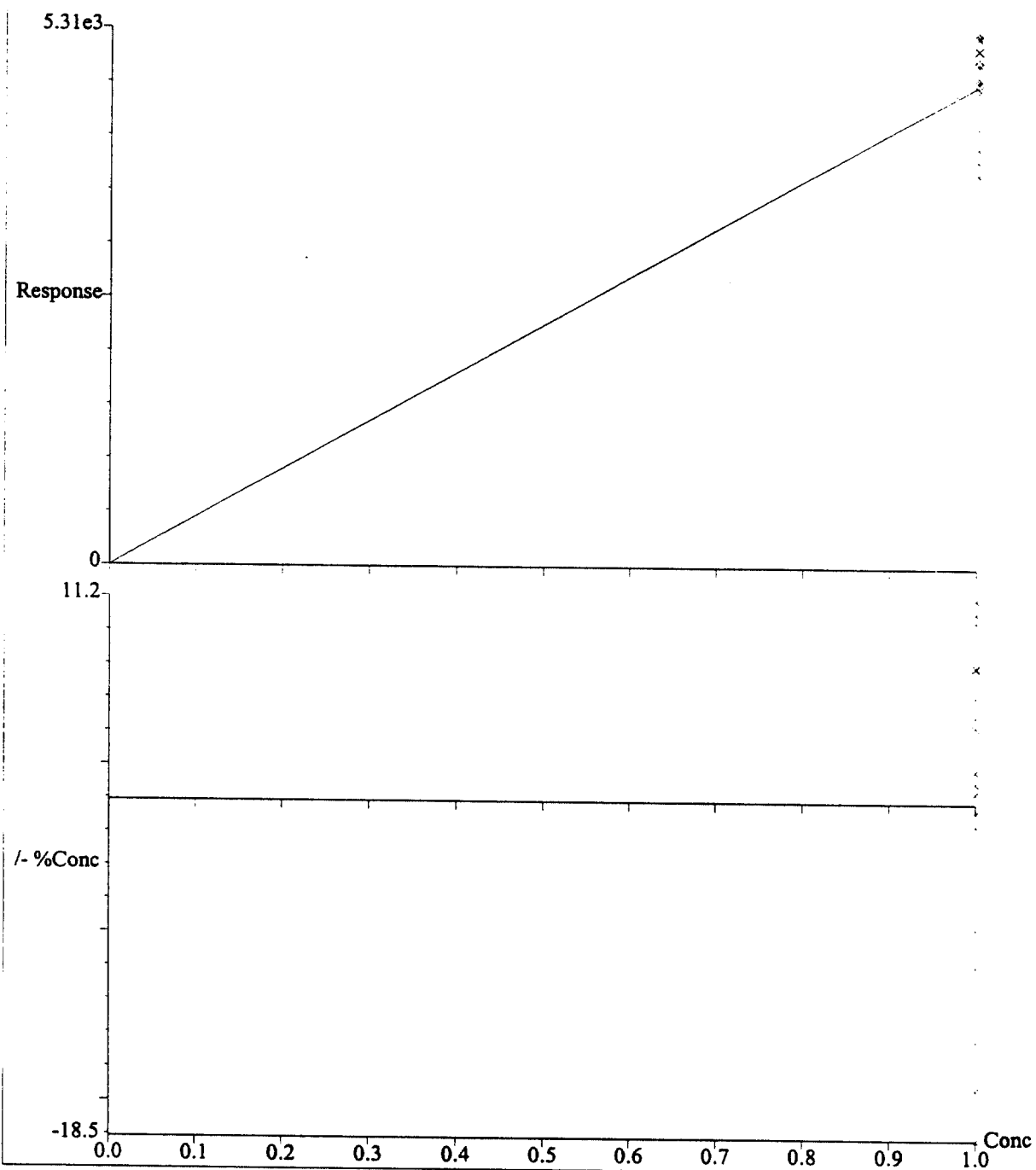
Compound 1 name: POAA (Ch1)
Coefficient of Determination: 0.989198
Calibration curve: $0.973462 * x + 119.913$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound 2 name: PFOS (Ch2)
Coefficient of Determination: 0.982749
Calibration curve: $9.51640 * x + 260.801$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



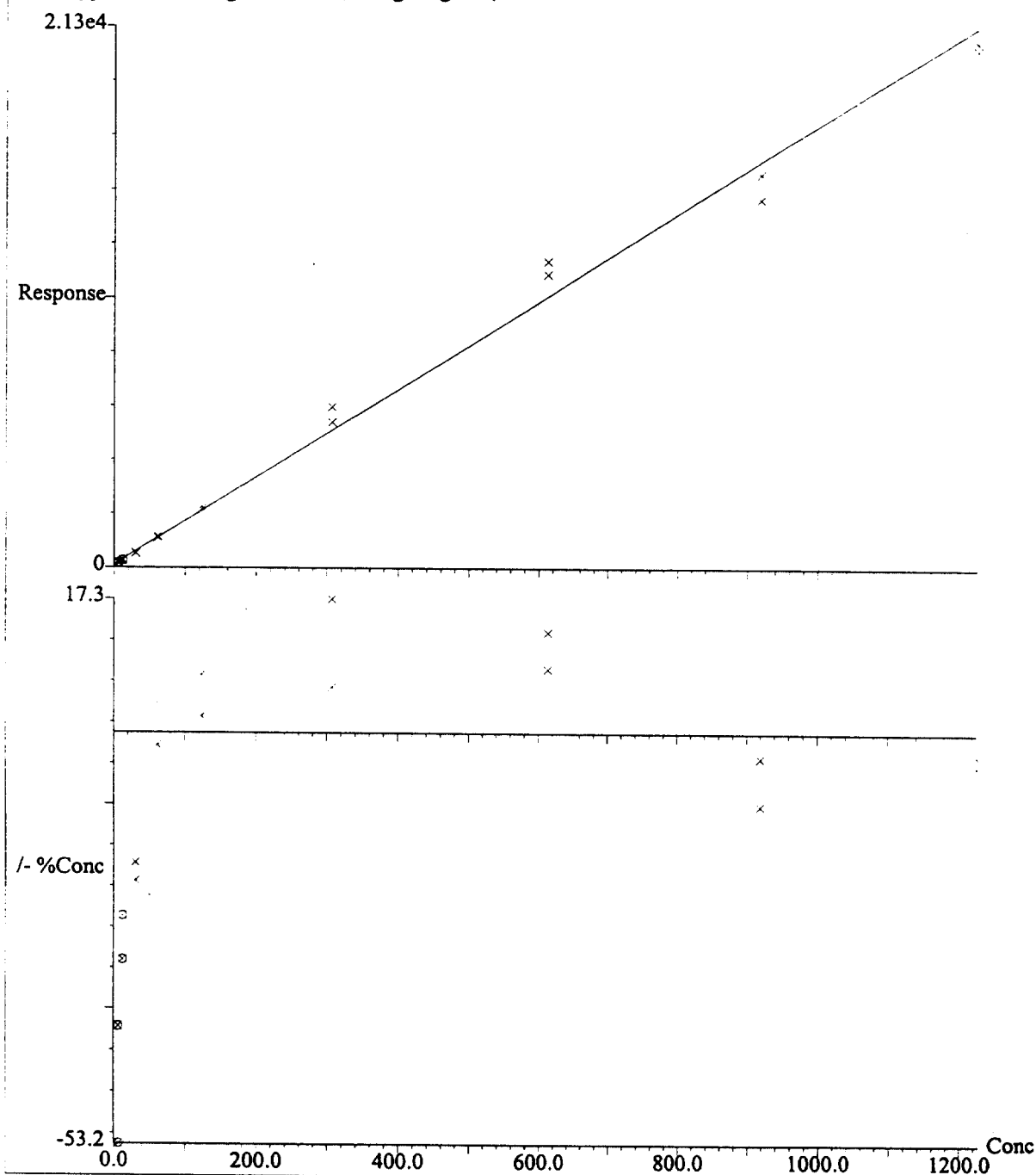
Compound 3 name: Surr (Ch3)
Response Factor: 4773.57
Response type: External Std, Area
Curve type: RF



Amelia 062498: Quantitated and reviewed by ias on 11/8/99

000781

Compound 4 name: PFOSA (Ch4)
Coefficient of Determination: 0.989845
Calibration curve: $17.2655 * x + 131.101$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Amelia 062498: Quantitated and reviewed by ias on 11/8/99

000782

Feb 11/02/99 p 1 of 4

Method Report

Page 1

Method file: F:\amelia backup sep99\amelia-rats.pro\MethDB\110599a.mdb
Last modified: Mon Nov 08 08:20:27 1999
Compound: 1: POAA (Ch1)
Printed: Mon Nov 08 08:56:38 1999

Compound Name: POAA (Ch1)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. A
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da
Peak Location

Retention Time (mins): 6.976, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.274

Peak Selection: Nearest
Response

Response Type: External
Response: Areas
Calibration Curves

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None
Concentration

Units:
Noise

Peak-to-peak amplitude: 530
Smooth

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Baselines

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Peak Separation

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00
Response Threshold

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000783

Method Report

Page 2

Method file: F:\amelia backup sep99\amelia-rats.pro\MethDB\110599a.mdb
Last modified: Mon Nov 08 08:20:27 1999C
Compound: 2: PFOS (Ch2)
Printed: Mon Nov 08 08:56:38 1999

Compound Name: PFOS (Ch2)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. B
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da
Peak Location

Retention Time (mins): 7.149, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.299

Peak Selection: Nearest
Response

Response Type: External
Response: Areas
Calibration Curves

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None
Concentration

Units:
Noise

Peak-to-peak amplitude: 56
Smooth

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:
Baselines

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00
Peak Separation

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00
Response Threshold

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000784

Method Report

Page 3

Method file: F:\amelia backup sep99\amelia-rats.pro\MethDB\110599a.mdb
Last modified: Mon Nov 08 08:20:27 1999C
Compound: J: Surr (Ch3)
Printed: Mon Nov 08 08:56:38 1999

Compound Name: Surr (Ch3)
Internal Ref.: (None)
Quantify Trace: TIC
Conc. of Standards: Fixed
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da
Peak Location

Retention Time (mins): 7.023, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: (None)
Time Window (mins): 0.310

Peak Selection: Nearest
Response
Response Type: External
Response: Areas
Calibration Curves

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None
Concentration

Units:
Noise

Peak-to-peak amplitude: 80
Smooth

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:
Baselines

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00
Peak Separation

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00
Response Threshold

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000785

Method Report

Page 4

Method file: F:\amelia backup sep99\amelia-rats.pro\MethDB\110599a.mdb
Last modified: Mon Nov 08 08:20:27 1999

Compound: 4: PFOSA (Ch4)

Printed: Mon Nov 08 08:56:38 1999

Compound Name: PFOSA (Ch4)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. C

User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da
Peak Location

Retention Time (mins): 7.770, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.286

Peak Selection: Nearest

Response

Response Type: External

Response: Areas

Calibration Curves

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Concentration

Units:

Noise

Peak-to-peak amplitude: 82

Smooth

Smoothing: Enabled

Window size (scans): 2

Number of smooths: 1

Smoothing method:

Baselines

Join valley threshold: 30.00

Reduce peak tailing: 200.00

Raise baseline maximum: 5.00

Peak Separation

Draw vertical threshold: 90.00

Detect shoulders: Disabled

Shoulder threshold: 30.00

Response Threshold

Relative height: 1.50, Disabled

Absolute height: 0.00, Disabled

Relative area: 2.00, Disabled

Absolute area: 0.00, Disabled

000786

JAS 11/5/99

AMELIA 062498: FACT Equipment Log 99114
Quattro II, #6448E

PAGE _____

DATE: 11/05/99

ANALYST: ias

STUDY NUMBER: TOX145 MATRIX: rat liver DESCRIPTION: Day 29 control and dose

FACT Method: ETS-8-7.0

Injection volume: 10 uL Cycle time: 10 min Electrode: Z-Spray

Mobile Phase: (A) 2.0 mM NH₄CH₃CO₂ (B) MeOH

TN-A- or stdrd number (A) 99085-11-6 (B) TN-A-2997

Prepared (date/by): (A) 10/30/99 sah (B) 10/29/99 mee

Expires: (A) 04/00 (B) 04/00

Store at room temp? ☒ Y or N Other: _____

Guard column: None

Part number: na Lot number: na

Analytical column: Keystone Betasil

Size: 2 x 50 mm Type: C18 Sphere size: 5 um

Part number: 055-701-2 Lot number: P9E11 Serial number: 089815 S

Analytical parameters:

Drying gas (L/hr): 300 ESI nebulizing gas (L/hr) 10 Sheath gas (L/hr) 0

LC Flow rate: 300 uL/min Back pressure: 62 bar

HPLC solvent gradient: 10min-10-95

Time (min.)	%B
1) <u>0.00</u>	<u>10.0</u>
2) <u>1.00</u>	<u>10.0</u>
3) <u>5.50</u>	<u>95.0</u>
4) <u>7.50</u>	<u>95.0</u>
5) <u>8.00</u>	<u>10.0</u>

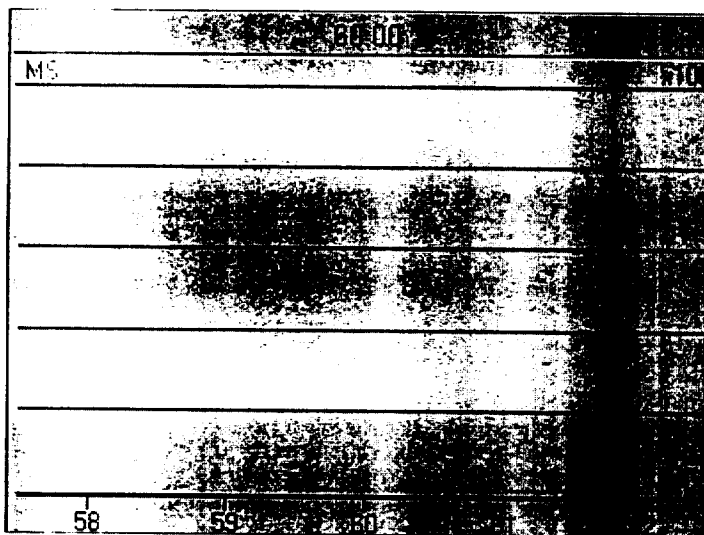
000787

Tuning Method Report

Page 1

Method: C:\MASSLYNX\AMELIA062498.PRO\ACQUDB\PFOS1-6KV

Printed: Fri Nov 05 09:43:09 1999



MS

SOURCE (ESP-)	Set	Rdbk	MS1	Set	Rdbk
Capillary	1.60	1.60	LM Resolution	15.0	
Cone	40	-40	HM Resolution	15.0	
Extractor	5	-3	Ion Energy	2.0	
RF Lens	0.2		Ion Energy Ramp	0.0	
Source Block Temp.	150	151	Lens 6	4	11
Desolvation Temp.	250	253	Multiplier	650	

Pressures	Rdbk	MS2	Set	Rdbk
Analyser Vacuum	1.2e-4	LM Resolution	14.0	
		HM Resolution	14.0	
Gas Cell	4.8e-3	Collision	10	12
		Ion Energy	2.0	
		Ion Energy Ramp	0.0	
		Lens 8	40	40
		Lens 9	0	10
		Multiplier	650	

000788

Scanning Method Report

Page 1

Method: C:\MASSLYNK\AMELIA\062498.PRO\ACQUDE\10MIN-PT06-POAA-18-PT06A

Last Modified: Mon Oct 11 07:22:17 1999

Printed: Fri Nov 05 09:26:14 1999

Solvent Delay (mins) : 0.00

Function : 1 MRM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	413.00	169.00	0.30	30	15

Function : 2 MRM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	499.00	99.00	0.30	45	60

Function : 3 MRM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	400.00	80.00	0.30	40	60

Function : 4 MRM of 1 Mass Pair (ESP-)

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)
1	435.00	78.00	0.40	45	60

000789

Method Report

Page 1

Method File: C:\MASSLYNX\AMELIA062498.PRO\ACQUDE\10min-10-95
Last Modified: Friday, November 05, 1999 06:22:24
Printed: Friday, November 05, 1999 09:26:28

HP1100 LC Pump Initial Conditions**Solvents**

A%	90.0
B%	10.0
C%	0.0
D%	0.0

Flow (ml/min)	0.300
Stop Time (mins)	10.0
Min Pressure (bar)	0
Max Pressure (bar)	400
Oven Temperature Left(°C)	30.0
Oven Temperature Right(°C)	30.0

HP1100 LC Pump Gradient Timetable

The gradient Timetable contains 5 entries which are :

Time	A%	B%	C%	D%	Flow	Pressure
0.00	90.0	10.0	0.0	0.0	0.300	400
1.00	90.0	10.0	0.0	0.0	0.300	400
5.50	5.0	95.0	0.0	0.0	0.300	400
7.50	5.0	95.0	0.0	0.0	0.300	400
8.00	90.0	10.0	0.0	0.0	0.300	400

HP1100 LC Pump External Event Timetable

The Timetable contains 3 entries which are :

Time	Column Switch	Contact1	Contact2	Contact3	Contact4
Initial	Off	Off	Off	Off	Off
0.00	Off	On	On	Off	On
0.10	Off	Off	Off	Off	Off

HP1100 Autosampler Initial Conditions

Draw Speed	200.0
Eject Speed (µl/min)	200
Draw Position (mm)	0.00
Stop Time (mins)	9.00
Injection Volume(µl)	10.0
Vial Number	32

000790

Masslynx - Sample List

Page 1

Sample List: C:\MASSLYNX\amelia062498.PRO\SampleDB\110599a.SPL
Printed: Fri Nov 05 09:25:34 1999

Page Position :)

	File Name	File Text	MS File
1	110599001	MeOH Blank	10MIN-PFOS-POAA-IS-PFOSA
2	110599002	MeOH Blank	10MIN-PFOS-POAA-IS-PFOSA
3	110599003	MeOH Blank	10MIN-PFOS-POAA-IS-PFOSA
4	110599004	TOX145-RBL11049-WBik-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
5	110599005	TOX145-RBL11049-RBL Bk-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
6	110599006	TOX145-RBL11049-6ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
7	110599007	TOX145-RBL11049-12ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
8	110599008	TOX145-RBL11049-30ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
9	110599009	TOX145-RBL11049-60ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
10	110599010	TOX145-RBL11049-120ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
11	110599011	TOX145-RBL11049-300ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
12	110599012	TOX145-RBL11049-600ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
13	110599013	TOX145-RBL11049-900ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
14	110599014	TOX145-RBL11049-1200ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
15	110599015	MeOH Blank	10MIN-PFOS-POAA-IS-PFOSA
16	110599016	TOX145-RBL, 9R03240-300ppb-MS, 11/4/99- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
17	110599017	TOX145-RBL, 9R03240-300ppb-MSD, 11/4/99 Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
18	110599018	TOX145-RBL, 9R03240/M/Day29/Control, 11/4/99	10MIN-PFOS-POAA-IS-PFOSA
19	110599019	TOX145-RBL, 9R03241/M/Day29/Control, 11/4/99	10MIN-PFOS-POAA-IS-PFOSA
20	110599020	TOX145-RBL, 9R03242/M/Day29/Control, 11/4/99	10MIN-PFOS-POAA-IS-PFOSA
21	110599021	TOX145-RBL11049-300ppb-CC1-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
22	110599022	MeOH Blank	10MIN-PFOS-POAA-IS-PFOSA
23	110599023	TOX145-RBL, 9R03243/M/Day29/Control, 11/4/99	10MIN-PFOS-POAA-IS-PFOSA
24	110599024	TOX145-RBL, 9R03244/M/Day29/Control, 11/4/99	10MIN-PFOS-POAA-IS-PFOSA
25	110599025	TOX145-RBL, 9R03255/M/Day29/5mg/kg, 11/4/99	10MIN-PFOS-POAA-IS-PFOSA
26	110599026	TOX145-RBL, 9R03256/M/Day29/5mg/kg, 11/4/99	10MIN-PFOS-POAA-IS-PFOSA
27	110599027	TOX145-RBL, 9R03257/M/Day29/5mg/kg, 11/4/99	10MIN-PFOS-POAA-IS-PFOSA
28	110599028	TOX145-RBL11049-300ppb-CC1-2, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
29	110599029	MeOH Blank	10MIN-PFOS-POAA-IS-PFOSA
30	110599030	TOX145-RBL, 9R03258/M/Day29/5mg/kg, 11/4/99	10MIN-PFOS-POAA-IS-PFOSA
31	110599031	TOX145-RBL, 9R03259/M/Day29/5mg/kg, 11/4/99	10MIN-PFOS-POAA-IS-PFOSA
32	110599032	TOX145-RBL11049-300ppb-CC1-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
33	110599033	MeOH Blank	10MIN-PFOS-POAA-IS-PFOSA
34	110599034	TOX145-RBL11049-WBik-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
35	110599035	TOX145-RBL11049-RBL Bk-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
36	110599036	TOX145-RBL11049-6ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
37	110599037	TOX145-RBL11049-12ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
38	110599038	TOX145-RBL11049-30ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
39	110599039	TOX145-RBL11049-60ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
40	110599040	TOX145-RBL11049-120ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
41	110599041	TOX145-RBL11049-300ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
42	110599042	TOX145-RBL11049-600ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
43	110599043	TOX145-RBL11049-900ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA
44	110599044	TOX145-RBL11049-1200ppb-1, RWW- Box 99-211	10MIN-PFOS-POAA-IS-PFOSA

000791

Masslynx - Sample List

Page 2

Sample List: C:\MASSLYNX\amelia062498.PRO\SampleDB\110599a.SPL
Printed: Fri Nov 05 09:25:34 1999

Page Position: (2, 1)

	Inlet File	Bottle	Inject Volume	Sample Type	Conc A	Conc B	Conc C	Conc D	Conc E	Conc F
1	10min-10-95	91	10	---	---	---	---	---	---	---
2	10min-10-95	91	10	---	---	---	---	---	---	---
3	10min-10-95	91	10	---	---	---	---	---	---	---
4	10min-10-95	1	10	---	---	---	---	---	---	---
5	10min-10-95	2	10	---	---	---	---	---	---	---
6	10min-10-95	3	10	---	---	---	---	---	---	---
7	10min-10-95	4	10	---	---	---	---	---	---	---
8	10min-10-95	5	10	---	---	---	---	---	---	---
9	10min-10-95	6	10	---	---	---	---	---	---	---
10	10min-10-95	7	10	---	---	---	---	---	---	---
11	10min-10-95	8	10	---	---	---	---	---	---	---
12	10min-10-95	9	10	---	---	---	---	---	---	---
13	10min-10-95	10	10	---	---	---	---	---	---	---
14	10min-10-95	11	10	---	---	---	---	---	---	---
15	10min-10-95	92	10	---	---	---	---	---	---	---
16	10min-10-95	12	10	---	---	---	---	---	---	---
17	10min-10-95	13	10	---	---	---	---	---	---	---
18	10min-10-95	14	10	---	---	---	---	---	---	---
19	10min-10-95	15	10	---	---	---	---	---	---	---
20	10min-10-95	16	10	---	---	---	---	---	---	---
21	10min-10-95	24	10	---	---	---	---	---	---	---
22	10min-10-95	92	10	---	---	---	---	---	---	---
23	10min-10-95	17	10	---	---	---	---	---	---	---
24	10min-10-95	18	10	---	---	---	---	---	---	---
25	10min-10-95	19	10	---	---	---	---	---	---	---
26	10min-10-95	20	10	---	---	---	---	---	---	---
27	10min-10-95	21	10	---	---	---	---	---	---	---
28	10min-10-95	25	10	---	---	---	---	---	---	---
29	10min-10-95	93	10	---	---	---	---	---	---	---
30	10min-10-95	22	10	---	---	---	---	---	---	---
31	10min-10-95	23	10	---	---	---	---	---	---	---
32	10min-10-95	24	10	---	---	---	---	---	---	---
33	10min-10-95	93	10	---	---	---	---	---	---	---
34	10min-10-95	1	10	---	---	---	---	---	---	---
35	10min-10-95	2	10	---	---	---	---	---	---	---
36	10min-10-95	3	10	---	---	---	---	---	---	---
37	10min-10-95	4	10	---	---	---	---	---	---	---
38	10min-10-95	5	10	---	---	---	---	---	---	---
39	10min-10-95	6	10	---	---	---	---	---	---	---
40	10min-10-95	7	10	---	---	---	---	---	---	---
41	10min-10-95	8	10	---	---	---	---	---	---	---
42	10min-10-95	9	10	---	---	---	---	---	---	---
43	10min-10-95	10	10	---	---	---	---	---	---	---
44	10min-10-95	11	10	---	---	---	---	---	---	---

000792

FACT: DATA REVIEW SUMMARY FORM

Date: 11/11/99
 Data Analyst: JAS
 FACT Study #: Tox 145
 Matrix/Timepoints: Pest Livir, Day 29 dose dilutions - ext 11/1/99
 Sample List: 110994
 Set-up Analysis Date/Analyst: JAS 11/10/99
 Instrument: Soup 020199

Data Review Sign-Off

Analyst Review
 Name / Date: JAS 11/11/99

Technical Review
 Name / Date: MEE 11/11/99 / kin Nov-15-99

Quality Assurance Review
 Name / Date: _____

Data Entry: LAC 11/15/99

Comments / Justifications: _____

Analyte: POAA (Func 1) ①
 Acceptable Linear Range: 123 ppb → 1227 ppb
 LOQ: 123 ppb Extraction Blank: less than LOQ
 R²: 0.980656 Matrix Blank: less than LOQ
 Calibration Checks: OK → -11%
 MS/MSD: Data not used - Not a target
 Data Status: analyte LAC 11/15/99

Analyte: PFOS (Func 2) ②
 Acceptable Linear Range: 12.30 ppb → 1228 ppb
 LOQ: 12.30 ppb Extracted Blank: less than LOQ
 R²: 0.991927 Matrix Blank: greater than LOQ
 Calibration Checks: OK → -17%
 MS/MSD: Not analyzed with these samples
 Data Status: OK Entered LAC 11/15/99

Analyte: PFOSA (Func 3)
 Acceptable Linear Range: 6.13 ppb → 1227 ppb
 LOQ: 6.13 ppb Extracted Blank: less than LOQ
 R²: 0.989396 Matrix Blank: less than LOQ
 Calibration Checks: OK → -13%
 MS/MSD: Data not used, entered from analysis
 Data Status: on 11/05/99 LAC 11/15/99

① Low level points, (6.13 ppb, 12.30 ppb, 30.70 ppb, & 61.30 ppb), were not 2x higher than the extraction blank; these points were excluded from the curve to disqualify a data range that may have been significantly affected by background levels of the analyte - JAS 11/11/99
 ② Low curve point, 6.14 ppb, was excluded as it was not within the ±30% accuracy requirements of the method when the curves were evaluated over a linear range appropriate to the data - JAS 11/11/99.

Comment to ①: POAA was not a target analyte in these diluted samples. kin Nov-15-99

LAC 11/15/99

JAS 11/11/99 p. 1 of 2

000793

FACT: DATA REVIEW SUMMARY FORM

Date: 11/11/99
 Data Analyst: IAS
 FACT Study #: TOX145
 Matrix/Timepoints: Ret Linic, Day 29 dose dilutions - ext 11/4/99
 Sample List: 111099a
 Set-up Analysis
 Date/Analyst: IAS 11/10/99
 Instrument: Samp 020199

Data Review Sign-Off

Analyst Review
 Name / Date: IAS 11/11/99

Technical Review
 Name / Date: Refer to page 1 of 2 UC 11/15/99

Quality Assurance Review
 Name / Date: _____

Data Entry: UC 11/15/99

Comments / Justifications: _____

Analyte: Surr (Furc 4) ③
 Acceptable Linear Range: NA
 LOQ: NA Extraction Blank: NA
 R²: NA Matrix Blank: NA
 Calibration Checks: NA
 MS/MSD: Not a target analyte - Data not used.
 Data Status: ↓ UC 11/15/99

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: IAS 11/11/99
 Data Status: _____

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: _____

③ Internal std levels were low in the dilutions due to the dilution factor from the spiked amount. - IAS 11/11/99

UC 11/15/99

IAS 11/11/99 p 2 of 2

000794

Quantify Compound Summary Report
TOX145-rat liver, day 29 dose dilutions- ext. 11/4/99 RW

Sample List: C:\MASSLYNX\scup020199.pro\SampleDB\111099a
Last modified: Thu Nov 11 07:09:30 1999
Method: C:\MASSLYNX\scup020199.pro\MethDB\111099a
Last modified: Thu Nov 11 07:08:22 1999
Job Code:

Printed: Thu Nov 11 07:26:35 1999

Compound 1: POAA (Func 1)

# Name	SampleList Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1 111099001	MeOH Blank							
2 111099002	MeOH Blank							
3 111099003	MeOH Blank							
4 111099004	TOX145-RBL11049-WBLk-2, RW			7.125	95	bb	0.00	
5 111099005	TOX145-RBL11049-RBLBk-2, RW			7.106	107	MM	0.00	
6 111099006	TOX145-RBL11049-6ppb-2, RW	Standard	6.13	7.110	116	MMX	6.46	5
7 111099007	TOX145-RBL11049-12ppb-2, RW	Standard	12.30	7.093	104	bbX	0.00	-100
8 111099008	TOX145-RBL11049-30ppb-2, RW	Standard	30.70	7.083	137	bbX	27.07	-12
9 111099009	TOX145-RBL11049-60ppb-2, RW	Standard	61.30	7.067	185	MMX	75.45	23
10 111099010	TOX145-RBL11049-120ppb-2, RW	Standard	123.00	7.069	214	bb	104.15	-15
11 111099011	TOX145-RBL11049-300ppb-2, RW	Standard	307.00	7.061	459	MM	350.86	14
12 111099012	TOX145-RBL11049-600ppb-2, RW	Standard	613.00	7.067	758	MM	651.92	6
13 111099013	TOX145-RBL11049-900ppb-2, RW	Standard	920.00	7.055	1097	MM	993.50	8
14 111099014	TOX145-RBL11049-1200ppb-2, RW	Standard	1227.00	7.067	1247	bb	1144.17	-7
15 111099015	MeOH Blank							
16 111099016	TOX145-RTL, 9R03255/M/Day29/5mg/kg-D100, 11/4/99							
17 111099017	TOX145-RTL, 9R03256/M/Day29/5mg/kg-D100, 11/4/99							
18 111099018	TOX145-RTL, 9R03257/M/Day29/5mg/kg-D100, 11/4/99							
19 111099019	TOX145-RTL, 9R03258/M/Day29/5mg/kg-D100, 11/4/99							
20 111099020	TOX145-RTL, 9R03259/M/Day29/5mg/kg-D100, 11/4/99							
21 111099021	TOX145-RBL11049-300ppb-CC1-2, RW	QC	307.00	7.089	382	bb	274.09	-11
22 111099022	MeOH Blank							
23 111099023	TOX145-RBL11049-WBLk-2, RW			7.106	99	bb	0.00	
24 111099024	TOX145-RBL11049-RBLBk-2, RW			7.105	91	MM	0.00	
25 111099025	TOX145-RBL11049-6ppb-2, RW	Standard	6.13	7.104	102	bbX	0.00	-100
26 111099026	TOX145-RBL11049-12ppb-2, RW	Standard	12.30	7.088	105	MMX	0.00	-100
27 111099027	TOX145-RBL11049-30ppb-2, RW	Standard	30.70	7.082	124	bbX	13.65	-56
28 111099028	TOX145-RBL11049-60ppb-2, RW	Standard	61.30	7.068	159	bbX	49.51	-19
29 111099029	TOX145-RBL11049-120ppb-2, RW	Standard	123.00	7.056	221	bb	111.55	-9
30 111099030	TOX145-RBL11049-300ppb-2, RW	Standard	307.00	7.064	444	bb	336.20	10
31 111099031	TOX145-RBL11049-600ppb-2, RW	Standard	613.00	7.066	693	bb	586.96	-4
32 111099032	TOX145-RBL11049-900ppb-2, RW	Standard	920.00	7.044	1071	bb	966.64	5
33 111099033	TOX145-RBL11049-1200ppb-2, RW	Standard	1227.00	7.064	1237	bb	1134.04	-8

Not a target analyte, data not used.

UC 11/15/99

Quantify Compound Summary Report
TOX145-rat liver, day 29 dose dilutions- ext. 11/4/99 RWW

Page 2

Sample List: C:\NASSELYNK\soup020199.pro\SampleDB\111099a
Last modified: Thu Nov 11 07:09:30 1999
Method: C:\NASSELYNK\soup020199.pro\MethDB\111099a
Last modified: Thu Nov 11 07:08:22 1999
Job Code:

Printed: Thu Nov 11 07:26:35 1999

Compound 2: PFOS (Punc 2)

# Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1 111099001	MeOH Blank			7.287	47	bb	0.00	
2 111099002	MeOH Blank			7.294	16	bb	0.00	
3 111099003	MeOH Blank			7.269	18	bb	0.00	
4 111099004	TOX145-RBL11049-RBL1k-2, RWW			7.263	115	bb	0.00	
5 111099005	TOX145-RBL11049-RBL1k-2, RWW			7.268	215	bb	1.86	
6 111099006	TOX145-RBL11049-6ppb-2, RWW	Standard	6.14	7.269	271	bbX	6.86	12
7 111099007	TOX145-RBL11049-12ppb-2, RWW	Standard	12.30	7.253	333	bb	12.47	1
8 111099008	TOX145-RBL11049-30ppb-2, RWW	Standard	30.70	7.244	550	bb	32.00	4
9 111099009	TOX145-RBL11049-60ppb-2, RWW	Standard	61.40	7.250	918	bb	65.16	6
10 111099010	TOX145-RBL11049-120ppb-2, RWW	Standard	123.00	7.230	1502	bb	117.79	-4
11 111099011	TOX145-RBL11049-300ppb-2, RWW	Standard	307.00	7.245	3576	bb	304.63	-1
12 111099012	TOX145-RBL11049-600ppb-2, RWW	Standard	614.00	7.251	7216	bb	632.53	3
13 111099013	TOX145-RBL11049-900ppb-2, RWW	Standard	921.00	7.238	11229	bb	994.02	8
14 111099014	TOX145-RBL11049-1200ppb-2, RWW	Standard	1228.00	7.229	13298	bb	1180.39	-4
15 111099015	MeOH Blank			7.264	27	bb	0.00	
16 111099016	TOX145-RTL, 9R03255/M/Day29/5mg/kg-D100, 11/4/99			7.254	2496	bb	207.31	
17 111099017	TOX145-RTL, 9R03256/M/Day29/5mg/kg-D100, 11/4/99			7.248	2774	bb	232.38	
18 111099018	TOX145-RTL, 9R03257/M/Day29/5mg/kg-D100, 11/4/99			7.241	2695	bb	225.25	
19 111099019	TOX145-RTL, 9R03258/M/Day29/5mg/kg-D100, 11/4/99			7.263	3176	bb	268.60	
20 111099020	TOX145-RTL, 9R03259/M/Day29/5mg/kg-D100, 11/4/99			7.267	3648	bb	311.12	
21 111099021	TOX145-RBL11049-300ppb-CC1-2, RWW	QC	307.00	7.250	3029	bb	255.32	-17
22 111099022	MeOH Blank			7.268	19	bb	0.00	
23 111099023	TOX145-RBL11049-RBL1k-2, RWW			7.268	109	bb	0.00	
24 111099024	TOX145-RBL11049-RBL1k-2, RWW			7.245	185	bb	0.00	
25 111099025	TOX145-RBL11049-6ppb-2, RWW	Standard	6.14	7.243	236	bbX	3.69	-40
26 111099026	TOX145-RBL11049-12ppb-2, RWW	Standard	12.30	7.249	335	bb	12.63	3
27 111099027	TOX145-RBL11049-30ppb-2, RWW	Standard	30.70	7.244	549	bb	31.93	4
28 111099028	TOX145-RBL11049-60ppb-2, RWW	Standard	61.40	7.251	833	bb	57.48	-6
29 111099029	TOX145-RBL11049-120ppb-2, RWW	Standard	123.00	7.240	1373	bb	106.17	-14
30 111099030	TOX145-RBL11049-300ppb-2, RWW	Standard	307.00	7.247	3580	bb	304.94	-1
31 111099031	TOX145-RBL11049-600ppb-2, RWW	Standard	614.00	7.228	6972	bb	610.55	-1
32 111099032	TOX145-RBL11049-900ppb-2, RWW	Standard	921.00	7.229	11295	bb	999.95	9
33 111099033	TOX145-RBL11049-1200ppb-2, RWW	Standard	1228.00	7.224	12763	bb	1132.17	-8

Entered UC
11/15/99

Entered UC
11/15/99

Quantify Compound Summary Report
TOX145-rat liver, day 29 dose dilutions- ext. 11/4/99 RWW

Page 3

Sample List: C:\MSL\SLYKX\scup020199.pro\SampleDB\111099a
Last modified: Thu Nov 11 07:09:30 1999
Method: C:\MSL\SLYKX\scup020199.pro\MethodB\111099a
Last modified: Thu Nov 11 07:08:22 1999
Job Code:

Printed: Thu Nov 11 07:26:35 1999

Compound 3: PFOSA (Func 3)

# Name	SampleList Text	Type	Std Conc	RT	Area	Flags	Conc. %Dev
1 111099001	MeOH Blank			7.848	276	NM	5.63
2 111099002	MeOH Blank			7.857	107	NM	0.29
3 111099003	MeOH Blank			7.830	55	NM	0.00
4 111099004	TOX145-RBL11049-WBLk-2, RWW			7.848	94	NM	0.00
5 111099005	TOX145-RBL11049-RBLBlk-2, RWW			7.829	122	NM	0.76
6 111099006	TOX145-RBL11049-6ppb-2, RWW	Standard	6.13	7.833	284	NM	5.85 -5
7 111099007	TOX145-RBL11049-12ppb-2, RWW	Standard	12.30	7.837	492	NM	12.42 1
8 111099008	TOX145-RBL11049-30ppb-2, RWW	Standard	30.70	7.825	1134	NM	32.63 6
9 111099009	TOX145-RBL11049-60ppb-2, RWW	Standard	61.30	7.811	2097	NM	62.98 3
10 111099010	TOX145-RBL11049-120ppb-2, RWW	Standard	123.00	7.812	3852	NM	118.26 -4
11 111099011	TOX145-RBL11049-300ppb-2, RWW	Standard	307.00	7.825	10288	NM	321.02 5
12 111099012	TOX145-RBL11049-600ppb-2, RWW	Standard	613.00	7.813	20144	NM	631.56 3
13 111099013	TOX145-RBL11049-900ppb-2, RWW	Standard	920.00	7.823	31969	NM	1004.08 9
14 111099014	TOX145-RBL11049-1200ppb-2, RWW	Standard	1227.00	7.835	35471	NM	1114.40 -9
15 111099015	MeOH Blank			7.848	44	NM	0.00
16 111099016	TOX145-RTL, 9R03255/W/Day29/5mg/kg-D100, 11/4/99			7.837	48	NM	0.00
17 111099017	TOX145-RTL, 9R03256/W/Day29/5mg/kg-D100, 11/4/99			7.854	73	NM	0.00
18 111099018	TOX145-RTL, 9R03257/W/Day29/5mg/kg-D100, 11/4/99			7.845	46	NM	0.00
19 111099019	TOX145-RTL, 9R03258/W/Day29/5mg/kg-D100, 11/4/99			7.847	45	NM	0.00
20 111099020	TOX145-RTL, 9R03259/W/Day29/5mg/kg-D100, 11/4/99			7.852	70	bb	0.00
21 111099021	TOX145-RBL11049-300ppb-CC1-2, RWW	QC	307.00	7.836	8529	NM	265.63 -13
22 111099022	MeOH Blank			7.828	21	NM	0.00
23 111099023	TOX145-RBL11049-WBLk-2, RWW			7.830	64	NM	0.00
24 111099024	TOX145-RBL11049-RBLBlk-2, RWW			7.830	80	bb	0.00
25 111099025	TOX145-RBL11049-6ppb-2, RWW	Standard	6.13	7.828	274	NM	5.54 -10
26 111099026	TOX145-RBL11049-12ppb-2, RWW	Standard	12.30	7.812	485	NM	12.19 -1
27 111099027	TOX145-RBL11049-30ppb-2, RWW	Standard	30.70	7.828	1022	NM	29.13 -5
28 111099028	TOX145-RBL11049-60ppb-2, RWW	Standard	61.30	7.813	2109	NM	63.35 3
29 111099029	TOX145-RBL11049-120ppb-2, RWW	Standard	123.00	7.825	3948	NM	121.29 -1
30 111099030	TOX145-RBL11049-300ppb-2, RWW	Standard	307.00	7.810	10281	NM	320.82 5
31 111099031	TOX145-RBL11049-600ppb-2, RWW	Standard	613.00	7.813	19121	NM	599.31 -2
32 111099032	TOX145-RBL11049-900ppb-2, RWW	Standard	920.00	7.812	31809	NM	999.04 9
33 111099033	TOX145-RBL11049-1200ppb-2, RWW	Standard	1227.00	7.832	36504	NM	1146.96 -7

Data entered from undiluted analysis on 11/05/99.
These data not used.

LAC 11/15/99

Quantify Compound Summary Report
TOX145-rat liver, day 29 dose dilutions- ext. 11/4/99 RWW

Sample List: C:\MSSLYNK\scup020199.pro\SampleDB\111099a
Last modified: Thu Nov 11 07:09:30 1999
Method: C:\MSSLYNK\scup020199.pro\MethDB\111099a
Last modified: Thu Nov 11 07:08:22 1999
Job Code:

Printed: Thu Nov 11 07:26:35 1999

Compound 4: SURT (Punc 4)

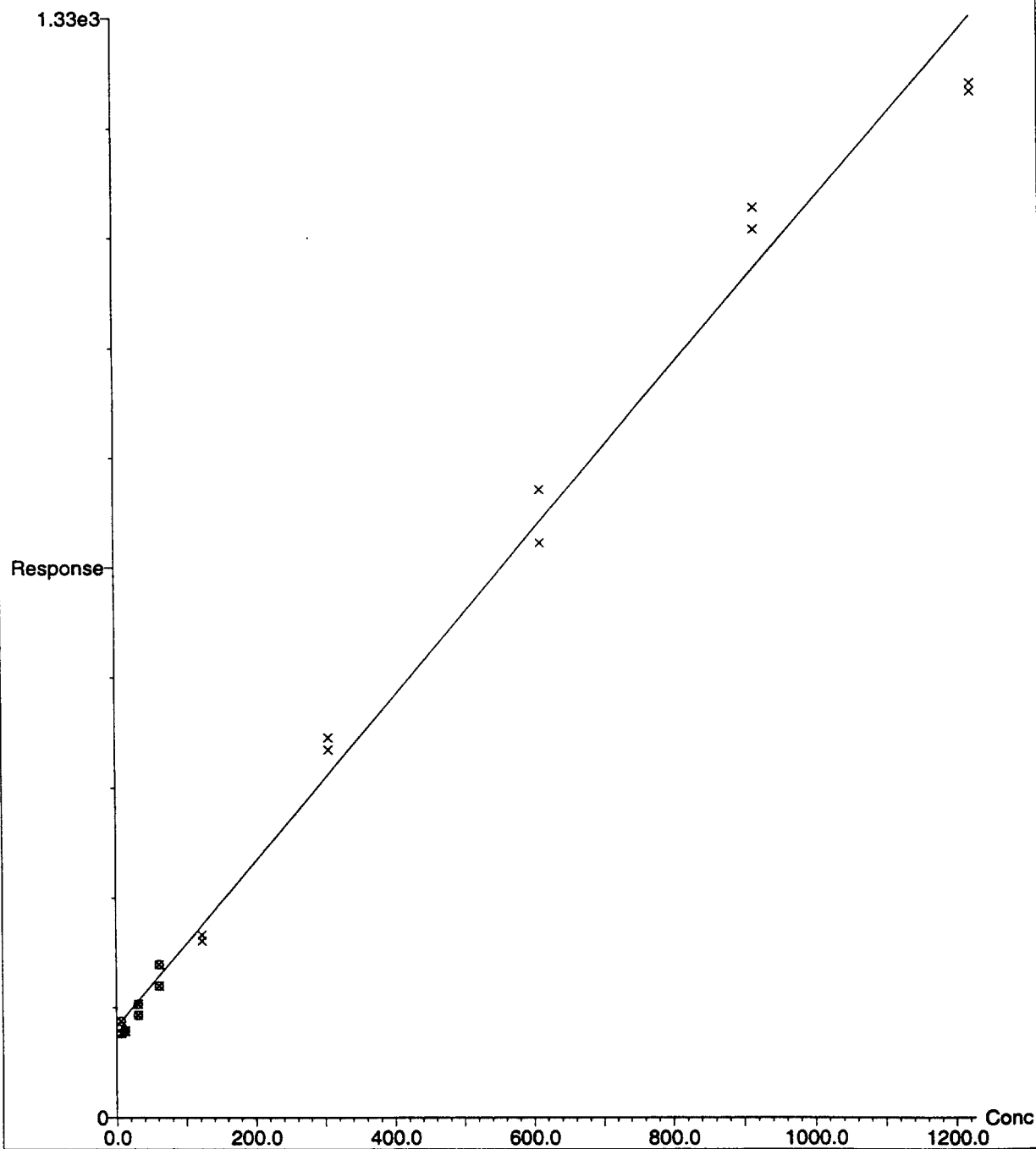
# Name	Samplelist Text	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1 111099001	MeOH Blank							
2 111099002	MeOH Blank							
3 111099003	MeOH Blank							
4 111099004	TOX145-RBL11049-WBLk-2, RWW			7.120	3832	bb	0.79	-21
5 111099005	TOX145-RBL11049-RBL1k-2, RWW			7.100	4057	bb	0.83	-17
6 111099006	TOX145-RBL11049-6ppb-2, RWW	Standard	1.00	7.103	4828	bb	0.99	-1
7 111099007	TOX145-RBL11049-12ppb-2, RWW	Standard	1.00	7.110	4818	bb	0.99	-1
8 111099008	TOX145-RBL11049-10ppb-2, RWW	Standard	1.00	7.099	5133	bb	1.05	5
9 111099009	TOX145-RBL11049-60ppb-2, RWW	Standard	1.00	7.083	4720	bb	0.97	-3
10 111099010	TOX145-RBL11049-120ppb-2, RWW	Standard	1.00	7.085	4662	bb	0.96	-4
11 111099011	TOX145-RBL11049-300ppb-2, RWW	Standard	1.00	7.100	4638	bb	0.95	-5
12 111099012	TOX145-RBL11049-600ppb-2, RWW	Standard	1.00	7.084	4860	bb	1.00	-0
13 111099013	TOX145-RBL11049-900ppb-2, RWW	Standard	1.00	7.094	4630	bb	0.95	-5
14 111099014	TOX145-RBL11049-1200ppb-2, RWW	Standard	1.00	7.083	4575	bb	0.94	-6
15 111099015	MeOH Blank			7.097	13	MM	0.00	-100
16 111099016	TOX145-RTL, 9R03255/M/Day29/5mg/kg-D100, 11/4/99			7.087	64	bb	0.01	-99
17 111099017	TOX145-RTL, 9R03256/M/Day29/5mg/kg-D100, 11/4/99			7.105	76	bb	0.02	-98
18 111099018	TOX145-RTL, 9R03257/M/Day29/5mg/kg-D100, 11/4/99			7.098	89	bb	0.02	-98
19 111099019	TOX145-RTL, 9R03258/M/Day29/5mg/kg-D100, 11/4/99			7.118	79	bb	0.02	-98
20 111099020	TOX145-RTL, 9R03259/M/Day29/5mg/kg-D100, 11/4/99			7.122	73	bb	0.01	-99
21 111099021	TOX145-RBL11049-300ppb-CC1-2, RWW	QC	1.00	7.106	3570	bb	0.73	-27
22 111099022	MeOH Blank			7.101	9	MM	0.00	-100
23 111099023	TOX145-RBL11049-WBLk-2, RWW			7.101	4042	bb	0.83	-17
24 111099024	TOX145-RBL11049-RBL1k-2, RWW			7.100	4274	bb	0.88	-12
25 111099025	TOX145-RBL11049-6ppb-2, RWW	Standard	1.00	7.099	5007	bd	1.03	3
26 111099026	TOX145-RBL11049-12ppb-2, RWW	Standard	1.00	7.082	5085	bb	1.04	4
27 111099027	TOX145-RBL11049-10ppb-2, RWW	Standard	1.00	7.100	5423	bb	1.11	11
28 111099028	TOX145-RBL11049-60ppb-2, RWW	Standard	1.00	7.084	5060	bb	1.04	4
29 111099029	TOX145-RBL11049-120ppb-2, RWW	Standard	1.00	7.074	4606	bd	0.94	-6
30 111099030	TOX145-RBL11049-300ppb-2, RWW	Standard	1.00	7.080	4718	bb	0.97	-3
31 111099031	TOX145-RBL11049-600ppb-2, RWW	Standard	1.00	7.084	5115	bd	1.05	5
32 111099032	TOX145-RBL11049-900ppb-2, RWW	Standard	1.00	7.084	5107	bb	1.05	5
33 111099033	TOX145-RBL11049-1200ppb-2, RWW	Standard	1.00	7.080	4777	bb	0.98	-2

Not a target analyte. data not used LAC 11/15/99

Inc 11/11/99 p. 1 of 4
Page 1

Quantify Calibration Report
TOX145-rat liver, day 29 dose dilutions- ext. 11/4/99 RMW

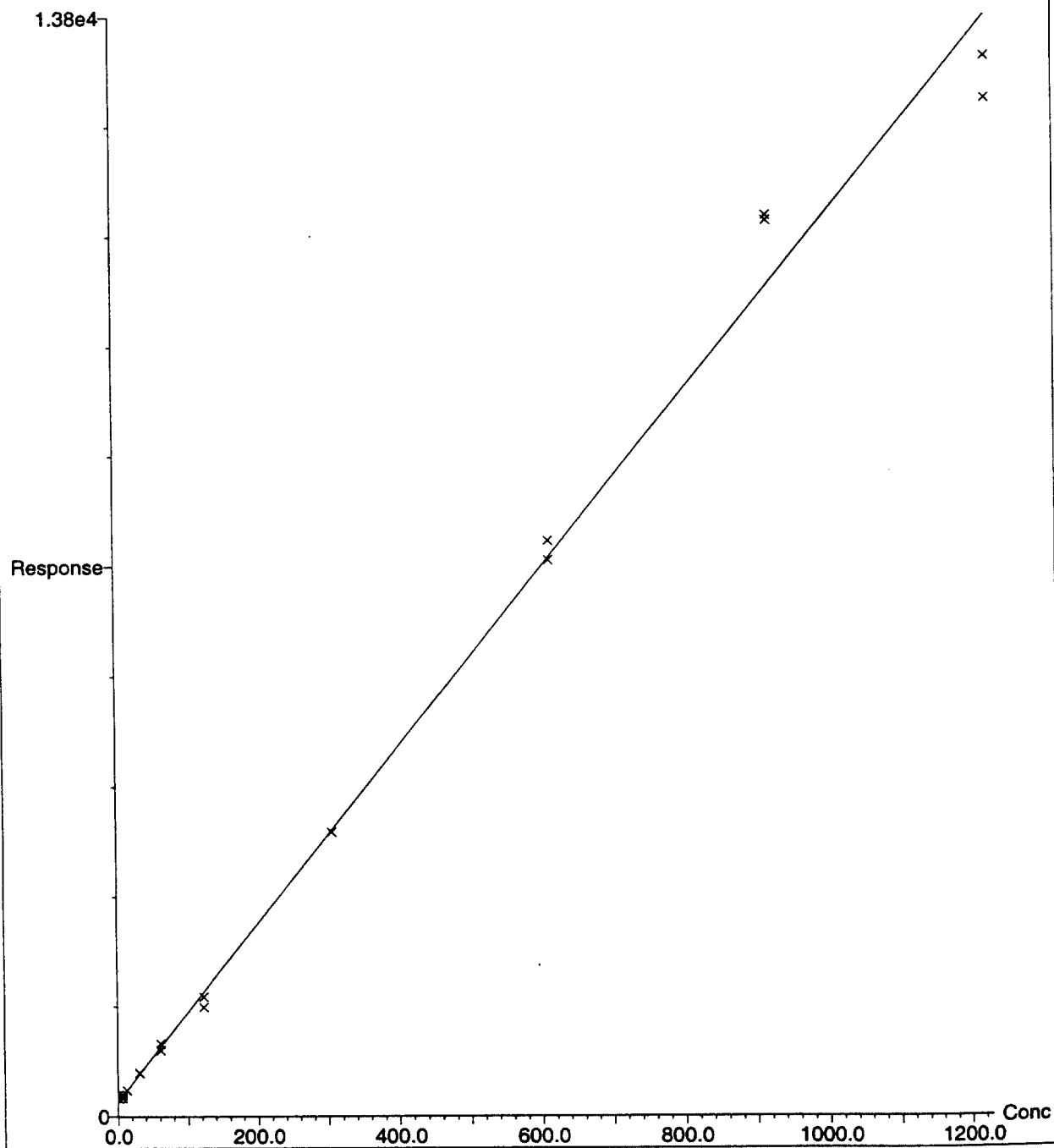
Compound 1 name: POAA (Func 1)
Coefficient of Determination: 0.980656
Calibration curve: $0.993942 \cdot x + 109.996$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Soup 020199: Quantitated and printed by ias on 11/11/99

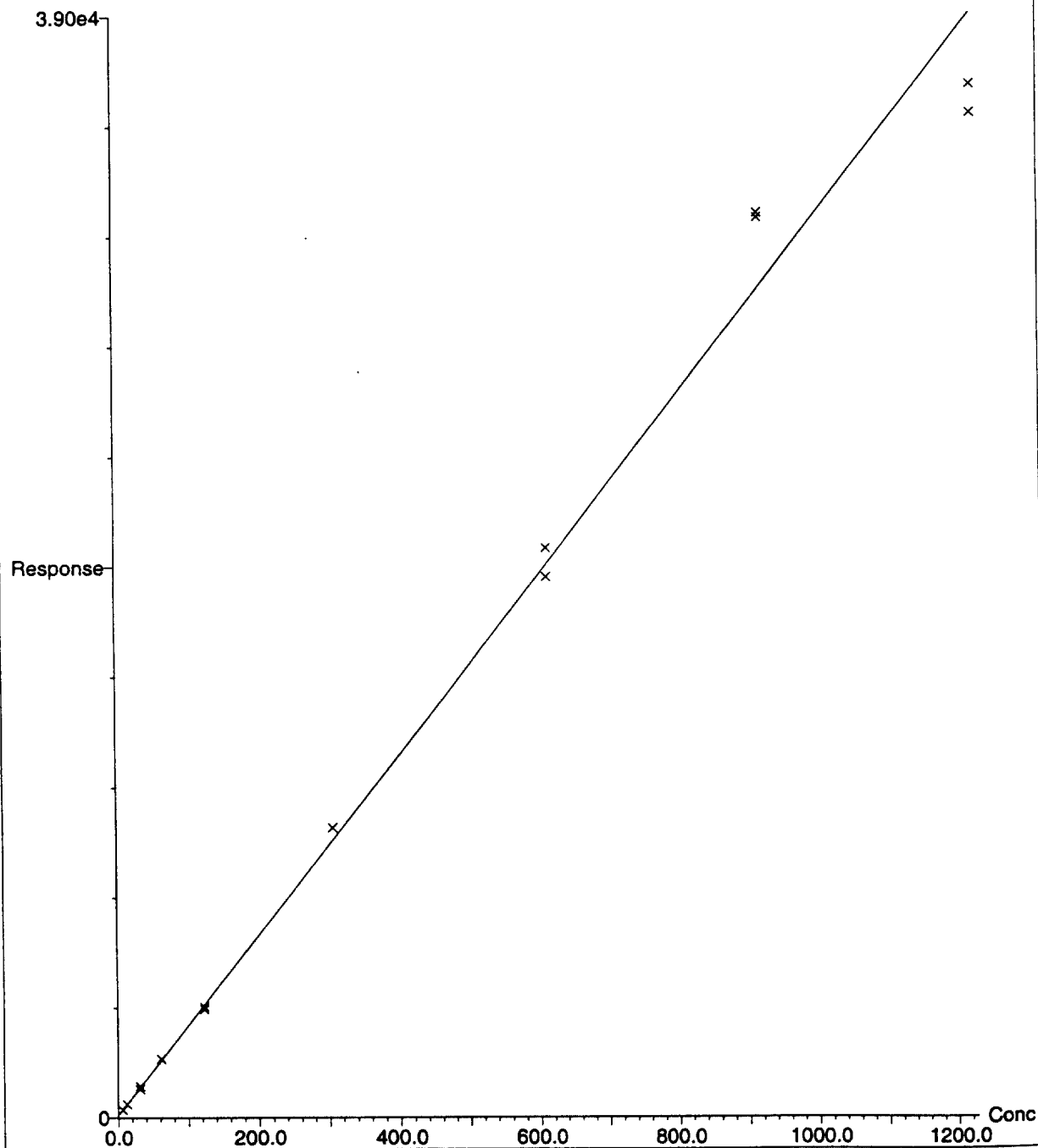
000799

Compound 2 name: PFOS (Func 2)
Coefficient of Determination: 0.991927
Calibration curve: $11.1011 * x + 194.545$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



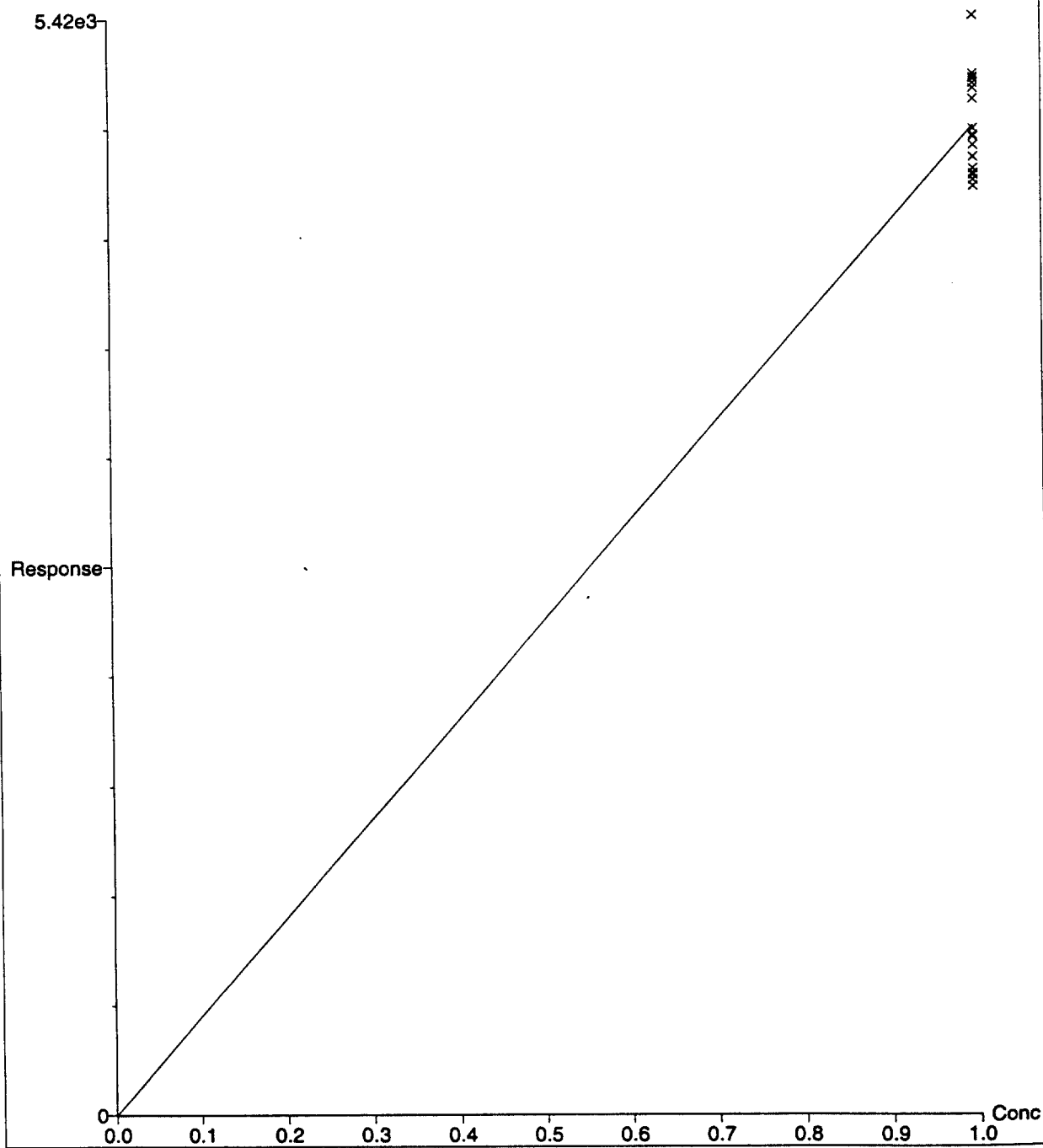
000800

Compound 3 name: PFOSA (Func 3)
Coefficient of Determination: 0.989396
Calibration curve: $31.7417 * x + 97.7287$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



000801

Compound 4 name: Surr (Func 4)
Response Factor: 4875.54
Response type: External Std, Area
Curve type: RF



Soup 020199: Quantitated and printed by ias on 11/11/99

000802

Method Report

Method file: C:\MASSLYNX\scup020199.pro\MethDB\111099a.mdb
 Last modified: Thu Nov 11 07:08:22 1999
 Compound: 1: POAA (Punc 1)
 Printed: Thu Nov 11 07:34:44 1999

Compound Name: POAA (Punc 1)
 Internal Ref.: [None]
 Quantify Trace: TIC
 Conc. of Standards: Conc. A
 User RF Value: 1.000000, Disabled
 User Peak Factor: 1.000000
 Integrate Window Factor: 2.000
 Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.067, Enabled
 Relative Retention Time: 0.000, Disabled
 RT Reference: [None]
 Time Window (mins): 0.246

Peak Selection: Nearest

Response Type: External
 Response: Areas

Polynomial Type: Linear
 Point of Origin: Exclude
 Fit Weighting: 1/X
 Axis Transformation: None

Units:

Peak-to-peak amplitude: 82

Smoothing: Enabled
 Window size (scans): 2
 Number of smooths: 1
 Smoothing method:

Join valley threshold: 30.00
 Reduce peak tailing: 200.00
 Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
 Detect shoulders: Disabled
 Shoulder threshold: 30.00

Relative height: 1.50, Disabled
 Absolute height: 0.00, Disabled
 Relative area: 2.00, Disabled
 Absolute area: 0.00, Disabled

Method Report

Method file: C:\MASSLYNK\soup020199.pro\MethDB\111099a.mdb
Last modified: Thu Nov 11 07:08:22 1999
Compound: 2: PFOS (Punc 2)
Printed: Thu Nov 11 07:34:44 1999

Compound Name: PFOS (Punc 2)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Conc. B
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.284, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.279

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 98

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000804

Method Report

Method file: C:\MASSLYNK\scup020199.pro\MethDB\111099a.mdb
Last modified: Thu Nov 11 07:08:22 1999
Compound: 4: Surr (Func 4)
Printed: Thu Nov 11 07:34:44 1999

Compound Name: Surr (Func 4)
Internal Ref.: [None]
Quantify Trace: TIC
Conc. of Standards: Fixed
User RF Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.161, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.278

Peak Selection: Nearest

Response Type: External
Response: Areas

Polynomial Type: Linear
Point of Origin: Exclude
Fit Weighting: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 92

Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

Join valley threshold: 30.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

Relative height: 1.50, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

000805

Jan 11/1/99

SOUP 020199: FACT Equipment
Log 99092 PAGE 1/1

Quattro II, #6414E

DATE: 11-10-99

ANALYST: ias

STUDY NUMBER: TOX145 MATRIX: rat liver DESCRIPTION: dilutions-day29

FACT Method: ETS-8-7.0

Injection volume: 10 uL Cycle time: 10 min. Electrode: Z-Spray

Mobile Phase: (A) 2.0 mM NH₄CH₃CO₂ (B) MeOH

TN-A- or stdrd number (A) 99085-11-21 (B) TN-A-3386

Prepared (date/by): (A) 11/6/99 sah (B) 11/6/99 sah

Expires: (A) 04/00 (B) 2010

Store at room temp? ☒ Y or N Other: _____

Guard column: None

Part number: na Lot number: na

Analytical column: Keystone Betasil

Size: 2 x 50 mm Type: C18 Sphere size: 5 um

Part number: 055-701-2 Lot number: P9E11 Serial number: 0591804Z

Analytical parameters:

Drying gas (L/hr): 350 ESI nebulizing gas (L/hr) 13 Sheath gas (L/hr) 0

LC Flow rate: 300 uL/min. Back pressure: 65 bar

HPLC solvent gradient: 10min-10-95

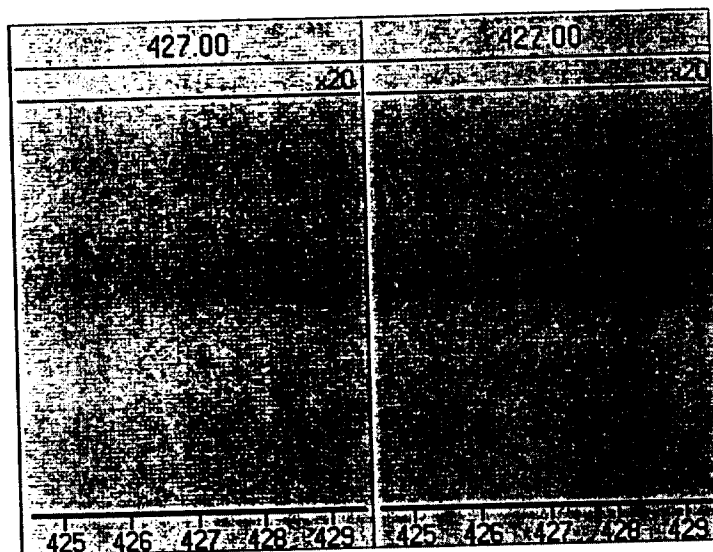
Time (min.)	%B
1) 0.00	10.0
2) 1.00	10.0
3) 5.50	95.0
4) 7.50	95.0
5) 8.00	10.0

000806

Tuning Method Report

Page 1

Method: C:\MASSLYNX\SOUP020199.PRO\ACQUDB\PFOS3
 Printed: Wed Nov 10 10:49:41 1999



MS

MS2

SOURCE (ESP-)	Set	Rdbk	MS1	Set	Rdbk
Capillary	1.60	1.61	LM Resolution	14.0	
Cone	40	-60	HM Resolution	14.0	
Extractor	5	-6	Ion Energy	0.9	
RF Lens	0.2		Ion Energy Ramp	0.0	
Source Block Temp.	150	150	Lens 6	4	3
Desolvation Temp.	250	250	Multiplier	650	

Pressures	Rdbk	MS2	Set	Rdbk
Analyser Vacuum	6.0e-5	LM Resolution	14.0	
		HM Resolution	14.0	
		Collision	10	37
Gas Cell	4.1e-3	Ion Energy	2.0	
		Ion Energy Ramp	0.0	
		Lens 8	40	38
		Lens 9	0	40
		Multiplier	650	

000807

Method Report

Page 1

Method File:
Last Modified:

C:\MASSLYNX\SOUP020199.PRO\ACQUDS\10min-10-95
Wednesday, November 10, 1999 10:43:07

Printed:

Wednesday, November 10, 1999 10:49:20

HP1100 LC Pump Initial Conditions

Solvents
A% 90.0
B% 10.0
C% 0.0
D% 0.0

Flow (ml/min) 0.300
Stop Time (mins) 10.0
Min Pressure (bar) 0
Max Pressure (bar) 400
Oven Temperature Left(°C) 30.0
Oven Temperature Right(°C) 30.0

HP1100 LC Pump Gradient Timetable

The gradient Timetable contains 5 entries which are :

Time	A%	B%	C%	D%	Flow	Pressure
0.00	90.0	10.0	0.0	0.0	0.300	400
1.00	90.0	10.0	0.0	0.0	0.300	400
5.50	5.0	95.0	0.0	0.0	0.300	400
7.50	5.0	95.0	0.0	0.0	0.300	400
8.00	90.0	10.0	0.0	0.0	0.300	400

HP1100 LC Pump External Event Timetable

The Timetable contains 3 entries which are :

Time	Column Switch	Contact1	Contact2	Contact3	Contact4
Initial	Off	Off	Off	Off	Off
0.00	Off	On	On	Off	On
0.10	Off	Off	Off	Off	Off

HP1100 Autosampler Initial Conditions

Draw Speed 200.0
Eject Speed (µl/min) 200
Draw Position (mm) 0.00
Stop Time (mins) 9.00
Injection Volume(µl) 10.0
Vial Number 91

000808

Sample List: C:\MASSLYNX\soup020199.pro\SampleDB\111099a.SPL
Printed: Wed Nov 10 10:48:56 1999

Page Position: (1, 1)

	File Name	File Text	MS File
1	111099001	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
2	111099002	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
3	111099003	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
4	111099004	TOX145-RBL11049-WBik-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
5	111099005	TOX145-RBL11049-RBLBik-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
6	111099006	TOX145-RBL11049-6ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
7	111099007	TOX145-RBL11049-12ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
8	111099008	TOX145-RBL11049-30ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
9	111099009	TOX145-RBL11049-60ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
10	111099010	TOX145-RBL11049-120ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
11	111099011	TOX145-RBL11049-300ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
12	111099012	TOX145-RBL11049-600ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
13	111099013	TOX145-RBL11049-900ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
14	111099014	TOX145-RBL11049-1200ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
15	111099015	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
16	111099016	TOX145-RTL, 9R03255/M/Day29/5mg/kg-D100, 11/4/99	10MIN-PFOS-POAA-PFOSA-IS
17	111099017	TOX145-RTL, 9R03256/M/Day29/5mg/kg-D100, 11/4/99	10MIN-PFOS-POAA-PFOSA-IS
18	111099018	TOX145-RTL, 9R03257/M/Day29/5mg/kg-D100, 11/4/99	10MIN-PFOS-POAA-PFOSA-IS
19	111099019	TOX145-RTL, 9R03258/M/Day29/5mg/kg-D100, 11/4/99	10MIN-PFOS-POAA-PFOSA-IS
20	111099020	TOX145-RTL, 9R03259/M/Day29/5mg/kg-D100, 11/4/99	10MIN-PFOS-POAA-PFOSA-IS
21	111099021	TOX145-RBL11049-300ppb-CC1-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
22	111099022	MeOH Blank	10MIN-PFOS-POAA-PFOSA-IS
23	111099023	TOX145-RBL11049-WBik-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
24	111099024	TOX145-RBL11049-RBLBik-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
25	111099025	TOX145-RBL11049-6ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
26	111099026	TOX145-RBL11049-12ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
27	111099027	TOX145-RBL11049-30ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
28	111099028	TOX145-RBL11049-60ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
29	111099029	TOX145-RBL11049-120ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
30	111099030	TOX145-RBL11049-300ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
31	111099031	TOX145-RBL11049-600ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
32	111099032	TOX145-RBL11049-900ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
33	111099033	TOX145-RBL11049-1200ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS
34	111099034	TOX145-RBL11049-900ppb-2, RWW	10MIN-PFOS-POAA-PFOSA-IS

000809

Masslynx - Sample List

Page 2

Sample List: C:\MASSLYNX\soup020199.pro\SampleDB\111099a.SPL
Printed: Wed Nov 10 10:48:56 1999

Page Position: (2, 1)

	Inlet File	Bottle	Inject Volume	Sample Type	Conc A	Conc B	Conc C
1	10min-10-95	91	10	---	---	---	---
2	10min-10-95	91	10	---	---	---	---
3	10min-10-95	91	10	---	---	---	---
4	10min-10-95	1	10	---	---	---	---
5	10min-10-95	2	10	---	---	---	---
6	10min-10-95	3	10	---	---	---	---
7	10min-10-95	4	10	---	---	---	---
8	10min-10-95	5	10	---	---	---	---
9	10min-10-95	6	10	---	---	---	---
10	10min-10-95	7	10	---	---	---	---
11	10min-10-95	8	10	---	---	---	---
12	10min-10-95	9	10	---	---	---	---
13	10min-10-95	10	10	---	---	---	---
14	10min-10-95	11	10	---	---	---	---
15	10min-10-95	92	10	---	---	---	---
16	10min-10-95	12	10	---	---	---	---
17	10min-10-95	13	10	---	---	---	---
18	10min-10-95	14	10	---	---	---	---
19	10min-10-95	15	10	---	---	---	---
20	10min-10-95	16	10	---	---	---	---
21	10min-10-95	17	10	---	---	---	---
22	10min-10-95	92	10	---	---	---	---
23	10min-10-95	1	10	---	---	---	---
24	10min-10-95	2	10	---	---	---	---
25	10min-10-95	3	10	---	---	---	---
26	10min-10-95	4	10	---	---	---	---
27	10min-10-95	5	10	---	---	---	---
28	10min-10-95	6	10	---	---	---	---
29	10min-10-95	7	10	---	---	---	---
30	10min-10-95	8	10	---	---	---	---
31	10min-10-95	9	10	---	---	---	---
32	10min-10-95	10	10	---	---	---	---
33	10min-10-95	11	10	---	---	---	---
34	10min-10-95	12	10	---	---	---	---

000810