

8EHQ-0904-15829

September 24, 2003 SEP 27 PM 9:15

**By Hand Delivery**

Document Processing Center (7407)
Office of Pollution, Prevention and Toxics
U.S. Environmental Protection Agency
1200 Pennsylvania Avenue, N. W.
Washington, DC 20460
Attention: Section 8(e) Coordinator

Re: **TSCA Section 8(e) Submissions**

Dear Sir/Madam:

3M Company ("3M") requests that EPA place the attached studies in the TSCA Section 8(e) docket. We have included a master index for these studies identifying the study title, test substance and CAS number. A Confidential Business Information (CBI) version of this index and the studies also is being submitted today pursuant to EPA procedures. 3M has not provided CBI substantiation with this submission, but would be willing to do so at the Agency's request.

3M has concluded that data in these studies may not be, strictly speaking, "corroborative" of previously reported or published information as defined in EPA's reporting guidance or otherwise potentially may warrant 8(e) submission based on EPA's reporting guidance.

3M appreciates EPA's attention to this matter. Please contact the undersigned if you have any questions or require further information regarding this submission.

Very truly yours,

Katherine E. Reed (g.e.r.)

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Study	Chemical	Accession Number
Primary Eye Irritation Study - Rabbits		
Guinea Pig Contact Dermal Irritation/Sensitization	20% solids (Ethomeen S/12 1.0M with diethyl sulfate 0.94M); 80% water [Ethomeen S/12 = R-N(Et)-(C ₂ H ₄ OH) ₂ where R=C18 with 1-2 double bonds]	20% (61791-24-0 with 64-67-5); 80% 7732-18-5
Primary Eye Irritation Study - Rabbits	Butanoic acid, heptafluoro-, calcium salt	2366-98-5
Acute Oral Toxicity Screen with T-2712CoC in Albino Rabbits	perfluorohexanoic acid	307-24-4
Primary Skin Irritation Test with T-2725Ec (Repeat Application) in Albino Rabbits		
Acute Ocular Irritation Test with T-2725Ec in Albino Rabbits		
Sensitization Study with T-2741AC in Albino Guinea Pigs		
Oral Rangefinder Study of T-3140BS in Pregnant Rats	1-[3'-(perfluorooctanesulfonate) anilino amide]-2-potassium 3,4,5,6-tetrachlorophthalate	57589-85-2
Oral Rangefinder Study of T-3139BS in Pregnant Rats	80% 1-[3'-(perfluorooctanesulfonate) anilino amide]-2-potassium 3,4,5,6-tetrachlorophthalate; 5% C7 homolog; 5% C5 homolog; 5% C4 homolog; 5% C6 homolog	80% 57589-85-2; 5% 68541-01-5 ; 5% 68541-02-6; 5% 68568-54-7; 5% 68815-72-5
Acute Ocular Irritation Test with T-2997CoC in Albino Rabbits	perfluoroethylcyclohexylsulfonic acid diethanol amine salt	salt of 133201-07-7 and 111-42-2
Sensitization Study with T-3386 in Albino Guinea Pigs		
In Vitro Microbiological Mutagenicity Assays of 3M Company's Compound T-3411		

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Study Title	Study Number	Study Status
Acute Oral Toxicity Screen with T-3448 in Albino Rats	68% poly(oxy-1,2-ethanediyl), alpha-[2-[ethyl[(heptadecafluorooctyl)sulfonyl]amino]ethyl]-omega-hydroxy-; 12% polyethylene glycol; 7% water; 4.86% poly(oxy-1,2-ethanediyl), alpha-[2-[ethyl[(pentadecafluoroheptyl)sulfonyl]amino]ethyl]-omega-hydroxy-; 4% residual organic fluorochemical; 3% heptadecafluoro-1-octanesulfonic acid; 0.81% poly(oxy-1,2-ethanediyl), alpha-[2-[ethyl[(undecafluoropentyl)sulfonyl]amino]ethyl]-omega-hydroxy-; 0.3% 1,4-dioxane; 0.2% n-ethylperfluorooctanesulfonamidoethyl alcohol; 0.03% linear n-ethyl perfluorooctanesulfonamide	68% 29117-08-6; 12% 25322-68-3; 7% 7732-18-5; 4.86% 56372-23-7; 4.05% 68298-79-3; 3.24% 68298-81-7; 3% 1763-23-1; 0.81% 68298-80-6; 0.3% 123-91-1; 0.2% 1691-99-2; 0.03% 4151-50-2
In Vitro Microbiological Mutagenicity Assays of 3M Company's Compound T-3516		
Acute Dermal Toxicity Study with T-3451 in Albino Rabbits	C8F17SO2N(CH3).Na	Unknown
Acute Oral Toxicity - Method, Summary, Pathology; Primary Dermal Irritation - Method, Summary; Primary Eye Irritation - Method, Summary; Guinea Pig Maximization - Method, Summary		
Acute Oral Toxicity - Method, Summary, Pathology; Primary Dermal Irritation - Method, Summary; Primary Eye Irritation - Method, Summary;		
Dermal Sensitization Study in Guinea Pigs, Maximization Test - Method, Summary		
4 Hour Acute Aerosol Inhalation Toxicity Study with T-3825 in Rats		
Primary Eye Irritation/Corrosion Study in Rabbits		
4-Hour Acute Aerosol Inhalation Toxicity Study with T-3825 in Rats		

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Study ID	Study Title	Study Date
T-3820: Acute Inhalation Toxicity Test		
T-3821: Acute Inhalation Toxicity Test		
T-3845 Acute Inhalation Toxicity Test	heptafluorobutyl chloride	375-16-6
Evaluation of the Acute Inhalation Toxicity of T-3920 in the Rat		
Primary Eye Irritation Study in Rabbits - Method, Summary	Decanoic acid, nonadecafluoro-, ammonium salt	3108-42-7
Acute Oral Toxicity Study in Rats (OECD Guidelines)	95% ammonium perfluorodecanoate; 5% ammonium perfluorooctanoate	5% 3825-26-1
Acute Inhalation Toxicity Study with T-4129 in the Rat		
Acute Inhalation Toxicity Study with T-4130 in the Rat		
Acute Oral Toxicity Study in Rats; Acute Dermal Irritation Study in Rabbits; Acute Eye Irritation Study in Rabbits		
Dermal Sensitization Study in Guinea Pigs - Maximization Test		
Mutagenicity Test on T- 4413 [] Mouse Lymphoma Forward Mutation Assay with Duplicate Cultures		
Acute Inhalation Toxicity Study with T-4354 in the Rat		
Primary Dermal Irritation/Corrosion Study in Rabbits		
Acute Inhalation Toxicity Study in the Rat with T-4397		
Primary Eye Irritation/Corrosion Study of T-5261 in Rabbits	lithium tetrafluoroethane-1,2-disulfonimide	Unknown
Acute Inhalation Toxicity Evaluation on T-5231 in Rats		
4-Hour, Acute Inhalation Toxicity Study with T-5305 in Rats		
4-Hour, Acute Inhalation Toxicity Study (Limit Test) with T-5343.1 in Rats		

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Study Title	Chemical Name	Study Number
4-Hour, Acute Inhalation Toxicity Study With T-5306 in Rats		
4-Hour, Acute Inhalation Toxicity Study (Limit Test) with T-5357.1		
Acute Dermal Toxicity Study of T-4201 in Rabbits	Lithium Bis(Trifluoromethanesulfonyl)imide	90076-65-6
Subacute 28-Day Oral Toxicity with T-2816 by Daily Gavage in the Rat Followed by a 14 Day Recovery Period		
Subacute 28-Day Oral Toxicity with T-2816 by Daily Gavage in the Rat Followed by a 14-Day Recovery Period		
Acute Inhalation Toxicity Evaluation on T-5187 in Rats		
T-4240 4-Week Oral Toxicity Study in Rats		
Dermal Sensitization Study of T-5473 in Guinea Pigs - Maximization Test		
4-Hour, Acute Inhalation Toxicity Study With T-5698 in Rats		
Acute Inhalation Toxicity Evaluation On T-5708 in Rats		
T-5486 Assessment of Cardiac Sensitization Potential in Dogs	octafluoropropane	76-19-7
Acute Inhalation Toxicity Evaluation on T-5655 in Rats		
T-4201 4 Week Oral Toxicity Study in Rats with 2-Week Recovery Period	Lithium Bis(Trifluoromethanesulfonyl)imide	90076-65-6
T-5658: Eye Irritation to the Rabbit		
Acute Inhalation Toxicity Evaluation on T-5715 in Rats		
Acute Inhalation Toxicity Evaluation on T-5716 in Rats		
Acute Inhalation Toxicity Study of T-5724 in Rats		
Acute Inhalation Toxicity Study of T-5725 (Resin Solution) in Rats		

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Study Title	Chemical Name	Study ID
Acute Inhalation Toxicity Study (Limit Test) of T-5927 in Rats		
Acute Inhalation Toxicity Study of T-5928 in Rats (LC50)		
Acute Inhalation Toxicity Evaluation on T-5829 in Rats		
Single-Dose Intravenous Pharmacokinetic Study of T-5963 in Rabbits		
Single-Dose Intravenous Pharmacokinetic Study of T-6030 in Rabbits		
5-Daily Dose Dermal Absorption/Toxicity Study of T-6029 and T-6032 in Rabbits	87-93% fluorinated alkyl alkoxyates; 4-10% linear N-ethyl perfluorooctanesulfonamide; 2-4% poly(oxy-1,2-ethanediyl).alpha.-[2-[ethyl[(pentadecafluoroheptyl)sulfonyl]amino]ethyl]-.omega.-methoxy-; 0-4% residual organic fluorochemicals; 0-2% c8 sulfonamide; 0.1-1% 1-heptanesulfonamide, N-ethyl-1,1,2,2,3,3,4,4,5,5,6,6,7,7,7-pentadecafluoro-; miscellaneous components (each less than 1%)	87-93% 68958-61-2; 4-10% 4151-50-2; 2-4% 68958-60-1; 0-2% 31506-32-8; 0.1-1% 68957-62-0
Single-Dose Intravenous Pharmacokinetic Study of T-6061 in Rabbits		
Single-Dose Intravenous Pharmacokinetic Study of T-6065 in Rabbits		
Single Dose Intravenous Pharmacokinetic Study of T-6063 in Rabbits		
Acute Inhalation Toxicity Study of T-6235 in Rats		
Primary Dermal Irritation/Corrosion Study of T-6402 in Rabbits		
Dermal Sensitization Study of T-6402 in Guinea Pigs- Maximization Test (EC Guidelines)		
Acute Eye Irritation/Corrosion Study with T-6318 in the Rabbit	1-Butanesulfinic acid, 1,1,2,2,3,3,4,4,4-nonafluoro-, Sodium Salt	102061-82-5

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Primary Skin Irritation / Corrosion Study with T-6567 in the Rabbit (4-Hour Semi-occlusive Application)		
Assessment of Contact Hypersensitivity to T-6318 in the Albino Guinea Pig (Maximization Test)	1-Butanesulfinic acid, 1,1,2,2,3,3,4,4,4-nonafluoro-, Sodium Salt	102061-82-5
Single-Dose Intravenous Pharmacokinetic Study of T-6502 in Rabbits		
Single-Dose Intravenous Pharmacokinetic Study of T-6504 in Rabbits		
Single Dose Intravenous Pharmacokinetic Study of T-6506 in Rabbits		
A Study for Effect on Embryofetal Development of the Rat (Inhalation Administration)	20-80% methyl nonafluoroisobutyl ether; 20-80% methyl nonfluorobutylether	20-80% 163702-08-7; 20-80% 163702-07-6
Bacterial Reverse Mutation Test of T-6695		
5-day Inhalation Toxicity of Perfluorocyclohexene ([]; T-6878) in Rats	70% crude perfluorocyclohexene; 30% perfluoromethylcyclopentene	70% 355-75-9
5-Daily Dose Dermal Absorption/Toxicity Study of T-6502 and T-6503 in Rabbits		
Primary Eye Irritation/Corrosion Study of T-6786 in Rabbits	Lithium Bis(perfluoroethylsulfonyl)imide	132843-44-8
Primary Dermal Irritation/Corrosion Study of T-6804 in Rabbits	Lithium Bis(perfluoroethylsulfonyl)imide	132843-44-8
5-Day Inhalation Toxicity Screen of HFE []	c-C6F11OCH3	4943-08-2
Primary Eye Irritation/Corrosion Study of T-6804 in a Rabbit (OECD Guidelines)	Lithium Bis(perfluoroethylsulfonyl)imide	132843-44-8
Acute Oral Toxicity Study of T-6804 in Rats (OECD Guidelines)	Lithium Bis(perfluoroethylsulfonyl)imide	132843-44-8
Dermal Sensitization Study of T-6908 in Guinea Pigs, Mazimization Test (EC Guidelines)		

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Study Title	Chemical Name	Study Number
Eye Irritation/Corrosion Study of T-4127 in the Rabbit	N-Me Fos Amide-Triphenylbenzyl Phosphonium Chloride Complex; D-1624	31506-32-8
Single-Dose Intravenous Pharmacokinetic Study of T-6924 in Rabbits		
Dermal Sensitization Study of T-6924 in Guinea Pigs- Maximization Test (EC Guidelines)		
Dermal Sensitization Study of T-7003 in Guinea Pigs - Maximization Test (EC Guidelines)		
Report of Sera and Liver Data for [] Monoester - Preliminary ADME Study in Rats	N-ethyl heptadecafluoro-N[2-(phosphonooxy)ethyl] octanesulfonamide diammonium salt	67969-69-1
[] Diester-Pharmacokinetic Study in Rats (Study No. T-7043.1, DT-26)	ammonium bis[ethyl(perfluorooctane)sulfonyl]phosphate	30381-98-7
Single Dose Intravenous Pharmacokinetic Study with T-7082 in Rabbits		
[] Monoester - Pharmacokinetic Study in Rats (Study No. T-6997.2)	N-ethyl heptadecafluoro-N[2-(phosphonooxy)ethyl] octanesulfonamide diammonium salt	67969-69-1
Determination of PFOS Presence and Concentration in Serum from the Dermal Absorption Studies of T-7106 and T-7107 in Hra:(NZW)SPF Rabbits		
Dermal Sensitization Study of T-7285.5 in Guinea Pigs - Maximization Test (EPA/OECD Guidelines)		
Twenty-eight Day Repeated-Dose Oral Toxicity Study of T-6861 in Rats	Lithium Bis(perfluoroethylsulfonyl)imide	132843-44-8
Twenty-eight Day Repeated Dose Oral Toxicity Study of T-7005 in Rats		

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Study Title	Chemical Name	Study Number
Acute (4-Hour) Inhalation Toxicity of Test Atmospheres Obtained after Heating [] in Rats	[]	[]
Toxicokinetic Study of Perfluorooctanesulfonamidoacetate ([]; T-7071.2) in Rats	perfluorooctanesulfonamido carboxylic acid	2806-24-8
Acute Nose-Only Inhalation Toxicity Study of T-7087, T-7088, T-7089 and T-7090 in Rats (Limit Test)	[]	[]
Acute Ocular Irritation Study of T-7485 Applied to New Zealand White Rabbits	potassium nonafluorobutanesulfonate	29420-49-3
Toxicokinetic Study of Perfluorooctane Sulfonamide (PFOSA; T-7132.2) in Rats	perfluorooctanesulfonamide	754-91-6
Acute Four-Hour Inhalation Study in Rats	Perfluorobutanesulfonyl Fluoride (96-98%) And Perfluorosulfolane (2-4%)	96-98% 375-72-4; 2-4% 42060-64-0
Primary Eye Irritation/Corrosion Study of T-7508.2 in Rabbits	[]	[]
MV31 K-Salz; Test for Primary Dermal Irritation in the Rabbit	[]	[]
Assessment of Acute Oral Toxicity with T-7560 In The Rat (Acute Toxic Class Method)	[]	[]
Acute Eye Irritation/Corrosion Study with T-7560 in the Rabbit	[]	[]
[] Potassium bis-(perfluorobutanesulfonyl)imide Repeat Dose ADME Study in Rats	Potassium bis(perfluorobutanesulfonyl)imide	129135-87-1
Toxicity Study by Repeat Dose Inhalation Administration to CD Rats for 4 Weeks	Perfluorobutanesulfonyl Fluoride (96-98%) And Perfluorosulfolane (2-4%)	96-98% 375-72-4; 2-4% 42060-64-0
A Sub-acute(28 Day) Inhalation Toxicity Study, Including a Recovery Study, with T-7479 in Rats	1,1,1,2,2,4,5,5,5-nonafluoro-4-(trifluoromethyl)-3-pentanone	756-13-8
Xenochemical Receptor trans-Activation by Perfluorooctane-based Chemicals	perfluorooctanesulfonamide	754-91-6

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Study Title	Chemical Name	Study Number
	84% 1-octanesulfonic acid, 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-, potassium salt; 5.5% potassium (perfluorohexyl)sulfonate; 4% potassium nonafluorobutanesulfonate; 4% potassium perfluoroheptanesulfonate; 2% potassium perfluoropentanesulfonate; 0.5% unknown	84% 2795-39-3; 5.5% 3871-99-6; 4% 29420-49-3; 4% 60270-55-5; 2% 3872-25-1
	95% N-ethylperfluorooctanesulfonamidoethyl alcohol; 5% 1-heptanesulfonamide N-ethyl-1,1,2,2,3,3,4,4,5,5,6,6,7,7,7-pentadecafluoro-N-(2-hydroxyethyl)-	95% 1691-99-2; 5% 68555-73-7
Acute Inhalation Toxicokinetic Study of Perfluorooctanesulfonyl Fluoride (POSF) T-7098.4	perfluorooctanesulfonyl fluoride	307-35-7
Five-Day Inhalation Toxicity Study of HFE [] in Male CD Rats	c-C6F11-CF2-O-CH3	181214-67-5
Acute Toxicity Screen of Perfluorocyclohexene (T-6878) in Rats	70% crude perfluorocyclohexene; 30% perfluoromethylcyclopentene	70% 355-75-9
[] (T-7056) Toxicokinetic Study in Rats	[]	[]
Assessment of Acute Oral Toxicity with T-7601.3 in the Rat (Acute Toxic Class Method)	N-Methyl Perfluorobutylsulfonamide = 95% 1-Butanesulfonamide, 1,1,2,2,3,3,4,4,4-Nonafluoro-n-Methyl; 5% N-Methyl-4-Hydroxy-Perfluorobutylsulfonamide	68298-12-4
Subchronic 90-Day Oral Toxicity Study with T-7320 By Daily Gavage in the Rat Followed by a 28-Day Recovery Period	[]	[]
Protein Binding of Perfluorobutane Sulfonate, Perfluorohexane Sulfonate, Perfluorooctane Sulfonate and Perfluorooctanoate to Plasma (Human, Rat, and Monkey), and Various Human-Derived Plasma Protein Fractions	84% 1-octanesulfonic acid, 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-, potassium salt; 5.5% potassium (perfluorohexyl)sulfonate; 4% potassium nonafluorobutanesulfonate; 4% potassium perfluoroheptanesulfonate; 2% potassium perfluoropentanesulfonate; 0.5% unknown	84% 2795-39-3; 5.5% 3871-99-6; 4% 29420-49-3; 4% 60270-55-5; 2% 3872-25-1
	potassium nonafluorobutanesulfonate	29420-49-3
	potassium (perfluorohexyl)sulfonate	3871-99-6

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Study Title	Chemical Name	Study Number
	potassium perfluorooctanoate	2395-00-8
Five Day Inhalation Toxicity Study of [] Monochloride, [], and HCFC225cb in Male CD Rats	C4F9-OCH2Cl	205367-42-6 (n-isomer) and 221617-86-3 (l-isomer)
	c-C6F11-CF2-O-CH3	181214-67-5
	CF2ClCF2CHClF	507-55-1
Toxicokinetic Screen of [] (T-7483) in Rats	C7F15C(O)N(H)CH3	89685-56-3
Low Level Oral Perfluorooctanesulfonate (PFOS) Dose Toxicokinetic Study in Rats: Serum and Liver PFOS	84% 1-octanesulfonic acid, 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-, potassium salt; 5.5% potassium (perfluorohexyl)sulfonate; 4% potassium nonafluorobutanesulfonate; 4% potassium perfluoroheptanesulfonate; 2% potassium perfluoropentanesulfonate; 0.5% unknown	84% 2795-39-3; 5.5% 3871-99-6; 4% 29420-49-3; 4% 60270-55-5; 2% 3872-25-1

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INFORMATION SUBJECT TO
PROTECTION UNDER THE
TOXIC SUBSTANCES CONTROL ACT
AND OTHER LAWS HAS BEEN
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FINAL REPORT

Study Title:

Toxicokinetic Study of Perfluorooctane Sulfonamide (PFOSA; T-7132.2) in Rats

Authors:

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Date: August 11, 2000

Testing Facility
3M Strategic Toxicology Laboratory
3M Center 270-3S-05
Saint Paul, MN 55144

Summary:

Purpose: The purpose of this study was to assess the relative absorption, metabolism, and elimination kinetics of Perfluorooctane Sulfonamide (PFOSA) in the rat following a single oral dose.

Methods: Two groups of fifteen male rats each received a single dose of either 2% Tween 80, as the vehicle control, or 5 mg/kg PFOSA suspended in 2% Tween 80 by oral gavage. Perfluorooctanoate (POAA) was found at a level of 2.5 ng/ml in the dosing solution. The PFOSA was found to be 96 % pure. Five rats from each dose group were sacrificed on days 1, 4, and 29 post dose. Liver and sera were analyzed for PFOSA and its predicted metabolites using high-pressure liquid chromatography/electrospray tandem mass spectrometry (HPLC/ESMSMS).

Results: The vehicle control group had no detectable levels of PFOSA or perfluorooctanesulfonate (PFOS) in liver or sera. The PFOSA dose group had decreasing levels of PFOSA, with evidence of conversion of PFOSA to PFOS in liver and sera throughout the study. Liver concentrations of PFOSA averaged 7.0 ppm, 3.7 ppm, and 0.1 ppm on days 1, 4, and 29 post-dose, respectively, with an apparent liver half-life of elimination of 5.2 days. Serum concentrations of PFOSA averaged 0.3 ppm and 0.1 ppm on days 1 and 4 post dose respectively, and were undetectable by day 29 post-dose, with an apparent serum half-life of elimination estimated to be less than 4 days. Liver PFOS concentrations were 28.2 ppm, 37.4 ppm and 23.8 ppm, representing 22.0%, 32.3%, and 25.2% of the PFOSA dosed, on days 1, 4 and 29 post-dose respectively. Sera PFOS averaged 8.2 ppm, 8.6 ppm, and 4.6 ppm on days 1, 4 and day 29 post dose respectively. The average total concentrations of PFOS equivalents (PFOS + PFOSA) in liver were 35.2, 41.1, and 23.9 ppm on days 1, 4, and 29 post-dose, respectively. The average concentrations of PFOS equivalents in the sera increased from 8.5 ppm on day 1 post dose to 8.7 ppm on day 4 post dose and decreased to 4.6 ppm on day 29 post dose. The liver to sera PFOS concentration ratio increased throughout the study with values of 4.2, 4.8, and 5.4 on days 1, 4, and 29 post dose, respectively. Total PFOS equivalents in the liver and sera peaked on day 4 post dose at 35.4% and 6.0 % of the PFOS equivalents dosed, respectively. An average of 0.1 ppm POAA was found in the sera on day 1 and day 4 post dose, and equaled 31.2% and 24.1% of the POAA residual present in the dose, respectively.

Conclusions: Perfluorooctanesulfonamide was readily absorbed from the gut and found in the liver and sera following an oral exposure. PFOSA was progressively metabolized to PFOS, which accumulated in the liver as evidenced by the high concentration of PFOS detected in the liver, representing 22.0% 32.3% and 25.2% of the PFOSA dosed, on days 1, 4 and 29 post dose respectively. The apparent half-life of elimination of PFOSA in the liver was approximately 5.2 days. The POAA present in the sera on days 1 and 4 post-dose could be accounted for by the residual POAA in the dosing solution, therefore, no direct evidence for metabolism of PFOSA to POAA was found in this study.

Introduction

The objective of this study was to assess the potential for oral absorption, urinary and fecal clearance, and biological persistence of Perfluorooctanesulfonamide (PFOSA) in male Sprague Dawley rats after a single oral dose. Analysis of the serum and liver for PFOSA and potential metabolites of PFOSA was performed by LCMS. Urine and feces were also collected and may be analyzed by LCMS and perhaps other methods as deemed necessary.

Previous studies on N-ethylperfluorooctanesulfonamide (1), the N-ethyl derivative of PFOSA, mistakenly concluded that PFOSA was the ultimate metabolite in rats (1,2); however, the gas chromatography analytical technique used in those studies was unable to detect PFOS. Dietary administration of N-ethylperfluorooctanesulfonamidoethanol (N-EtFOSE) in rats, and *in-vitro* metabolism by hepatocytes results primarily in the formation of PFOS with a minor amount of PFOSA (3, 4, and 5). Furthermore, PFOSA has been identified as a potent direct uncoupler of mitochondrial respiration *in-vitro* (6). Prior to the current study, direct metabolism of PFOSA to PFOS had not been demonstrated and the relative toxicokinetics of PFOSA were unknown. Methods were recently validated for the quantitation of PFOSA and its potential metabolite, PFOS, in serum and liver down to the low part per billion level (7). *In-vivo* studies were, thus, warranted to investigate the toxicokinetics of PFOSA.

Materials

The test material was identified as Perfluorooctanesulfonamide (PFOSA) [redacted]. Initial analysis by GCMS determined that the starting material was over 99% pure (8). Qualitative and quantitative compositional results derived from ¹H and ¹⁹F-NMR analysis revealed that the isomer distribution was approximately 65.8% CF₃ (CF₂) x-SO₂-NH₂ (Normal chain), 18.7% internal monomethyl branch and 11.2% Isopropyl branch (9). HPLC/MS characterization revealed low-level impurities of 9,600 ppm PFOS (0.96 %), and lower concentrations of several other amides (10). Perfluorooctanoate (POAA) was found in the dosing solution at a level of 2.5 µg/ml (0.25% of the nominal dose) (12). Based on the sum of the impurities, the purity of the PFOSA sample was determined to be approximately 96 % (8, 9 & 10).

Methods

The protocol (11) and analytical methods (12) used in this study are briefly described below.

Dose and Dosing Procedures:

A single 5mg/kg dose of PFOSA was administered via oral gavage to 15 male Sprague Dawley rats (obtained from Harlan Laboratories) on day zero of the study. The PFOSA was prepared as a 1% (1 mg/ml) uniform suspension in 2% Tween 80. A volume of 5 ml suspension / kg body weight was administered to each rat. The vehicle control group, consisting of 15 male Sprague Dawley rats (also obtained from Harlan Laboratories), received 2% Tween 80 in deionized water at a volume of 5 ml/kg on day zero of the study.

Specimen Collection:

Urine and feces collections were made on days 1 – 4 post dose from the animals designated for sacrifice on day 29 post dose group. Five animals from each dose group were euthanized by CO₂ on days 1, 4, and 29 post dose and gross necropsy was performed. Sera and liver were collected from each animal and flash-frozen in liquid nitrogen.

Specimen Handling:

Specimens (urine, feces, liver, and serum) were maintained at -70°C and sent to Kris Hansen, Ph.D., 3M Environmental Technology and Safety Services, for analysis. Liver and sera samples were analyzed by 3M Environmental for the parent compound and metabolites. The limits of detection (LODs) for PFOSA were 3.5 ng/ml in the liver and 1.51ng/ml in the sera. The LODs for PFOS were 8.45 ng/ml in liver and 1.74 ng/ml in the sera (12). Urine and feces were retained for possible future analysis as appropriate.

Results

Control Groups:

Body and Liver Weight

The average initial body weight $\pm$ SD of the fifteen vehicle control male rats was 273.7 ± 7.3 g. These rats lost an average of 2.2 g body weight from day zero to day 1 post dose. From day zero to day 4 post dose, the average body weight gain was 24.2 g and from day zero to day 29 post dose, body weight increased an average of 114.1 g. Liver weights and relative liver to body weight percentages are shown in Table 1A.

Liver and Sera Concentrations

Control animals had no detectable levels of PFOSA or PFOS in liver or sera at any time during this study (data not shown).

PFOSA Dose Groups:

Body and Liver Weight

The average initial body weight $\pm$ SD of the fifteen PFOSA dosed male rats was 270.0 ± 3.6 g. These rats lost an average of 4.5 g body weight from day zero to day 1 post dose. From day zero to day 4 post dose, the average body weight gain was 20.0 g and from day zero to day 29 post dose, body weight increased an average of 100.9 g. Liver weights and relative liver to body weight percentages were equivalent to control values (Table 1B).

Liver and Sera Concentrations

PFOSA and PFOS were detected in the liver and sera of all animals dosed with PFOSA. Liver and sera levels of PFOSA were highest on day 1 post dose and dropped significantly between

days 1 and 4 post dose and again between days 4 and 29 post dose (Tables 2 and 3). The average liver concentrations of PFOSA decreased from approximately 7.0 ppm to 3.7 ppm to 0.1 ppm from days 1 to 4, to 29 post dose, respectively (Table 2). The half-life of elimination of PFOSA in the liver was calculated from a log linear regression of the liver PFOSA concentrations to be 5.2 days. The sera concentrations of PFOSA were 0.3 ± 0.1 ppm, 0.1 ± 0.0 and <LOD (1.74 ng/ml) on days 1, 4, and 29 post dose, respectively (Table 3). The half-life of elimination of PFOSA in the serum cannot be calculated from this data, but is estimated to be less than 4 days. Average PFOS liver and sera levels peaked on day 4 post dose and declined by day 29 post dose to lower levels than found on day one. Average $\pm$ SD liver PFOS levels increased significantly from 28.2 ± 6.1 ppm on day 1 post dose to 37.4 ± 3.6 ppm on day 4 post dose. Levels then decreased significantly to 23.8 ± 4.1 ppm by day 29 post dose (Table 2). Average sera PFOS values were approximately 8 ppm on day 1 and day 4 post dose, then decreased significantly to approximately 4.6 ppm on day 29 post dose (Table 3).

Liver and Sera Concentrations as a Percentage of Dose

The percentage of PFOSA dosed detected as PFOSA in the liver and sera peaked on day one post dose at 5.4% and 0.2 % respectively. By day 4 post dose, these levels had dropped to 3.2% and 0.0 % and by day 29 post dose the liver levels dropped to 0.1 % and the sera levels remained at 0.0% (Table 6).

The percent PFOS equivalents dosed detected as PFOS in the liver and sera was 22.0% and 5.2 % on day 1 post dose and increased to 32.3% and 6.0% by day 4 post dose. On day 29 post dose, these levels had dropped to 25.2% and 4.1%, respectively (Table 6). Residual PFOS in the dose could account for 3.5%, 2.5%, and 5.6% of the PFOS found in sera, and 3.6%, 2.7%, and 3.9 % of the PFOS found in liver on days 1, 4, and 29 post dose, respectively (Tables 4 and 5).

The percent PFOS equivalents dosed detected as PFOS equivalents in the liver and sera was 27.2 % and 5.3 % on day 1 post dose and increased to 35.4 % and 6.0 % by day 4 post dose. On day 29 post dose these levels decreased to 25.4 % and 4.1 % in the liver and sera respectively (Table 6).

Sera POAA Concentrations

Perfluorooctanoate (POAA) was found at a level of 2.5 ng/ml in the dosing solution (12). Sera samples in the PFOSA dose group contained POAA at average values of 0.1 and 0.8 ppm POAA on days 1 and 4 post-dose respectively. These values equal 30.8% and 23.5%, respectively, of the POAA dosed (Table 7).

Conclusions:

No compound related effects were observed on body weight, liver weight, or liver/body weight ratios under treatment conditions.

Both PFOSA and PFOS were found at high concentrations in the liver and sera of rats one day following an oral dose. Approximately 38% of the PFOSA dosed was converted to PFOS four

days post-dose. PFOSA levels diminished in both sera and liver over time, with a liver half-life of approximately 5.2 days, and a much shorter residence period in the sera. Significantly more PFOS was found in the liver and sera at all time points than were present in the dose, therefore metabolic conversion of PFOSA to PFOS occurred. No direct evidence for metabolism of PFOSA to POAA was found in this study. The route of elimination of PFOSA in this study was not determined. No glucuronide or other conjugates were analyzed for, and further investigation of possible conjugates in the urine and feces is warranted. It is possible that some of the PFOSA was eliminated through the lung via expiration, but this has not been determined. The present results suggest that the PFOSA from the dose is progressively converted to PFOS and that the resulting PFOS is accumulated in the liver.

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Table 1A: Biological Parameters. Control Group							
Time	Dose	animal # 9R03-xxx	Initial BW (g)	Terminal BW (g)	BW Gain (g)	Liver wt (g)	Liver wt as % of BW
Day 1	Control	230	282.5	282.0	-0.5	12.0	4.3%
Day 1	Control	231	272.1	268.4	-3.7	11.2	4.2%
Day 1	Control	232	277.4	277.1	-0.3	12.4	4.5%
Day 1	Control	233	274.1	274.8	0.7	11.5	4.2%
Day 1	Control	234	267.6	260.5	-7.1	11.2	4.3%
	Avg		274.7	272.6	-2.2	11.7	4.3%
	SD		5.6	8.3	3.2	0.5	0.1%
Day 4	Control	235	266.9	294.5	27.6	11.6	3.9%
Day 4	Control	236	275.8	301.8	26.0	11.4	3.8%
Day 4	Control	237	261.9	283.4	21.5	11.9	4.2%
Day 4	Control	238	279.0	302.7	23.7	11.3	3.7%
Day 4	Control	239	268.3	291.2	22.9	10.7	3.7%
	Avg		270.4	294.7	24.3	11.4	3.9%
	SD		6.9	8.0	2.4	0.4	0.2%
Day 29	Control	240	273.5	371.8	98.3	13.9	3.7%
Day 29	Control	241	270.5	380.5	110.0	14.4	3.8%
Day 29	Control	242	280.2	389.4	109.2	14.2	3.6%
Day 29	Control	243	289.9	444.0	154.1	17.5	3.9%
Day 29	Control	244	266.3	365.2	98.9	14.3	3.9%
	Avg		276.1	390.2	114.1	14.9	3.8%
	SD		9.2	31.4	23.0	1.5	0.1%

Table 1B: Biological Parameters. PFOSA Dose Group							
Time	Dose	animal # 9R03-xxx	Initial BW (g)	Terminal BW (g)	BW Gain (g)	Liver wt (g)	Liver wt as % of BW
Day 1	5 mg/kg	245	263.2	257.8	-5.4	10.4	4.0%
Day 1	5 mg/kg	246	266.1	259.2	-6.9	10.2	3.9%
Day 1	5 mg/kg	247	266.5	264.1	-2.4	10.9	4.1%
Day 1	5 mg/kg	248	267.2	264.0	-3.2	10.1	3.8%
Day 1	5 mg/kg	249	268.7	264.3	-4.4	10.7	4.0%
	Avg		266.3	261.9	-4.5	10.5	4.0%
	SD		2.0	3.1	1.8	0.3	0.1%
Day 4	5 mg/kg	250	266.6	286.3	19.7	11.3	3.9%
Day 4	5 mg/kg	251	272.1	294.6	22.5	11.7	4.0%
Day 4	5 mg/kg	252	271.7	294.5	22.8	12.1	4.1%
Day 4	5 mg/kg	253	271.8	291.4	19.6	11.3	3.9%
Day 4	5 mg/kg	254	272.9	288.4	15.5	11.7	4.1%
	Avg		271.0	291.0	20.0	11.6	4.0%
	SD		2.5	3.7	2.9	0.3	0.1%
Day 29	5 mg/kg	255	275.2	385.4	110.2	16.7	4.3%
Day 29	5 mg/kg	256	273.6	380.3	106.7	14.4	3.8%
Day 29	5 mg/kg	257	271.4	373.9	102.5	13.9	3.7%
Day 29	5 mg/kg	258	274.5	371.4	96.9	14.6	3.9%
Day 29	5 mg/kg	259	268.7	356.7	88.0	12.7	3.6%
	Avg		272.7	373.5	100.9	14.5	3.9%
	SD		2.6	10.9	8.7	1.5	0.3%

Table 2: Liver PFOSA and metabolites in the PFOSA dose Group

Time	Dose	animal # 9R03- xxx	Liver [PFOS] ppm		Liver [PFOSA] ppm		Liver PFOSA PFOS eq ppm ¹
Day 1	5 mg/kg	245	23.7		6.02		6.02
Day 1	5 mg/kg	246	32.3		7.02		7.02
Day 1	5 mg/kg	247	36.2		6.24		6.24
Day 1	5 mg/kg	248	21.2		6.66		6.66
Day 1	5 mg/kg	249	27.8		8.81		8.81
	Avg		28.2		6.95		6.95
	SD		6.1		1.11		1.11
	Range	Min	21.2		6.02		6.02
		Max	36.2		8.81		8.81
	n	5	5		5		5
Day 4	5 mg/kg	250	36.8		3.52		3.52
Day 4	5 mg/kg	251	32.6		3.47		3.47
Day 4	5 mg/kg	252	42.6		3.96		3.96
Day 4	5 mg/kg	253	36.9		3.45		3.45
Day 4	5 mg/kg	254	38.3		4.11		4.11
	Avg		37.4 *		3.70 *		3.70
	SD		3.6		0.31		0.31
	Range	Min	32.6		3.45		3.45
		Max	42.6		4.11		4.11
	n	5	5		5		5
Day 29	5 mg/kg	255	20.3		0.09		0.09
Day 29	5 mg/kg	256	20.6		0.02		0.02
Day 29	5 mg/kg	257	22.0		0.10		0.10
Day 29	5 mg/kg	258	26.2		0.14		0.14
Day 29	5 mg/kg	259	29.9		0.30		0.30
	Avg		23.8 **		0.13 * / **		0.13
	SD		4.1		0.11		0.11
	Range	Min	20.3		0.02		0.02
		Max	29.9		0.30		0.30
	n	5	5		5		5

¹ Assuming a 1:1 ratio of PFOSA:PFOS equivalents; Liver PFOSA PFOS equivalents = Liver PFOSA

* = significantly different from day 1 values by a two tailed T-test (p<0.05)

** = significantly different from day 4 values by a two tailed T-test (p<0.05)

PQL = practical quantitation limit in liver (PFOS = 30 ng/g; PFOSA 10ng)

MDL = method detection limit in liver: (PFOS = 15 ng/g; PFOSA = 5 ng/g)

Table 3: Serum PFOSA and metabolites in the PFOSA Group .						
Time	Dose	animal # 9R03- xxx	Sera [PFOS] ppm		Sera [PFOSA] ppm	Sera PFOSA PFOS eq ppm ¹
Day 1	5 mg/kg	245	6.9		0.26	0.26
Day 1	5 mg/kg	246	10.8		0.44	0.44
Day 1	5 mg/kg	247	9.4		0.26	0.26
Day 1	5 mg/kg	248	6.7		0.33	0.33
Day 1	5 mg/kg	249	7.3		0.28	0.28
	Avg		8.2		0.31	0.31
	SD		1.8		0.08	0.08
	Range	Min	6.7		0.26	0.26
		Max	10.8		0.44	0.44
	n	5	5		5	5
Day 4	5 mg/kg	250	7.8		0.06	0.06
Day 4	5 mg/kg	251	7.5		0.06	0.06
Day 4	5 mg/kg	252	11.3		0.08	0.08
Day 4	5 mg/kg	253	8.6		0.06	0.06
Day 4	5 mg/kg	254	7.9		0.06	0.06
	Avg		8.6		0.06 *	0.06
	SD		1.6		0.01	0.01
	Range	Min	7.5		0.06	0.06
		Max	11.3		0.08	0.08
	n	5	5		5	5
Day 29	5 mg/kg	255	3.6		<LOD	
Day 29	5 mg/kg	256	4.0		<LOD	
Day 29	5 mg/kg	257	4.0		<LOD	
Day 29	5 mg/kg	258	4.1		<LOD	
Day 29	5 mg/kg	259	7.4		0.0	
	Avg		4.6	* / **	0.0	
	SD		1.5			
	Range	Min	3.59			
		Max	7.36			
	n		5		5	5
1. Assuming a 1:1 ratio of PFOSA :PFOS equivalents, Sera PFOSA PFOS equivalents =Sera PFOSA concentration * = significantly different from day 1 values by a two tailed T-test (p<0.05) ** = significantly different from day 4 values by a two tailed T-test (p<0.05) MDL = method detection limit (PFOS = 4.75 ng/ml; PFOSA = 2.89 ng/ml) LOD = limit of detection (PFOS = 1.74 ng/ml; PFOSA = 1.51 ng/ml)						

Table 4: Percent Dose Liver PFOS equivalents in PFOSA Group

Time	Dose	animal # 9R03-xxx	PFOS eq concentration in liver (ppm) ¹	Total PFOS eq in whole liver (ug) ²	PFOS eq in dose (ug) ³	% PFOS eq dosed found in liver ⁴	Residual PFOS in dose (ug) ⁵	% of PFOS in liver from residual PFOS in dose (%) ⁶
Day 1	5 mg/kg	245	29.7	309.1	1316.0	23.5	12.6	4.09%
Day 1	5 mg/kg	246	39.3	401.1	1330.5	30.1	12.8	3.18%
Day 1	5 mg/kg	247	42.4	462.6	1332.5	34.7	12.8	2.77%
Day 1	5 mg/kg	248	27.9	281.4	1336.0	21.1	12.8	4.56%
Day 1	5 mg/kg	249	36.6	391.7	1343.5	29.2	12.9	3.29%
	Avg		35.2	369.2	1331.7	27.7	12.8	3.58%
	SD		6.2	73.4	10.1	5.5	0.1	0.73%
	Range	Min	27.9	281.4	1316.0	21.1	12.6	2.77%
		Max	42.4	462.6	1343.5	34.7	12.9	4.56%
	n	5	5	5	5	5	5	5
Day 4	5 mg/kg	250	40.3	455.6	1333.0	34.2	12.8	2.81%
Day 4	5 mg/kg	251	36.1	422.0	1360.5	31.0	13.1	3.09%
Day 4	5 mg/kg	252	46.6	563.4	1358.5	41.5	13.0	2.31%
Day 4	5 mg/kg	253	40.4	456.0	1359.0	33.6	13.0	2.86%
Day 4	5 mg/kg	254	42.4	496.2	1364.5	36.4	13.1	2.64%
	Avg		41.1	478.6 *	1355.1	35.3 *	13.0	2.74%
	SD		3.8	54.2	12.6	3.9	0.1	0.29%
	Range	Min	36.1	422.0	1333.0	31.0	12.8	2.31%
		Max	46.6	563.4	1364.5	41.5	13.1	3.09%
	n	5	5.0	5.0	5.0	5.0	5	5
Day 29	5 mg/kg	255	20.4	340.4	1376.0	24.7	13.2	3.88%
Day 29	5 mg/kg	256	20.6	296.9	1368.0	21.7	13.1	4.42%
Day 29	5 mg/kg	257	22.1	307.2	1357.0	22.6	13.0	4.24%
Day 29	5 mg/kg	258	26.3	384.6	1372.5	28.0	13.2	3.43%
Day 29	5 mg/kg	259	30.2	383.6	1343.5	28.6	12.9	3.36%
	Avg		23.9 **	342.5 **	1363.4	25.1 **	13.1	3.87%
	SD		4.2	41.2	13.2	3.1	0.1	0.47%
	Range	Min	20.4	296.9	1343.5	21.7	12.9	3.36%
		Max	30.2	384.6	1376.0	28.6	13.2	4.42%
	n		5	5	5	5	5	5

1. PFOS equivalents in liver (ppm) = Liver PFOS (ppm) + Liver PFOSA PFOS equivalents (ppm); 2. Total PFOS equivalents in whole liver (ug) = PFOS equivalent concentration in liver (ppm) * liver weight (g); 3. Assuming a 1:1 ratio of PFOSA:PFOS equivalents, PFOS equivalents in dose (ug) = Initial BW (g) * dose (mg/kg) = BW*5; 4. % PFOS eq dosed as PFOSA in liver = (PFOS equivalents in whole liver (ug) / PFOS equivalents in dose) * 100; 5. Residual PFOS in dose (ug) = dose * 0.96% = (5mg/kg * BW) * 0.0096; 6. % of PFOS in liver from residual PFOS in dose = (residual PFOS in dose / total PFOS eq in liver + V14) * 100; * Significantly different from day 1 values by a two tailed T-test (p<0.05); ** Significantly different from day 4 values by a two tailed T-test (p<0.05); PQL = practical quantitation limit (PFOS = 30 ng/g; PFOSA 10ng)

Table 5: Percent Dose: Serum PFOS equivalents in PFOSA Group

Time	Dose	animal # 9R03-xxx	PFOS eq conc in Serum (ppm) ¹	PFOS eq conc in liver (ppm)	Liver/serum PFOS ratio (ppm/ppm)	Total PFOS Eq in whole liver (ug)	Serum volume (ml) ²	Total PFOS Eq in Serum (ug) ³	Liver/ Serum PFOS Eq ratio ⁴	PFOS eq in dose (ug) ⁵	% PFOS eq dosed found in serum ⁶	Residual PFOS in dose (ug) ⁷	% of PFOS in serum from residual PFOS in dose (%) ⁸
Day 1	5 mg/kg	245	7.2	29.7	4.1	309.3	8.3	60.0	5.2	1316.0	4.6	12.6	21.1%
Day 1	5 mg/kg	246	11.2	39.3	3.5	401.4	8.4	94.1	4.3	1330.5	7.1	12.8	13.6%
Day 1	5 mg/kg	247	9.6	42.5	4.4	462.9	8.5	81.9	5.6	1332.5	6.1	12.8	15.6%
Day 1	5 mg/kg	248	7.0	27.9	4.0	281.7	8.5	59.6	4.7	1336.0	4.5	12.8	21.5%
Day 1	5 mg/kg	249	7.6	36.6	4.8	392.1	8.5	65.0	6.0	1343.5	4.8	12.9	19.8%
	Avg		8.5	35.2	4.2	369.5	8.5	72.1	5.2	1331.7	5.4	12.8	17.7%
	SD		1.8	6.2	0.5	73.4	0.1	15.3	0.7	10.1	1.1	0.1	3.5%
	Range	Min	7.0	27.9	3.5	281.7	8.3	59.6	4.3	1316.0	4.5	12.6	13.6%
		Max	11.2	42.5	4.8	462.9	8.5	94.1	6.0	1343.5	7.1	12.9	21.5%
	N	5	5	5.00	5	5	5	5	5	5	5	5	5
Day 4	5 mg/kg	250	7.8	40.3	5.1	455.8	9.2	72.5	6.3	1333.0	5.4	12.8	17.6%
Day 4	5 mg/kg	251	7.5	38.1	4.8	422.2	9.5	71.7	5.9	1360.5	5.3	13.1	18.2%
Day 4	5 mg/kg	252	11.4	48.6	4.1	563.6	9.5	108.2	5.2	1358.5	8.0	13.0	12.1%
Day 4	5 mg/kg	253	8.7	40.4	4.7	456.1	9.4	81.6	5.8	1359.0	6.0	13.0	16.0%
Day 4	5 mg/kg	254	8.0	42.4	5.3	496.4	9.3	74.2	6.7	1364.5	5.4	13.1	17.6%
	Avg		8.7	41.2	4.8	478.8	9.4	81.7	5.9	1355.1	6.0	13.0	15.9%
	SD		1.6	3.8	0.5	54.2	0.1	15.3	0.6	12.6	1.1	0.1	2.5%
	Range	Min	7.5	36.1	4.1	422.2	9.2	71.7	5.2	1333.0	5.3	12.8	12.1%
		Max	11.4	48.6	5.3	563.6	9.5	108.2	6.7	1364.5	8.0	13.1	18.2%
	N	5	5	5	5	5	5	5	5	5	5	5	5
Day 29	5 mg/kg	255	3.6	20.4	5.7	340.4	12.4	44.7	7.8	1376.0	3.2	13.2	29.6%
Day 29	5 mg/kg	256	4.0	20.6	5.2	296.9	12.3	48.9	6.1	1368.0	3.6	13.1	26.9%
Day 29	5 mg/kg	257	4.0	22.1	5.5	307.3	12.1	48.8	6.3	1357.0	3.6	13.0	26.7%
Day 29	5 mg/kg	258	4.1	26.3	6.4	384.6	12.0	49.5	7.8	1372.5	3.6	13.2	26.6%
Day 29	5 mg/kg	259	7.4	30.2	4.1	383.6	11.5	84.8	4.5	1343.5	6.3	12.9	15.2%
	Avg		4.6**	23.9**	5.4	342.6	12.1	55.3	6.5	1363.4	4.1	13.1	23.7%
	SD		1.5	4.2	0.8	41.2	0.4	16.6	1.3	13.2	1.3	0.1	5.6%
	Range	Min	3.6	20.4	4.1	296.9	11.5	44.7	4.5	1343.5	3.2	12.9	15.2%
		Max	7.4	30.2	6.4	384.6	12.4	84.8	7.8	1376.0	6.3	13.2	29.6%
	N	5	5	5	5	5	5	5	5	5	5	5	5

1. PFOS equivalents in sera (ppm) = sera PFOS (ppm) + sera PFOSA PFOS equivalents (ppm); 2. There are 32.3 mL Plasma per Kg of rat. (9); 3. Total PFOS equivalents in sera (ug) = PFOS equivalent concentration in sera (ppm) * serum volume (ml); 4. Liver/ Serum total PFOS Eq ratio = total PFOS equivalents in whole liver (ug)/ total PFOS equivalents in serum (ug); 5. Assuming a 1:1 ratio of PFOSA:PFOS PFOS equivalents, PFOS equivalents in dose (ug) = Initial BW (g) * dose (mg/kg) = BW*5; 6. % PFOS equivalents dosed in serum = (PFOS equivalents in sera (ug)/ PFOS equivalents in dose (ug)) *100; 7. Residual PFOS in dose (ug) = dose*0.96% = (5mg/kg*BW)0.0096; 8. % of PFOS in sera from residual PFOS in dose = (residual PFOS in dose / total PFOS eq in sera)*100; ** Sign. different from dy 4 values by a two tailed T-test (p<0.05); MDL = method detection limit in sera: (PFOS = 4.75 ng/ml; PFOSA = 2.89 ng/ml)

Table 6 - Summary: % + SD of Dose or of PFOS Equivalents Dosed Detected in Liver & Sera			
	Day 1 post dose	day 4 post dose	day 29 post dose
% PFOSA dosed detected as PFOSA in:			
liver	5.4 ± 0.9	3.2 ± 0.3	0.1 ± 0.1
sera	0.2 ± 0.1	0.0 ± 0.0	0.0 ± 0.0
liver & sera	5.6 ± 0.9	3.2 ± 0.3	0.1 ± 0.1
% PFOS Equivalents dosed detected as PFOS in:			
liver	22.0 ± 5.2	32.3 ± 3.7	25.2 ± 3.0
sera	5.2 ± 1.1	6.0 ± 1.1	4.1 ± 1.2
liver & sera	27.1 ± 6.2	38.3 ± 4.8	29.3 ± 3.8
NA = not analyzed			
PFOS equivalents (ppm) = PFOS (ppm) + PFOSA PFOS equivalents (ppm)			

3M Strategic Toxicology ST39
T-7132.2 PFOSA PK study in rats

Table 7: Serum POAA in the PFOSA Group.

Time	Dose	animal # 9R03- xxx	Sera POAA (ppm)	Total POAA in total Serum (ug)	*POAA in dose (ug)	% POAA dosed found as POAA in sera
Day 1	5 mg/kg	245	0.11	0.90	3.29	27.3%
Day 1	5 mg/kg	246	0.15	1.23	3.33	37.0%
Day 1	5 mg/kg	247	0.13	1.09	3.33	32.8%
Day 1	5 mg/kg	248	0.10	0.88	3.34	26.3%
Day 1	5 mg/kg	249	0.12	1.02	3.36	30.5%
	Avg		0.12	1.02	3.33	30.8%
	SD		0.02	0.15	0.03	4.3%
	Range	Min	0.10	0.88	3.29	26.3%
		Max	0.15	1.23	3.36	37.0%
	n	5	5	5	5	5
Day 4	5 mg/kg	250	0.07	0.63	3.33	18.9%
Day 4	5 mg/kg	251	0.07	0.64	3.40	18.9%
Day 4	5 mg/kg	252	0.11	1.00	3.40	29.4%
Day 4	5 mg/kg	253	0.10	0.95	3.40	28.0%
Day 4	5 mg/kg	254	0.08	0.76	3.41	22.2%
	Avg		0.08	0.80	3.39	23.5%
	SD		0.02	0.17	0.03	5.0%
	Range	Min	0.07	0.63	3.33	18.9%
		Max	0.11	1.00	3.41	29.4%
	n	5	5	5	5	5

Limit of detection (LOD): POAA 24.7 ng/ml

NA: Not analyzed

* based on work by Kris Hansen finding 2.5 ug/ml POAA in dosing solution

¹ There are 32.3 mL Plasma per Kg of rat.(13)

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, $C_8F_{15}CO_2^-$, 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 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. 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° 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 $\pm 30\%$ 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 Hz
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

[illegible]

Donor Summary: 10/1/99, 10/2/99, 11/10/99 LAC, 11/10/99 CHL.
Donor Verdict: 11/10/99 CHL.



Laboratory Report
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Attachment B
Table of Results - Liver Analysis

FACT-TOX-145

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

TOX-145 Pharmacokinetic Study of PFOSA in Rats
7/2/1122
Ballmer
ETS-4.6 and ETS-4.7.0
Samp 001199, Analyt 062498
Mixture 3.3
101199, 110499 SEBR/VW/SAL
101199, 101199 M04E, 110599 LAS, 111099 LAS
101199, 102099, 110499, 111199 LAS/M04E

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

See Attachments
See Attachments
See Attachments
See Attachments

RAT LIVER

Group	Sample #	PFOS Calc. Conc.	Total Amount of PFOS	Average PFOS	MSD	PFOSA Calc. Conc.	Total Amount of PFOSA	Average PFOSA	MSD
Method BIR	H2O BIR-11	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
	H2O BIR-11	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
	H2O BIR-2	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
	H2O BIR-2	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
Matrix BIR	BAL10199 Liver BIR-11	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
	BAL10199 Liver BIR-11	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
	BAL10199 Liver BIR-2	1.54	<LOD	<LOD	<LOD	0.1760	<LOD	<LOD	<LOD
	BAL10199 Liver BIR-2	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
QC	BAL10199 Liver BIR-11	216	95%	90%	5%	313	104%	99%	10%
	BAL10199 Liver BIR-2	244	87%	73%	5%	283	94%	74%	7%
	PFOS1394L Day14S-11	234	73%	73%	5%	229	71%	74%	7%
	PFOS1394L Day14S-12	216	73%	73%	5%	215	71%	74%	7%
	PFOS1394L Day14S-13	212	73%	73%	5%	352	119%	116%	7%
	PFOS1394L Day14S-14	191	65%	66%	10%	330	112%	116%	7%
Group 1	PFOS1394L Day1	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
	PFOS1394L Day1	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
	PFOS1394L Day1	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
	PFOS1394L Day1	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
Group 2	PFOS1394L Day4	0.00	<LOD	<LOD	<LOD	2.35	<LOD	<LOD	<LOD
	PFOS1394L Day4	0.00	<LOD	<LOD	<LOD	6.00	<LOD	<LOD	<LOD
	PFOS1394L Day4	29.3	0.005	<LOD	<LOD	22.8	0.0220	<LOD	<LOD
	PFOS1394L Day4	2.0	<LOD	<LOD	<LOD	4.66	<LOD	<LOD	<LOD
	PFOS1394L Day4	0.00	<LOD	<LOD	<LOD	0.00	<LOD	<LOD	<LOD
Group 3	PFOS1394L Day7	0.00	<LOD	<LOD	<LOD	3.27	<LOD	<LOD	<LOD
	PFOS1394L Day7	0.00	<LOD	<LOD	<LOD	1.71	<LOD	<LOD	<LOD
	PFOS1394L Day7	0.00	<LOD	<LOD	<LOD	0.714	<LOD	<LOD	<LOD
	PFOS1394L Day7	0.00	<LOD	<LOD	<LOD	0.142	<LOD	<LOD	<LOD
	PFOS1394L Day7	0.00	<LOD	<LOD	<LOD	0.0047	<LOD	<LOD	<LOD
Group 4	PFOS1394L Day1	23.640	21.7	<LOD	<LOD	20.51	6.02	<LOD	<LOD
	PFOS1394L Day1	123.640	31.3	<LOD	<LOD	70.15	7.02	<LOD	<LOD
	PFOS1394L Day1	34.179	34.2	<LOD	<LOD	42.39	6.34	<LOD	<LOD
	PFOS1394L Day1	21.200	31.2	<LOD	<LOD	44.07	6.44	<LOD	<LOD
	PFOS1394L Day1	27.729	27.8	<LOD	<LOD	48.11	6.81	<LOD	<LOD
Group 5	PFOS1394L Day4	34.791	34.8	<LOD	<LOD	33.32	3.22	<LOD	<LOD
	PFOS1394L Day4	124.19	31.6	<LOD	<LOD	34.70	3.47	<LOD	<LOD
	PFOS1394L Day4	42.006	41.6	<LOD	<LOD	39.59	3.86	<LOD	<LOD
	PFOS1394L Day4	34.945	34.9	<LOD	<LOD	34.54	3.45	<LOD	<LOD
	PFOS1394L Day4	13.641	31.3	<LOD	<LOD	41.05	4.11	<LOD	<LOD
Group 6	PFOS1394L Day7	30.035	20.3	<LOD	<LOD	13.5	0.0353	<LOD	<LOD
	PFOS1394L Day7	206.34	20.6	<LOD	<LOD	175	0.175	<LOD	<LOD
	PFOS1394L Day7	22.025	22.0	<LOD	<LOD	104	0.104	<LOD	<LOD
	PFOS1394L Day7	24.172	24.2	<LOD	<LOD	142	0.142	<LOD	<LOD
	PFOS1394L Day7	29.67	29.9	<LOD	<LOD	303	0.303	<LOD	<LOD

Limit of Quantitation (LOQ): PFOS = 3.5 ng/g PFOSA = 1.1 ng/g

Limit of Detection (LOD): PFOS = 1.41 ng/g PFOSA = 1.59 ng/g

PFOS = Perfluorooctanesulfonic acid
PFOSA = Perfluorooctanesulfonamide
* Samples numbers may have been switched. 3137 should be 3130
* These samples may be slightly high, due to carryovers from accidentally switching 3137 and 3130. CML 11/11/99

Date Entered/Analysis: 10/21/99, 10/22/99, 11/15/99 LAC, 11/06/99 GML
Date Verified/Analysis: 11/16/99 GML



Laboratory Report
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(Sera and Liver)

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Requester Project No. T-7132.2

Attachment C

Sample Receipt

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

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

Chain of Custody Record
3M Environmental Laboratory

09303

Form 35178 - PWO

3M Facility Corporate Tox	3M Project Title PFOSA PK, TOX-145, T-7132.2 IN RATS	3M Project Number 7-7132.2 0010535009
Address 220-25-02	3M Facility Contact Deanna Lucker	3M Department Number 0227
City, State, Zip Code	3M Telephone Number 7-1374	Lab Request/Template Number W2814

Sampled By (Print) Deanna Lucker		Contractor		Number Containers of each		Total Containers of Each Item	☐ Samples Split See Attachment	Comments			
Sampler Signature <i>Deanna Lucker</i>		Date 10/6/99		Preservatives							
Item #	Sample Description	Date	Time	Matrix	None	HNO ₃	H ₂ SO ₄	NaOH	VOC		
1	Dy1 Post dose Urine	10/5	1pm	Urine							10
2	Dy2 " " "	10/10	8am	Urine							"
3	Dy1 Post Dose Feces	10/5	1pm	Feces							"
4	Dy2 " " "	10/10	8am	Feces							"
5	Dy1 Post dose Liver	10/5	2pm	Liver							"
6	Dy1 Post dose Sera	10/5	2pm	Sera							"
7											
8											
9											
10											
11											
12											
13											

Item #	Relinquished By/Affiliation	Time	Date	Shipped Via	Received By/Affiliation	Time	Date
1-13	Deanna Lucker <i>KL</i>		10/6/99		Barb Brannan <i>az</i>	12:45	10/6/99

Original Accompanying Samples

Field Notes - Completed

See Reverse Side For Instructions

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

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	②9R03254

Chain of Custody Record
3M Environmental Laboratory

09600

Form 35176 - PWO

3M Facility Corporate Tox	3M Project Title PFOA PK IN RATS	3M Project Number T-7132.2
Address 2202E-02	3M Facility Contact Deanna	3M Project Lead Andrew Seacat
City, State, Zip Code	3M Telephone Number 7-1374	3M Department Number 62270
		Lab Request/Template Number W2814

Sampled By (Print) Deanna Liebker		Contractor		Number Containers of each		Total Containers of Each Item	☐ Samples Split See Attachment					
Sampler Signature <i>[Signature]</i>		Date 10/18/99		Preservatives								
Item #	Sample Description	Date	Time	Matrix	None	HNO ₃	H ₂ SO ₄	NaOH	VOC			Comments
1	Dx3 Post Dose Urine	10/17	2pm	Urine							10	Vehicle Control 9203240-44 Smg/kg PFOA 9203255-54
2	" " " " feces	10/17	2pm	Feces							10	"
3	Dx4 " " Urine	10/18	9am	Urine							10	"
4	" " " " feces	10/18	9am	Feces							10	"
5	Dx4 Post dose liver	10/18	2pm	Liver							10	Vehicle Control 9203235-39 Smg/kg PFOA 9203250-54
6	" " " " sera	10/18	2pm	Sera							10	"
7											10	
8												
9												
10												
11												
12												
13												

Item #	Relinquished By/Affiliation	Time	Date	Shipped Via	Received By/Affiliation	Time	Date
1-6	Deanna Liebker / Tox	3:45 PM	10/19/99	Back Runner	Jim A. Clamer / 3M	4:00 PM	10/19/99

Original - Accountability Switch

Lab Request/Template

(1) RE UAC 10/19/99

See Reverse Side For Instructions

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

3M Environmental Laboratory

Form 35176-PWD

09305

3M Requester	Deanna Luebker
3M Project Lead	Andrew Seacat
3M Project Number	DO10535009
3M Department Number	7432-2
Lab Request Template Number	227 4291
	W2814

Sampled By (Print)		Contractor		Number Containers of each							Total Containers of Each Item	☐ Samples Split See Attachment	
Sampler Signature		Date			Preservatives							Comments	
Item #	Sample Description	Date	Time	Matrix	None	HNO ₃	H ₂ SO ₄	NaOH	VOC				
1	Rat Liver vehic control	11/2/99	10a	LIVER								5	
2	Rat LIVER Smg/Kg PFOSA	"	"	LIVER								5	
3	Rat GRA vehic control	"	"	SRA								5	
4	Rat GRA Smg/Kg PFOSA	"	"	SRA								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	Dorinda Walker / TOF	12:15p	11/2/99	RoadRunner	Burt G. / Nene 13	1:30p	11/2/99



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

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

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

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 _a	% Recovery of M	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 _a	% Recovery of M	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 _a	% Recovery of M	Date of Extraction
Tween dose-200ppb	265.65	200	100	385.42	(133)	(120)	10/29/99

FACT: DATA REVIEW SUMMARY FORM

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

Data Review Sign-Off

Analyst Review
 Name / Date: JAS 11/01/99

Technical Review
 Name / Date: qh 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

FACT: DATA REVIEW SUMMARY FORM

Data Review Sign-Off

Date: 11/1/99
 Data Analyst: IAS
 FACT Study #: TOX145
 Matrix/Timepoints: Tissue 80 dose confirmation sample
 Sample List: 107799a
 Set-up Analysis
 Date/Analyst: 11/29/99 IAS
 Instrument: Simp 920199

Analyst Review
 Name / Date: IAS 11/21/99

Technical Review
 Name / Date: kjh 11/1/99

Quality Assurance Review
 Name / Date: _____

Data Entry: _____

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 11/1/99
 MS/MSD: _____
 Data Status: _____

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

③ (cont) curve points were greater than $\pm 30\%$ when keeping the upper linear range. — IAS 11/1/99
 Carryover was noticed in the Meth Blks. — IAS 11/21/99.
 ④ ISTD area counts varied throughout the sample run. Deviations were as high as 54% and as low as -65%. — IAS 11/1/99

Quantity Confirmation Summary Report

TOX15:twan 80 dose confirmation for POA, PTOS, & POA-ant. 10/23/99 las

Sample List: C:\MSD22\Twin\comp20199\pro\sample.htm\102399a

Lab Method: GC/MS/MS 28.10.21.59 1999

Method: C:\MSD22\Twin\comp20199\pro\method.htm\102399a

Last modified: Mon Nov 01 06:21:40 1999

Job Code:

Printed: Mon Nov 01 06:47:34 1999

Compound 1: POA (Ch1)

Sample Name	Sample List	Type	Std Conc	RT	Area	Flags	Conc.	Adv
1 102399001	MEOH BLK	Standard	1.00	7.134	42	bb	3.66	
2 102399002	FCMIX-99086-43-1. 1ppb- 9/10/99 RMW	Standard	1.00	7.088	16	MM	1.02	2
3 102399003	FCMIX-99086-43-2. 5ppb- 9/10/99 RMW	Standard	5.00	7.112	53	MM	1.02	2
4 102399004	FCMIX-99086-43-3. 10ppb- 9/10/99 RMW	Standard	10.00	7.101	108	bb	10.22	2
5 102399005	FCMIX-99086-43-4. 25ppb- 9/10/99 RMW	Standard	25.10	7.082	244	bb	23.74	5
6 102399006	FCMIX-99086-43-5. 50ppb- 9/10/99 RMW	Standard	50.20	7.087	471	bb	46.43	8
7 102399007	FCMIX-99086-43-6. 100ppb- 9/10/99 RMW	Standard	100.00	7.084	981	bb	97.28	3
8 102399008	FCMIX-99086-43-7. 250ppb- 9/10/99 RMW	Standard	251.00	7.113	2481	bbx	246.70	2
9 102399009	FCMIX-99086-43-8. 500ppb- 9/10/99 RMW	Standard	502.00	7.103	4234	bbx	421.45	10
10 102399010	FCMIX-99086-43-9. 750ppb- 9/10/99 RMW	Standard	752.00	7.114	5343	bbx	532.73	2
11 102399011	MEOH BLK	Standard	1.00	7.117	14	MM	0.97	2
12 102399012	TOX145-PTOS/POA in Tween-dose-2ppb- 10/23/99 IAS	QC	25.10	7.132	22	MM	1.67	
13 102399013	TOX145-PTOS/POA in Tween-dose-4ppb- 10/23/99 IAS	QC	25.10	7.134	40	MM	3.51	1
14 102399014	FCMIX-99086-43-4. 25ppb- 9/10/99 RMW	Standard	25.10	7.117	359	bb	25.32	1
15 102399015	MEOH BLK	Standard	1.00	7.131	12	MM	0.70	
16 102399016	TOX145-PTOS in Tween-dose-20ppb- 10/23/99 IAS	QC	251.00	7.112	11	MM	0.61	
17 102399017	TOX145-PTOS in Tween-dose-40ppb- 10/23/99 IAS	QC	251.00	7.128	236	bb	234.37	-6
18 102399018	FCMIX-99086-43-7. 250ppb- 9/10/99 RMW	Standard	251.00	7.110	13	MM	0.73	-27
19 102399019	MEOH BLK	Standard	1.00	7.110	13	MM	0.73	-27
20 102399020	FCMIX-99086-43-1. 1ppb- 9/10/99 RMW	Standard	1.00	7.110	13	MM	0.73	-27
21 102399021	FCMIX-99086-43-2. 5ppb- 9/10/99 RMW	Standard	5.02	7.169	67	MM	6.15	23
22 102399022	FCMIX-99086-43-3. 10ppb- 9/10/99 RMW	Standard	10.00	7.174	113	MM	10.72	7
23 102399023	FCMIX-99086-43-4. 25ppb- 9/10/99 RMW	Standard	25.10	7.180	273	MM	26.64	6
24 102399024	FCMIX-99086-43-5. 50ppb- 9/10/99 RMW	Standard	50.20	7.129	542	bb	51.45	6
25 102399025	FCMIX-99086-43-6. 100ppb- 9/10/99 RMW	Standard	100.00	7.110	1024	MM	101.22	2
26 102399026	FCMIX-99086-43-7. 250ppb- 9/10/99 RMW	Standard	250.00	7.110	1024	MM	101.22	2
27 102399027	FCMIX-99086-43-8. 500ppb- 9/10/99 RMW	Standard	502.00	7.154	418	bbx	418.97	-17
28 102399028	FCMIX-99086-43-9. 750ppb- 9/10/99 RMW	Standard	752.00	7.129	5411	bbx	538.72	-28
29 102399029	FCMIX-99086-43-9. 750ppb- 9/10/99 RMW	Standard	752.00	7.129	5411	bbx	538.72	-28

Soup 020199: Quantitated and printed by ias on 11/01/99

Scanned and printed by law on 11/01/99

Quantity Compound Summary Report

TD115-tween 80 dose confirmation for POLA, PROS, & POLA-enc. 10/29/99 las

Sample List: C:\MSAS\DATA\comp020199\pros\comp020199\10299003

Date modified: Fri Oct 29 10:13:58 1999

Last modified: Mon Nov 01 06:21:40 1999

Job Code: MON NOV 01 06:47:34 1999

Compound 2: PROS (CNS)

# Name	Sample/Label Text	Type	Std Conc	Rf	Area Flag	Conc. Value
1 10299002	MEOH BLK					
2 10299003	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	1.01	7.295	302 bb	1.56
3 10299004	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	5.03	7.231	42 bb	0.00
4 10299005	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	10.00	7.231	148 RM	0.93
5 10299006	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.20	7.246	476 RM	4.74
6 10299007	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	50.30	7.246	913 RM	9.81
7 10299008	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	10.00	7.246	2108 bb	21.71
8 10299009	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	25.20	7.246	432 RM	47.57
9 10299010	FCMIX-99086-43-3, 25ppb- 9/10/99 RMW	Standard	25.20	7.271	4843 RM	48.91
10 10299011	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.20	7.271	32878 bbX	245.04
11 10299012	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	50.30	7.265	41708 bbX	483.87
12 10299013	MEOH BLK					
13 10299014	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	755.00	7.289	56437 bbX	655.48
14 10299015	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	15.20	7.313	170 RM	1.18
15 10299016	FCMIX-99086-43-3, 25ppb- 9/10/99 RMW	Standard	35.20	7.340	424 bb	4.13
16 10299017	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	7.313	7.340	619 bb	6.40
17 10299018	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	7.313	7.318	3288 bb	25.75
18 10299019	MEOH BLK					
19 10299020	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	10.00	7.318	373 RM	0.00
20 10299021	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	25.20	7.318	373 RM	0.00
21 10299022	FCMIX-99086-43-3, 25ppb- 9/10/99 RMW	Standard	25.20	7.318	373 RM	0.00
22 10299023	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.20	7.318	373 RM	0.00
23 10299024	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	25.20	7.318	373 RM	0.00
24 10299025	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	10.00	7.318	373 RM	0.00
25 10299026	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	10.00	7.318	373 RM	0.00
26 10299027	FCMIX-99086-43-3, 25ppb- 9/10/99 RMW	Standard	10.00	7.318	373 RM	0.00
27 10299028	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	10.00	7.318	373 RM	0.00
28 10299029	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	10.00	7.318	373 RM	0.00
29 10299030	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	755.00	7.291	55588 bbX	645.14
30 10299031	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	15.20	7.291	170 RM	1.18
31 10299032	FCMIX-99086-43-3, 25ppb- 9/10/99 RMW	Standard	35.20	7.291	424 bb	4.13
32 10299033	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	7.313	7.291	619 bb	6.40
33 10299034	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	7.313	7.291	3288 bb	25.75
34 10299035	MEOH BLK					
35 10299036	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	10.00	7.295	91 bb	0.37
36 10299037	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	25.20	7.272	158 bb	1.04
37 10299038	FCMIX-99086-43-3, 25ppb- 9/10/99 RMW	Standard	25.20	7.310	510 bb	5.37
38 10299039	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.20	7.315	989 bb	10.70
39 10299040	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	25.20	7.342	2289 bb	25.40
40 10299041	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	10.00	7.295	476 RM	4.74
41 10299042	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	913 RM	9.81
42 10299043	FCMIX-99086-43-3, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	2108 bb	21.71
43 10299044	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	432 RM	47.57
44 10299045	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	4843 RM	48.91
45 10299046	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	25.20	7.295	32878 bbX	245.04
46 10299047	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	41708 bbX	483.87
47 10299048	FCMIX-99086-43-3, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	56437 bbX	655.48
48 10299049	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	170 RM	1.18
49 10299050	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	424 bb	4.13
50 10299051	MEOH BLK					
51 10299052	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	10.00	7.295	619 bb	6.40
52 10299053	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	3288 bb	25.75
53 10299054	FCMIX-99086-43-3, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	424 bb	4.13
54 10299055	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	619 bb	6.40
55 10299056	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	3288 bb	25.75
56 10299057	MEOH BLK					
57 10299058	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	10.00	7.295	91 bb	0.37
58 10299059	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	158 bb	1.04
59 10299060	FCMIX-99086-43-3, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	510 bb	5.37
60 10299061	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	989 bb	10.70
61 10299062	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	2289 bb	25.40
62 10299063	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	10.00	7.295	476 RM	4.74
63 10299064	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	913 RM	9.81
64 10299065	FCMIX-99086-43-3, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	2108 bb	21.71
65 10299066	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	432 RM	47.57
66 10299067	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	4843 RM	48.91
67 10299068	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	25.20	7.295	32878 bbX	245.04
68 10299069	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	41708 bbX	483.87
69 10299070	FCMIX-99086-43-3, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	56437 bbX	655.48
70 10299071	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	170 RM	1.18
71 10299072	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	424 bb	4.13
72 10299073	MEOH BLK					
73 10299074	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	10.00	7.295	619 bb	6.40
74 10299075	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	3288 bb	25.75
75 10299076	FCMIX-99086-43-3, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	424 bb	4.13
76 10299077	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	619 bb	6.40
77 10299078	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	3288 bb	25.75
78 10299079	MEOH BLK					
79 10299080	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	10.00	7.295	91 bb	0.37
80 10299081	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	158 bb	1.04
81 10299082	FCMIX-99086-43-3, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	510 bb	5.37
82 10299083	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	989 bb	10.70
83 10299084	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	2289 bb	25.40
84 10299085	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	10.00	7.295	476 RM	4.74
85 10299086	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	913 RM	9.81
86 10299087	FCMIX-99086-43-3, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	2108 bb	21.71
87 10299088	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	432 RM	47.57
88 10299089	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	4843 RM	48.91
89 10299090	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	25.20	7.295	32878 bbX	245.04
90 10299091	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	41708 bbX	483.87
91 10299092	FCMIX-99086-43-3, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	56437 bbX	655.48
92 10299093	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	170 RM	1.18
93 10299094	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	424 bb	4.13
94 10299095	MEOH BLK					
95 10299096	FCMIX-99086-43-1, 10ppb- 9/10/99 RMW	Standard	10.00	7.295	619 bb	6.40
96 10299097	FCMIX-99086-43-2, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	3288 bb	25.75
97 10299098	FCMIX-99086-43-3, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	424 bb	4.13
98 10299099	FCMIX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.20	7.295	619 bb	6.40
99 10299100	FCMIX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	25.20	7.295	3288 bb	25.75
100 10299101	MEOH BLK					

Quantify Compound Summary Report
 TOX145-tween 80 dose confirmation for PCOA, PFOS, & PFOA- sec. 10/29/99 IAS

Sample List: C:\MSASLTX\comp02199-pro\sample001\102999a
 Last Modified: Fri Oct 28 10:23:59 1999
 Method: C:\MSASLTX\comp02199-pro\methods\102999a
 Last Modified: Mon Nov 01 04:22:40 1999
 Job Code:

Printed: Mon Nov 01 06:47:34 1999

Compound 1: PFOS (CH3)

B Name	Sample/Lot Text	Type	Std Conc	RT	Area	Flags	Conc	Udev
1 102999002	MeOH Bk			7.831	18206	MM	6.15	
2 102999002	MeOH Bk			7.730	435	MM	2.12	
3 102999003	FCMx-99086-43-1, 1ppb- 9/10/99 RMW	Standard	1.01	7.741	1102	MMX	3.10	237
4 102999004	FCMx-99086-43-2, 5ppb- 9/10/99 RMW	Standard	5.03	7.809	1881	MMX	6.16	23
5 102999005	FCMx-99086-43-3, 10ppb- 9/10/99 RMW	Standard	10.10	7.803	2710	MM	9.22	-9
6 102999006	FCMx-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.10	7.805	6408	MM	22.40	-9
7 102999007	FCMx-99086-43-5, 50ppb- 9/10/99 RMW	Standard	50.10	7.786	10741	MM	78.71	-7
8 102999008	FCMx-99086-43-6, 100ppb- 9/10/99 RMW	Standard	100.10	7.766	21619	MM	178.14	-7
9 102999009	FCMx-99086-43-7, 250ppb- 9/10/99 RMW	Standard	250.10	7.857	48815	MM	252.44	-7
10 102999010	FCMx-99086-43-8, 500ppb- 9/10/99 RMW	Standard	500.10	7.895	139095	MM	510.28	-5
11 102999011	FCMx-99086-43-9, 750ppb- 9/10/99 RMW	Standard	750.00	7.941	199487	MM	712.15	-5
12 102999012	MeOH Bk			7.982	2140	MM	7.12	
13 102999013	TCX145-PFOS/PCOA in Tween-dose-2ppb- 10/29/99 IAS			7.920	72407	bb	265.27	
14 102999014	TCX145-PFOS/PCOA in Tween-dose-4ppb- 10/29/99 IAS			7.969	71318	bb	268.62	
15 102999015	FCMx-99086-43-4, 25ppb- 9/10/99 RMW	QC	25.10	7.984	8797	MM	31.57	26
16 102999016	MeOH Bk			7.989	435	MM	2.12	
17 102999017	TCX145-PFOA in Tween-dose-200ppb- 10/29/99 IAS			7.943	72318	MM	265.45	
18 102999018	TCX145-PFOA in Tween-dose-300ppb- 10/29/99 IAS			7.950	105109	db	385.42	
19 102999019	FCMx-99086-43-7, 250ppb- 9/10/99 RMW	QC	251.00	7.954	75327	MM	276.00	10
20 102999020	MeOH Bk			7.990	870	MM	2.45	
21 102999021	FCMx-99086-43-1, 1ppb- 9/10/99 RMW	Standard	1.01	7.947	783	MMX	2.13	111
22 102999022	FCMx-99086-43-2, 5ppb- 9/10/99 RMW	Standard	5.03	8.003	1994	MMX	6.58	31
23 102999023	FCMx-99086-43-3, 10ppb- 9/10/99 RMW	Standard	10.10	7.988	3433	MM	13.87	17
24 102999024	FCMx-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.10	7.943	14768	MM	53.97	7
25 102999025	FCMx-99086-43-5, 50ppb- 9/10/99 RMW	Standard	50.10	7.965	30309	MM	110.62	10
26 102999026	FCMx-99086-43-6, 100ppb- 9/10/99 RMW	Standard	100.10	7.965	76998	MM	282.14	12
27 102999027	FCMx-99086-43-7, 250ppb- 9/10/99 RMW	Standard	250.10	7.995	140797	MM	516.53	3
28 102999028	FCMx-99086-43-8, 500ppb- 9/10/99 RMW	Standard	500.10	7.989	201774	MM	740.56	-2
29 102999029	FCMx-99086-43-9, 750ppb- 9/10/99 RMW	Standard	750.00	7.937				

Soup 020199. Quantitated and printed by ias on 11/01/99

Scup 020199: Quantitated and printed by las on 11/01/99

Quantity Compound Summary Report

TOX145-Tween 80 dose confirmation for POLA, PROA, & POLA- sec. 10/29/99 148

Sample List: C:\MSD\TNC\temp020199-proa\temp020199-sec\102999a

Last modified: Fri Oct 29 10:23:59 1999

Method: C:\MSD\TNC\temp020199-proa\method\102999a

Last modified: Mon Nov 01 06:11:40 1999

Job Code:

Printed: Mon Nov 01 06:47:34 1999

Compound 4: IS (CNS)

# Name	Sample/Lab Test	Type	Std Conc	RT	Area Flag	Conc. Nbr
1 102999001	NEOH B1x					
2 102999002	NEOH B1x					
3 102999003	NEOH B1x					
4 102999004	FCMx-99086-43-1. 10ppb- 9/10/99 RMW	Standard	1.00	7.187	4292 bb	1.48 48
5 102999005	FCMx-99086-43-1. 10ppb- 9/10/99 RMW	Standard	1.00	7.187	4269 bb	1.48 48
6 102999006	FCMx-99086-43-1. 10ppb- 9/10/99 RMW	Standard	1.00	7.095	4424 bb	1.55 52
7 102999007	FCMx-99086-43-1. 25ppb- 9/10/99 RMW	Standard	1.00	7.099	4486 bb	1.54 54
8 102999008	FCMx-99086-43-1. 50ppb- 9/10/99 RMW	Standard	1.00	7.104	4486 bb	1.53 53
9 102999009	FCMx-99086-43-1. 100ppb- 9/10/99 RMW	Standard	1.00	7.106	4093 bb	1.41 41
10 102999010	FCMx-99086-43-1. 250ppb- 9/10/99 RMW	Standard	1.00	7.118	4830 bb	1.41 41
11 102999011	FCMx-99086-43-1. 500ppb- 9/10/99 RMW	Standard	1.00	7.126	4830 bb	0.95 19
12 102999012	FCMx-99086-43-1. 750ppb- 9/10/99 RMW	Standard	1.00	7.145	1842 bb	0.43 17
13 102999013	NEOH B1x					
14 102999014	TOX145-9705/POLA in Tween-dose-2ppb- 10/29/99 IAS					
15 102999015	TOX145-9705/POLA in Tween-ma-10ppb- 10/29/99 IAS					
16 102999016	FCMx-99086-43-1. 25ppb- 9/10/99 RMW	OC	1.00	7.150	2631 bb	0.99 -10
17 102999017	NEOH B1x					
18 102999018	TOX145-9705A in Tween-dose-200ppb- 10/29/99 IAS					
19 102999019	TOX145-9705A in Tween-ma-100ppb- 10/29/99 IAS					
20 102999020	FCMx-99086-43-1. 250ppb- 9/10/99 RMW	OC	1.00	7.146	3529 bb	0.87 41
21 102999021	FCMx-99086-43-1. 10ppb- 9/10/99 RMW	Standard	1.00	7.137	2120 bb	0.80 -20
22 102999022	FCMx-99086-43-1. 5ppb- 9/10/99 RMW	Standard	1.00	7.163	2161 bb	0.81 -19
23 102999023	FCMx-99086-43-1. 10ppb- 9/10/99 RMW	Standard	1.00	7.168	2196 bb	0.83 -17
24 102999024	FCMx-99086-43-1. 25ppb- 9/10/99 RMW	Standard	1.00	7.174	2276 bb	0.78 -22
25 102999025	FCMx-99086-43-1. 50ppb- 9/10/99 RMW	Standard	1.00	7.145	2435 bb	0.84 16
26 102999026	FCMx-99086-43-1. 100ppb- 9/10/99 RMW	Standard	1.00	7.145	2435 bb	0.84 16
27 102999027	FCMx-99086-43-1. 250ppb- 9/10/99 RMW	Standard	1.00	7.174	2059 bb	0.71 -25
28 102999028	FCMx-99086-43-1. 500ppb- 9/10/99 RMW	Standard	1.00	7.166	1934 bb	0.67 -31
29 102999029	FCMx-99086-43-1. 750ppb- 9/10/99 RMW	Standard	1.00	7.146	1818 bb	0.63 -37

Method Report

Page 1

Method file: C:\MASSLYNX\group020199.pro\MethDB\102999a.mdb
Last modified: Mon Nov 01 06:21:40 1999

Compound: 1: POAA (Ch1)

Printed: Mon Nov 01 07:16:09 1999

Compound Name: POAA (Ch1)
Internal Ref.: [None]
Quantity 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): 0.112

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: 90

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

Join valley threshold: 10.00
Reduce peak tailing: 100.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 10.00

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\scup020199.pro\Meth08\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

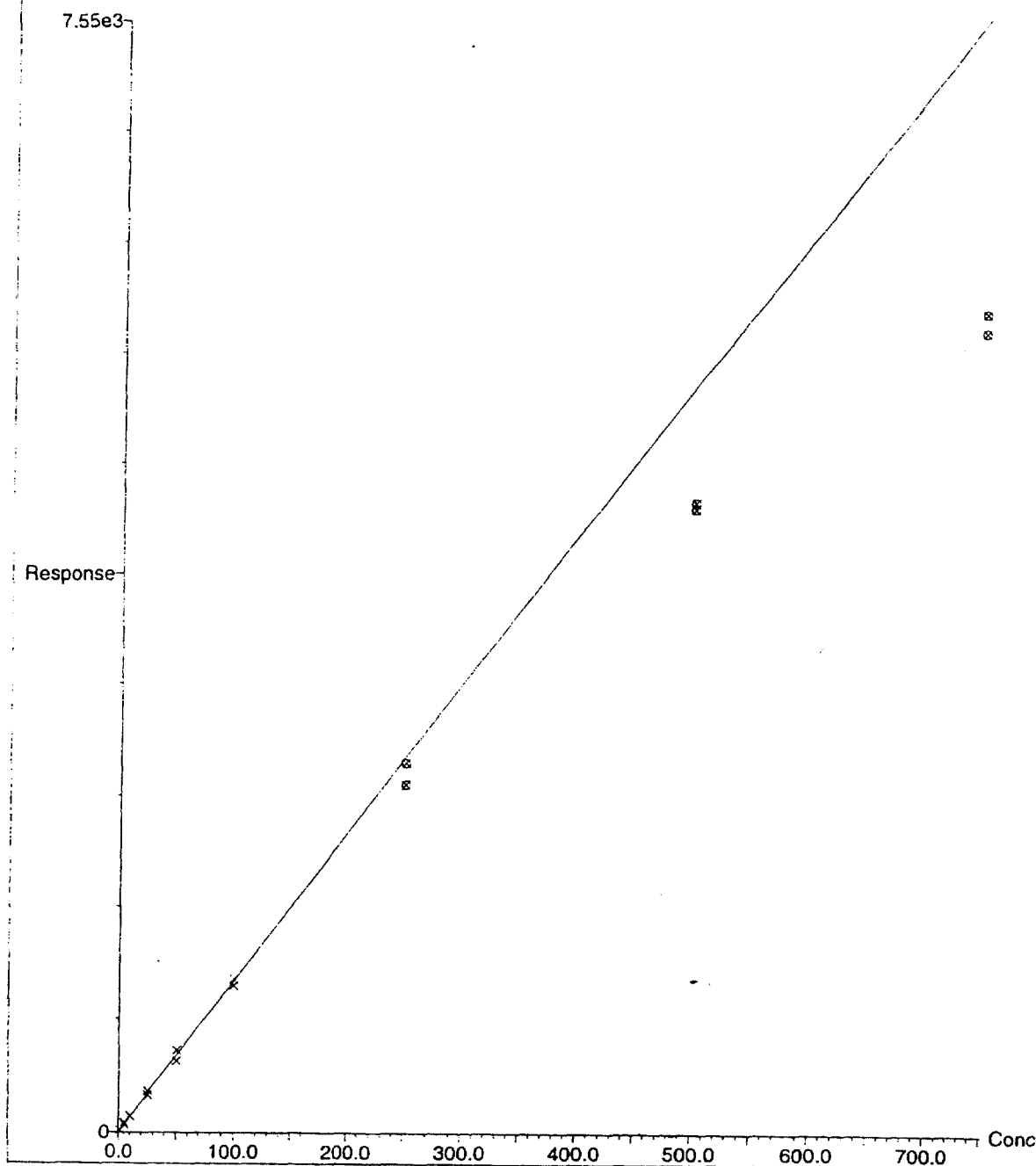
Smoothing: Enabled
Window size (scans): 2
Number of smooths: 1
Smoothing method:

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

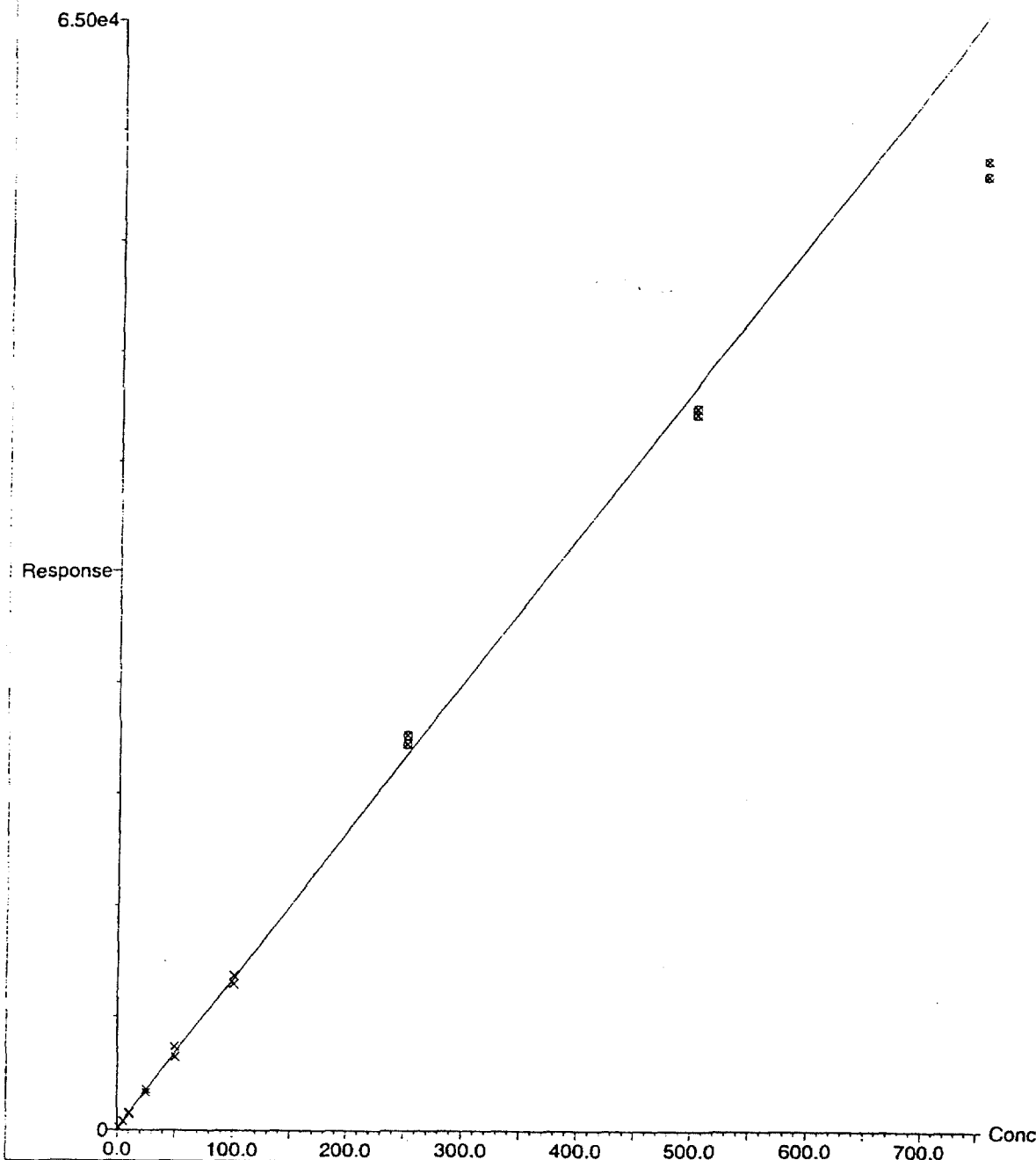
Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 30.00

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

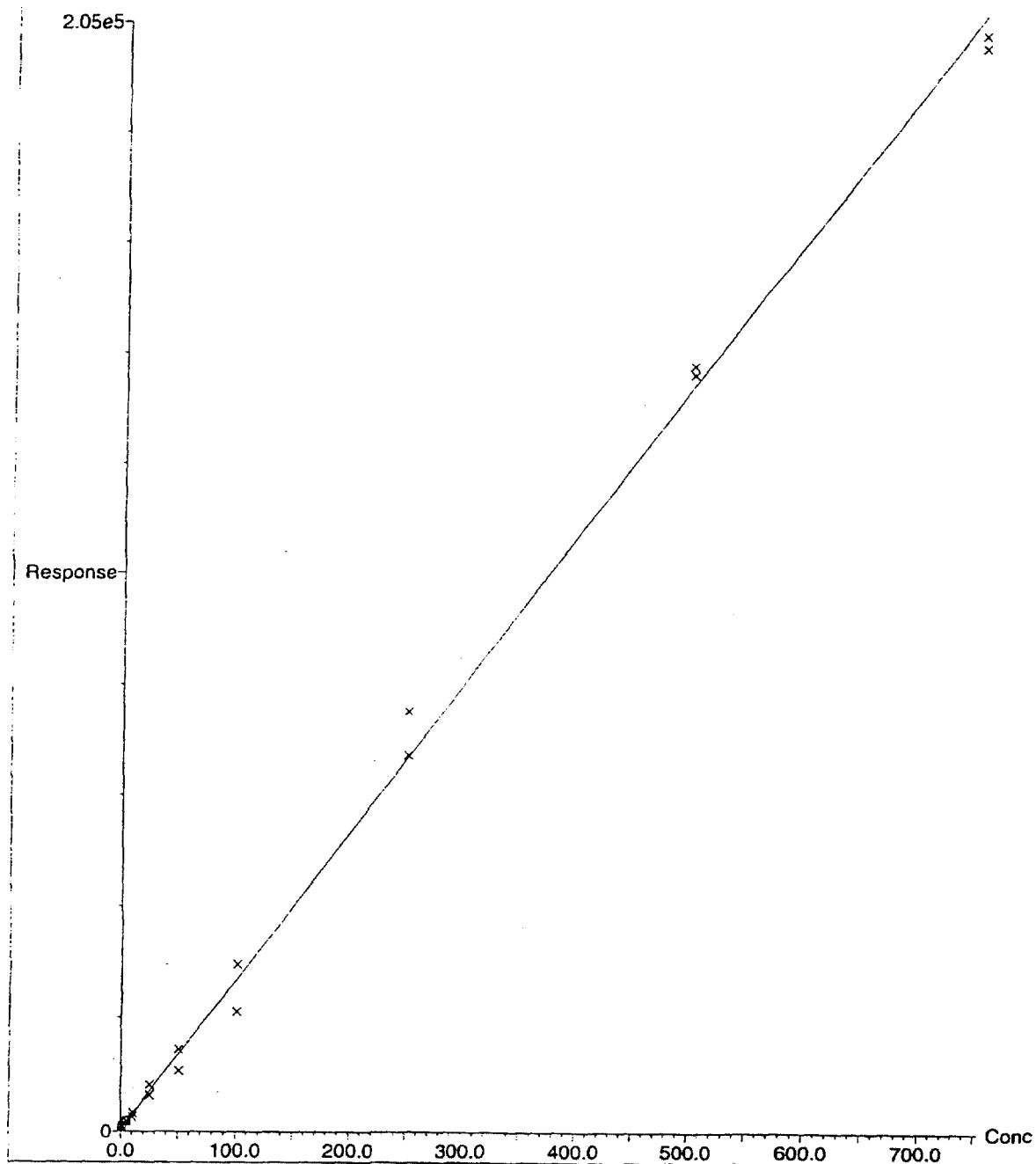
Compound 1 name: POAA (Ch1)
Coefficient of Determination: 0.997121
Calibration curve: $10.0347 * x + 5.23213$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



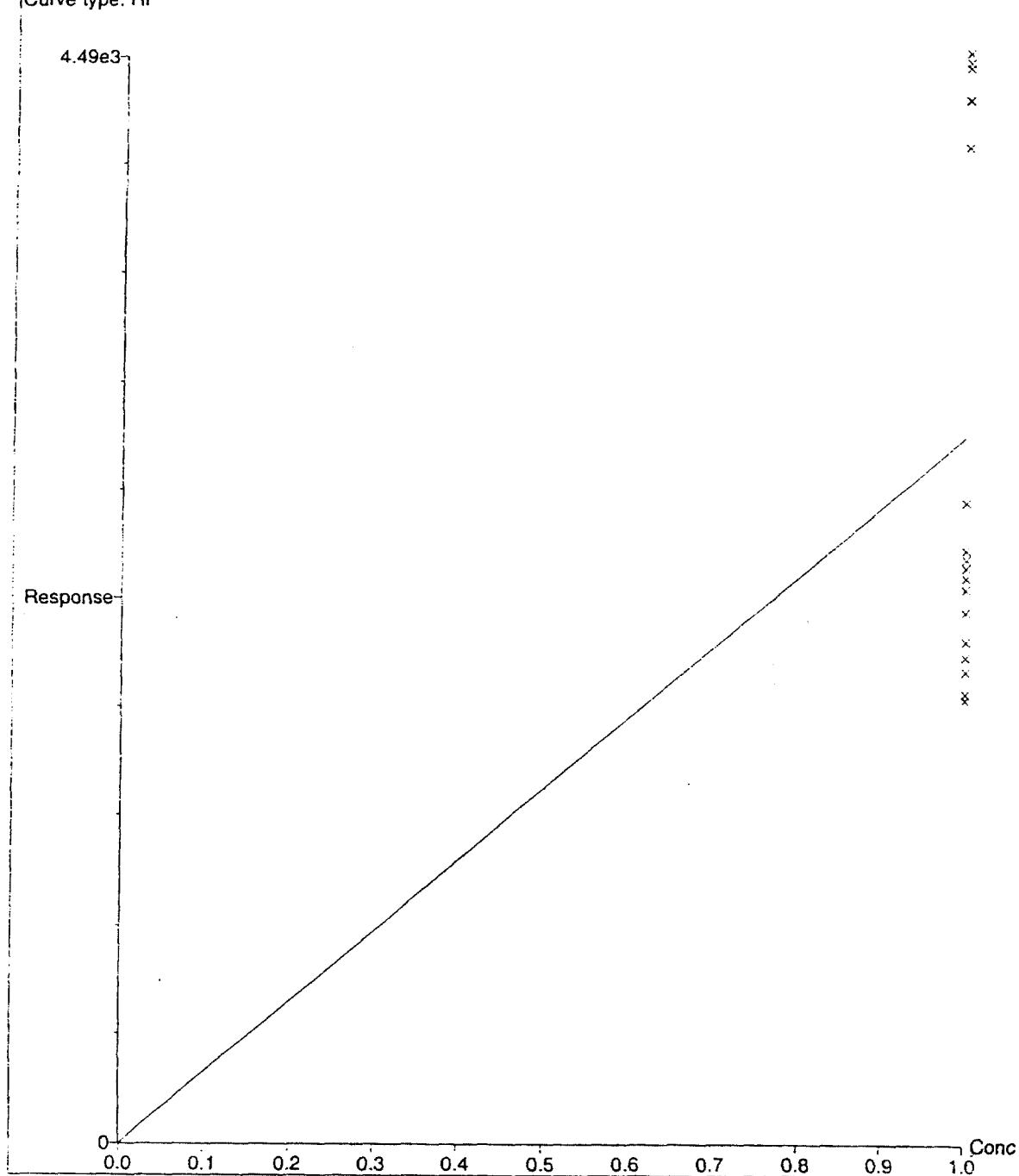
Compound 2 name: PFOS (Ch2)
Coefficient of Determination: 0.996935
Calibration curve: $86.0577 \cdot x + 68.0228$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



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





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>Tax-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>dy 1 & 4</u>	# <u>99086-13-11</u>	# <u>99086-47-2</u>	# <u>99086-47-1</u>	# <u>99095-83</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	-	10-13-99/SL	N/A
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	-	-	-		3AL 10/13/99
9R03232M, Dy1, Control	5 uL	-	-	-		N/A
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 - 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.			10-13-99/SL			
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	10-13-99/SL	Std. # 99086-13-14				10/13/99
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					N/A
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	10-14-99/SL			
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]

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-Sc	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	1.0127			
9R03245M.Dy1.5mg/kg	1.0171		Mult 10/12/99	
9R03246M.Dy1.5mg/kg	1.0074		N/A	
9R03247M.Dy1.5mg/kg	1.0101			
9R03248M.Dy1.5mg/kg	1.0169			
9R03249M.Dy1.5mg/kg	0.9918			
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	1.0187			
		10-12-99	Sc	

Summary of method:

Notes:

Review Date _____

RU10139

Form Approved by/Date:

Form Completion
 Verified By: SAR 10/14/99

Ion Pair Standard Curves--Tissues

Prep Date(s): 10/14/99
 Analyst(s): SEE
 Sample Matrix: R.M. Liver
 Method/Revision: ETS-8-0
 Target Analyte(s): Fc-Nit
 Box Number: 90-200

Study Number: TOX-145
 Equipment Number: 0222-9900
 Final Solvent & TN Number: MeOH/TN-A-2945
 Blank Tissue Identifier: Rabbit Liver/9906-13-5

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	MSS6 Std Conc ug/mL	Am't Spiked mL	Am't Spiked mL	Density g	Am't Spiked mL
0.5000	0.5005	0.4997	0.4999	0.5005	0.5010	0.002	0.002	0.163	1.000
0.5000	0.5050	0.4997	0.4999	0.5005	0.5010	0.004	0.004	0.163	1.000
0.5000	0.5050	0.4997	0.4999	0.5005	0.5010	0.010	0.010	0.163	1.000
0.5000	0.5050	0.4997	0.4999	0.5005	0.5010	0.020	0.020	0.163	1.000
0.5000	0.5050	0.4997	0.4999	0.5005	0.5010	0.040	0.040	0.163	1.000
5.000	5.005	4.997	4.999	5.005	5.010	0.010	0.010	0.163	1.000
5.000	5.005	4.997	4.999	5.005	5.010	0.020	0.020	0.163	1.000
5.000	5.005	4.997	4.999	5.005	5.010	0.030	0.030	0.163	1.000
50.00	50.05	49.97	49.99	50.05	50.10	0.004	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	MSS6 Std Conc ng/g	Surrogate Std Conc ug/mL	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		
30.7	31.0	30.7	30.7	30.7	30.7		
61.3	62.0	61.3	61.3	61.4	61.5		
123	124	123	123	123	123		
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	MSS6 Std Conc ng/mL	Surrogate Std Conc ug/mL	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		
5.00	5.05	5.00	5.00	5.01	5.01		
10.0	10.1	10.0	10.0	10.0	10.0		
20.0	20.2	20.0	20.0	20.0	20.0		
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: 9902230M, 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	MSS6 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	MSS6 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

Sample used for MS/MSD: 9902235M, 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	MSS6 Std Conc ng/g
301	301	301	301	301	302
301	301	301	301	301	302

Sample used for MS/MSD: 03235M, 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	MSS6 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

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 Run 10/18/99

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

Matrix/Timepoint: Rat Liver

[illegible]

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

Form Completion
Verified By: 10-17-99 / Ser

Extraction Worksheet ETS-8-4.1

[illegible]

Extraction Worksheet ETS-8-4.1

Study # <u>713-145</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>02 104</u>	# <u>99062-13-11</u>	# <u>99062-47-2</u>	# <u>99062-47-1</u>	# <u>99062-33</u>		
Date Spiked/Analyst	<u>10-15-99/SK</u>	<u>10-17-99/SK</u>	<u>10-18-99/SK</u>	<u>10-19-99/SK</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	-	-	-		
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</u> Serum Std# <u>99062-34</u> amount = <u>1.0 ml</u>						
Serum Extraction Method <u>ETS-8-4.1</u>					Date & Initials	
Vortex 15 Sec.					<u>10-19-99/SK</u>	
Pipette Matrix, spike with appropriate surrogate or FC-Mix Volume <u>1.0 ml</u>						
Pipette 1 ml of 0.5 M TBA, pH 10. pH= <u>10-10/19/99</u>						
Pipette 2 ml of 0.25 M Na ₂ CO ₃ /0.25 M NaHCO ₃ buffer Std. # <u>99062-23-12</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</u> TN-A- <u>2945</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.						

FN-1: The rat serum blanks + MS/D were from control rat serum 99062/87 10-17-99/SK

Extraction Worksheet ETS-8-4.1

[illegible]

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

[illegible]

Summary of method:

Notes:

Review Date _____

RLS10199

Form Completion
Verified By: MAJ 10/19/99

1st Year Standard Curve-Fluor

Prep Batch: 101000
 Analyte: SE
 Sample Matrix: Rat Serum
 Method/Reagent: ETS-4.1
 Target Analyte(s): PTOS, PTOSA
 Lot Number: 99-206

QC Met Std Approx. 0.500 ppm: 9906-17.2
 QC Met Std Approx. 1.00 ppm: 9906-17.1
 QC Met Std Approx. 50.0 ppm: 9905-1.1
 Storage Std Approx. 100 ppm: 9906-1.11

Study Number: TOX-143
 Equipment Number: MCHVTA-2945
 Fluid Solvent & TV Reservoir: Rat Serum/PTOS-2.15
 Blank Time/Reservoir: Rat Serum/PTOS-2.15
 Rat Serum/PTOS-2.15

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

PTOS	PTOSA	PTOS/A	PTOS/A	EPTOS/OB	MESA	All	All	Final Vol
Std Conc	Std Conc	Std Conc	Std Conc	Std Conc	Std Conc	Am't Spiked	Std Volume	Am't
ug/mL	ug/mL	ug/mL	ug/mL	ug/mL	ug/mL	mL	mL	mL
0.5000	0.5105	0.4997	0.4999	0.5005	0.5010	0.010	1.015	1.00
5.000	5.005	4.997	4.999	5.005	5.010	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
50.00	50.05	49.97	49.99	50.05	50.10	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	0.015	1.015	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

PTOS	PTOSA	PTOS/A	PTOS/A	EPTOS/OB	MESA	Surrogate	All
Std Conc	Std Conc	Std Conc	Std Conc	Std Conc	Std Conc	Am't Spiked	Am't
ug/mL	ug/mL	ug/mL	ug/mL	ug/mL	ug/mL	mL	mL
4.95	4.95	4.95	4.95	4.95	4.95	1.01	0.005
9.76	9.76	9.76	9.76	9.76	9.76	1.01	0.005
24.8	24.8	24.8	24.8	24.8	24.8	1.01	0.005
48.3	48.3	48.3	48.3	48.3	48.3	1.01	0.005
97.6	97.6	97.6	97.6	97.6	97.6	1.01	0.005
248	248	248	248	248	248	1.01	0.005
497	497	497	497	497	497	1.01	0.005
715	715	715	715	715	715	1.01	0.005
976	976	976	976	976	976	1.01	0.005

Calculated Concentrations of Standards in the Sample Matrix

PTOS	PTOSA	PTOS/A	PTOS/A	EPTOS/OB	MESA	Surrogate	All
Std Conc	Std Conc	Std Conc	Std Conc	Std Conc	Std Conc	Am't Spiked	Am't
ug/mL	ug/mL	ug/mL	ug/mL	ug/mL	ug/mL	mL	mL
3.00	3.00	3.00	3.00	3.00	3.00	1.01	0.005
10.0	10.0	10.0	10.0	10.0	10.0	1.01	0.005
33.0	33.0	33.0	33.0	33.0	33.0	1.01	0.005
50.0	50.0	50.0	50.0	50.0	50.0	1.01	0.005
100	100	100	100	100	100	1.01	0.005
250	250	250	250	250	250	1.01	0.005
500	500	500	500	500	500	1.01	0.005
750	750	750	750	750	750	1.01	0.005
1000	1000	1000	1000	1000	1000	1.01	0.005

Sample used for MS/MSD: Rat control serum/PTOS-2.15

Approx. Std Conc: 50.0 ug/mL

PTOS	PTOSA	PTOS/A	PTOS/A	EPTOS/OB	MESA
Final Conc	Final Conc	Final Conc	Final Conc	Final Conc	Final Conc
ug/mL	ug/mL	ug/mL	ug/mL	ug/mL	ug/mL
248	248	248	248	248	248
497	497	497	497	497	497

Am't Spiked

Val (mL)

0.005

Sample used for MS/MSD: Rat control serum/PTOS-2.15

Approx. Std Conc: 50.0 ug/mL

PTOS	PTOSA	PTOS/A	PTOS/A	EPTOS/OB	MESA
Std Conc	Std Conc	Std Conc	Std Conc	Std Conc	Std Conc
ug/mL	ug/mL	ug/mL	ug/mL	ug/mL	ug/mL
250	250	250	250	250	250
500	500	500	500	500	500
750	750	750	750	750	750
1000	1000	1000	1000	1000	1000

Am't Spiked

Val (mL)

0.005

Rat10199

Form Completion
 Verified By: MW 10/19/99

DILUTIONS SUMMARY SHEET

Study: TOX-145

Dilution Date/Analyst: MCH, 10/21/99 *muu*
Box #: 99-206 *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	-	-	-	-	-	-	↓	↓
10/21/99									

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

Form Completion
Verified By: GML 10/21/97

Extraction Worksheet ETS-8-6.0

Q1 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/ul ppm	actual ppm	actual ppm	actual ppm		
WK/Day	#	#	#	#		
Date Spiked/Analyst	10/22/99-RWN	10/22/99-RWN	10/22/99-RWN	10/22/99-RWN		
RAL10225-H2OBlk -1	5.0 ul					
-LiverBlk -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. # 99086-13-4				
Pipette 2 ml of 0.25 M Na ₂ CO ₃ /0.25 M NaHCO ₃ buffer		Std. # 99086-13-28				
Dispense 5 ml of methyl-t-butyl ether		TN-A- 2946				
Shake 20 min	Shaker speed: 500					
Centrifuge 20-25 min.	Centrifuge speed: 1500					
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

Calculated Concentrations of Standards in the 1.0 mL Final volume vial

PFOS	PFOSA	ETFOSE-OH	MISF	MSTF	MeFOSE-OH	POAA	PFHS	Surrogate Std Conc. ug/mL	Anti Spiked (mL)	All
Final Conc. ug/mL	Final Conc. ug/mL	Final Conc. ug/mL	Final Conc. ug/mL	Final Conc. ug/mL	Final Conc. ug/mL	Final Conc. ug/mL	Final Conc. ug/mL	Final Conc. ug/mL	Final Conc. ug/mL	all
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	Surrogate		
5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	Final Conc. ug/mL		
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

PFOS	PFOSA	ETFOSE-OH	MISF	MSTF	MeFOSE-OH	POAA	PFHS
Final Conc. ug/L	Final Conc. ug/L	Final Conc. ug/L	Final Conc. ug/L	Final Conc. ug/L	Final Conc. ug/L	Final Conc. ug/L	Final Conc. ug/L
300	300	300	300	300	300	300	300
300	300	300	300	300	300	300	300

Approx. Std Conc.: 1.00 ug/mL
Anti Spiked (mL): 0.010
Density: 0.167

Sample used for MS/MSD: No MS/MSD

MS/MSD calc. in matrix

PFOS	PFOSA	ETFOSE-OH	MISF	MSTF	MeFOSE-OH	POAA	PFHS
Final Conc. ug/L	Final Conc. ug/L	Final Conc. ug/L	Final Conc. ug/L	Final Conc. ug/L	Final Conc. ug/L	Final Conc. ug/L	Final Conc. ug/L
50	50	50	50	50	50	50	50
50	50	50	50	50	50	50	50

Approx. Std Conc.: 1.00 ug/mL
Anti Spiked (mL): 0.010
Final Vol.: 1.000

195E-10259L-9908-44-1 sh

See 10/25/99

Ion Pair Standard Curves--Tissues

Prep Date: 10/25/99
 Analyst: RWH
 Sample Matrix: Rabbit Liver
 Method/Review: ETS-4-6-0
 Target Analyte(s): See below
 Bag Number: 99-206

Study Number: FOX145
 Equipment Number:
 Final Solvent & TV Number: MCH/NTX-1345
 Rabbit Liver/9906-135

EC Mix Std Apprxl. 0.500 ppm:
 EC Mix Std Apprxl. 1.000 ppm:
 EC Mix Std Apprxl. 50.0 ppm:
 Surrogate Std Apprxl. 101 ppm:

9906-14-1
 9906-14-1
 9906-14-1
 9906-13-11

Actual Concentrations of Standards in the EC Mix

PFOS	PFOSA	EtFOSE-OH	MESA	MSTB	MeFOSE-OH	PDAA	PFBS
Std Conc. ug/mL	Std Conc. ug/mL	Std Conc. ug/mL	Std Conc. ug/mL	Std Conc. ug/mL	Std Conc. ug/mL	Std Conc. ug/mL	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
3.0004	3.0000	3.0000	3.0000	3.0000	3.0000	3.0000	3.0000
3.0004	3.0000	3.0000	3.0000	3.0000	3.0000	3.0000	3.0000
50.04	50.00	50.00	50.00	50.00	50.00	50.00	50.00

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

0.163
 1.000

All	All	All
Am's Spiked	Density	Vol.
mL	g	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
0.100	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	PFOSA	EtFOSE-OH	MESA	MSTB	MeFOSE-OH	PDAA	PFBS
Std Conc. ug/g	Std Conc. ug/g	Std Conc. ug/g	Std Conc. ug/g	Std Conc. ug/g	Std Conc. ug/g	Std Conc. ug/g	Std Conc. ug/g
6.14	6.13	6.13	6.13	6.13	6.13	6.13	6.13
123	123	123	123	123	123	123	123
307	307	307	307	307	307	307	307
614	613	613	613	613	613	613	613
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

Surrogate	All
Std Conc. ug/mL	Am's Spiked
101	mL
3098	0.005

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						
R4510229	H2OBik -1	5.0 ul	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	
10/22/99 RWW						
Blank R4510229 Std# TNA-2382 amount = 1.0 ml						
Serum Extraction Method ETS-8-4.1 Date & Initials						
Vortex 15 Sec. 10/23/99 - RWW						
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 10/23/99-RWW Std. # 95086-15-16						
Pipette 2 ml of 0.25 M Na ₂ CO ₃ /0.25 M NaHCO ₃ buffer Std. # 95086-14-08						
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 - RWW						
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—Fluors

Prep Date: 10/25/99
 Analyst: RWW
 Sample Matrix: Rabbit Serum
 Method/Revision: ETS-8-1
 Target Analyte(s): Below
 Box Number: 99-206

Study Number: TOX145
 Equipment Number:
 Final Solvent & TN Number: MeOH/TNA-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	MSSA Std Conc. ug/mL	MSTB 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!	#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	MSSA Std Conc. ng/mL	MSTB Std Conc. ng/mL	MeFOSEOH Std Conc. ng/mL	POAA Std Conc. ng/mL	PFHS Std Conc. ng/mL	Surrogate Std Conc. ng/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	MSSA Final Conc. ng/mL	MSTB Final Conc. ng/mL	MeFOSEOH Final Conc. ng/mL	POAA Final Conc. ng/mL	PFHS Final Conc. ng/mL	Surrogate Final Conc. ng/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	505	
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

PFOS Final Conc. ng/mL	PFOSA Final Conc. ng/mL	EtFOSEOH Final Conc. ng/mL	MSSA Final Conc. ng/mL	MSTB Final Conc. ng/mL	MeFOSEOH Final Conc. ng/mL	POAA Final Conc. ng/mL	PFHS Final Conc. ng/mL
248	248	248	248	248	248	248	248
248	248	248	248	248	248	248	248

Approx. Std Conc.: 50 ug/ml

Am't Spiked
Vol. (mL)
0.005

Sample used for MS/MSD: No MS/MSD

MS/MSD Conc. in the final solvent

PFOS Std Conc. ug/mL	PFOSA Std Conc. ug/mL	EtFOSEOH Std Conc. ug/mL	MSSA Std Conc. ug/mL	MSTB 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

Approx. Std Conc.: 50ug/ml

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

See
10/25/99

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: 70X145
 Equipment Number:
 Notebook# and Page:
 Final Solvent: MESH TN-A-2117

Procedure Detail

A PFOS Sample, TN-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.

Std's used

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

ISTD - 99086-13-11, 101 ppm

PFOS/POAA

(amt of PFOS in sample)

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

Dose + MS :

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

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

> 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): 10/29/11
 Analyst(s): IAS
 Sample Matrix: Tween 80
 Method/Revision:
 Target Analyte(s): PFOS/PAA
 PFOSA

Study Number: T0X145
 Equipment Number:
 Notebook# and Page:
 Final Solvent: MeOH - DNA-2547

Procedure Detail

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

Std used:

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

PFOS/PAA

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

$$\begin{aligned} \text{Dose + MS} : & 1000 \times 10 \text{ ppm} \times \frac{5 \text{ mL}}{25 \text{ mL}} = 2 \text{ ppb} \\ & + 1000 \times 0.100 \text{ mL} = 2 \text{ ppb} \end{aligned} \rightarrow 4 \text{ ppb}$$

PFOSA:

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

$$\begin{aligned} \text{Dose + MS} : & 100 \text{ ppm} \times \frac{0.025 \text{ mL}}{25 \text{ mL}} \times 1000 = 100 \text{ ppb} \\ & + 100 \text{ ppm} \times \frac{5 \text{ mL}}{25 \text{ mL}} \times 1000 = 200 \text{ ppb} \end{aligned} \rightarrow 300 \text{ ppb}$$

Matrix Spikes IAS 10/29/11 RE

* All samples were spiked with surrogate 99086-13-11, 101 ppm.
 The spike was 50 µL of 100 ppm diluted to 1000 µL to give final conc. in the MS at 202 ppb. 25 mL IAS RE 10/29/11
 This Dose was corrected with a 0.100 spike to give a final conc. of 60.5 ppb.

Spiked incorrectly
 IAS 11/26/11

IAS 11/19/11

Sample Extraction Sheet

Prep Date(s): 12/22/99

Analyst(s): JAS

Sample Matrix: TERN

Method/Revision:

Target Analyte(s): PFOSA
POAA/POA

Study Number: T-145

Equipment Number:

Notebook and Page

Final Solvent: Hex: TN-A-797

Procedure Detail

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

SHE USED

PFOSA = 99086-12-14 at 100.1 ppm - cap 3/60

POA/POAA = 99086-37-3 at 0.5 ppm - cap 3/60

PFOS/POAA =

Dose: 100 ppb x (0.20 mL) = 20 ppb
10 mL

Dose + MS: 2.50 ppm x 40 µL x 1000 = 20 ppb
10 mL
+ 100 ppb x (0.20 mL) = 20 ppb
(PFOS/POAA std) 10 mL
40 ppb

PFOSA =

Dose: 10 ppm x 0.20 mL x 1000 = 200 ppb
10 mL

Dose + MS: 10 ppm x 0.20 mL x 1000 = 200 ppb
10 mL
+ 100 ppm x 0.02 mL x 1000 = 100 ppb
(PFOSA std) 10 mL
300 ppb
FAS @ 10/2/99
A 300 ppb

* All samples were spiked with surrogate 99086-17-11, 10 ppm.
The spike was blank + a 100 ppm diluted in 10 mL to give a final conc at 555 ppb

JAS 11/19/99

Form Approved by Date:

Extraction-I Template

Review Date

Extraction.xls

Page 1 of

Extraction Worksheet ETS-8-4.1

Study # TOX 145	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: ppm	actual: ppm	actual: ppm	actual: ppm		
WK/Day: Day 29	# 99086-13-11	# 99086-47-2	# 99086-47-1	# 99086-33		
Date Spiked/Analyst	11/11/99				11/11/99	
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
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# 99086-94	amount = 1.0 ml				
Serum Extraction Method ETS-8-4.1					Date & Initials	
Vortex 15 Sec.					11/11/99	
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	Std. # 99086-14-15					11/3/99
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-28416					
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
Add methanol	Volume 1.0 ml	TN-A-28416				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 99086-33C. 11/3/99

Ion Pair Standard Curves--Serum

Prep Date(s): 11/03/99
 Analyst(s): MCH
 Sample Matrix: Rat Sera
 Method/Revision: ETS-4-1
 Target Analyte(s): PFOS, PFOA
 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	PFOA Std Conc. ug/mL	PFOSAA Std Conc. ug/mL	EtFOSE Std Conc. ug/mL	PFOSEA Std Conc. ug/mL	MSS6 Std Conc. ug/mL	All Am't Spiked mL	All Initial Volume 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	PFOA Std Conc. ng/mL	PFOSAA Std Conc. ng/mL	EtFOSE Std Conc. ng/mL	PFOSEA Std Conc. ng/mL	MSS6 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 Final Conc. ng/mL	
24.8	24.8	24.7	24.7	24.8	24.8	498	
49.3	49.5	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	PFOA Final Conc. ng/mL	PFOSAA Final Conc. ng/mL	EtFOSE Final Conc. ng/mL	PFOSEA Final Conc. ng/mL	MSS6 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 Final Conc. ng/mL	
25.0	25.0	25.0	25.0	25.0	25.1	505	
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: TCR99062-30

MS/MSD Conc. in the sample matrix

Initial Volume (mL): 1.00

PFOS Final Conc. ng/mL	PFOA Final Conc. ng/mL	PFOSAA Final Conc. ng/mL	EtFOSE Final Conc. ng/mL	PFOSEA Final Conc. ng/mL	MSS6 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

Initial Volume (mL): 1.00

PFOS Std Conc. ug/mL	PFOA Std Conc. ug/mL	PFOSAA Std Conc. ug/mL	EtFOSE Std Conc. ug/mL	PFOSEA Std Conc. ug/mL	MSS6 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

Ver. hi.
by 4/03/99
Jen

DILUTIONS SUMMARY SHEET

Study: TOX145

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

sample number or description	DILUTIONS							Comments	Verified By
	1:10	1: NA	1: NA	1: NA	1: NA	1: NA	1: NA		
9R03255-5mg/kg-Day 29	D10	-	-	-	-	-	-	N/A	See 11/5/99
9R03256-5mg/kg-Day 29	D10	-	-	-	-	-	-		N/A
9R03257-5mg/kg-Day 29	D10	-	-	-	-	-	-		
9R03258-5mg/kg-Day 29	D10	-	-	-	-	-	-		
9R03259-5mg/kg-Day 29	D10	-	-	-	-	-	-		

Notes: D10=100 uL sample + 900 uL MeOH. μ ex 11 5 99

Form Completion
Verified By: Sul 11/15/99

Extraction Worksheet ETS-8-6.0

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	# 99086-14-2	# 99086-14-2	# 99086-14-1		
Date Spiked/Analyst	11/1/99 - R.W.	11/1/99 - R.W.	11/1/99 - R.W.	11/1/99 - R.W.	11/1/99 - R.W.	
RAL11049 - CCV-1-1-2	5.0 ul		10 ul			NA
99086-140 -MS-	5.0 ul		10 ul			
MSD-	5.0 ul		10 ul			
RAL11049 -H2OBik-1&2	5.0 ul					11/1/99
-LiverBik-1&2	5.0 ul					NA
-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.						
11/1/99 11/1/99						
Blank - R.B. + Liver Std# 99086-13-5 amount = 1.0 ml 0.163 g/ml NA						
Liver Extraction Method ETS-8-6.0 Date & Initials						
Vortex 15 Sec. 11/1/99 - R.W.						
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. # 99086-14-15 11/1/99 - R.W.						
Pipette 2 ml of 0.25 M Na ₂ CO ₃ /0.25 M NaHCO ₃ buffer Std. # 99086-14-01						
Dispense 5 ml of methyl-1-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-2992						
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.						

Sample Weights/ Volumes

Prep Date(s): 11/3/99
Analyst(s): RWW
Sample Matrix: Rat Liver
Method/Revision: ETS-8-6.0
Target Analyte(s): PFOS, PFOSA

Study Number: TOX145
Equipment Number:
Final Solvent & TN Number: MQ Water
Matrix Blank/Identifier: Rabbit Liver/99086-13-5
Box: 99-211

[illegible]

Summary of method:

Notes:

Form Approved by/Date: _____

Review Date _____

Weight Volume Sheet-101299.xls

Form Completion
Verified By: MU 11/04/99

[illegible]

Serraglate Std Conc. µg/mL	All Aunt Spiked mL
101	0.003
Serraglate Final Conc. µg/mL	
505	

MS/MISD code. In practice

Initial Volume (mL): 5.00

Approx. Std Conc.: 5.00 ug/mL
Amt Spiked (mL)

Spiked (mL)	Density
0.010	0.169

0.010 0.169

MS/MSD calc. for solvent

Initial Volume (mL): 5.00

Approx. Std Conc.: 5.00 ug/mL
Aven't Spilled (mL):

Spiked (mL)	Final Volume (mL)
0.010	1.000

	1.000	0.010
...	...	...

IPSE-10259Liv-99086-44-1.xls

with a by
Alist 11/11/97

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

sample number or description	DILUTIONS							Comments	Verified By
	1: 100	1:	1:	1:	1:	1:	1:		
9RO3255/M/Day29/5mg/kg	0100							N/A	MM 11/5/99
9RO3256/M/Day29/5mg/kg	↓							↓	↓
9RO3257/M/Day29/5mg/kg									
9RO3258/M/Day29/5mg/kg									
9RO3259/M/Day29/5mg/kg	↓							↓	↓
5mg 11/5/99									

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

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

FACT: DATA REVIEW SUMMARY FORM

Data Review Sign-Off

Date: 10/20/99
 Data Analyst: JAS
 FACT Study #: FOXMS
 Matrix/Timepoints: Ext Sers - Day 144 - Control 1/2 dose - ext 10/19/99 SEE
 Sample List: 101988h
 Set-up Analysis
 Date/Analyst: 10/19/99 MMH
 Instrument: Samp 070199

Analyst Review
 Name / Date: JAS 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: PFOA (CH2) ②
 Acceptable Linear Range: 9.76 ppb → 976 ppb
 LOQ: 9.76 ppb Extracted Blank: less than LOQ
 R²: 0.998442 Matrix Blank: less than LOQ
 Calibration Checks: OK - Range from -16% → 16%
 MS/MSD: OK
 Data Status: Dilutions needed for dosed animals LAC 10/21/99

Analyte: PFOA (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 LAC 10/21/99

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

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

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

FACT: DATA REVIEW SUMMARY FORM

Date: 10/2/99
 Data Analyst: IAS
 FACT Study #: Tox145
 Matrix/Timepoints: Plasma - Day 14 - Control & dose - Oct. 7/11/99 SEE
 Sample List: 101996
 Set-up Analysis
 Date/Analyst: 10/19/99 MMH
 Instrument: Samp 020195

Data Review Sign-Off

Analyst Review
 Name / Date: IAS 10/2/99

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) 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 10/2/99
 MS/MSD: _____
 Data Status: _____

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

① Internal std area counts look
 consistent. All samples, curves, & cal
 checks had deviations of 30% or less.
 — IAS 10/2/99

LAC 10/21/99

Quantify Compound Summary Report
 TOX145-ret sera, day 1 & 4, Control & dose- ext. 10/19/99 SEH

Page 1

Sample List: C:\MSD\SL\TK\comp-race.pro\SampleData\101999b
 Last modified: Wed Oct 20 07:37:23 1999
 Method: C:\MSD\SL\TK\comp-race.pro\Method\101999b
 Last modified: Wed Oct 20 07:36:40 1999
 Job Code:
 Printed: Wed Oct 20 08:00:10 1999

Compound 1: PMAA (Chl)

# Name	Sample/Ret	Type	Std Conc	RT	Area	Flags	Conc.	%Dev
1 101999012	MeOH Blank							
2 101999013	MeOH Blank							
3 101999014	MeOH Blank							
4 101999015	TOX145-RAS10199-MCO Blank-11, SEH			7.085	37 MM		0.88	-12
5 101999016	TOX145-RAS10199-Sera Blank-11, SEH			7.123	38 MM		0.51	-9
6 101999017	TOX145-RAS10199-Sera Blank-11, SEH			7.121	34 MM		0.81	-19
7 101999018	TOX145-RAS10199-Sera Blank-12, SEH			7.112	38 MM		0.90	-10
8 101999019	TOX145-RAS10199-50ppb-11, SEH	Standard	1.00	7.084	35 MM		0.83	-17
9 101999020	TOX145-RAS10199-50ppb-11, SEH	Standard	1.00	7.111	42 MM		0.99	-1
10 101999021	TOX145-RAS10199-250ppb-11, SEH	Standard	1.00	7.109	53 MM		0.77	-23
11 101999022	TOX145-RAS10199-50ppb-11, SEH	Standard	1.00	7.108	42 MM		1.00	0
12 101999023	TOX145-RAS10199-100ppb-11, SEH	Standard	1.00	7.112	39 MM		0.92	-8
13 101999024	TOX145-RAS10199-250ppb-11, SEH	Standard	1.00	7.110	39 MM		0.93	-7
14 101999025	TOX145-RAS10199-500ppb-11, SEH	Standard	1.00	7.111	51 MM		1.21	21
15 101999026	TOX145-RAS10199-750ppb-11, SEH	Standard	1.00	7.112	42 MM		1.00	0
16 101999027	TOX145-RAS10199-1000ppb-11, SEH	Standard	1.00	7.111	60 MM		1.41	41
17 101999028	MeOH Blank							
18 101999029	TOX145-RTS, 9803234M, Dy1, Control, -10/19/99 SEH			7.090	30 MM		0.71	-29
19 101999030	TOX145-RTS, 9803232M, Dy1, Control, -10/19/99 SEH			7.121	37 MM		0.87	-13
20 101999031	TOX145-RTS, 9803232M, Dy1, Control, -10/19/99 SEH			7.110	36 MM		0.85	-15
21 101999032	TOX145-RTS, 9803233M, Dy1, Control, -10/19/99 SEH			7.086	43 MM		1.00	-0
22 101999033	TOX145-RTS, 9803234M, Dy1, Control, -10/19/99 SEH			7.113	43 MM		1.02	2
23 101999034	TOX145-RAS10199-250ppb-CCV-11-1, SEH	QC	1.00	7.089	34 MM		0.81	-19
24 101999035	MeOH Blank							
25 101999036	TOX145-RTS, 9803233M, Dy4, Control, -10/19/99 SEH			7.133	29 MM		0.64	-32
26 101999037	TOX145-RTS, 9803234M, Dy4, Control, -10/19/99 SEH			7.112	37 MM		0.88	-12
27 101999038	TOX145-RTS, 9803237M, Dy4, Control, -10/19/99 SEH			7.129	34 MM		0.81	-19
28 101999039	TOX145-RTS, 9803232M, Dy4, Control, -10/19/99 SEH			7.112	33 MM		0.77	-23
29 101999040	TOX145-RTS, 9803235M, Dy4, Control, -10/19/99 SEH			7.106	30 MM		0.71	-28
30 101999041	TOX145-RAS10199-250ppb-CCV-11-2, SEH	QC	1.00	7.105	40 MM		0.96	-4
31 101999042	MeOH Blank							
32 101999043	TOX145-RAS10199-250ppb-RSD-11, 10/19/99 SEH			7.122	56 MM		1.32	32
33 101999044	TOX145-RAS10199-250ppb-RSD-11, 10/19/99 SEH			7.123	35 MM		0.83	-17
34 101999045	TOX145-RTS, 9803235M, Dy1, 5 mg/kg 10/19/99 SEH			7.090	308 MM		7.28	626
35 101999046	TOX145-RTS, 9803234M, Dy1, 5 mg/kg 10/19/99 SEH			7.111	425 MM		10.06	906
36 101999047	TOX145-RTS, 9803237M, Dy1, 5 mg/kg 10/19/99 SEH			7.112	360 MM		8.52	752
37 101999048	TOX145-RAS10199-250ppb-CCV-11-3, SEH	QC	1.00	7.107	42 MM		1.00	-0
38 101999049	MeOH Blank							
39 101999050	TOX145-RTS, 9803234M, Dy1, 5 mg/kg 10/19/99 SEH			7.104	391 MM		6.88	588
40 101999051	TOX145-RTS, 9803235M, Dy1, 5 mg/kg 10/19/99 SEH			7.090	340 MM		8.04	704
41 101999052	TOX145-RTS, 9803235M, Dy4, 5 mg/kg 10/19/99 SEH			7.108	210 MM		4.97	397
42 101999053	TOX145-RTS, 9803231M, Dy4, 5 mg/kg 10/19/99 SEH			7.103	209 MM		4.93	393
43 101999054	TOX145-RTS, 9803232M, Dy4, 5 mg/kg 10/19/99 SEH			7.110	260 MM		6.15	515
44 101999055	TOX145-RAS10199-250ppb-CCV-11-4, SEH	QC	1.00	7.113	31 MM		0.73	-27
45 101999056	MeOH Blank							
46 101999057	TOX145-RTS, 9803234-1M, Dy4, 5 mg/kg 10/19/99 SEH			7.110	248 MM		5.88	488
47 101999058	TOX145-RTS, 9803234-2M, Dy4, 5 mg/kg 10/19/99 SEH			7.113	234 MM		5.55	455
48 101999059	TOX145-RAS10199-250ppb-CCV-11-5, SEH	QC	1.00	7.111	39 MM		0.92	-8
49 101999060	MeOH Blank							
50 101999061	TOX145-RAS10199-MCO Blank-11, SEH			7.109	28 MM		0.66	-34
51 101999062	TOX145-RAS10199-Sera Blank-11, SEH			7.129	38 MM		0.89	-11
52 101999063	TOX145-RAS10199-Sera Blank-11, SEH			7.110	28 MM		0.66	-34
53 101999064	TOX145-RAS10199-Sera Blank-12, SEH			7.091	44 MM		1.04	4
54 101999065	TOX145-RAS10199-50ppb-11, SEH	Standard	1.00	7.113	31 MM		0.73	-27
55 101999066	TOX145-RAS10199-100ppb-11, SEH	Standard	1.00	7.134	37 MM		0.87	-13
56 101999067	TOX145-RAS10199-250ppb-11, SEH	Standard	1.00	7.122	34 MM		0.81	-19
57 101999068	TOX145-RAS10199-500ppb-11, SEH	Standard	1.00	7.111	40 MM		0.95	-5
58 101999069	TOX145-RAS10199-1000ppb-11, SEH	Standard	1.00	7.126	37 MM		0.88	-12
59 101999070	TOX145-RAS10199-250ppb-11, SEH	Standard	1.00	7.111	45 MM		1.07	7
60 101999071	TOX145-RAS10199-500ppb-11, SEH	Standard	1.00	7.111	37 MM		0.87	-13
61 101999072	TOX145-RAS10199-750ppb-11, SEH	Standard	1.00	7.107	53 MM		1.26	26
62 101999073	TOX145-RAS10199-1000ppb-11, SEH	Standard	1.00	7.110	64 MM		1.51	51

Quantity Compound Summary Report

TUM-100-000, day 3 & 4, control & dose, em. 10/15/99 SEE

Sample Size: C:\MSD\TUM-100-000\data\sum\sum\sum\101599b

Last Modified: Wed Oct 20 07:31:33 1999

Created: C:\MSD\TUM-100-000\data\sum\sum\sum\101599b

Modified: Wed Oct 20 07:36:48 1999

Job Code:

Printed: Wed Oct 20 06:08:16 1999

Compound 3: 9900 (Ch3)

9	101599012	MOON Blank	Sampling Test	Type	Std Conc	RT	Area Peak	Conc. (ug/g)
1	101599012	MOON Blank				7.274	134 MB	0.00
2	101599012	MOON Blank				7.269	11 MB	0.00
3	101599012	MOON Blank				7.269	11 MB	0.00
4	101599012	MOON Blank				7.269	11 MB	0.00
5	101599012	MOON Blank				7.269	11 MB	0.00
6	101599012	MOON Blank				7.269	11 MB	0.00
7	101599012	MOON Blank				7.269	11 MB	0.00
8	101599012	MOON Blank				7.269	11 MB	0.00
9	101599012	MOON Blank				7.269	11 MB	0.00
10	101599012	MOON Blank				7.269	11 MB	0.00
11	101599012	MOON Blank				7.269	11 MB	0.00
12	101599012	MOON Blank				7.269	11 MB	0.00
13	101599012	MOON Blank				7.269	11 MB	0.00
14	101599012	MOON Blank				7.269	11 MB	0.00
15	101599012	MOON Blank				7.269	11 MB	0.00
16	101599012	MOON Blank				7.269	11 MB	0.00
17	101599012	MOON Blank				7.269	11 MB	0.00
18	101599012	MOON Blank				7.269	11 MB	0.00
19	101599012	MOON Blank				7.269	11 MB	0.00
20	101599012	MOON Blank				7.269	11 MB	0.00
21	101599012	MOON Blank				7.269	11 MB	0.00
22	101599012	MOON Blank				7.269	11 MB	0.00
23	101599012	MOON Blank				7.269	11 MB	0.00
24	101599012	MOON Blank				7.269	11 MB	0.00
25	101599012	MOON Blank				7.269	11 MB	0.00
26	101599012	MOON Blank				7.269	11 MB	0.00
27	101599012	MOON Blank				7.269	11 MB	0.00
28	101599012	MOON Blank				7.269	11 MB	0.00
29	101599012	MOON Blank				7.269	11 MB	0.00
30	101599012	MOON Blank				7.269	11 MB	0.00
31	101599012	MOON Blank				7.269	11 MB	0.00
32	101599012	MOON Blank				7.269	11 MB	0.00
33	101599012	MOON Blank				7.269	11 MB	0.00
34	101599012	MOON Blank				7.269	11 MB	0.00
35	101599012	MOON Blank				7.269	11 MB	0.00
36	101599012	MOON Blank				7.269	11 MB	0.00
37	101599012	MOON Blank				7.269	11 MB	0.00
38	101599012	MOON Blank				7.269	11 MB	0.00
39	101599012	MOON Blank				7.269	11 MB	0.00
40	101599012	MOON Blank				7.269	11 MB	0.00
41	101599012	MOON Blank				7.269	11 MB	0.00
42	101599012	MOON Blank				7.269	11 MB	0.00
43	101599012	MOON Blank				7.269	11 MB	0.00
44	101599012	MOON Blank				7.269	11 MB	0.00
45	101599012	MOON Blank				7.269	11 MB	0.00
46	101599012	MOON Blank				7.269	11 MB	0.00
47	101599012	MOON Blank				7.269	11 MB	0.00
48	101599012	MOON Blank				7.269	11 MB	0.00
49	101599012	MOON Blank				7.269	11 MB	0.00
50	101599012	MOON Blank				7.269	11 MB	0.00
51	101599012	MOON Blank				7.269	11 MB	0.00
52	101599012	MOON Blank				7.269	11 MB	0.00
53	101599012	MOON Blank				7.269	11 MB	0.00
54	101599012	MOON Blank				7.269	11 MB	0.00
55	101599012	MOON Blank				7.269	11 MB	0.00
56	101599012	MOON Blank				7.269	11 MB	0.00
57	101599012	MOON Blank				7.269	11 MB	0.00
58	101599012	MOON Blank				7.269	11 MB	0.00
59	101599012	MOON Blank				7.269	11 MB	0.00
60	101599012	MOON Blank				7.269	11 MB	0.00
61	101599012	MOON Blank				7.269	11 MB	0.00
62	101599012	MOON Blank				7.269	11 MB	0.00

Sample 920100: Quantified and printed by LMS on 10/15/99

Quantify Compound Summary Report

Page 3

TCX145-rat sera, day 1 & 4, control & dose- cont. 10/19/99 SER

Sample List: C:\MSDCHEM\Source\sera\Sample00\101999a

Last modified: Wed Oct 20 07:37:33 1999

Method: C:\MSDCHEM\Source\sera\Method00\101999b

Last modified: Wed Oct 20 07:34:48 1999

Job Code:

Printed: Wed Oct 20 08:01:10 1999

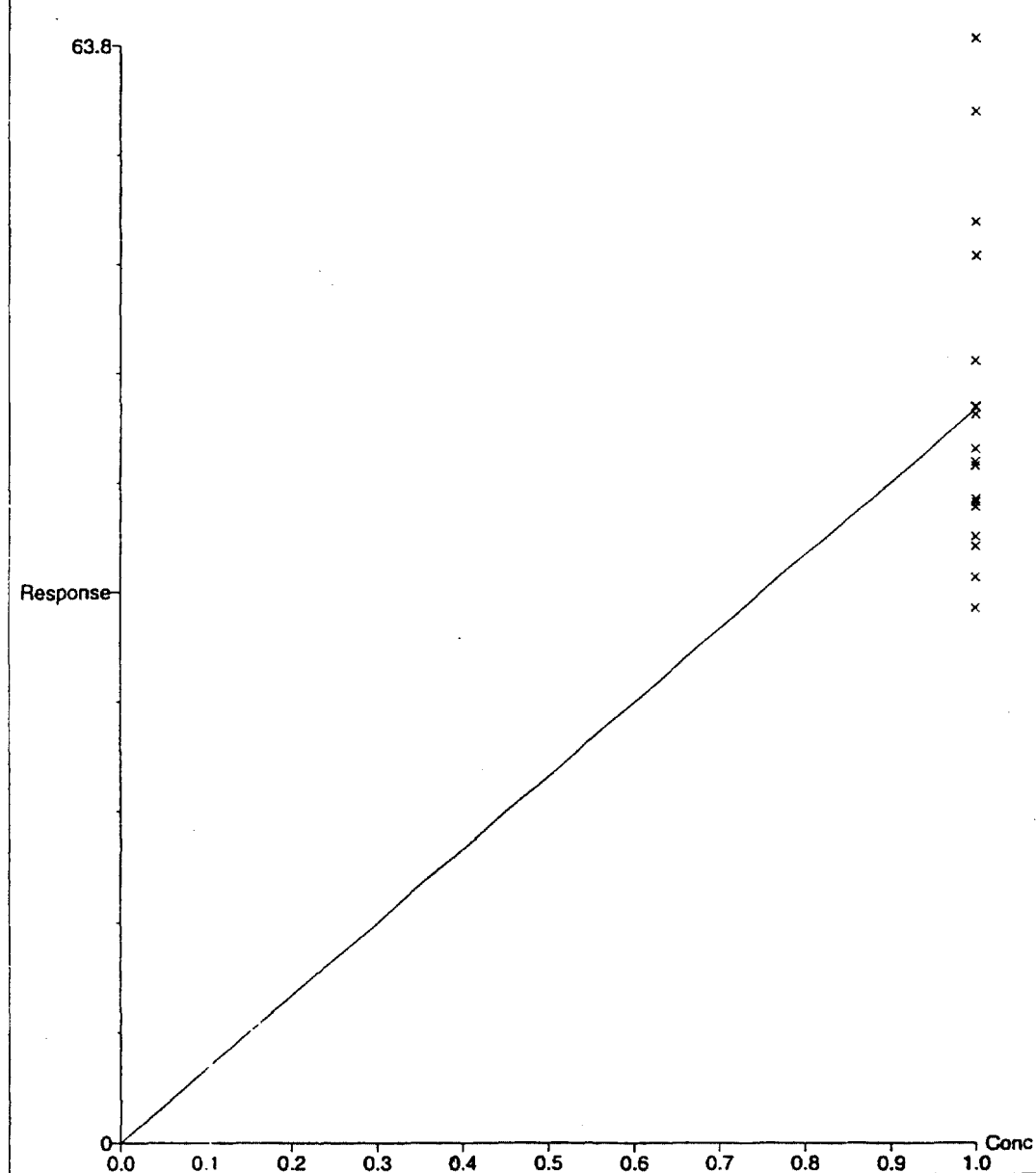
Compound 3: PFOSA (CH3)

#	Name	Sample List Text	Type	Std Conc	RT	Area	Flags	Conc.	Adov
1	101999012	MeCN Blank			7.836	2725	MM	11.20	
2	101999013	MeCN Blank			7.806	519	MM	2.28	
3	101999014	MeCN Blank			7.853	294	MM	1.06	
4	101999015	TCX145-RAS10159-MCN Blank-11, SER			7.833	171	MM	0.45	
5	101999016	TCX145-RAS10159-Sera Blank-11, SER			7.839	154	MM	0.36	
6	101999017	TCX145-RAS10159-Sera Blank-11, SER			7.822	159	MM	0.39	
7	101999018	TCX145-RAS10159-Sera Blank-12, SER			7.834	145	MM	0.22	
8	101999019	TCX145-RAS10159-50ppb-11, SER			7.808	1074	MM	4.56	1
9	101999020	TCX145-RAS10159-50ppb-11, SER	Standard	4.93	7.813	2016	MM	9.76	-0
10	101999021	TCX145-RAS10159-50ppb-11, SER	Standard	9.77	7.810	5196	MM	25.54	3
11	101999022	TCX145-RAS10159-50ppb-11, SER	Standard	24.80	7.802	9515	MM	47.10	-4
12	101999023	TCX145-RAS10159-100ppb-11, SER	Standard	49.30	7.814	14241	MM	80.69	-17
13	101999024	TCX145-RAS10159-250ppb-11, SER	Standard	97.70	7.811	49679	MM	247.45	-0
14	101999025	TCX145-RAS10159-500ppb-11, SER	Standard	246.00	7.809	100839	MM	503.11	2
15	101999026	TCX145-RAS10159-750ppb-11, SER	Standard	493.00	7.814	117401	MM	585.80	-20
16	101999027	TCX145-RAS10159-1000ppb-11, SER	Standard	736.00	7.813	149225	MM	744.71	-24
17	101999028	MeCN Blank			7.877	593	MM	3.55	
18	101999029	TCX145-RTS, 983321LM, Dyl. Control.-10/19/99 SER			7.836	161	MM	1.43	
19	101999030	TCX145-RTS, 983321LM, Dyl. Control.-10/19/99 SER			7.834	226	MM	0.72	
20	101999031	TCX145-RTS, 983321LM, Dyl. Control.-10/19/99 SER			7.808	147	MM	0.33	
21	101999032	TCX145-RTS, 983321LM, Dyl. Control.-10/19/99 SER			7.812	150	MM	0.34	
22	101999033	TCX145-RTS, 983321LM, Dyl. Control.-10/19/99 SER			7.837	142	MM	0.30	
23	101999034	TCX145-RAS10159-250ppb-CCV-11-1, SER	QC	248.00	7.812	49041	MM	244.47	-1
24	101999035	MeCN Blank			7.854	312	MM	1.15	
25	101999036	TCX145-RTS, 983321LM, Dyl. Control.-10/19/99 SER			7.856	168	MM	0.43	
26	101999037	TCX145-RTS, 983321LM, Dyl. Control.-10/19/99 SER			7.837	139	MM	0.29	
27	101999038	TCX145-RTS, 983321LM, Dyl. Control.-10/19/99 SER			7.852	167	MM	0.43	
28	101999039	TCX145-RTS, 983321LM, Dyl. Control.-10/19/99 SER			7.858	146	MM	0.32	
29	101999040	TCX145-RTS, 983321LM, Dyl. Control.-10/19/99 SER			7.852	136	MM	0.27	
30	101999041	TCX145-RAS10159-250ppb-CCV-11-2, SER	QC	248.00	7.806	43165	MM	215.13	-13
31	101999042	MeCN Blank			7.854	315	MM	1.17	
32	101999043	TCX145-RTS10159-250ppb-RS-11, 10/19/99 SER			7.789	48811	MM	243.32	
33	101999044	TCX145-RTS10159-250ppb-RS-11, 10/19/99 SER			7.803	52881	MM	263.64	
34	101999045	TCX145-RTS, 983321LM, Dyl. 5 ug/kg 10/19/99 SER			7.791	52155	MM	260.01	
35	101999046	TCX145-RTS, 983321LM, Dyl. 5 ug/kg 10/19/99 SER			7.790	87504	MM	436.52	
36	101999047	TCX145-RTS, 983321LM, Dyl. 5 ug/kg 10/19/99 SER			7.814	51116	MM	254.81	
37	101999048	TCX145-RAS10159-250ppb-CCV-11-3, SER	QC	248.00	7.809	49994	MM	248.74	0
38	101999049	MeCN Blank			7.881	384	MM	1.51	
39	101999050	TCX145-RTS, 983321LM, Dyl. 5 ug/kg 10/19/99 SER			7.805	67032	MM	334.30	
40	101999051	TCX145-RTS, 983321LM, Dyl. 5 ug/kg 10/19/99 SER			7.793	55570	MM	277.07	
41	101999052	TCX145-RTS, 983321LM, Dyl. 5 ug/kg 10/19/99 SER			7.810	12158	MM	61.30	
42	101999053	TCX145-RTS, 983321LM, Dyl. 5 ug/kg 10/19/99 SER			7.804	11759	MM	58.31	
43	101999054	TCX145-RTS, 983321LM, Dyl. 5 ug/kg 10/19/99 SER			7.789	15156	MM	75.28	
44	101999055	TCX145-RAS10159-250ppb-CCV-11-4, SER	QC	248.00	7.814	46425	MM	232.41	-6
45	101999056	MeCN Blank			7.856	307	MM	1.13	
46	101999057	TCX145-RTS, 983321LM, Dyl. 5 ug/kg 10/19/99 SER			7.813	12235	MM	60.49	
47	101999058	TCX145-RTS, 983321LM, Dyl. 5 ug/kg 10/19/99 SER			7.792	11895	MM	58.99	
48	101999059	TCX145-RAS10159-250ppb-CCV-11-5, SER	QC	248.00	7.811	50283	MM	250.67	1
49	101999060	MeCN Blank			7.857	348	MM	1.33	
50	101999061	TCX145-RAS10159-MCN Blank-11, SER			7.854	145	MM	0.32	
51	101999062	TCX145-RAS10159-Sera Blank-11, SER			7.851	126	MM	0.23	
52	101999063	TCX145-RAS10159-Sera Blank-11, SER			7.833	120	MM	0.19	
53	101999064	TCX145-RAS10159-Sera Blank-11, SER			7.859	148	MM	0.34	
54	101999065	TCX145-RAS10159-50ppb-11, SER	Standard	4.93	7.814	1139	MM	5.28	7
55	101999066	TCX145-RAS10159-50ppb-11, SER	Standard	9.77	7.813	2063	MM	9.90	1
56	101999067	TCX145-RAS10159-50ppb-11, SER	Standard	24.80	7.811	5615	MM	27.63	11
57	101999068	TCX145-RAS10159-50ppb-11, SER	Standard	49.30	7.813	10041	MM	49.73	1
58	101999069	TCX145-RAS10159-50ppb-11, SER	Standard	97.70	7.805	18028	MM	89.61	-8
59	101999070	TCX145-RAS10159-50ppb-11, SER	Standard	246.00	7.813	51869	MM	254.60	3
60	101999071	TCX145-RAS10159-50ppb-11, SER	Standard	493.00	7.791	100103	MM	499.43	1
61	101999072	TCX145-RAS10159-50ppb-11, SER	Standard	736.00	7.786	122134	MM	609.44	-17
62	101999073	TCX145-RAS10159-50ppb-11, SER	Standard	977.00	7.811	151334	MM	765.23	-22

Quantity: Command Summary Page 1
 TOLU-10C data, day 1 & 4, control & expo. on: 10/19/99
 Sample List: C:\MSDCHEM\1001-PC-1-Data\SampleList\101999
 Date modified: Wed Oct 20 07:23:33 1999
 Datafile: 1001-PC-1-Data\101999-1001-PC-1-Data\101999
 Datafile modified: Wed Oct 20 07:23:33 1999
 Job Name: 1001-PC-1-Data\101999-1001-PC-1-Data\101999
 Printed: Wed Oct 20 08:11:10 1999

Compound 1: 26 (C04)									
Sample	Sample Name	Type	Ref Conc	IR	Area	Wt%	Conc	Unit	Notes
1	101999013	Blank							
2	101999013	Blank							
3	101999013	Blank							
4	101999013	Blank							
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57	101999013	Blank							
58	101999013	Blank							
59	101999013	Blank							
60	101999013	Blank							
61	101999013	Blank							

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



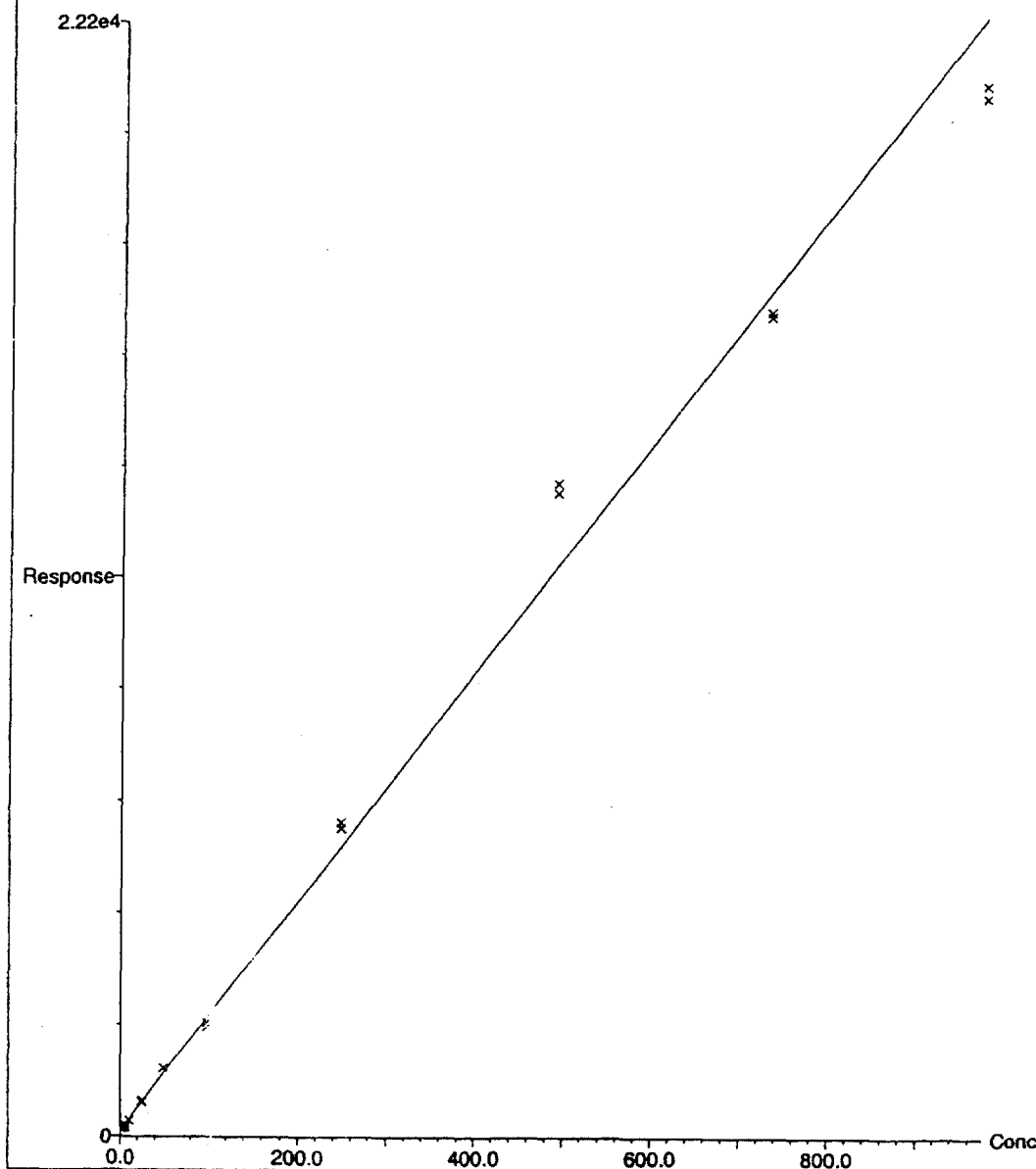
Compound 2 name: PFOS (Ch2)

Coefficient of Determination: 0.988440

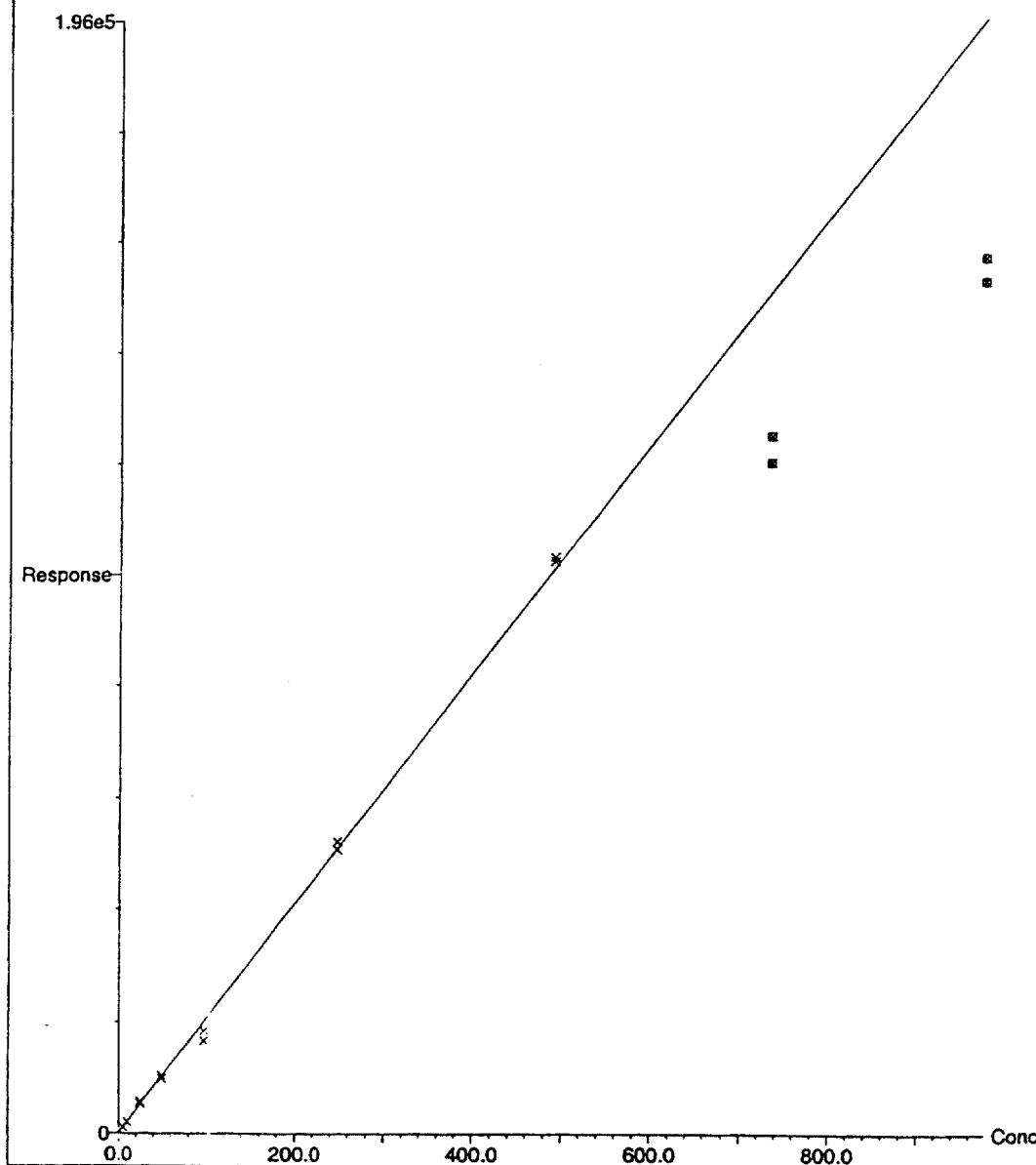
Calibration curve: $22.5689 \cdot x + 138.989$

Response type: External Std, Area

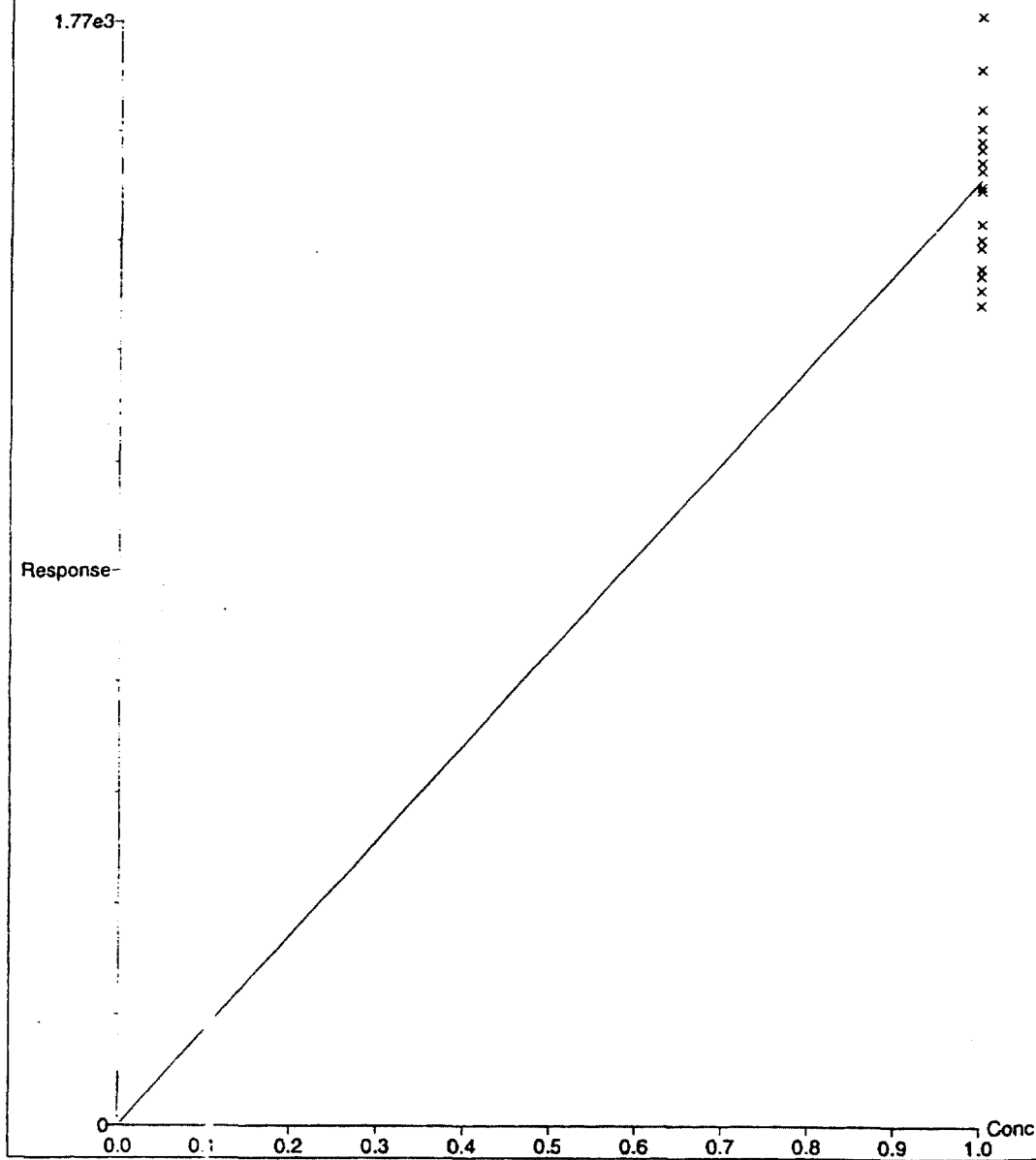
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



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



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



Main Report

Method: SELECT
Last Modified: 10/10/1999
Comp. ID: 10000000

Printed: 10/10/1999 11:10:35

Comp. ID: 10000000
User: J. J. J.
Quantity: 1000000
Conc. of Standards: 1.000000
User RF Value: 1.000000
Integer Width Factor: 1.000000
Chromatogram Data Width: 1.000000

Retention Time (min): 1.111, Disabled
Peak Selection: 1.111, Disabled
Time: 1.111

Peak Selection: 1.111, Disabled
Mass: 1.111, Disabled
Mass: 1.111, Disabled

Polynomial Type: Linear
Point of Origin: 1/X
File Name: none
Axis Transformation: none

Notes:

Peak Data:

Peak: 1.111, Disabled
Width: 1.111, Disabled
Height: 1.111, Disabled
Area: 1.111, Disabled

Join: 1.111, Disabled
Mass: 1.111, Disabled
Mass: 1.111, Disabled

Mass: 1.111, Disabled
Mass: 1.111, Disabled
Mass: 1.111, Disabled

Method Report Page 2
 Method file: C:\MASSLYN\sup-rats.ppt\Meth08\101999b.mdb
 Last modified: Wed Oct 2 13:48 1999
 Compound: 20 PFOS (C)
 Printed: Wed Oct 2 14:05 1999

Concentration: 705 (C2)
 Internal Std.: (none)
 Quantify Peak: 100
 Control Standard: 000. 8
 User AF Value: 1.000000, Disabled
 User Peak Factor: 1.000000
 Interference Window Factor: 1.000
 Chromatogram Scale Multiplier: 1.000000

Retention Time (min): 109, Enabled
 Retention Time (min): 110, Disabled
 RT Alignment: (none)
 Time (min): 111

Peak Selection: nearest

Response Type: external
 Response: none

Polynomial Type: linear
 Points to Exclude: include
 Fit Weighting: 1/X
 Axis Transformation: none

Unit:

Peak-to-peak Amplitude: 1

Selection: enabled
 Warns if file is open: 1
 Warns if file is open: 1
 Warns if file is open: 1

Justification: 1.00
 As: 1.00
 As: 1.00

Draw: 1.00
 Draw: 1.00, disabled
 Show: 1.00

Relax: 1.00, Disabled
 Abort: 1.00, Disabled
 Relax: 1.00, Disabled
 Abort: 1.00, Disabled

[illegible]

[illegible]

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 μ L 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 μ m

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 μ L/min. Back pressure: 65 bar

HPLC solvent gradient: 10min-10-95

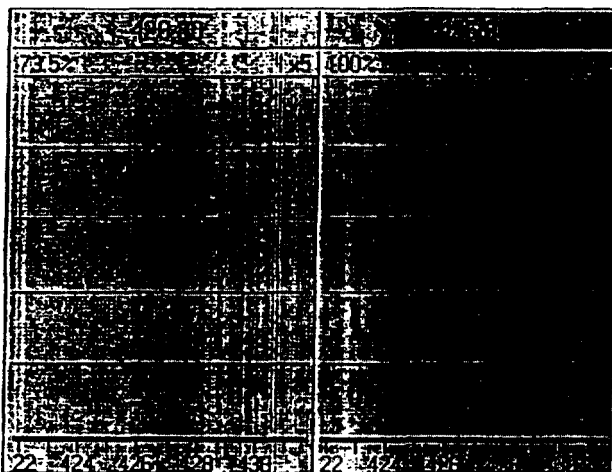
Time (min.)	%B
1) 0.00	10.0
2) 1.00	10.0
3) 5.50	95.0
4) 7.50	95.0
5) 8.00	10.0

Tuning Method Report

Page 1

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

Printed: Tue Oct 19 15:40:03 1999



MS

MS2

SOURCE (ESP-)	Set	Rdbk	MS1	Set	Rdbk
Capillary	3.00	3.01	LM Resolution	14.0	
Cone	40	-41	HM Resolution	14.0	
Extractor	5	-8	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	251	Multiplier	650	

Pressures	Rdbk	MS2	Set	Rdbk
Analysar Vacuum	5.5e-5	LM Resolution	14.0	
		HM Resolution	14.0	
Gas Cell	4.1e-3	Collision	10	8
		Ion Energy	2.0	
		Ion Energy Ramp	0.0	
		Lens 8	40	38
		Lens 9	0	1
		Multiplier	650	

Examining Method Report

Method: C:\MSDCHEM\SOEP-MATHS\PRO\ACQUO\11\SOEP-PROD-POOL-PP00A-25a,
 Last Modified: Fri Oct 15 11:31:03 1999
 Printed: Tue Oct 19 15:40:29 1999

Solvent Delay (mins) : 0.00

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

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) Cose (V)

1 413.00 169.00 0.30 30 15

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

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) Cose (V)

1 459.00 99.00 0.30 45 60

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

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) Cose (V)

1 498.00 78.00 0.30 45 60

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

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) Cose (V)

1 427.00 80.00 0.30 40 60



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

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	---	---

Masslynx - Sample List

Page 3

Sample List: C:\MASSLYNX\oup-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

Masslynx - Sample List**Page 4****Sample List:** C:\MASSLYNX\scup-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 --	--	--

FACT: DATA REVIEW SUMMARY FORM

Date: 10/26/99
 Data Analyst: Jas
 FACT Study #: Tox 145
 Matrix/Timepoints: Rat Sera Dilutions - Day 1 & 4, cat 17/10/99
 Sample List: 1022994
 Set-up Analysis Date/Analyst: 10/22/99 Jas
 Instrument: AMEL 062498

Data Review Sign-Off

Analyst Review
 Name / Date: Jas 10/26/99

Technical Review
 Name / Date: MEE 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

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

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: ↓

② Calibration checks for PFOS failed. It is suspected that injections failed for cal checks due to lack of sample. The 735 ppb & 974 ppb 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.

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: ↓

③ SUGGEST DETERMINING REASON FOR CAL CHECK FAILURE AND THEN REPEAT ANALYSIS OF THE SAMPLE SET. MEE 10/25/99.

④ 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.

FACT: DATA REVIEW SUMMARY FORM

Data Review Sign-Off

Date: 10/25/99
 Data Analyst: Ias
 FACT Study #: Tox 145
 Matrix/Timepoints: Ref. Sere Dilutions. Day 1 & 4, cat 1/11/99
 Sample List: 102299a ③
 Set-up Analysis Date/Analyst: 10/22/99 Ias
 Instrument: Amelia 062498

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

Technical Review
 Name / Date: Refer to page 1-2

Quality Assurance Review
 Name / Date: _____

Data Entry: Not entered. need to reanalyze UC

Comments / Justifications: _____ 10/25/99

Analyte: IS (CH3)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: _____
 MS/MSD: Ias 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 Prosa channel. Peaks should be investigated. - Ias 10/25/99

③ The 913 ppb, 756 ppb, and 977 ppb curve 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 Prosa channel and should be investigated - Ias 10/25/99

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

Amis 04498: Quantified and Printed by las 10/28/99

Quality: Compounded Summary Report
 TON115-RAT serum dilutions, day 1, 6, 4 - out: 10/19/99 SEE
 Sample List: C:\MSD\TNC\Amate-mts.FMO\Sample001\1027996
 Last modified: Mon Oct 25 06:42:18 1999
 Job Code: TON115-RAT serum dilutions, day 1, 6, 4 - out: 10/19/99
 Last modified: Mon Oct 25 06:42:18 1999
 Job Code:

Printed: Mon Oct 25 06:39:42 1999

Compound 2: FPOB (CH3)

6 Name	Sample/Lot Text	Type	Std Conc	AV	Area	Conc. Flags Show
1 102799001	MeOM Blank			1.190	118	0.24 M
2 102799002	MeOM Blank			7.162	112	0.00 M
3 102799003	TON115-RAS10199-R2D B1k-12, SEE			7.162	66	0.00 M
4 102799004	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
5 102799005	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
6 102799006	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
7 102799007	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
8 102799008	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
9 102799009	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
10 102799010	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
11 102799011	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
12 102799012	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
13 102799013	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
14 102799014	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
15 102799015	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
16 102799016	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
17 102799017	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
18 102799018	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
19 102799019	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
20 102799020	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
21 102799021	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
22 102799022	MeOM Blank			7.162	66	0.00 M
23 102799023	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
24 102799024	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
25 102799025	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
26 102799026	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
27 102799027	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
28 102799028	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
29 102799029	MeOM Blank			7.162	66	0.00 M
30 102799030	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
31 102799031	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
32 102799032	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
33 102799033	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
34 102799034	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
35 102799035	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
36 102799036	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
37 102799037	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
38 102799038	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
39 102799039	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
40 102799040	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M
41 102799041	TON115-RAS10199-Serum B1k-12, SEE			7.162	66	0.00 M

Amelia 042498: Quantitated and Printed by LSC 10/23/99

Quantity Compared Primary Report
 T0X145-RTS serum dilution, day 1 & 4 - ant. 10/19/99 MSE
 Sample List: C:\MSDCHEM\amala-rts.MPO\sample.mh\102259a
 Last modified: Mon Oct 25 09:18:56 1999
 Job Code: T0X145-RTS serum dilution, day 1 & 4 - ant. 10/19/99 MSE
 Last modified: Mon Oct 25 09:30:53 1999
 Job Code:
 Printed: Mon Oct 25 06:39:42 1999

Compound 3: PBOA (Chd)

Q Name	Sample/Std Test	Type	Std Conc	RT	Area	Conc.	Flags	Idov
1 102259001	MOB Blank			7.846	445	4.23 bb		
2 102259002	MOB Blank			7.846	121	3.42 bb		
3 102259003	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	151	2.33 bb		
4 102259004	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	95	1.89 bb		29
5 102259005	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	815	6.36 bb		29
6 102259006	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1144	9.37 bb		-2
7 102259007	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		-2
8 102259008	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		-28
9 102259009	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		13
10 102259010	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		-2
11 102259011	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		9
12 102259012	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
13 102259013	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
14 102259014	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
15 102259015	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
16 102259016	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
17 102259017	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
18 102259018	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
19 102259019	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
20 102259020	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
21 102259021	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
22 102259022	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
23 102259023	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
24 102259024	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
25 102259025	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
26 102259026	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
27 102259027	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
28 102259028	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
29 102259029	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
30 102259030	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
31 102259031	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
32 102259032	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
33 102259033	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
34 102259034	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
35 102259035	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
36 102259036	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
37 102259037	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
38 102259038	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
39 102259039	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
40 102259040	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6
41 102259041	T0X145-RTS-090324-0M, Day1, Smg/kg, D50-ant10/19/99			7.846	1624	13.44 bb		6

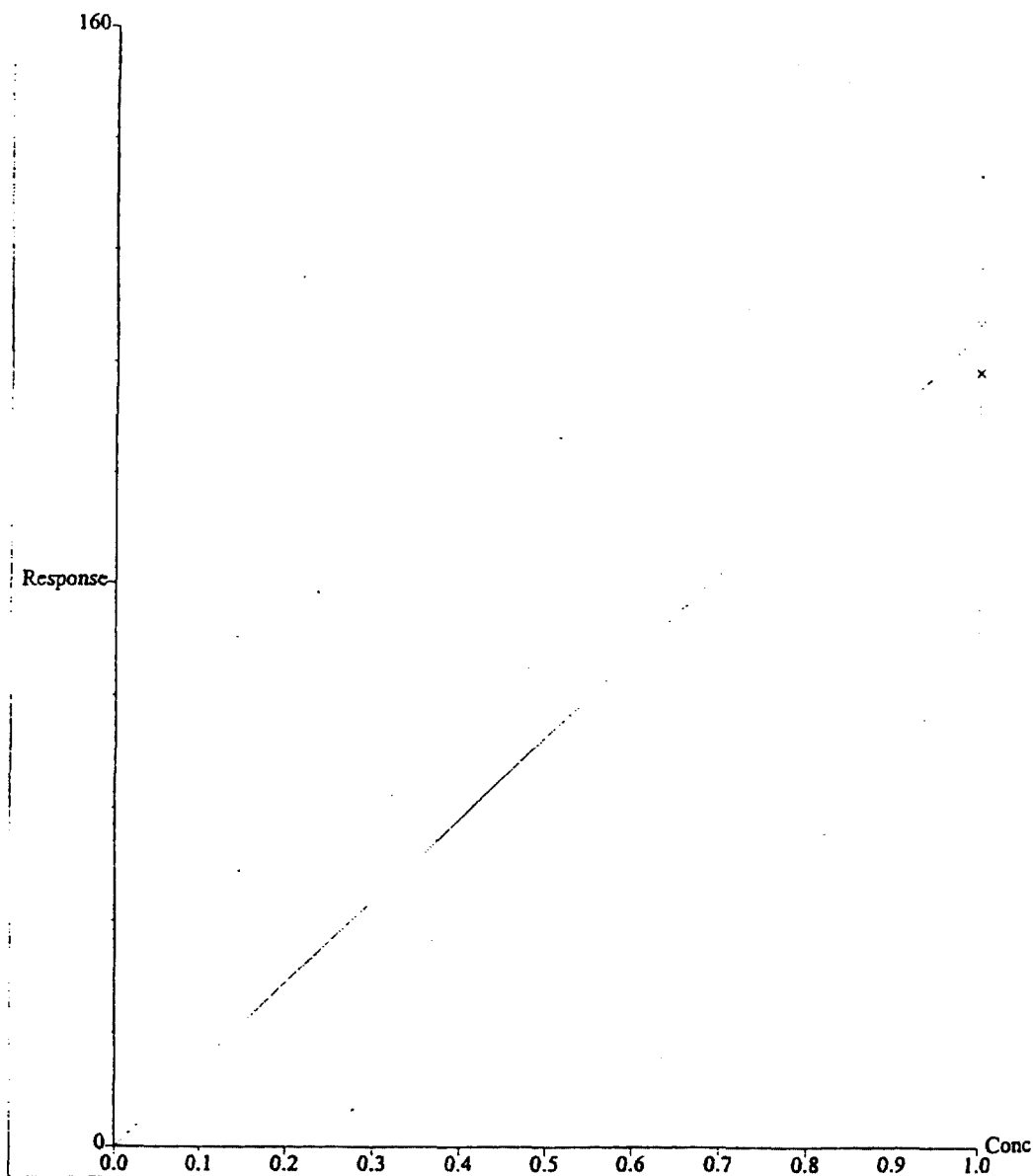
Amelia 82189: Quantitated and Printed by Loe 10/23/99

Amelia 052498: Quantitated and Printed by Leo 10/23/99

Compound 4: IS (CH)

4 Name	Sample List Text	Type	Std Conc	RT	Area	Conc. Stage 0509
1 10229001	Mock Blank					
2 10229002	TK0145-TRE-M021019P-100p-11, SEE			7.061	2016	1.11 Bd
3 10229003	TK0145-TRE-M021019P-100p-11, SEE			6.964	2291	0.96 Bd
4 10229004	TK0145-TRE-M021019P-100p-11, SEE			7.002	2311	1.06 Bd
5 10229005	TK0145-TRE-M021019P-100p-11, SEE			7.014	2303	1.04 Bd
6 10229006	TK0145-TRE-M021019P-100p-11, SEE	Standard	1.00	6.997	2281	0.92 Bd
7 10229007	TK0145-TRE-M021019P-100p-11, SEE	Standard	1.00	7.003	2334	0.96 Bd
8 10229008	TK0145-TRE-M021019P-100p-11, SEE	Standard	1.00	6.999	2322	1.06 Bd
9 10229009	TK0145-TRE-M021019P-100p-11, SEE	Standard	1.00	6.985	2031	1.11 Bd
10 10229010	TK0145-TRE-M021019P-100p-11, SEE	Standard	1.00	6.993	2075	1.11 Bd
11 10229011	TK0145-TRE-M021019P-100p-11, SEE	Standard	1.00	6.994	2017	1.11 Bd
12 10229012	TK0145-TRE-M021019P-100p-11, SEE					
13 10229013	TK0145-TRE-M021019P-100p-11, SEE					
14 10229014	TK0145-TRE-M021019P-100p-11, SEE					
15 10229015	Mock Blank					
16 10229016	TK0145-TRE-M021019P-100p-11, SEE			6.984	89	0.03 Bd
17 10229017	TK0145-TRE-M021019P-100p-11, SEE			6.985	83	0.03 Bd
18 10229018	TK0145-TRE-M021019P-100p-11, SEE			6.982	76	0.03 Bd
19 10229019	TK0145-TRE-M021019P-100p-11, SEE			6.982	76	0.03 Bd
20 10229020	TK0145-TRE-M021019P-100p-11, SEE			6.983	2260	0.83 Bd
21 10229021	Mock Blank					
22 10229022	TK0145-TRE-M021019P-100p-11, SEE					
23 10229023	TK0145-TRE-M021019P-100p-11, SEE			6.982	84	0.03 Bd
24 10229024	TK0145-TRE-M021019P-100p-11, SEE			6.983	88	0.03 Bd
25 10229025	TK0145-TRE-M021019P-100p-11, SEE			6.987	38	0.03 Bd
26 10229026	TK0145-TRE-M021019P-100p-11, SEE			7.003	70	0.03 Bd
27 10229027	TK0145-TRE-M021019P-100p-11, SEE			6.983	70	0.03 Bd
28 10229028	TK0145-TRE-M021019P-100p-11, SEE			6.985	1669	0.70 Bd
29 10229029	Mock Blank					
30 10229030	TK0145-TRE-M021019P-100p-11, SEE			6.981	2796	1.04 Bd
31 10229031	TK0145-TRE-M021019P-100p-11, SEE			6.987	2329	0.83 Bd
32 10229032	TK0145-TRE-M021019P-100p-11, SEE			7.001	2321	0.94 Bd
33 10229033	TK0145-TRE-M021019P-100p-11, SEE	Standard	1.00	6.984	2315	0.92 Bd
34 10229034	TK0145-TRE-M021019P-100p-11, SEE	Standard	1.00	6.989	2315	0.94 Bd
35 10229035	TK0145-TRE-M021019P-100p-11, SEE	Standard	1.00	7.009	2391	0.89 Bd
36 10229036	TK0145-TRE-M021019P-100p-11, SEE	Standard	1.00	7.007	2392	0.81 Bd
37 10229037	TK0145-TRE-M021019P-100p-11, SEE	Standard	1.00	7.012	2435	1.06 Bd
38 10229038	TK0145-TRE-M021019P-100p-11, SEE	Standard	1.00	7.008	2435	0.89 Bd
39 10229039	TK0145-TRE-M021019P-100p-11, SEE	Standard	1.00	7.009	2895	1.06 Bd
40 10229040	TK0145-TRE-M021019P-100p-11, SEE	Standard	1.00	7.004	2813	1.04 Bd
41 10229041	TK0145-TRE-M021019P-100p-11, SEE	Standard	1.00	6.991	2833	1.04 Bd

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



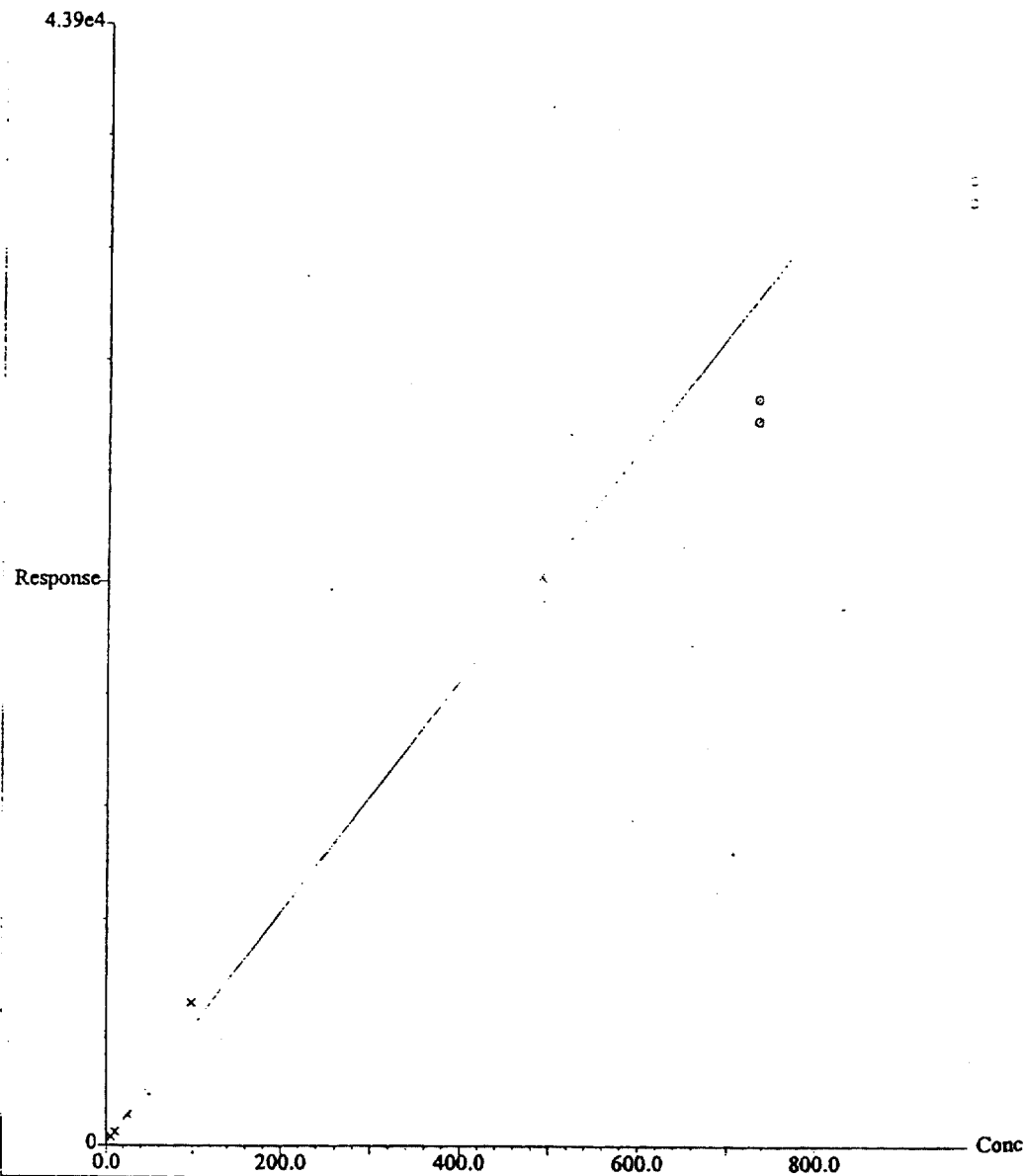
Compound 2 name: PFOS (Ch2)

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



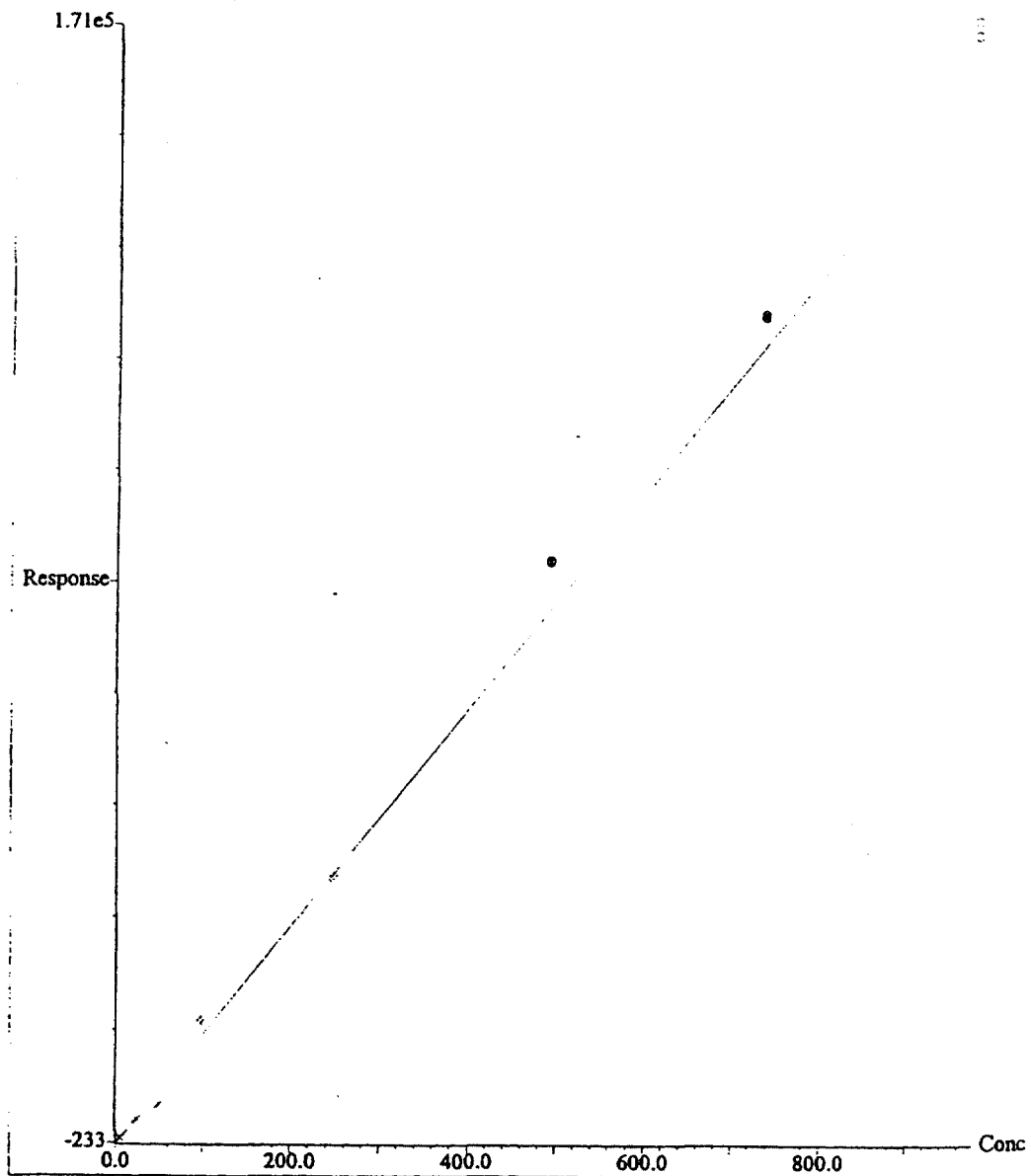
Compound 3 name: PFOSA (Ch4)

Coefficient of Determination: 0.989156

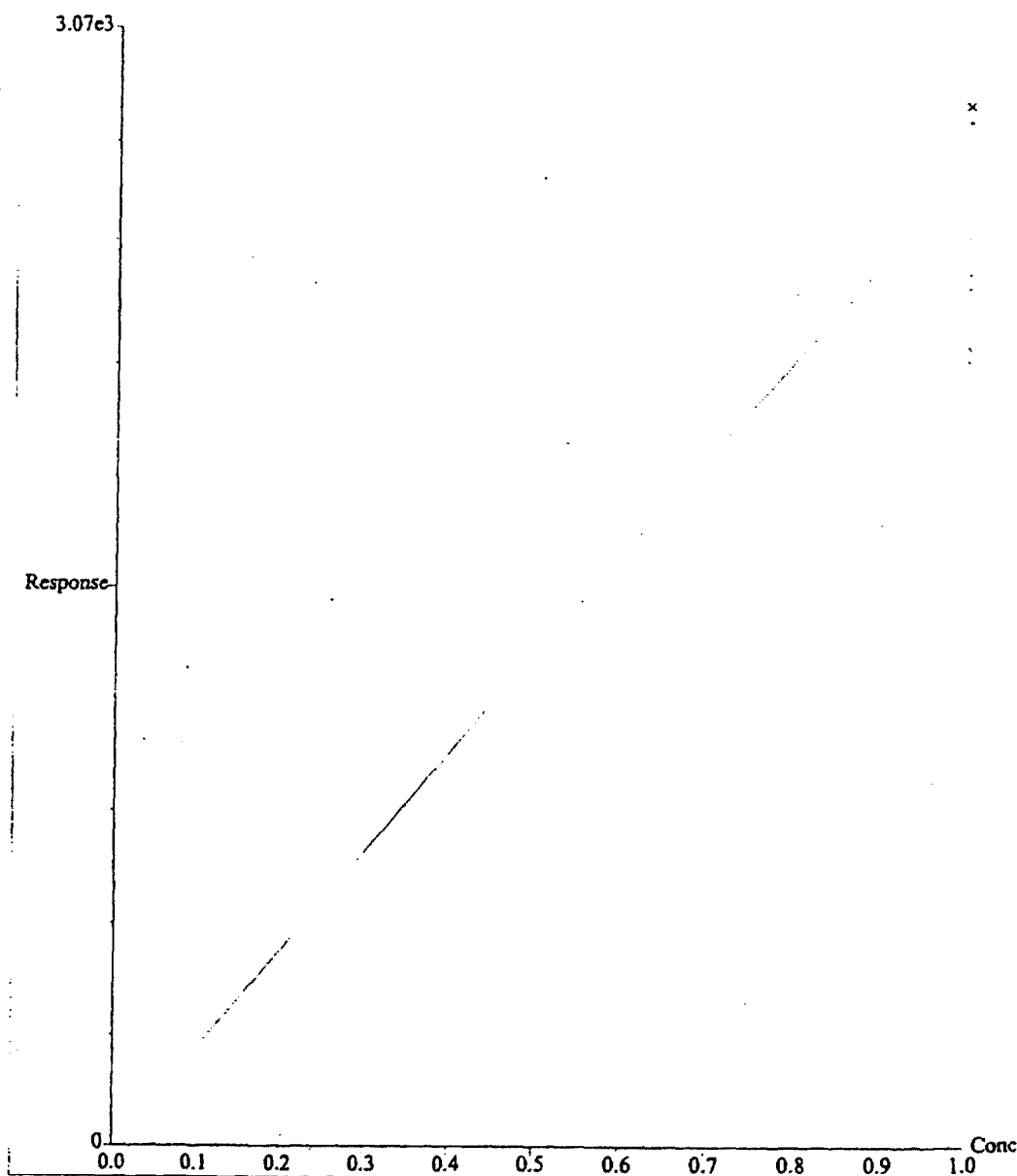
Calibration curve: $164.764 * x + -232.515$

Response type: External Std, Area

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



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



Over vertical threshold:	99.00
Under vertical threshold:	Disabled
Shoulder threshold:	35.00
<u>Response threshold</u>	
Relative height:	1.50, Disabled
Relative width:	9.00, Disabled
Relative area:	2.00, Disabled
Absolute area:	0.00, Disabled

Method Report

Method file: C:\MSDCHEM\anal-software\PROTECTION\102298a.mdb
 Last modified: Mon Oct 26 06:39:55 1999

Compound: 1: POLA (CA1)

Printed: Mon Oct 25 07:03:23 1999

Compound Name: POLA (CA1)
 Internal Name: (none)
 Quantity Traced: TIC
 Conc. of Standards: Fixed

User RF Value: 1.00000, Disabled
 User RF Standard: 1.00000
 Integration Window Factor: 1.00000
 Chromatogram mass window: 1.00000

Peak Location
 Retention Time (min): 6.384, Enabled
 Retention Time (min): 6.384, Disabled
 RT Reference: (none)
 Time Window (min): 0.202

Peak Selection: Manual
 Response: Manual

Response Type: External
 Response: Areas

Calibration Curve
 Polynomial Type: Linear
 Point of Origin: Excluded
 Fit Weibling: 1/X
 Axis Transformation: None

Units:

None

Peak-to-peak amplitude: 95
 Smooth

Smoothing: Disabled
 Window size (scan): 2
 Number of scans: 1
 Smoothing method: Baseline

Join valley threshold: 30.00
 Join valley valley: 200.00
 Peak threshold: 3.00
 Peak separation: 3.00

Method Report

Method file: C:\MSDCHEM\SampleData\102299a.mdb
 Last modified: Mon Oct 25 06:28:55 1999
 Compound: 2: PFOS (CH2)
 Printed: Mon Oct 25 07:03:23 1999

Compound Name: PFOS (CH2)
 Internal Ref.: [None]
 Quantity Traced: TIC
 Calc. of Standards: Calc. A
 User RT Value: 1.000000, Disabled
 Integrate Window Factor: 2.000000
 Chromatogram mask window: 1.000000
 Peak Location:

Retention Time (min): 7.176, Enabled
 Relative Retention Time: 0.000, Disabled
 RT Reference: [None]
 Time Window (min): 0.262

Peak Selection: Weighted
 Response

Response Type: External
 Calibration: Area
 Calibration Curves

Polynomial Type: Linear
 Point of Origin: Exclude
 Fit Weighting: 1/X
 Area Normalization: None
 Concentration

Units: Moles

Peak-to-peak amplitude: 2300
 Smooth

Smoothing: Disabled
 Number of scans: 2
 Number of methods: 1
 Smoothing method: Baseline

Join valley threshold: 30.00
Reduce peak calling: 200.00
Reduce baseline maxima: 5.00
Peak suppression
Dew 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: 1.00, Disabled
Absolute area: 0.00, Disabled

Draw vertical Ebramoid: 39.00
Detect shoulders: Disabled
Shoulder thresholds: 39.00
RESPONSE THRESHOLD
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:\MSDCHEM\analyse-rsca\PROVMETHOD\102298a.mtd

Last modified: Mon Oct 23 06:39:55 1995

Compound: 3: PTMBA (CM4)

Printed: Mon Oct 23 07:03:23 1995

Compound Name:

PTMBA (CM4)

Internal Ref.:

(None)

Quantity Traced:

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:

1.000000

Peak Detection

Retention Time (mins):

7.466, Enabled

Relative Retention Time:

0.000, Disabled

RT Reference:

(None)

TIC Window (mins):

0.213

Peak Selection:

Nearest

Response

Response Type:

External

Response:

Area

Calibration Curve

Polynomial Type:

Linear

Point of Origin:

Exclude

RT Reference:

None

Axis Transformation:

None

Concentration

Units:

None

Noise

None

Peak-to-peak amplitude:

161

Smooth

None

Smoothing:

Enabled

Window size (seconds):

2

Number of smooths:

1

Smoothing method:

None

Baseline

None

Baseline

None

Auto valley threshold:

20.00

Reduce peak calling:

200.00

Raise baseline minimum:

5.00

Peak Selection

None

Method Report

Method file: C:\MSDCHEM\SampleData\refc\PROVMethod102599.mdb
 Last modified: Mon Oct 25 07:31:35 1999

Compound: 4: IS (CH3)

Printed: Mon Oct 25 07:03:23 1999

Compound Name: IS (CH3)
 Internal Ref.: [None]
 Quantity Rec.: None
 Conc. of Standards: Fixed
 User RF Value: 1.000000, Disabled
 User Peak Factor: 1.000000
 Integration Method: 1.000000
 Chromatogram Mass Window: 1.000000
 Peak Location
 Retention Time (mins): 7.960, Enabled
 Retention Time (mins): 7.960, Disabled
 RT Reference: [None]
 Time Window (mins): 0.239
 Peak Selection: Nearest
 Response: External
 Response Type: External
 Response: Areas
 Calibration Curve
 Polynomial Type: Linear
 Point of Origin: Exclude
 Fit Weighting: 1/K
 Axis Transformation: None
 Concentration
 Units:
 Noise
 Peak-to-peak amplitude: 99
 Smooth
 Smoothing: Disabled
 Window size (secs): 2
 Number of scans: 1
 Smoothing method: Baseline
 Join valley threshold: 30.00
 Peak-to-peak minimum: 30.00
 Base baseline minimum: 5.00
 Peak separation
 Draw vertical threshold: 90.00
 Draw horizontal threshold: 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

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? ☒ 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

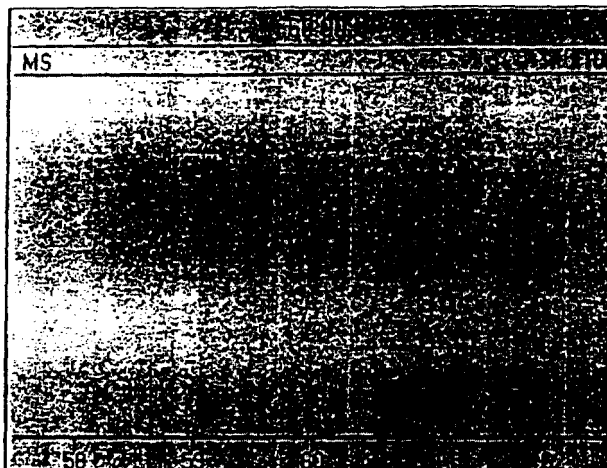
Time (min.)	%B
1) <u>0.00</u>	<u>10.0</u>
2) <u>1.00</u>	<u>10.0</u>
3) <u>5.50</u>	<u>95.0</u>
4) <u>7.50</u>	<u>95.0</u>
5) <u>8.00</u>	<u>10.0</u>

Tuning Method Report

Page 1

Method: C:\MASSLYNX\AMELIA-RATS.PRO\ACQ0DB\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
Analyser 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	

Scanning Method Report

Page 1

Method: C:\MSDCHEM\AMELIA-RATE.FTO\ACQUIS\10MIN-PTOS-POAA-16-PTOIA
 Last Modified: Mon Oct 11 08:22:17 1999
 Printed: Fri Oct 22 09:50:09 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)

1 413.00 149.00 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)

1 439.00 99.00 0.30 45 40

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 60.00 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)

1 436.00 76.00 0.40 45 60

Method Report

Page 1

Method File:
Last Modified:c:\masslynx\amelia-rats.pro\acqddb\10min-10-95
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

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

Sample List: C:\MASSLYNX\data\rats\PRO\Samp\081022\288.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	--	--	--	--	--	--	--

FACT: DATA REVIEW SUMMARY FORM

Date: 10-26-99
 Data Analyst: mmx
 FACT Study #: Tox 145
 Matrix/Timepoints: Bot Src, Day 1 & 4 Dilutions
 Sample List: 1025996
 Set-up Analysis Date/Analyst: 10-25-99 mmx
 Instrument: SOUP 120199

Data Review Sign-Off

Analyst Review Name / Date: 10-26-99 mmx
 Technical Review Name / Date: MEE 10/26/99 | kh 10/27/99
 Quality Assurance Review Name / Date: _____
 Data Entry: LAC 10/29/99

Analyte: PEOS 4.93 at 2.1 mmx 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: PEOSA
 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: GUR
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: _____

- Comments / Justifications:
- ① Excluded 2nd run since it was divergent from the 1st mmx 10/26/99
 - ② Lowest ex point dropped, it was not 2X matrix blank. mmx 10/26/99
 - ③ ~~2X~~ Excluded lowest point to meet less than 30% deviation criteria mmx 10/26/99
 - ④ did not set up method to screen for PEOSA since only dilutions of samples were analyzed mmx 10/26/99
 - ⑤ lowest point can be included since it is 2X RBS blank so LOQ is 4.93 ppb mmx 10/27/99

LAC 10/29/99

COMP 020123: Quantified and printed by IBM on 10/26/78

Quantity Compound Primary Report
 Test 1651 Test Date: May 1st 1999 Date: 10/15/99
 Sample Name: C:\MSDCHEM\comp215199.prn\SampleName10235908
 Date Modified: Mon Oct 25 16:32:42 1999
 Method: C:\MSDCHEM\comp215199.prn\Method10235908
 Last Modified: Tue Oct 26 09:11:31 1999
 Job Code:

Printed: Tue Oct 26 10:11:29 1999

Compound 1: PROD

9 Name	Empirical	Year	Type	Ref	Chem. Shift
1 102359019	PROD				
2 102359019	PROD				
3 102359019	PROD				
4 102359019	PROD				
5 102359019	PROD				
6 102359019	PROD				
7 102359019	PROD				
8 102359019	PROD				
9 102359019	PROD				
10 102359019	PROD				
11 102359019	PROD				
12 102359019	PROD				
13 102359019	PROD				
14 102359019	PROD				
15 102359019	PROD				
16 102359019	PROD				
17 102359019	PROD				
18 102359019	PROD				
19 102359019	PROD				
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25 102359019	PROD				
26 102359019	PROD				
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28 102359019	PROD				
29 102359019	PROD				
30 102359019	PROD				
31 102359019	PROD				
32 102359019	PROD				
33 102359019	PROD				
34 102359019	PROD				
35 102359019	PROD				
36 102359019	PROD				
37 102359019	PROD				
38 102359019	PROD				
39 102359019	PROD				
40 102359019	PROD				
41 102359019	PROD				
42 102359019	PROD				
43 102359019	PROD				
44 102359019	PROD				
45 102359019	PROD				

Quantity Compound Summary Report
 Run 161: Max Area, Day 114 dillwoma, Run 16/19/99
 Sample Name: C:\MSDCHEM\comp20109.prm\sample\0103599b
 Last Modified: Mon Oct 25 16:12:42 1999
 Method: C:\MSDCHEM\comp20109.prm\method\103599b
 Last Modified: Tue Oct 26 09:11:11 1999
 Job Order:
 Printed: Tue Oct 26 16:51:19 1999

Compound 2: PFOMA

9 Name	Sample/Ink Test	Type	Std Conc	SP	Area Flag	Conc, Mgr
1 102359039	MeOH Blank			6.155	101 M	1.13
2 102359040	MeOH Blank			8.175	153 M	1.28
3 102359041	TU0145-MS10139-820-BLK-12, SEE			8.175	153 M	1.28
4 102359042	TU0145-MS10139-820-BLK-12, SEE			8.210	154 M	1.30
5 102359043	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
6 102359044	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
7 102359045	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
8 102359046	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
9 102359047	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
10 102359048	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
11 102359049	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
12 102359050	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
13 102359051	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
14 102359052	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
15 102359053	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
16 102359054	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
17 102359055	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
18 102359056	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
19 102359057	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
20 102359058	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
21 102359059	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
22 102359060	TU0145-MS10139-820-BLK-12, SEE			8.240	243 M	1.35
23 102359061	MeOH Blank			8.135	211 M	9.52
24 102359062	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
25 102359063	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
26 102359064	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
27 102359065	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
28 102359066	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
29 102359067	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
30 102359068	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
31 102359069	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
32 102359070	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
33 102359071	MeOH Blank			8.135	211 M	9.52
34 102359072	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
35 102359073	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
36 102359074	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
37 102359075	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
38 102359076	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
39 102359077	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
40 102359078	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
41 102359079	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
42 102359080	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
43 102359081	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
44 102359082	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52
45 102359083	TU0145-MS10139-820-BLK-12, SEE			8.135	211 M	9.52

NOV 03 1999: Quantified and printed by NIST on 10/26/99

Quantify Compound Summary Report
 Run 145: Rat Sero, Day 144 Dilutions, Ext 10/19/99

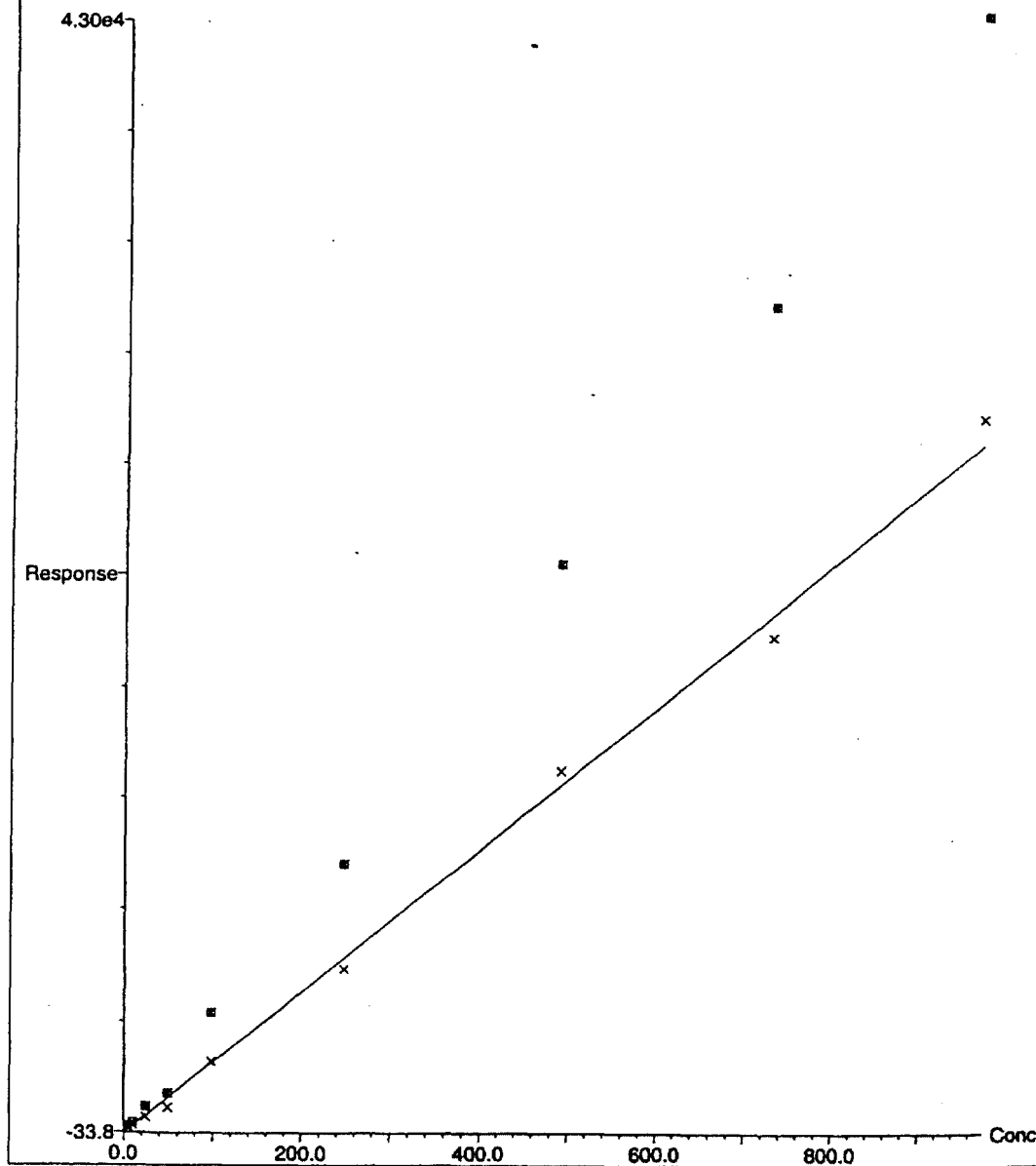
Sample List: C:\MSDCHEM\seup020199.pro\SampleDB\102599b
 Last modified: Mon Oct 25 16:31:42 1999
 Method: C:\MSDCHEM\seup020199.pro\MethodDB\102599b
 Last modified: Tue Oct 26 09:11:31 1999
 Job Code:

Printed: Tue Oct 26 10:51:19 1999

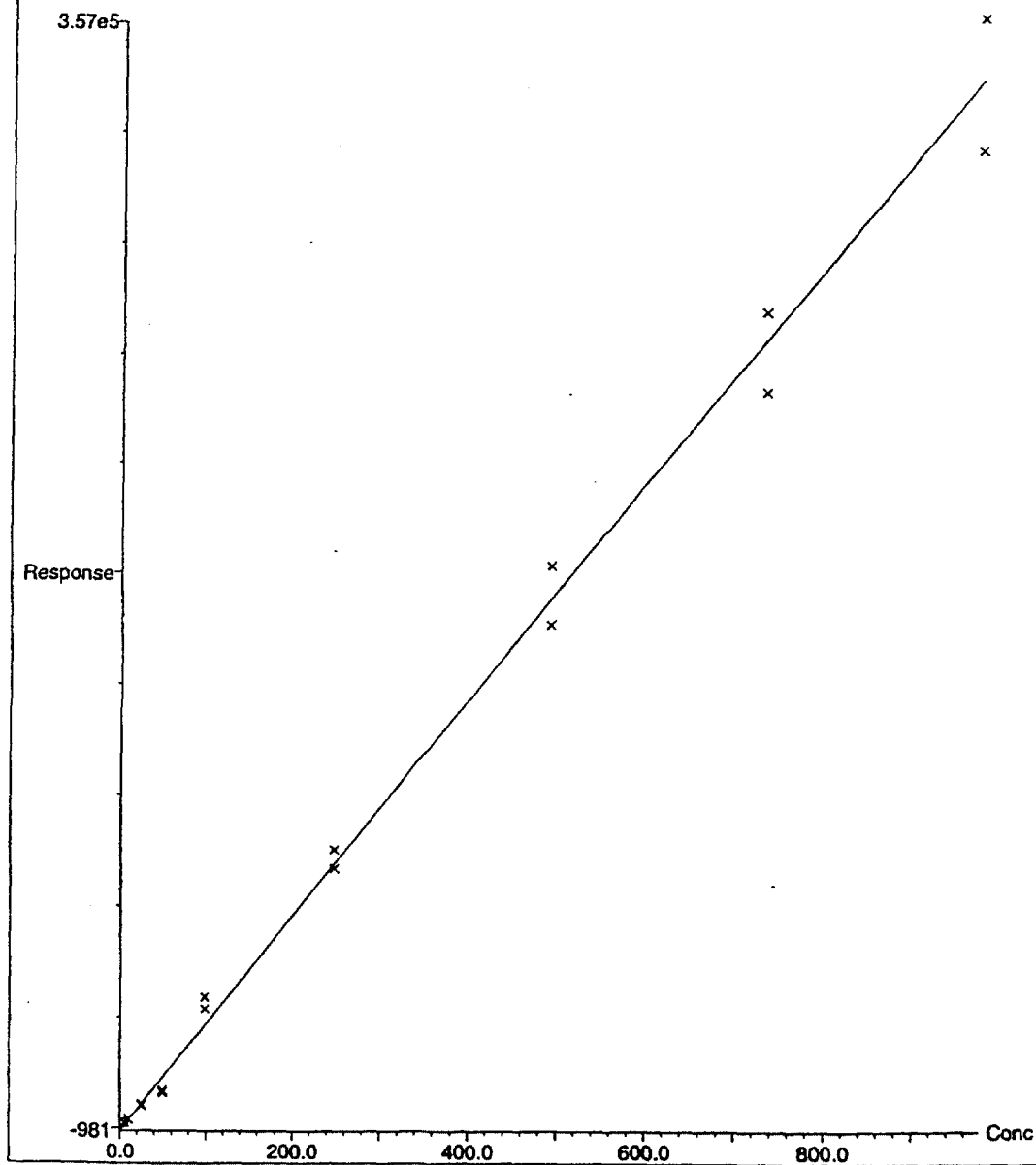
Compound 3: Start

#	Name	Sample/Unit Test	Type	Std Conc	RT	Area	Flags	Conc.	Abov
1	102599039	MeOH Blank			7.101	14	bb	0.00	-100
2	102599040	MeOH Blank							
3	102599041	TOX145-RAS10199-KO Btk-12, SER			7.213	3451	bb	1.11	11
4	102599042	TOX145-RAS10199-Sarum Btk-12, SER			7.213	2874	bb	0.91	-9
5	102599043	TOX145-RAS10199-5ppb-12, SER			7.230	2230	bb	0.71	-29
6	102599044	TOX145-RAS10199-5ppb-12, SER	Standard	1.00	7.227	3080	bb	0.98	-2
7	102599045	TOX145-RAS10199-10ppb-12, SER	Standard	1.00	7.225	2937	bb	0.93	-7
8	102599046	TOX145-RAS10199-25ppb-12, SER	Standard	1.00	7.201	2890	bb	0.92	-8
9	102599047	TOX145-RAS10199-50ppb-12, SER	Standard	1.00	7.211	2576	bb	0.82	-18
10	102599048	TOX145-RAS10199-100ppb-12, SER	Standard	1.00	7.230	2965	bb	0.94	-6
11	102599049	TOX145-RAS10199-100ppb-12, SER	Standard	1.00	7.253	3053	MM	0.97	-3
12	102599050	TOX145-RAS10199-250ppb-12, SER	Standard	1.00	7.229	3171	bb	1.01	1
13	102599051	TOX145-RAS10199-750ppb-12, SER	Standard	1.00	7.205	3030	bb	0.96	-4
14	102599052	TOX145-RAS10199-1000ppb-12, SER	Standard	1.00	7.214	3041	MM	0.96	-4
15	102599053	MeOH Blank			7.231	9	bb	0.00	-100
16	102599054	TOX145-RTS-980224SM, Day1, Smg/kg, D50-ant10/19/99			7.163	69	bb	0.02	-96
17	102599055	TOX145-RTS-980224SM, Day1, Smg/kg, D50-ant10/19/99			7.251	66	MM	0.02	-96
18	102599056	TOX145-RTS-980224SM, Day1, Smg/kg, D50-ant10/19/99			7.211	71	bb	0.02	-94
19	102599057	TOX145-RTS-980224SM, Day1, Smg/kg, D50-ant10/19/99			7.171	43	bb	0.01	-99
20	102599058	TOX145-RTS-980224SM, Day1, Smg/kg, D50-ant10/19/99			7.146	49	bb	0.02	-98
21	102599059	TOX145-RAS10199-250ppb-CCV-11-1, SER	QC	1.00	7.183	2685	bb	0.91	-9
22	102599060	TOX145-RAS10199-250ppb-12, SER	QC	1.00	7.148	2800	bb	0.89	-11
23	102599061	MeOH Blank			7.160	3	bb	0.00	-100
24	102599062	TOX145-RTS-980225SM, Day4, Smg/kg, D50-ant10/19/99			7.163	47	bb	0.02	-98
25	102599063	TOX145-RTS-980225SM, Day4, Smg/kg, D50-ant10/19/99			7.119	45	bb	0.01	-99
26	102599064	TOX145-RTS-980225SM, Day4, Smg/kg, D50-ant10/19/99			7.074	60	bb	0.02	-98
27	102599065	TOX145-RTS-980225SM, Day4, Smg/kg, D50- 10/19/99			7.093	60	MM	0.02	-98
28	102599066	TOX145-RTS-980225SM, Day4, Smg/kg, D50- 10/19/99			7.074	55	MM	0.02	-98
29	102599067	TOX145-RAS10199-250ppb-CCV-11-3, SER	QC	1.00	7.056	1907	MM	0.60	-40
30	102599068	TOX145-RAS10199-250ppb-12, SER	QC	1.00	7.076	2628	bd	0.90	-10
31	102599069	TOX145-RAS10199-250ppb-CCV-11-3, SER	QC	1.00	7.102	2949	bb	0.93	-7
32	102599070	TOX145-RAS10199-250ppb-CCV-11-4, SER	QC	1.00	7.080	2911	bb	0.92	-8
33	102599071	MeOH Blank			7.100	8	MM	0.00	-100
34	102599072	TOX145-RAS10199-KO Btk-12, SER			7.182	3418	bb	1.15	15
35	102599073	TOX145-RAS10199-Sarum Btk-12, SER			7.114	3157	bd	1.00	0
36	102599074	TOX145-RTS10199-Sarum Btk-12, SER			7.088	2944	bd	0.93	-7
37	102599075	TOX145-RAS10199-5ppb-12, SER	Standard	1.00	7.101	3177	bd	1.01	1
38	102599076	TOX145-RAS10199-10ppb-12, SER	Standard	1.00	7.092	3076	MM	0.98	-2
39	102599077	TOX145-RAS10199-25ppb-12, SER	Standard	1.00	7.121	2960	MM	0.94	-6
40	102599078	TOX145-RAS10199-50ppb-12, SER	Standard	1.00	7.092	2748	bb	0.87	-11
41	102599079	TOX145-RAS10199-100ppb-12, SER	Standard	1.00	7.099	3443	bd	1.09	9
42	102599080	TOX145-RAS10199-250ppb-12, SER	Standard	1.00	7.102	3418	bb	1.09	9
43	102599081	TOX145-RAS10199-500ppb-12, SER	Standard	1.00	7.082	3715	bb	1.18	18
44	102599082	TOX145-RAS10199-750ppb-12, SER	Standard	1.00	7.101	3771	bb	1.20	20
45	102599083	TOX145-RAS10199-1000ppb-12, SER	Standard	1.00	7.098	3655	MM	1.17	17

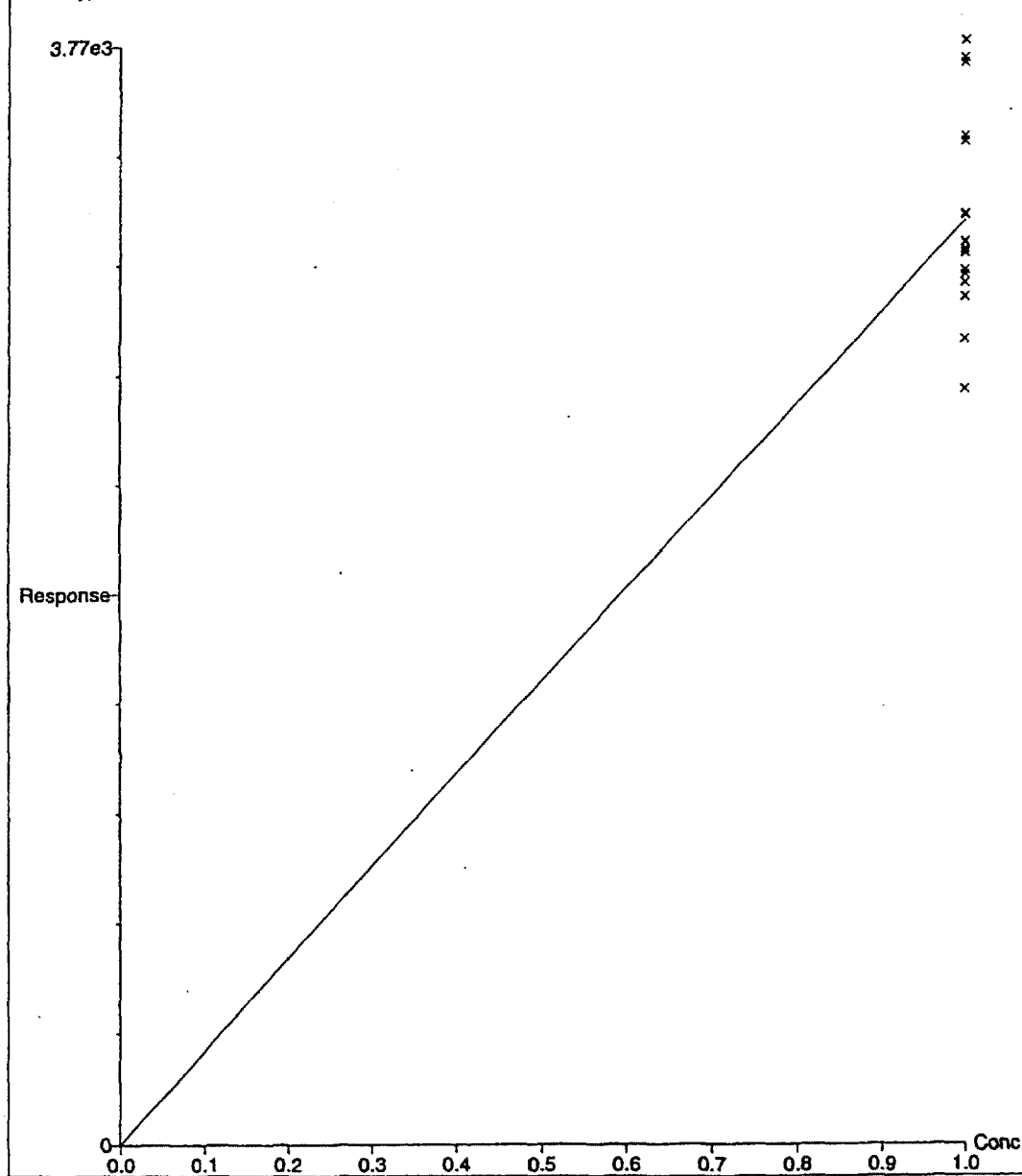
Compound 1 name: PFOS
Coefficient of Determination: 0.997063
Calibration curve: $27.1171 \cdot x + -33.7747$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound 2 name: PFOSA
Coefficient of Determination: 0.992571
Calibration curve: $345.863 \cdot x + -981.494$
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



Method Report
 Method File: C:\MSDCHEM\MSDCHEM\102399b.mdb
 Date modified: Tue Oct 26 09:11:31 1999
 Compound: 1: PYOS
 Printed: Tue Oct 26 10:48:14 1999

Compound Name: PYOS
 Internal Ref.: (None)
 Quantity: 1.000000
 Conc. of Standard: Conc. A

User RF Value: 1.000000, Disabled
 User Peak Factor: 1.000000
 Reference Window Factor: 2.000
 Chromatogram Scale Window: 1.000000

Retention Time (min): 7.450, Disabled
 Retention Time (min): 7.450, Disabled
 RT Reference: (None)
 Time Window (min): 0.121

Peak Selection: Manual

Response Type: Retention
 Response: Area

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

Units:

Peak-to-peak amplitude: 76

Smoothing: Disabled
 Window size (scans): 2
 Smoothing method: 1

Join valley threshold: 30.00
 Minimum baseline: 20.00
 Noise baseline minimum: 5.00

Draw vertical threshold: 50.00
Detect shoulders: Disabled
Shoulder threshold: 10.00

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\comp620199.prp\MethDB\102599b.mdb
Last modified: Tue Oct 26 09:11:11 1999
Compound: 2: PFOSA
Printed: Tue Oct 26 10:48:16 1999

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

Retention Time (min): 7.966, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (min): 0.000

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): 3
Number of smooths: 1
Smoothing method:

Join valley threshold: 10.00
Reduce peak calling: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 10.00

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:\MSDCHEM\msd010199.pro\MethodM102397b.mdb
 Last modified: Tue Oct 26 09:11:11 1999
 Compound: 1: Butt
 Printed: Tue Oct 26 10:48:14 1999

Compound Name: Butt
 Internal Ref.: (none)
 Quantify Trace: TIC
 Conc. of Standards: 9.100
 User RP Value: 1.000000, Disabled
 User Peak Factor: 1.000000
 Integrate Window Factor: 2.000
 Chromatogram Mass Window: 1.00000e

Retention Time (min): 7.245, Enabled
 Relative Retention Time: 0.000, Disabled
 RT Reference: (none)
 Time Window (min): 0.301

Peak Selection: Nearest

Response Type: External
 Response: Area

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

Units:

Peak-to-peak amplitude: 45
 Smoothing: Enabled
 Window size (scan): 2
 Number of scans: 1
 Smoothing method:

Join valley threshold: 30.00
 Join peak threshold: 200.00
 Baseline threshold: 2.00

Draw vertical threshold:
Draw threshold:
Shoulder threshold:

99.00
99.00
30.00

Relative height:
Absolute height:
Relative area:
Absolute area:

1.35 Disabled
0.00 Disabled
2.00 Disabled
0.00 Disabled

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

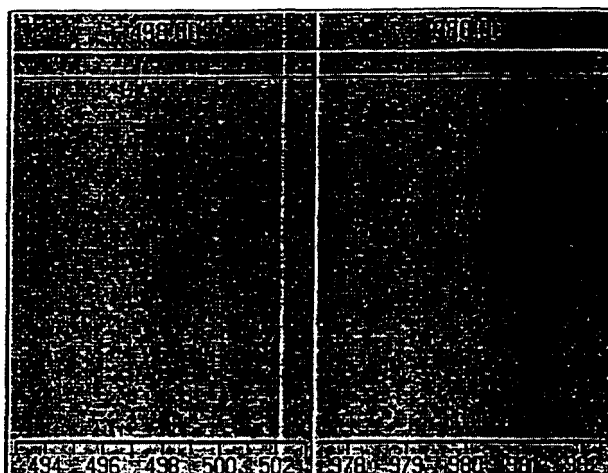
Time (min.)	%B
1) <u>0.00</u>	<u>10.0</u>
2) <u>1.00</u>	<u>10.0</u>
3) <u>5.50</u>	<u>95.0</u>
4) <u>7.50</u>	<u>95.0</u>
5) <u>8.00</u>	<u>10.0</u>

Tuning Method Report

Page 1

Method: C:\MASSLYNX\SOUP-RATS.PRO\ACQUDB\PPOS3

Printed: Mon Oct 25 16:37:32 1999



MS

MS

SOURCE (ZSP-)	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
Analyser 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	

Scanning Method Report

Page 1

Method: C:\MSDCHEM\SCUP-RATE.PRO\ACQDATA\18KHZ-PT04-FOAA-PT04A-18
Last Modified: Fri Oct 15 11:35:03 1999
Printed: Mon Oct 25 16:28:24 1999

Solvent Delay (mins) : 0.00

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

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 (MSF-)

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	412.00	99.00	0.30	45	60

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

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	418.00	78.00	0.30	45	60

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

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	88.00	0.30	40	60

Method Report

Page 1

Method File: c:\masslynx\scup-rate.pro\acqdb\10min-10-95
 Last Modified: Monday, October 25, 1999 16:29:58
 Printed: Monday, October 25, 1999 16:34:46

MP1100 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

MP1100 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

MP1100 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

MP1100 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

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

Sample List: C:\MASSLYNX\soup-raiis.pro\SampleDB\102599b.SPL
 Printed: Mon Oct 25 18:32:57 1999

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	493	493
7	10min-10-95	25	10	Standard	976	977
8	10min-10-95	26	10	Standard	248	248
9	10min-10-95	27	10	Standard	493	493
10	10min-10-95	28	10	Standard	976	977
11	10min-10-95	29	10	Standard	493	493
12	10min-10-95	30	10	Standard	735	736
13	10min-10-95	31	10	Standard	976	977
14	10min-10-95	32	10	--	--	--
15	10min-10-95	82	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	OC	248	248
22	10min-10-95	29	10	OC	248	248
23	10min-10-95	82	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	OC	248	248
30	10min-10-95	29	10	OC	248	248
31	10min-10-95	45	10	OC	248	248
32	10min-10-95	46	10	OC	248	248
33	10min-10-95	83	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	493	493
38	10min-10-95	25	10	Standard	976	977
39	10min-10-95	26	10	Standard	248	248
40	10min-10-95	27	10	Standard	493	493
41	10min-10-95	28	10	Standard	976	977
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

FACT: DATA REVIEW SUMMARY FORM

Date: 10-28-99
 Data Analyst: mmn
 FACT Study #: Tox145
 Matrix/Timepoints: Int. Liver, day 144
 Sample List: 102799.6
 Set-up Analysis Date/Analyst: 10-27-99 mmn
 Instrument: DAIY 070799

Data Review Sign-Off

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

Technical Review Name / Date: MEC 10/28/99 / k/h 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: <1/200
 R²: 0.9952 Matrix Blank: <1/200
 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: SUV
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: _____

- ① excluded and curve due to divergence. mmn 10-28-99
- ② excluded bottom 4 points to meet 3σ Deviation Criteria and bottom 3 points did not have area counts that were 2x matrix blank. mmn 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. mmn 10-28-99
- ④ the analysis of 980 was for screening purposes only. mmn 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

Sample List: C:\MSI\TNC\Down\070798, PNO\Sample\01079808
 Last modified: Thu Oct 28 09:14:03 1999
 Method: C:\MSI\TNC\Down\070798, PNO\MSI\TNC\01079808
 Last modified: Thu Oct 28 09:17:12 1999
 Data Code:

Printed: Thu Oct 28 09:17:12 1999

Component 1: PNOA (Crew 1)

Sample	Sample	Std Comp	EC	Area	Comp (ppm)	Elaps	Moist
1 102798013	Sample	6.421	6.421	342	0.00	100	
2 102798013	Moist Blank	6.793	6.793	315	0.00	100	
3 102798015	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
4 102798015	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
5 102798017	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
6 102798018	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
7 102798019	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
8 102798020	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
9 102798021	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
10 102798022	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
11 102798023	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
12 102798024	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
13 102798025	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
14 102798026	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
15 102798027	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
16 102798028	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
17 102798029	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
18 102798030	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
19 102798031	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
20 102798032	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
21 102798033	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
22 102798034	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
23 102798035	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
24 102798036	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
25 102798037	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
26 102798038	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
27 102798039	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
28 102798040	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
29 102798041	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
30 102798042	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
31 102798043	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
32 102798044	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
33 102798045	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
34 102798046	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
35 102798047	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
36 102798048	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
37 102798049	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
38 102798050	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
39 102798051	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
40 102798052	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
41 102798053	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
42 102798054	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
43 102798055	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
44 102798056	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
45 102798057	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
46 102798058	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
47 102798059	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
48 102798060	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
49 102798061	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
50 102798062	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
51 102798063	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
52 102798064	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
53 102798065	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
54 102798066	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
55 102798067	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
56 102798068	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
57 102798069	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
58 102798070	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	
59 102798071	TOX15-MS10223-620 B1-1, MW	6.421	6.421	3022	0.00	100	

Quality Control Summary Report
 Test 145: Fat, Serum and Liver, Day 1 & 2

Sample List: C:\MSR\IN\Q\Q00707799.m0\SampleList\1077906
 Last Modified: Thu Oct 28 09:27:15 1999
 Sample List: C:\MSR\IN\Q\Q00707799.m0\SampleList\1077906
 Last Modified: Thu Oct 28 09:27:15 1999
 Job Code:

Printed: Thu Oct 28 09:27:15 1999

Component 2: 980 (Item 2)									
6 Phase	Sample List Text	Std Curve	RT	Area	Conc. (ppb)	Flags	Obs		
1 10279013	MoCh Blank								
2 10279013	MoCh Blank								
3 10279013	TOX145-MS10229-420 BL-1, MW								
4 10279014	TOX145-MS10229-420 BL-1, MW	0.88	7.542	24	0.88 MW		1		
5 10279017	TOX145-MS10229-420 BL-1, MW	1.93	7.582	64	1.93 MW		-7		
6 10279017	TOX145-MS10229-420 BL-1, MW	8.76	7.584	173	8.76 MW		-2		
7 10279018	TOX145-MS10229-420 BL-1, MW	24.80	7.586	396	24.80 MW		-4		
8 10279020	TOX145-MS10229-420 BL-1, MW	49.30	7.594	649	49.30 MW		6		
9 10279021	TOX145-MS10229-420 BL-1, MW	97.40	7.596	1336	97.40 MW		0		
10 10279022	TOX145-MS10229-420 BL-1, MW	248.00	7.598	3918	248.00 MW		-5		
11 10279023	TOX145-MS10229-420 BL-1, MW	493.00	7.599	6110	493.00 MW		-26		
12 10279023	TOX145-MS10229-420 BL-1, MW	976.00	7.599	9343	976.00 MW		-21		
13 10279027	MoCh Blank								
14 10279028	TOX145-RT-980323MW, Day1, control, -act10/19/99								
15 10279029	TOX145-RT-980323MW, Day1, control, -act10/19/99								
16 10279030	TOX145-RT-980323MW, Day1, control, -act10/19/99								
17 10279030	TOX145-RT-980323MW, Day1, control, -act10/19/99								
18 10279030	TOX145-RT-980323MW, Day1, control, -act10/19/99								
19 10279031	TOX145-RT-980323MW, Day1, control, -act10/19/99								
20 10279032	TOX145-RT-980323MW, Day1, control, -act10/19/99								
21 10279033	TOX145-MS10229-420 BL-1, MW	248.00	7.593	4604	248.00 MW		-3		
22 10279033	MoCh Blank								
23 10279033	TOX145-RT-980323MW, Day1, control, -act10/19/99								
24 10279036	TOX145-RT-980323MW, Day1, control, -act10/19/99								
25 10279037	TOX145-RT-980323MW, Day1, control, -act10/19/99								
26 10279038	TOX145-RT-980323MW, Day1, control, -act10/19/99								
27 10279039	TOX145-RT-980323MW, Day1, control, -act10/19/99								
28 10279040	TOX145-MS10229-420 BL-1, MW	248.00	7.588	4224	248.00 MW		2		
29 10279041	MoCh Blank								
30 10279042	TOX145-RT-980323MW, Day1, control, -act10/19/99								
31 10279043	TOX145-RT-980323MW, Day1, control, -act10/19/99								
32 10279044	TOX145-RT-980323MW, Day1, control, -act10/19/99								
33 10279045	TOX145-RT-980323MW, Day1, control, -act10/19/99								
34 10279046	TOX145-RT-980323MW, Day1, control, -act10/19/99								
35 10279047	TOX145-MS10229-420 BL-1, MW	248.00	7.593	3343	248.00 MW		-2		
36 10279048	MoCh Blank								
37 10279049	TOX145-RT-980323MW, Day1, control, -act10/19/99								
38 10279050	TOX145-RT-980323MW, Day1, control, -act10/19/99								
39 10279051	TOX145-RT-980323MW, Day1, control, -act10/19/99								
40 10279052	TOX145-RT-980323MW, Day1, control, -act10/19/99								
41 10279053	TOX145-RT-980323MW, Day1, control, -act10/19/99								
42 10279054	TOX145-MS10229-420 BL-1, MW	248.00	7.598	4020	248.00 MW		-3		
43 10279055	MoCh Blank								
44 10279056	TOX145-RT-980323MW, Day1, control, -act10/19/99								
45 10279057	TOX145-RT-980323MW, Day1, control, -act10/19/99								
46 10279058	TOX145-MS10229-420 BL-1, MW	248.00	7.591	3935	248.00 MW		-10		
47 10279059	MoCh Blank								
48 10279060	TOX145-MS10229-420 BL-1, MW								
49 10279061	TOX145-MS10229-420 BL-1, MW	0.99	7.525	23	0.99 MW		-5		
50 10279062	TOX145-MS10229-420 BL-1, MW	4.93	7.580	100	4.93 MW		12		
51 10279063	TOX145-MS10229-420 BL-1, MW	9.76	7.578	152	9.76 MW		-11		
52 10279064	TOX145-MS10229-420 BL-1, MW	21.80	7.586	427	21.80 MW		1		
53 10279065	TOX145-MS10229-420 BL-1, MW	49.30	7.597	818	49.30 MW		-1		
54 10279066	TOX145-MS10229-420 BL-1, MW	97.40	7.599	1608	97.40 MW		11		
55 10279067	TOX145-MS10229-420 BL-1, MW	194.80	7.598	2612	194.80 MW		2		
56 10279068	TOX145-MS10229-420 BL-1, MW	389.60	7.598	4312	389.60 MW		-19		
57 10279069	TOX145-MS10229-420 BL-1, MW	779.20	7.596	6918	779.20 MW		8		
58 10279070	TOX145-MS10229-420 BL-1, MW								
59 10279071	TOX145-MS10229-420 BL-1, MW	976.00	7.596	11346	976.00 MW		-30		

Quantity Compound Summary Report

Run Date and Time: Day 1 & 4

Sample List: C:\MSDCHEM\Sample070799.Pro\SampleList\102799b

Last Modified: Thu Oct 28 09:14:01 1999

Method: C:\MSDCHEM\Sample070799.Pro\Method\102799b

Last Modified: Thu Oct 28 09:17:32 1999

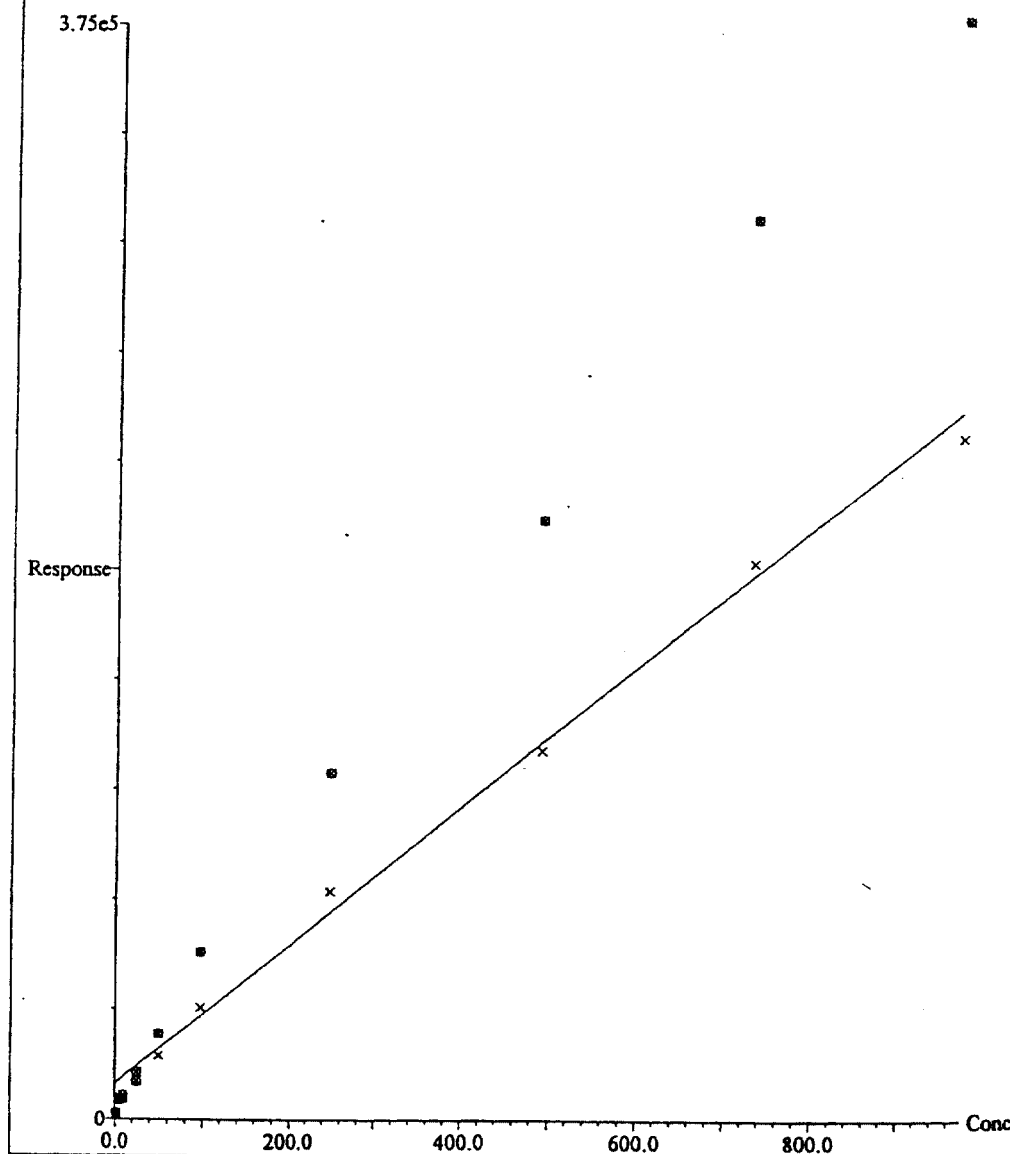
Job Code:

Printed: Thu Oct 28 09:27:15 1999

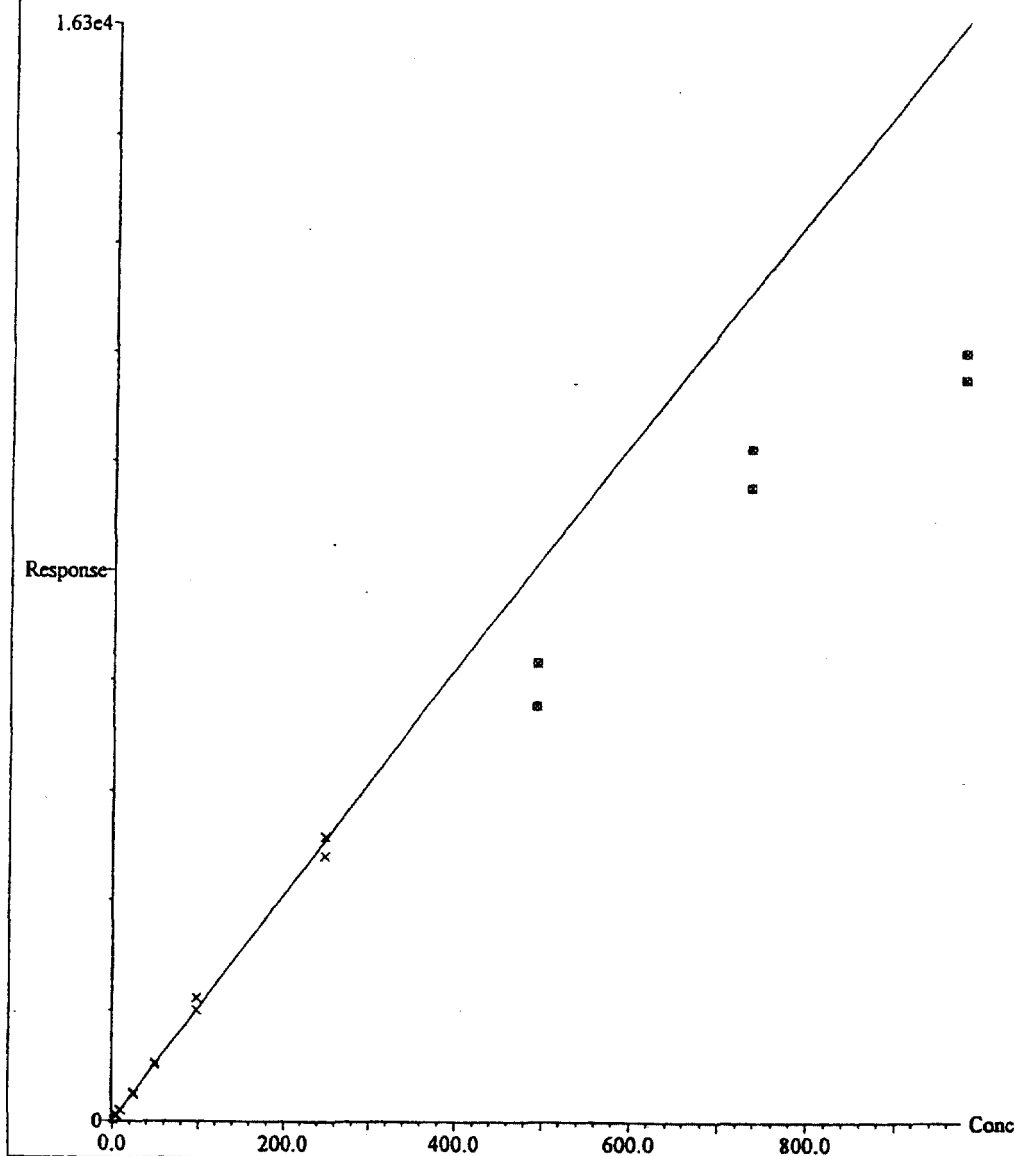
Compound 3: pure (raw 3)

Peak	Sample List	814 Count	RT	Area	Conc. (ppb)	Flags	Qty
1	102799013						
2	102799014						
3	102799015						
4	102799016						
5	102799017						
6	102799018						
7	102799019						
8	102799020						
9	102799021						
10	102799022						
11	102799023						
12	102799024						
13	102799025						
14	102799026						
15	102799027						
16	102799028						
17	102799029						
18	102799030						
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22	102799034						
23	102799035						
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25	102799037						
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230	102799242						

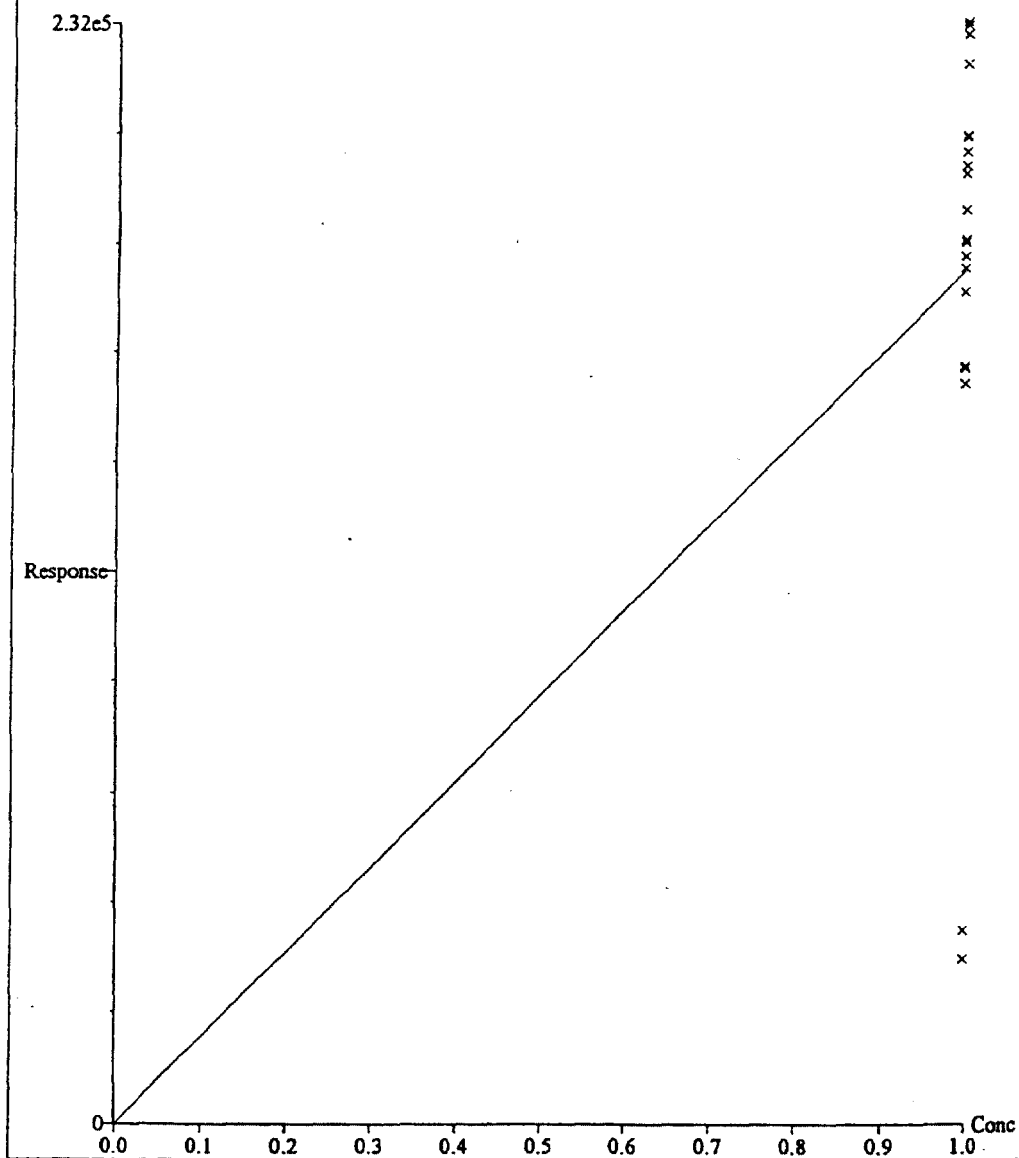
Compound 1 name: POAA (func 1)
Coefficient of Determination: 0.995226
Calibration curve: $234.909 * x + 12236.1$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



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



Method Report

Page 1

Method file: C:\MASSLYN\Devey\070799.PQA\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): 6.812, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.114

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

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

Polymer type: Linear
Point of origin: Exclude
File weighting: 1/x
Axis transformation: None

Units:

Peak-to-peak amplitude:

444

Smoother:

Disabled

Window size (scans):

1

Number of scans:

1

Smoother method:

Sevitzky Golay

John valley threshold:

1.00

Peak-to-peak valley:

3.00

Base baseline maximum:

10.00

Drew 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

Method Report

Page 2

Method file: C:\MASSLIT\Devego70799.PRO\MSDC08\102799b.mtd
 Last modified: Thu Oct 28 08:57:32 1999

Compound: Z: 980 (Func 2)

Printed: Thu Oct 28 12:41:01 1999

Compound Name: 980 (Func 2)
 Internal Ref.: [None]
 Quantity Traced: TIC
 Conc. of Standards: Conc. A

User RF Value: 1.00000, Disabled
 User Peak Factor: 1.00000
 Integrate Window Factor: 2.000
 Chromatogram Mass Window: 1.00000Da

Retention Time (min): 7.620, Enabled
 Relative Retention Time: 0.800, Disabled
 RT Reference: [None]
 Time Window (min): 0.359

Peak Selection:

None

Response Type:

External Areas

Method Report

Method file: C:\MASSLYTK\Desy070799\FMO\Method08\102799b.mdb

Last modified: Thu Oct 28 08:57:32 1999

Compound: 3: sucr (func 3)

Printed: Thu Oct 28 12:11:03 1999

Compound Name: sucr (func 3)
 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: 1.00000e

Retention Time (mins): 6.629, Enabled
 Relative Retention Time: 0.000, Disabled
 RT Reference: (None)
 Time Window (mins): 0.265

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: 629

Soothing: Disabled
 Smoothing (waves): 1
 Number of samples: 1
 Smoothing method: Savitzky Golay

Join valley threshold:
Detect peak falling:
Raise baseline maximum:

5.00
30.00
10.00

Draw vertical threshold:
Detect shoulders:
Shoulder threshold:

25.00
Disabled
5.00

Relative height:
Absolute height:
Relative area:
Absolute area:

1-10, Disabled
0.00, Disabled
2.00, Enabled
0.00, Disabled

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) m-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

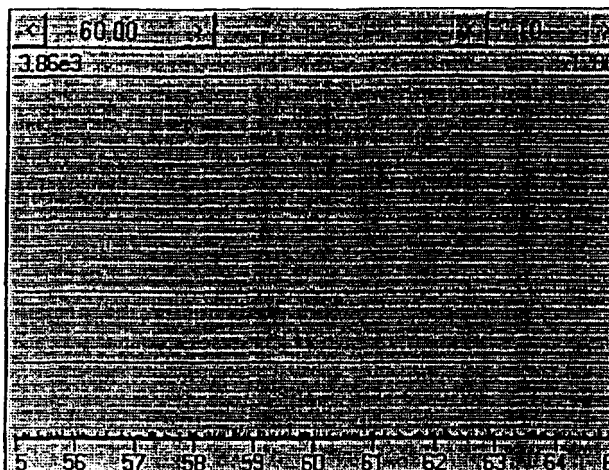
Time (min.)	%B
1) <u>0.00</u>	<u>10.0</u>
2) <u>1.00</u>	<u>10.0</u>
3) <u>5.50</u>	<u>95.0</u>
4) <u>7.50</u>	<u>95.0</u>
5) <u>8.00</u>	<u>10.0</u>

Tuning Method Report

Page 1

Method: C:\MASSLYNX\DAVEY BACKUP AUG99\DAVEY-RATS.PRO\ACQUD8\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			Gas Flows		
	Rdbk			Rdbk	
Analysr Vacuum	OFF		Cone Gas	57.8	
Gas Cell	4.3e-3		Desolvation	484.8	

Scanning Method Report

Page 1

Method: C:\MSI\NET\DAVEY\070789.990\ACQ\DS\10MCM-90AA-990-12
Last Modified: Wed Oct 27 14:18:51 1999
Printed: Wed Oct 27 14:22:05 1999

Solvent Delay (mins) : 0.00

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

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 (ESI-)

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 (ESI-)

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

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

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

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	--

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

59	102799071	TOX145-RBS10229-1000ppb-1, RWW

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	—

FACT: DATA REVIEW SUMMARY FORM

Data Review Sign-Off

Date: 11/05/99
 Data Analyst: JAS
 FACT Study #: Tox 145
 Matrix/Timepoints: Pol/Sun - Day 21 dose and control - ext 11/5/99 MCH
 Sample List: 110199a
 Set-up Analysis Date/Analyst: JAS 11/04/99
 Instrument: Sup 020199

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: 9.76ppb → 735ppb
 LOQ: 9.76ppb Extracted Blank: less than LOQ
 R²: 0.980777 Matrix Blank: less than LOQ
 Calibration Checks: OK - Range from -7% → -17%
 MS/MSD: OK - one value at 69% still reported ^{unc}
 Data Status: dosed samples need 1:10 dilution ^{11/05/99}

Analyte: PFOSA (CH3)
 Acceptable Linear Range: 4.93ppb → 977ppb
 LOQ: 4.93ppb Extracted Blank: less than LOQ
 R²: 0.997747 Matrix Blank: less than LOQ
 Calibration Checks: OK - Range from -5% → -13%
 MS/MSD: OK ^{unc} 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, 4.93ppb, 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 976ppb 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

FACT: DATA REVIEW SUMMARY FORM

Data Review Sign-Off

Date: 11/05/99
 Data Analyst: JAS
 FACT Study #: Tox 145
 Matrix/Timepoints: Pat Sera - Day 29 dose and control - cel 11/3/99 MCH
 Sample Lot: 110499a
 Set-up Analysis Date/Analyst: Jas 11/04/99
 Instrument: Soup 020199

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

Technical Review
 Name / Date: See page one LAC 11/05/99

Quality Assurance Review
 Name / Date: _____

Data Entry: LAC 11/05/99

Comments / Justifications: _____

Analyte: <u>Serr (CH4)</u>	
Acceptable Linear Range: <u>NA</u>	
LOQ: <u>NA</u>	Extraction Blank: <u>NA</u>
R ² : <u>NA</u>	Matrix Blank: <u>NA</u>
Calibration Checks: <u>NA</u>	
MS/MSD: <u>NA</u>	
Data Status: <u>NA</u>	

Analyte: _____	
Acceptable Linear Range: _____	
LOQ: _____	Extracted Blank: _____
R ² : _____	Matrix Blank: _____
Calibration Checks: <u>Jas 11/05/99</u>	
MS/MSD: _____	
Data Status: _____	

Analyte: _____	
Acceptable Linear Range: _____	
LOQ: _____	Extracted Blank: _____
R ² : _____	Matrix Blank: _____
Calibration Checks: _____	
MS/MSD: _____	
Data Status: _____	

[Large diagonal line across the right side of the page, with handwritten "LAC 11/05/99" written vertically along it.]

Quantify Compound Recovery Report
 TQ0113-Rat case, day 29 nose and control - cont. 11/13/99 MCM

C:\msd1\TQX1\comp\2010129_grow\temp\comp\11040994
 Last modified: Fri Mar 05 06:20:33 1999
 Method: C:\msd1\TQX1\comp\2010129_grow\MethodM\11040994
 Last modified: Fri Mar 05 06:20:44 1999
 Job Code:

Printed: Fri Nov 85 06:12:31.1999						
Completed: 1: P004 (ChL)						
0 Name	Sample/Inst Event	Type	Old Count	RT	Airway Phase	Count/Day
1 110499001	MOON Blank					
2 110499002	MOON Blank					
3 110499003	MOON Blank					
4 110499004	VOYL145-ASR11039-4000-8-11/3/99 MCH			7.047	102 MB	1.49 48
5 110499005	VOYL145-ASR11039-4000-8-11/3/99 MCH			7.043	114 MB	1.67 67
6 110499006	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.102	94 MB	1.37 37
7 110499007	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.128	44 MB	1.16 36
8 110499008	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.128	44 MB	1.16 36
9 110499009	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.128	44 MB	1.16 36
10 110499010	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.111	71 MB	1.47 7
11 110499011	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.113	71 MB	1.06 6
12 110499012	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.151	85 MB	0.95 5
13 110499013	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.151	85 MB	0.95 5
14 110499014	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.113	86 MB	1.28 28
15 110499015	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.113	86 MB	1.28 28
16 110499016	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.090	103 MB	1.50 50
17 110499017	VOYL145-ASR11039-4000-8-11/3/99 MCH			7.113	54 MB	0.92 18
18 110499018	VOYL145-ASR11039-4000-8-11/3/99 MCH			7.113	54 MB	0.92 18
19 110499019	VOYL145-ASR11039-4000-8-11/3/99 MCH			7.113	54 MB	0.92 18
20 110499020	VOYL145-ASR11039-4000-8-11/3/99 MCH			7.129	57 MB	0.83 18
21 110499021	VOYL145-ASR11039-4000-8-11/3/99 MCH			7.129	57 MB	0.83 18
22 110499022	VOYL145-ASR11039-4000-8-11/3/99 MCH	QC	1.00	7.132	43 MB	0.80 20
23 110499023	VOYL145-ASR11039-4000-8-11/3/99 MCH			7.132	43 MB	0.83 17
24 110499024	VOYL145-ASR11039-4000-8-11/3/99 MCH			7.150	54 MB	0.78 22
25 110499025	VOYL145-ASR11039-4000-8-11/3/99 MCH			7.134	54 MB	0.95 15
26 110499026	VOYL145-ASR11039-4000-8-11/3/99 MCH			7.151	54 MB	0.82 18
27 110499027	VOYL145-ASR11039-4000-8-11/3/99 MCH			7.135	113 MB	1.45 45
28 110499028	VOYL145-ASR11039-4000-8-11/3/99 MCH			7.135	113 MB	1.45 45
29 110499029	VOYL145-ASR11039-4000-8-11/3/99 MCH	QC	1.00	7.086	61 MB	0.98 11
30 110499030	MOON Blank					
31 110499031	VOYL145-ASR11039-4000-8-11/3/99 MCH			7.108	113 MB	1.43 43
32 110499032	VOYL145-ASR11039-4000-8-11/3/99 MCH			7.089	113 MB	1.52 52
33 110499033	VOYL145-ASR11039-4000-8-11/3/99 MCH	QC	1.00	7.106	48 MB	0.49 11
34 110499034	VOYL145-ASR11039-4000-8-11/3/99 MCH			7.127	94 MB	1.31 31
35 110499035	VOYL145-ASR11039-4000-8-11/3/99 MCH			7.159	52 MB	0.76 24
36 110499036	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.137	46 MB	0.68 12
37 110499037	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.147	38 MB	0.67 11
38 110499038	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.147	38 MB	0.67 11
39 110499039	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.156	43 MB	0.85 13
40 110499040	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.085	47 MB	0.69 11
41 110499041	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.105	50 MB	0.73 27
42 110499042	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.111	55 MB	0.66 12
43 110499043	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.111	55 MB	0.66 12
44 110499044	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.111	55 MB	0.66 12
45 110499045	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.113	70 MB	1.03 3
46 110499046	VOYL145-ASR11039-4000-8-11/3/99 MCH	Standard	1.00	7.136	92 MB	1.34 34

SONG 82019: Quantized and printed by LAS on 11/05/89

Quantify Compound Summary Report
TOL143-rat sera, day 29 dose and control- ext. 11/3/99 MCH

Page 3

Sample List: C:\MSD\SLV\comp620199.pro\SampleDB\110499a
Last modified: Fri Nov 05 06:30:11 1999
Method: C:\MSD\SLV\comp620199.pro\Method\110499a
Last modified: Fri Nov 05 06:30:14 1999
Job Code:

Printed: Fri Nov 05 06:32:31 1999

Compound 3: PFOS (Ch3)

#	Name	Sample/Lot	Type	Std Conc	SE	Area	Flags	Conc.	%Dev
1	110499001	MeOH Blank			7.220	53	bb	0.00	
2	110499002	MeOH Blank			7.220	41	bb	0.00	
3	110499003	MeOH Blank			7.291	26	bb	0.00	
4	110499004	TOL143-RS11039-RTG Blk-9, ext. 11/3/99 MCH			7.106	108	bb	0.00	
5	110499005	TOL143-RS11039-Sera Blk-9, ext. 11/3/99 MCH			7.100	72	bb	0.00	
6	110499006	TOL143-RS11039-Sera Blk-9, ext. 11/3/99 MCH			7.242	84	bb	0.00	
7	110499007	TOL143-RS11039-10ppb-9, ext. 11/3/99 MCH	Standard	4.93	7.267	208	bbX	4.89	-1
8	110499008	TOL143-RS11039-10ppb-9, ext. 11/3/99 MCH	Standard	9.76	7.267	233	bb	9.02	-8
9	110499009	TOL143-RS11039-25ppb-9, ext. 11/3/99 MCH	Standard	24.80	7.245	327	bb	20.47	-17
10	110499010	TOL143-RS11039-50ppb-9, ext. 11/3/99 MCH	Standard	49.26	7.250	995	bb	43.28	-12
11	110499011	TOL143-RS11039-100ppb-9, ext. 11/3/99 MCH	Standard	97.60	7.251	1931	bb	88.51	-9
12	110499012	TOL143-RS11039-250ppb-9, ext. 11/3/99 MCH	Standard	248.00	7.291	4504	bb	214.64	-13
13	110499013	TOL143-RS11039-500ppb-9, ext. 11/3/99 MCH	Standard	493.00	7.244	8983	bb	433.34	-12
14	110499014	TOL143-RS11039-750ppb-9, ext. 11/3/99 MCH	Standard	735.00	7.252	13842	bb	670.62	-9
15	110499015	TOL143-RS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	976.00	7.220	17240	bbX	636.93	-14
16	110499016	MeOH Blank			7.253	193	bb	4.12	
17	110499017	TOL143-RS11039-250ppb-MS9, ext. 11/3/99 MCH			7.250	3926	bb	186.40	
18	110499018	TOL143-RS11039-250ppb-MSD16, ext. 11/3/99 MCH			7.222	3624	bb	172.14	
19	110499019	TOL143-RTG-PR03243M-0-Day 29, ext. 11/3/99 MCH			7.270	57	bb	0.00	
20	110499020	TOL143-RTG-PR03243M-0-Day 29, ext. 11/3/99 MCH			7.260	54	bb	0.00	
21	110499021	TOL143-RTG-PR03243M-0-Day 29, ext. 11/3/99 MCH			7.268	51	bb	0.00	
22	110499022	TOL143-RTG-PR03243M-0-Day 29, ext. 11/3/99 MCH	QC	248.00	7.248	4344	bb	105.81	-17
23	110499023	MeOH Blank			7.259	81	bb	0.00	
24	110499024	TOL143-RTG-PR03243M-0-Day 29, ext. 11/3/99 MCH			7.251	39	bb	0.00	
25	110499025	TOL143-RTG-PR03243M-0-Day 29, ext. 11/3/99 MCH			7.251	40	bb	0.00	
26	110499026	TOL143-RTG-PR03243M-0-Day 29, ext. 11/3/99 MCH			7.248	52663	bb	2164.17	
27	110499027	TOL143-RTG-PR03243M-0-Day 29, ext. 11/3/99 MCH			7.275	38663	bb	2159.14	
28	110499028	TOL143-RTG-PR03243M-0-Day 29, ext. 11/3/99 MCH			7.267	61721	bb	3008.48	
29	110499029	TOL143-RS11039-250ppb-CCV10, ext. 11/3/99 MCH	QC	248.00	7.225	4581	bb	218.39	-12
30	110499030	MeOH Blank			7.271	123	bb	1.19	
31	110499031	TOL143-RTG-PR03243M-0-Day 29, ext. 11/3/99 MCH			7.246	56708	bb	2763.89	
32	110499032	TOL143-RTG-PR03243M-0-Day 29, ext. 11/3/99 MCH			7.251	98061	bb	4782.90	
33	110499033	TOL143-RS11039-250ppb-CCV9, ext. 11/3/99 MCH	QC	248.00	7.224	4843	bb	231.17	-7
34	110499034	MeOH Blank			7.106	182	bb	3.59	
35	110499035	TOL143-RS11039-RTG Blk-9, ext. 11/3/99 MCH			7.266	87	bb	0.00	
36	110499036	TOL143-RS11039-Sera Blk-9, ext. 11/3/99 MCH			7.298	61	bb	0.00	
37	110499037	TOL143-RS11039-Sera Blk-9, ext. 11/3/99 MCH			7.334	107	bb	0.00	
38	110499038	TOL143-RS11039-10ppb-9, ext. 11/3/99 MCH	Standard	4.93	7.248	183	bbX	3.65	-26
39	110499039	TOL143-RS11039-25ppb-9, ext. 11/3/99 MCH	Standard	9.76	7.247	326	bb	10.65	9
40	110499040	TOL143-RS11039-50ppb-9, ext. 11/3/99 MCH	Standard	24.80	7.289	656	bb	26.74	8
41	110499041	TOL143-RS11039-100ppb-9, ext. 11/3/99 MCH	Standard	49.10	7.224	1268	bb	56.44	15
42	110499042	TOL143-RS11039-250ppb-9, ext. 11/3/99 MCH	Standard	97.60	7.244	2489	bb	116.27	13
43	110499043	TOL143-RS11039-500ppb-9, ext. 11/3/99 MCH	Standard	248.00	7.271	5716	bb	273.70	10
44	110499044	TOL143-RS11039-750ppb-9, ext. 11/3/99 MCH	Standard	493.00	7.269	11073	bb	535.41	9
45	110499045	TOL143-RS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	735.00	7.250	16812	bb	615.64	11
46	110499046	TOL143-RS11039-1000ppb-9, ext. 11/3/99 MCH	Standard	976.00	7.277	21935	bbX	1114.59	14

Box 30181: Quantified and printed by LRS on 11/05/99

Quantity Compound Density Report
 T0010-001 Data, day 23 data and control, ant. 11/3/99 MCH

Sample List: C:\MSADSL\T0010-001\T0010-001\1104994

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

Method: C:\MSADSL\T0010-001\T0010-001\1104994

Unit: mg/kg

Unit Code: 11/3/99 MCH

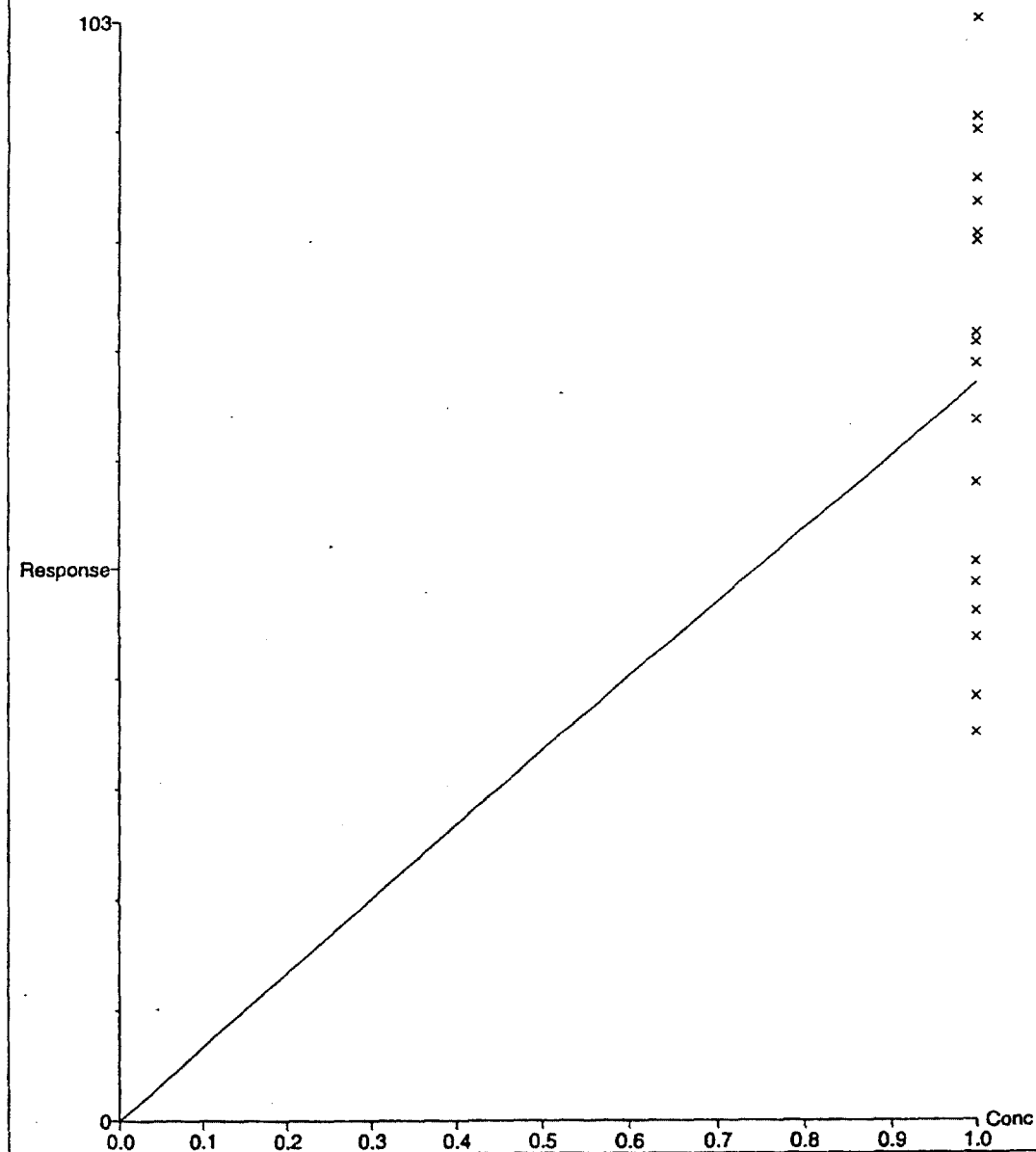
Printed: Fri Nov 05 06:30:18 1999

Compound 4: Pure (Ch)

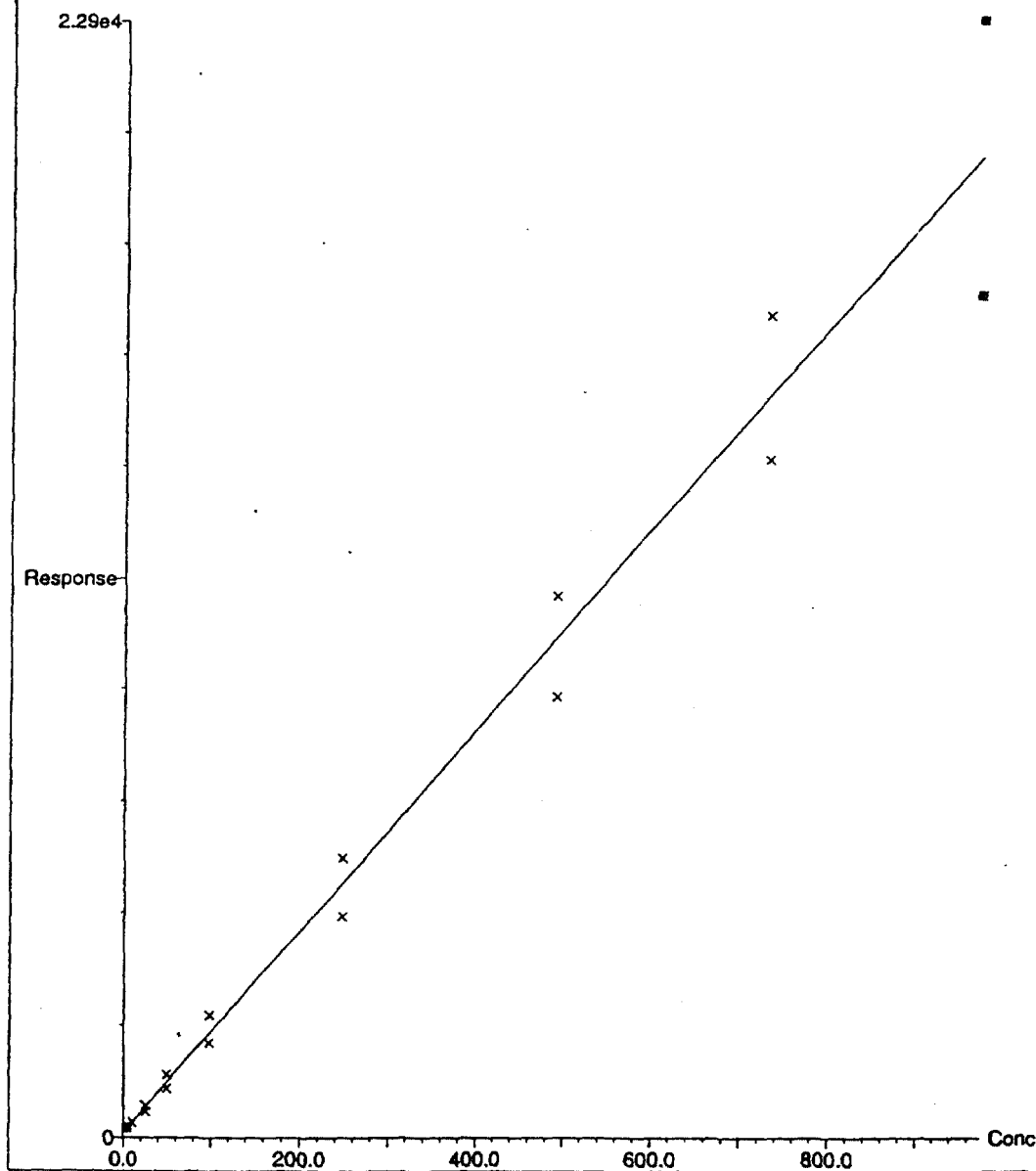
Q Name	Sample/Label	Type	Std. Chd	RT	Area	Peak	Q Name
1 110499001	Blank						0.01 -99
2 110499002	Blank						
3 110499003	Blank						
4 110499004	Blank						
5 110499005	Blank						
6 110499006	Blank						
7 110499007	Blank						
8 110499008	Blank						
9 110499009	Blank						
10 110499010	Blank						
11 110499011	Blank						
12 110499012	Blank						
13 110499013	Blank						
14 110499014	Blank						
15 110499015	Blank						
16 110499016	Blank						
17 110499017	Blank						
18 110499018	Blank						
19 110499019	Blank						
20 110499020	Blank						
21 110499021	Blank						
22 110499022	Blank						
23 110499023	Blank						
24 110499024	Blank						
25 110499025	Blank						
26 110499026	Blank						
27 110499027	Blank						
28 110499028	Blank						
29 110499029	Blank						
30 110499030	Blank						
31 110499031	Blank						
32 110499032	Blank						
33 110499033	Blank						
34 110499034	Blank						
35 110499035	Blank						
36 110499036	Blank						
37 110499037	Blank						
38 110499038	Blank						
39 110499039	Blank						
40 110499040	Blank						
41 110499041	Blank						
42 110499042	Blank						
43 110499043	Blank						
44 110499044	Blank						
45 110499045	Blank						
46 110499046	Blank						

Group 830193: Quantitated and printed by IAS on 11/03/93

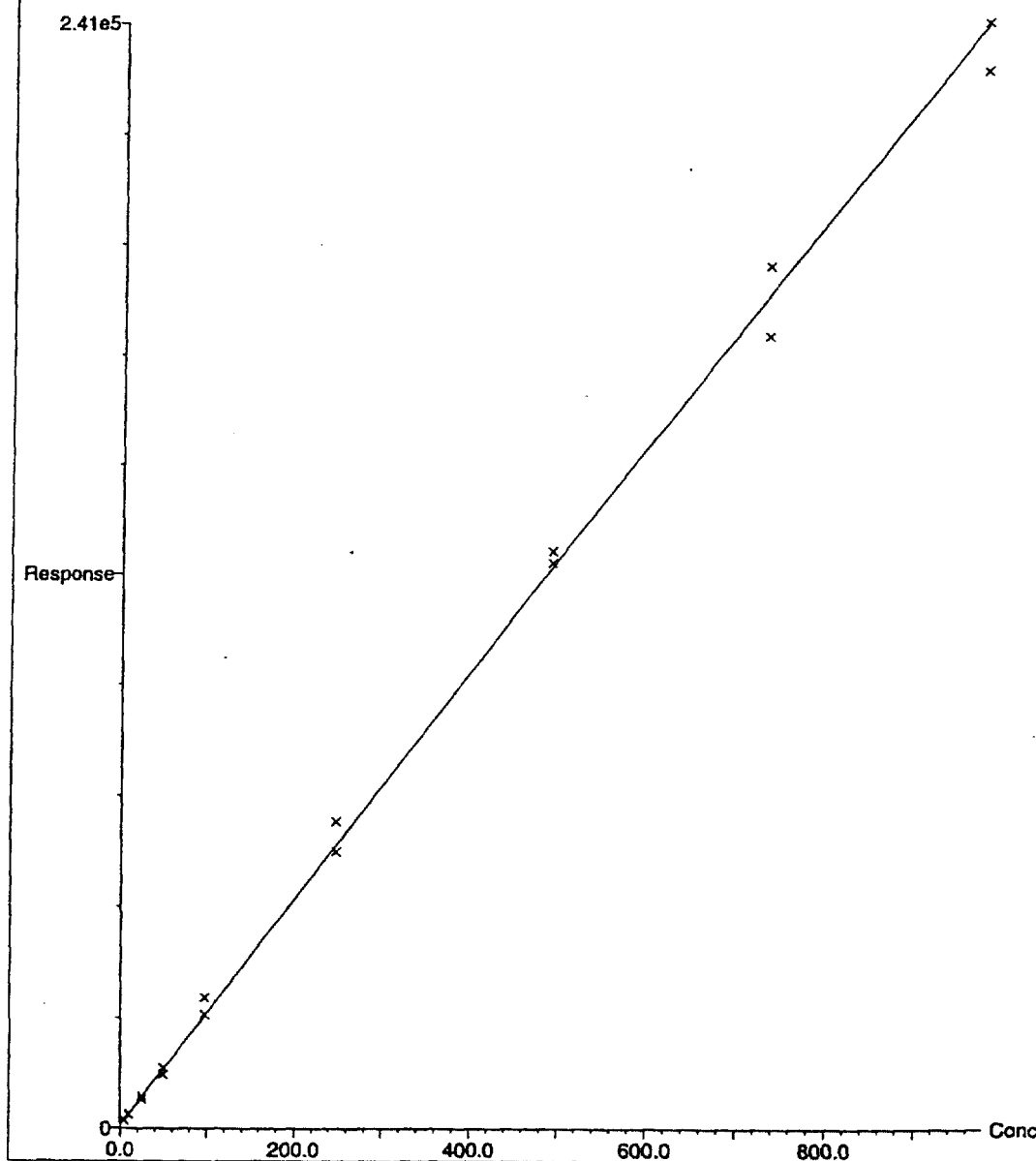
Compound 1 name: POAA (Ch1)
Response Factor: 68.6538
Response type: External Std, Area
Curve type: RF



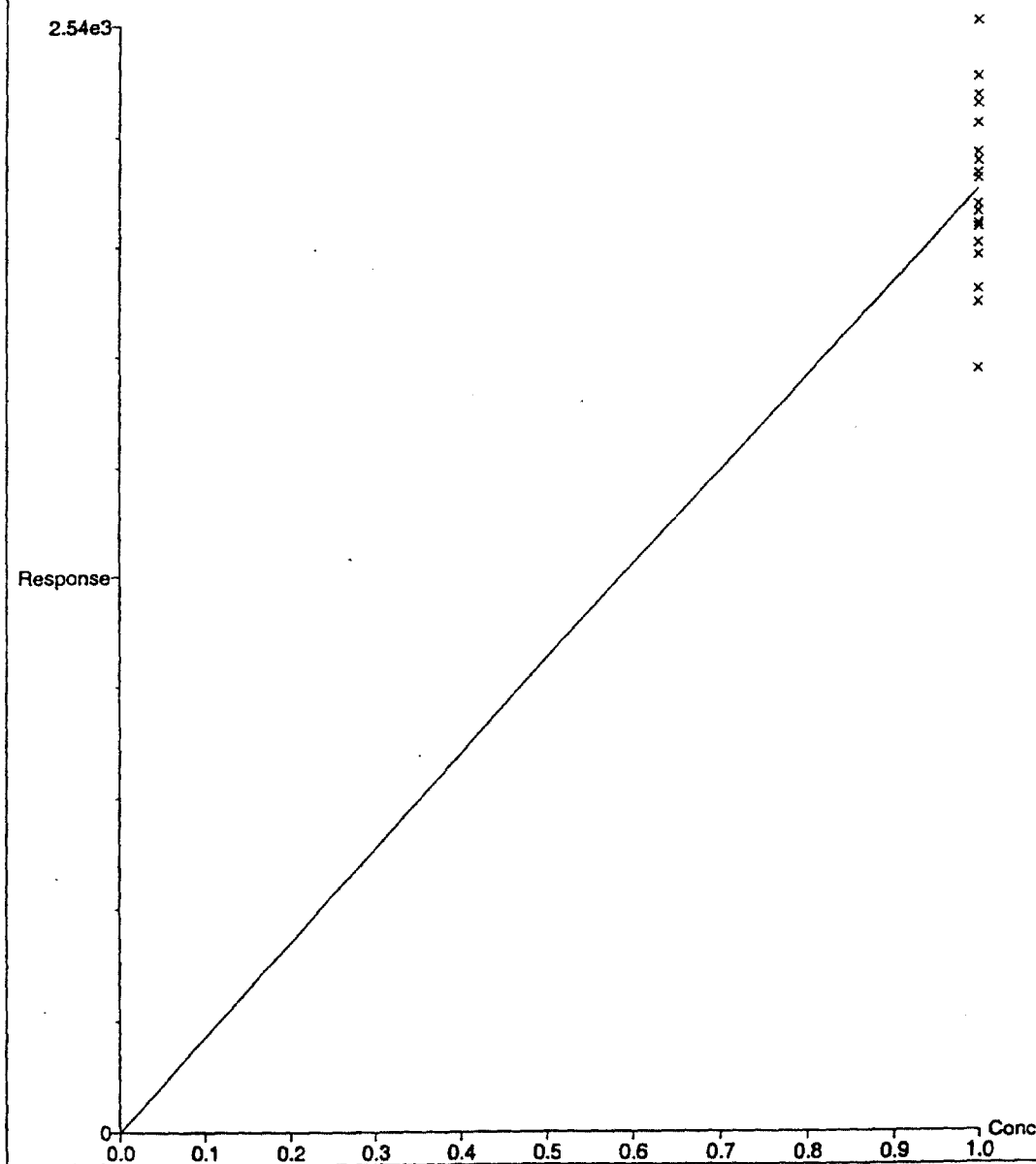
Compound 2 name: PFOS (Ch2)
Coefficient of Determination: 0.980777
Calibration curve: $20.4798 \cdot x + 108.210$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound 3 name: PFOSA (Ch3)
Coefficient of Determination: 0.997747
Calibration curve: $245.706 \cdot x + 643.062$
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



Method Report

Method file: C:\MSDCHEM\comp030199.prc\Method0110499a.mxd
 Last modified: Fri Nov 95 08:25:46 1995

Compound: 1. POLA (Chl)

Printed: Fri Nov 95 06:15:16 1995

Compound Name: POLA (Chl)
 Internal Ref.: (None)
 Quantity Traced: TIC
 Conc. of Standards: Fixed
 User RF Value: 1.00000, Disabled
 User Peak Factor: 1.00000
 Integrate window factor: 2.000
 Chromatogram area window: 1.000Da

Retention Time (min): 7.147, Enabled
 Relative Retention Time: 0.000, Disabled
 RT Standard Deviation: 0.345
 Time Window (min): 0.345

Peak Selection: Nearest

Response Type: External
 Response: Area

Polynomial Type: Linear
 Point of Origin: Baseline
 Fit Weights: 1/2
 Axis Transformation: None

Units:

Peak-to-peak amplitude: 81

Smoothing: Enabled
 Window size (scans): 2
 Smoothing method: Savitzky-Golay

2-sigma valley threshold: 10.00
 2-sigma peak height: 10.00
 Baseline maximum: 5.00

30.00
Disabled
30.00

Draw vertical threshold:
Detect shoulders:
Shoulder threshold:

1.50, Disabled
0.00, Disabled
2.00, Disabled
0.00, Disabled

Relative height:
Absolute height:
Relative area:
Absolute area:

Method Report

Method file: C:\MSDCHEM\comp020199.pse\Method0116499a.mtd

Last modified: Fri Mar 03 06:37:40 1999

Compound: 2: PYOS (C3)

Printed: Fri Mar 05 06:36:36 1999

Compound Name: PYOS (C3)

Internal Ref.: [None]

Quantity Trace: TIC

Conc. of Standards: Conc.: A

User N° Value: 1.000000, Disabled

Dead Peak Factor: 1.000000

Interference Window Factor: 2.000

Chromatogram mass Window: 1.000000

Detection Time (min): 7.033, Enabled

Relative Retention Time: 0.000, Disabled

Peak Retention Time (min): 7.033

Time Window (min): 0.437

Peak Selection: Nearest

Response Type: External

Response: Area

Polynomial Type: Linear

Point of Origin: Endpoint

Fit Weighting: 1/2

Axis Transformation: None

Units:

Peak-to-peak amplitude: 81

Soothing: Enabled

Window size (scan): 2

Number of scans: 1

Soothing method:

Join valley threshold: 30.00

Join peak valley: 30.00

Align baseline: 5.00

Draw vertical threshold: 30.00

Draw horizontal threshold: 30.00

Shoulder threshold: 30.00

Relative height:
1.00, Disabled
1.00, Disabled
2.00, Disabled
0.00, Disabled

Relative height:
1.00, Disabled
1.00, Disabled
2.00, Disabled
0.00, Disabled

Relative area:
1.00, Disabled
1.00, Disabled
2.00, Disabled
0.00, Disabled

Absolute area:
1.00, Disabled
1.00, Disabled
2.00, Disabled
0.00, Disabled

Relative height:
Relative height:
Relative area:
Absolute area:

1.50 Disabled
1.50 Disabled
2.50 Disabled
2.50 Disabled
6.00 Disabled

Method Report

Page 4

Method file: C:\MSST\MSK\comp\20179.ppt\MethDB\110499a.mdb
Last modified: Fri Nov 05 06:39:44 1999

Compound: 4: Surr (CH4)

Printed: Fri Nov 05 06:56:54 1999

Compound Name: Surr (CH4)
Internal Ref.: [None]
Quantify Trace: TIC
Comp. of 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 (mins): 7.218, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.375

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: 41

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

Join valley threshold: 10.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00

Draw vertical threshold: 10.00
Detect shoulders: Disabled
Shoulder threshold: 10.00

Relative height: 1.00, Disabled
Absolute height: 0.00, Disabled
Relative area: 1.00, Disabled
Absolute area: 0.00, Disabled

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

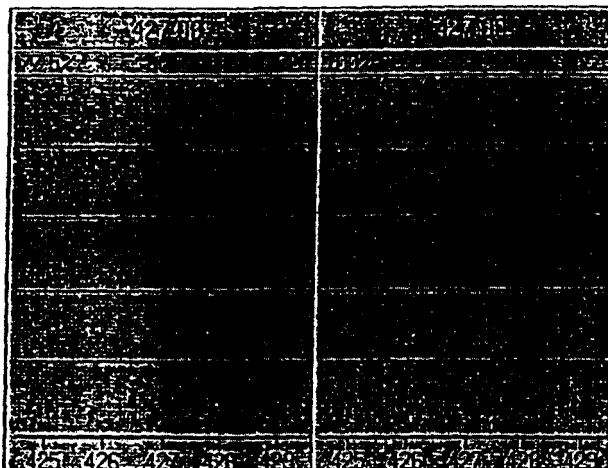
Time (min.)	%B
1) <u>0.00</u>	<u>10.0</u>
2) <u>1.00</u>	<u>10.0</u>
3) <u>5.50</u>	<u>95.0</u>
4) <u>7.50</u>	<u>95.0</u>
5) <u>8.00</u>	<u>10.0</u>

Tuning Method Report

Page 1

Method: C:\MASSLYNX\SOUP020199.PRO\ACQ00B\PF0S3

Printed: Thu Nov 04 14:02:27 1999



MS

MS2

SOURCE (ESP-)	Set	Rdbk	MS1	Set	Rdbk
Capillary	1.60	1.62	LM Resolution	14.0	
Cone	40	-41	HM Resolution	14.0	
Extractor	5	-7	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
Analysar Vacuum	5.1e-5	LM Resolution	14.0	
		HM Resolution	14.0	
		Collision	10	7
Gas Cell	4.0e-3	Ion Energy	2.0	
		Ion Energy Ramp	0.0	
		Lens 8	40	36
		Lens 9	0	1
		Multiplier	650	

Examining Method Report

Method: C:\MSDAS\NRI\00099\0010109.PHOLOGEN\10000-0700-0000-0000-20

Last Modified: Fri Oct 15 10:25:00 1999

Printed: Thu Nov 04 13:01:26 1999

Solvent Delay (mins) : 0.00

Function : 1 MM 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) Call Masses (eV) Cone (V)

1 413.00 169.00 0.30 30 15

Function : 2 MM 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) Call Masses (eV) Cone (V)

1 435.00 99.00 0.30 45 60

Function : 3 MM 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) Call Masses (eV) Cone (V)

1 458.00 70.00 0.30 45 60

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

Inter Channel Delay (Secs) : 0.03

Span (Daltons) : 0.00

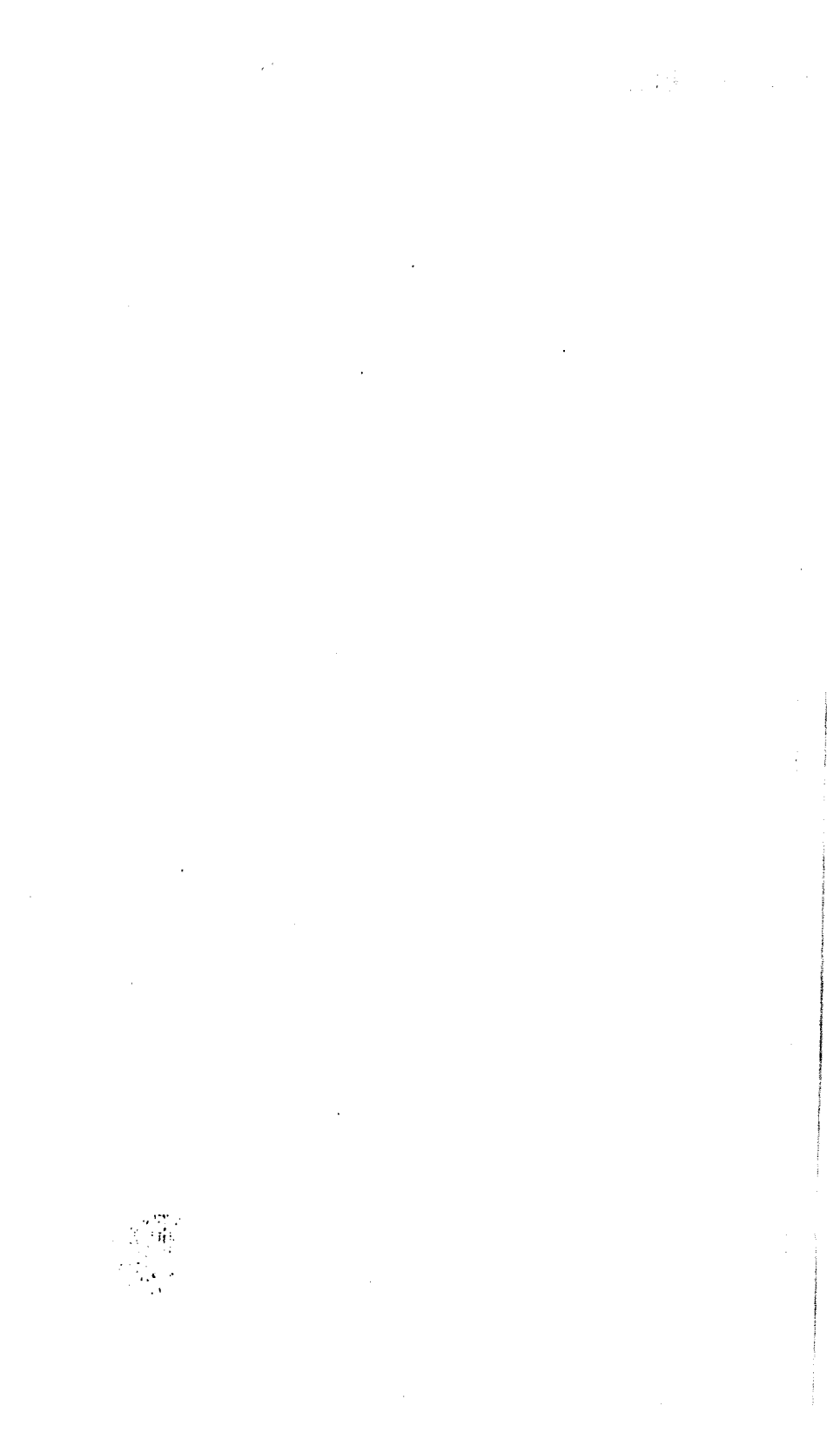
Start Time (mins) : 0.00

End Time (mins) : 10.00

Repeats : 1

Channel Parent Daughter Dwell (Secs) Call Masses (eV) Cone (V)

1 427.00 80.00 0.30 40 60



Method Report

Page 1

Method File:
Last Modified:C:\MASSELYNEX\SCUP020199.PRO\ACQUDS\10min-10-95
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

Sample List: C:\MASSLYNX\oup020199.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\lsoup020199.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	--	--	--	--

FACT: DATA REVIEW SUMMARY FORM

Date: 11/08/99
 Data Analyst: JAS
 FACT Study #: Tox IVS
 Matrix/Timepoints: Ref Sam - day 29 control / dose - cat 11/3/99
 Sample List: 1105996
 Set-up Analysis Date/Analyst: MMH 11/05/99
 Instrument: Simp 020199

Data Review Sign-Off

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

Technical Review
 Name / Date: MMH 11/2/99 / Kih 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 11/18/99

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, DIL 1110 GML 11/01/99

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 11/18/99

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 conc points 7.89 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

FACT: DATA REVIEW SUMMARY FORM

Date: 11/08/99
 Data Analyst: JAS
 FACT Study #: For MS
 Matrix/Timepoints: Ref. Sera - day 29 dose 1 Control - Oct 11/3/99
 Sample List: 405996
 Set-up Analysis
 Date/Analyst: MMH 11/05/99
 Instrument: Sup 020199

Data Review Sign-Off

Analyst Review
 Name / Date: JAS 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. 121
 Acceptable Linear Range: NA
 LOQ: NA Extraction Blank: NA
 R²: NA Matrix Blank: NA
 Calibration Check: NA
 MS/MSD: _____
 Data Status: _____

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: _____

Good
11/08/99

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Keep 220191: Quantified and printed by lsm on 11/06/95

Form 02039, Qualified and printed by Lee on 11/08/99

Quantity Computed Summary Report
 TOL45-day29 interval and deer - wt. 11/7/99

Sample Size: C:\data\TOL45\day29\day29\summary\summary110899b
 Date modified: Fri Nov 05 18:16:11 1999

Method: C:\data\TOL45\day29\day29\summary\summary110899b
 Last modified: Mon Nov 08 07:01:28 1999

Job Code:

Printed: Mon Nov 08 07:26:43 1999

Computed 1: TOL45 (CAL)

	Sample Size	Sample Size	Type	Pre-Cause	SE	Area Flag	Count	Mean
0 Name	11059028	MOCK BLANK						
1 11059028	MOCK BLANK							
2 11059028	MOCK BLANK							
3 11059028	MOCK BLANK							
4 11059031	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
5 11059031	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
6 11059031	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
7 11059034	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
8 11059034	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
9 11059034	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
10 11059037	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
11 11059038	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
12 11059040	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
13 11059040	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
14 11059042	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
15 11059042	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
16 11059043	MOCK BLANK							
17 11059044	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
18 11059045	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
19 11059045	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
20 11059047	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
21 11059048	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
22 11059048	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
23 11059048	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
24 11059050	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
25 11059051	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
26 11059052	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
27 11059054	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
28 11059054	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
29 11059057	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
30 11059058	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
31 11059058	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
32 11059059	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
33 11059061	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
34 11059061	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							
35 11059062	TOL45-MSL1039-deer B1k-9, wt. 11/7/99 MCM							

Quantity Component Summary Report

T0015-day75 control and decon- ent. 11/3/99

Sample List: C:\MSD\T0015\comp20159-pro\data\comp20159\10395D

Last Modified: Fri Nov 05 12:26:11 1999

Method: C:\MSD\T0015\comp20159-pro\Method\10395B

Last Modified: Mon Nov 08 07:49:50 1999

Job Code:

Printed: Mon Nov 08 07:28:43 1999

Component 3: P0004 (C3)

#	Name	SampleList Text	Type	Std Conc	MC	Area Flag	Conc. Mkr
1	110590628	SampleList Text			7.432	2756	0.00
2	110590629	NaOH Blank			7.432	2756	0.00
3	110590630	NaOH Blank			7.432	2756	0.00
4	110590631	NaOH Blank			7.432	2756	0.00
5	110590632	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
6	110590633	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
7	110590634	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
8	110590635	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
9	110590636	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
10	110590637	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
11	110590638	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
12	110590639	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
13	110590640	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
14	110590641	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
15	110590642	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
16	110590643	NaOH Blank			7.432	2756	0.00
17	110590644	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
18	110590645	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
19	110590646	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
20	110590647	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
21	110590648	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
22	110590649	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
23	110590650	NaOH Blank			7.432	2756	0.00
24	110590651	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
25	110590652	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
26	110590653	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
27	110590654	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
28	110590655	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
29	110590656	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
30	110590657	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
31	110590658	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
32	110590659	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
33	110590660	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
34	110590661	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00
35	110590662	T0015-RTS-100ppb-9, ent. 11/3/99 MCH	Standard	4.31	7.432	2756	0.00

Doc 020139: Quantified and printed by las on 11/08/99

11/11/79

Comp 020251 Qualified and printed by law on 11/08/79

Quantity Compound Summary Report
 TOL15-dry23 control and cover, etc. 11/3/79
 Sample Name: C:\MS61EXE\temp020139-jerry\temp020139020
 Date: 10/1/81
 Date: 11/3/79
 Method: C:\MS61EXE\temp020139-jerry\temp020139020
 Last modified: Nov Nov 08 07:09:56 1999
 Job Code:
 Printed: Nov Nov 08 07:28:42 1999

Compound 61: PAHs (C64)

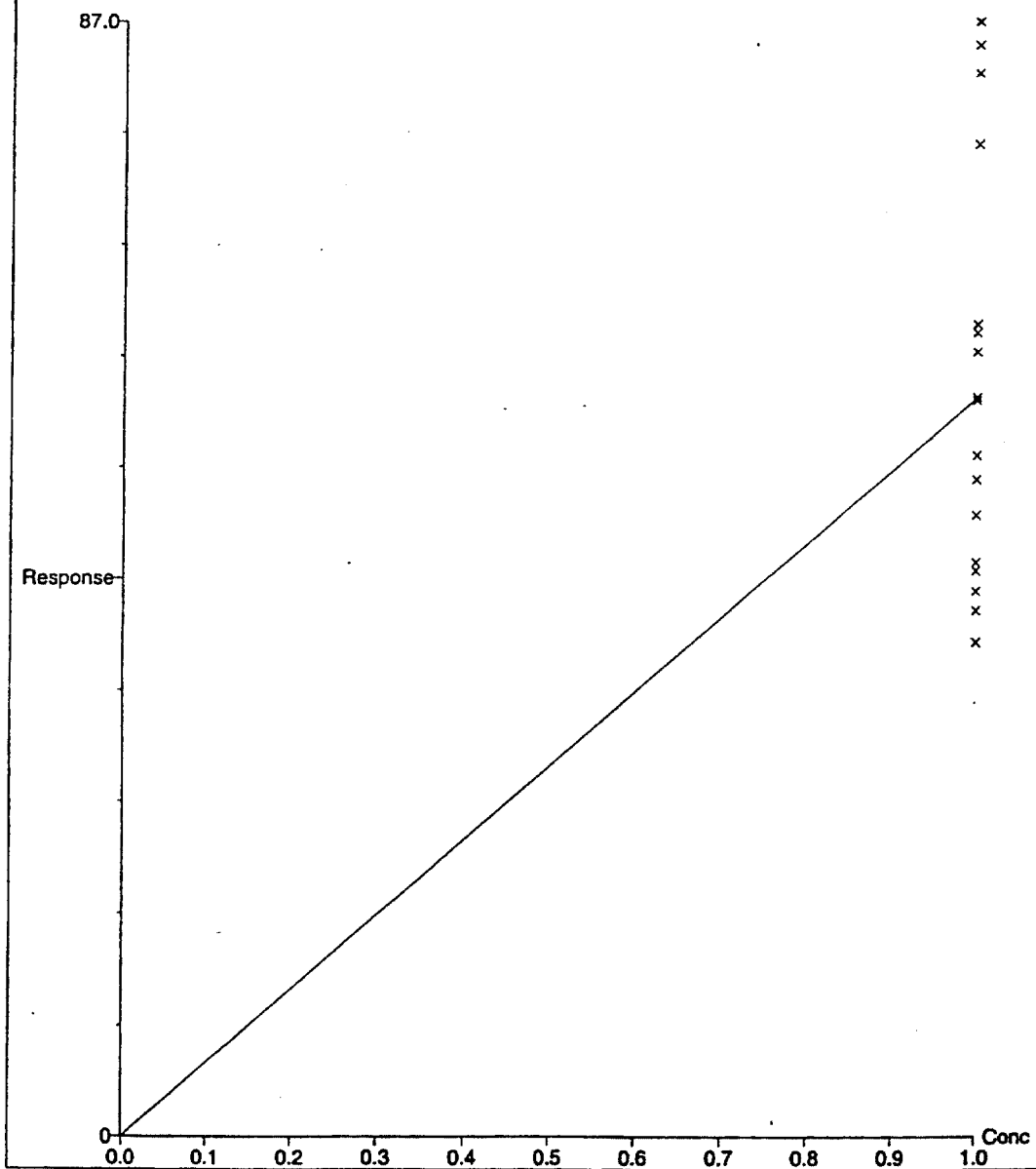
Sample	Sample Name	Type	Std Conc	RT	Area	Conc	Wt%
0	Blank						
1	11059028	Blank		7.171	1793	0.43	17
2	11059029	Blank		7.167	1826	0.45	15
3	11059030	Blank		7.164	1826	0.45	15
4	11059031	Blank		7.161	2046	0.85	-5
5	11059032	Blank		7.163	2266	1.05	5
6	11059033	Blank		7.165	2115	1.01	1
7	11059034	Blank		7.162	2115	1.01	1
8	11059035	Blank		7.163	2115	1.01	1
9	11059036	Blank		7.164	2115	1.01	1
10	11059037	Blank		7.165	2115	1.01	1
11	11059038	Blank		7.166	2115	1.01	1
12	11059039	Blank		7.167	2115	1.01	1
13	11059040	Blank		7.168	2115	1.01	1
14	11059041	Blank		7.169	2115	1.01	1
15	11059042	Blank		7.170	2115	1.01	1
16	11059043	Blank		7.171	2115	1.01	1
17	11059044	Blank		7.172	2115	1.01	1
18	11059045	Blank		7.173	2115	1.01	1
19	11059046	Blank		7.174	2115	1.01	1
20	11059047	Blank		7.175	2115	1.01	1
21	11059048	Blank		7.176	2115	1.01	1
22	11059049	Blank		7.177	2115	1.01	1
23	11059050	Blank		7.178	2115	1.01	1
24	11059051	Blank		7.179	2115	1.01	1
25	11059052	Blank		7.180	2115	1.01	1
26	11059053	Blank		7.181	2115	1.01	1
27	11059054	Blank		7.182	2115	1.01	1
28	11059055	Blank		7.183	2115	1.01	1
29	11059056	Blank		7.184	2115	1.01	1
30	11059057	Blank		7.185	2115	1.01	1
31	11059058	Blank		7.186	2115	1.01	1
32	11059059	Blank		7.187	2115	1.01	1
33	11059060	Blank		7.188	2115	1.01	1
34	11059061	Blank		7.189	2115	1.01	1
35	11059062	Blank		7.190	2115	1.01	1

Ins 4/08/99 p. 1.14

Quantity Calibration Report
POE145-day19 control and dose- ent. 11/3/99

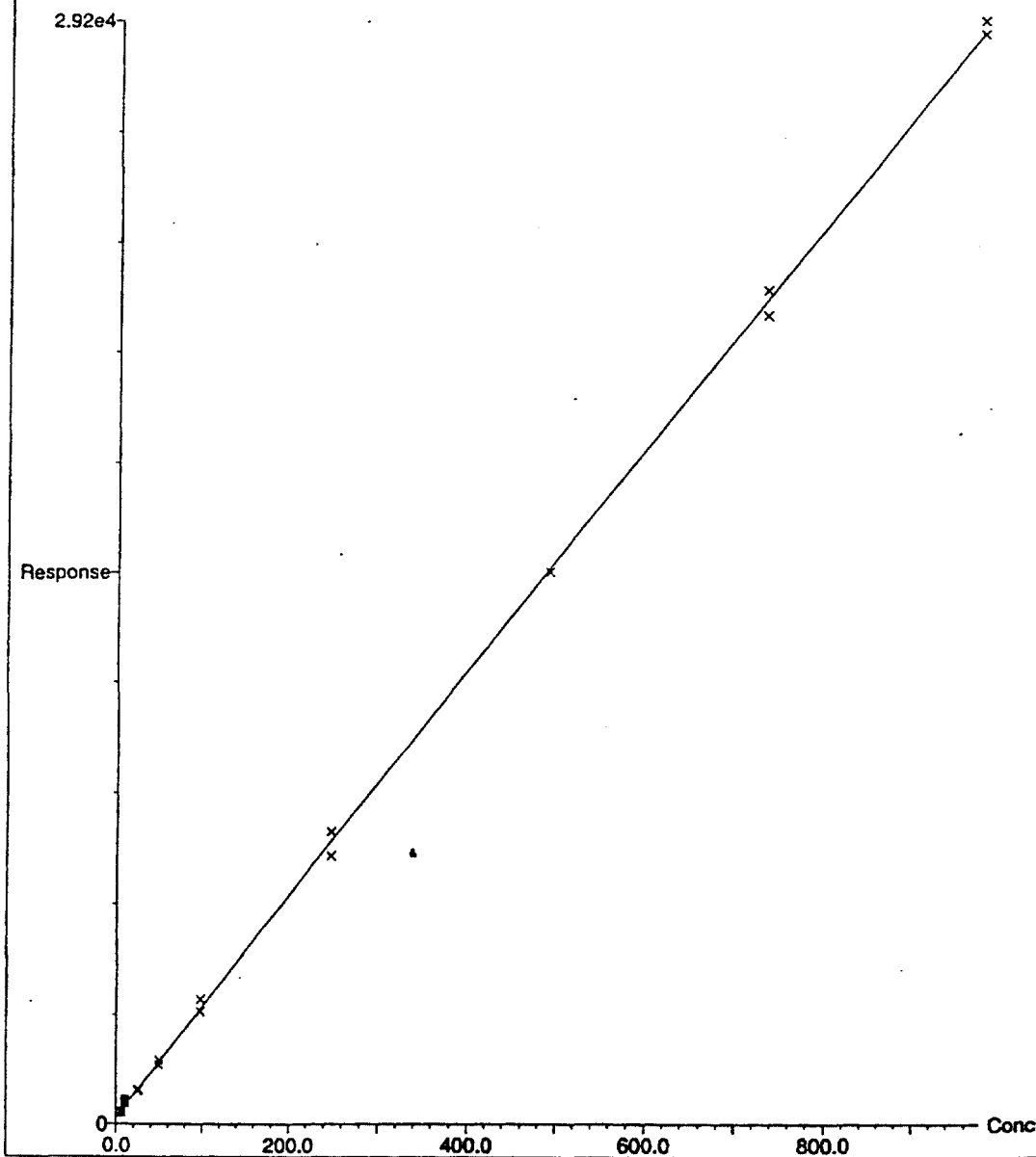
Page 1

Compound 1 name: POAA (Ch1)
Response Factor: 57.5599
Response type: External Std, Area
Curve type: RF

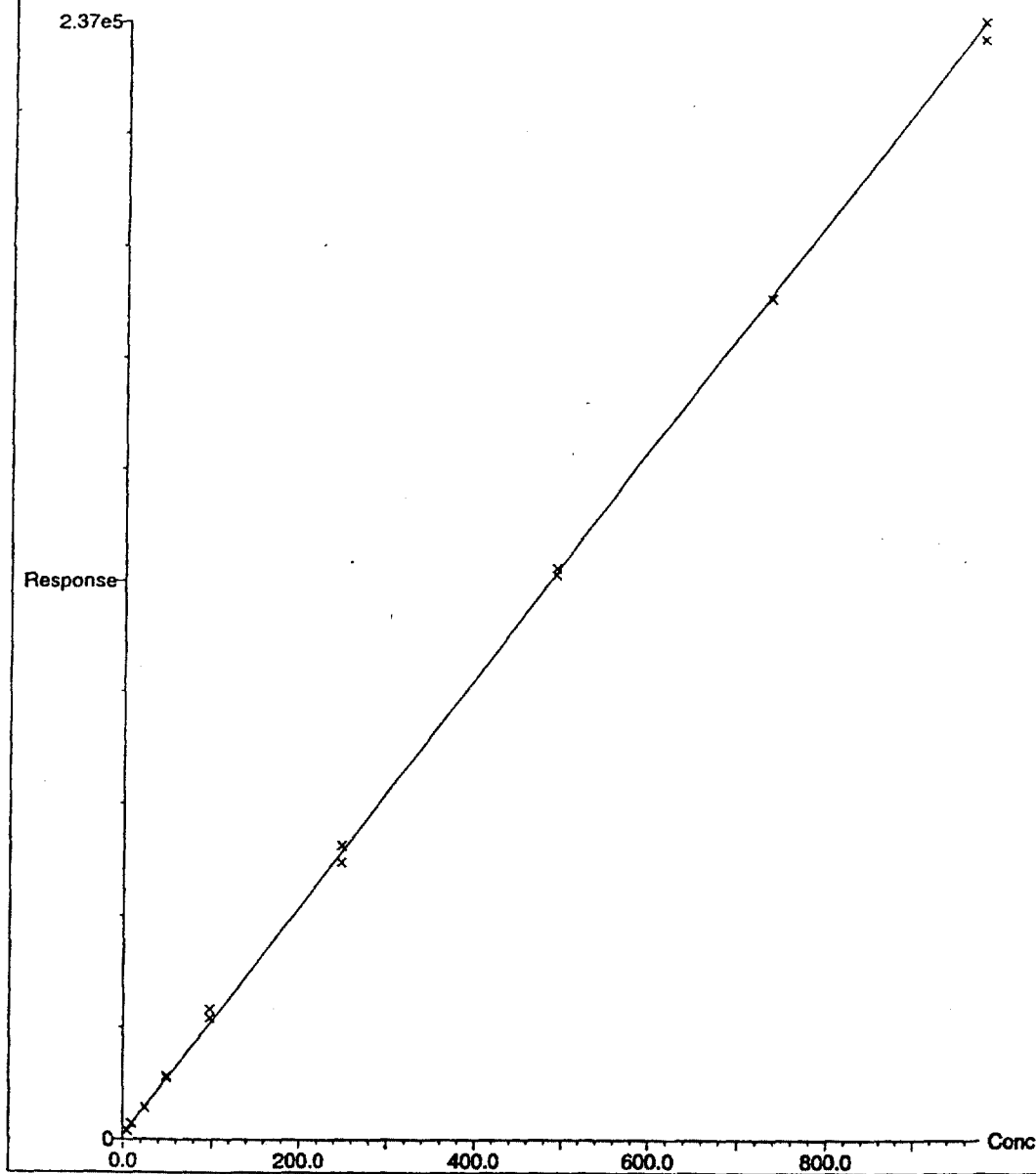


Group 020129: Quantitated and printed by ins on 11/08/99

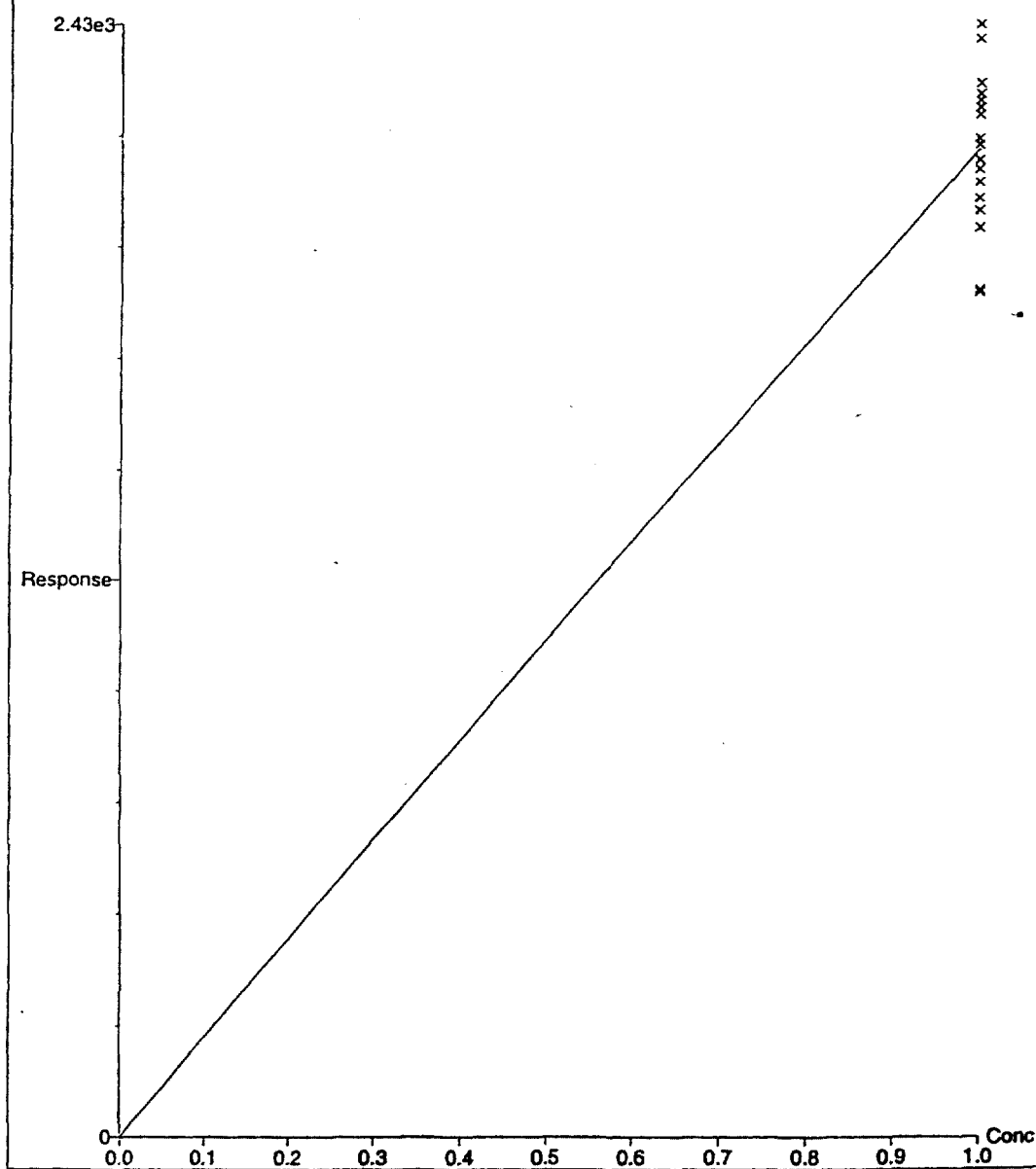
Compound 2 name: PFOS (Ch2)
Coefficient of Determination: 0.999549
Calibration curve: $29.3773 * x + 191.305$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound 3 name: PFOSA (Ch3)
Coefficient of Determination: 0.999708
Calibration curve: $241.363 \cdot x + 800.807$
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



Relative % ONH:
Relative % ONH:
Relative % ONH:
Absolute % ONH:

Relative % ONH:
Relative % ONH:
Relative % ONH:
Absolute % ONH:

Method Report

Method File: C:\MSDCHEM\MSDCHEM\110519B.meth
 Last modified: Mon May 02 07:05:18 1999

Compound: 21 210A (C6H11)

Printed: Mon May 03 07:05:55 1999

Compound Name: 21 210A (C6H11)
 Internal Ref.: 110519B
 Quantity Traced: 1.00000
 Conc. of Standards: Fixed
 User SP Value: 1.00000 Disabled
 User Peak Factor: 1.00000
 Intersect Window Factor: 2.000
 Chromatogram Mass Window: 1.0000A

Retention Time (min): 1.111 Enabled
 Relative Retention Time: 1.111 Disabled
 RT Meter On: (None)
 Time Wt. % (min): 1.111

Peak Selection: Standard

Response Type: External
 Response: Area

Polynomial Type: Linear
 Point of Origin: Exclude
 Fit Method: LR
 Axis Transformation: None

Units:

Peak-to-Peak Amplitude: 10

Smoothing: 2000
 Number of Points (Spectrum): 2000
 Number of Channels: 1
 Smoothing Method: 1

Gain Valley Threshold: 10.00
 Reduce Peak Calling: 10.00
 Raise Baseline Maximum: 10.00

Draw Vertical Threshold: 10.00
 Detect Amplitude: Disabled
 Shoulder Threshold: 10.00

Method Report

Page 2

Method file: C:\MSISL\MSL\supp\19199.pro\Meth08\110599b.mdb
 Last modified: Mon Nov 08 07:19:13 1999
 Compound: 2: PFCS (CH2)
 Printed: Mon Nov 08 17:29:14 1999

Compound Name: PFCS (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: 1.000
 Chromatogram mass window: 1.000000e

Retention Time (min): 7.111, Enabled
 Relative Retention Time: 1.000, Disabled
 RT Reference: None!
 Time Window (min): 3.100

Peak Selection: Nearest

Response Type: External
 Response: Atrial

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

Units:

Peak-to-peak amplitude: 100

Smoothing: Enabled
 Window size (scans): 7
 Number of smooths: 1
 Smoothing method:

Join valley threshold: 10.00
 Reduce peak tailing: 100.00
 Raise baseline minimum: 5.00

Draw vertical threshold: 10.00
 Detect shoulders: 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

Relative height:
Absolute height:
Relative area:
Absolute area:

Method Report

Method File: C:\MSI\DATA\250201899\proj\method08110399b.mtd
 Date Modified: Mon Nov 08 07:00:38 1999

Compound: 3: PDSA (CNS)

Printed: Mon Nov 08 07:00:38 1999

Compound Name: PDSA (CNS)
 Internal Ref.: (None)
 Quantity Tested: 1.00
 Conc. of Standards: Conc. 8

Dear AF Value: 1.00000, Disabled
 Near Peak Factor: 1.00500
 Integrate Window Factor: 1.00500
 Chromatogram Peak Window: 1.00500

Retention Time (min): 3.771, Enabled
 Relative Retention Time: 1.000, Disabled
 Time Window (min): 2.548

Peak Selection: Nearest

Response Type: External
 Response: Area

Polynomial Type: Linear
 Point of Origin: 1.000
 Fit Weights: 1/2
 Axis Transformation: None

Units:

Peak-to-Peak Amplitude: 1.00

Smoothing: 1
 Window Size (Points): 2
 Number of Scans: 1
 Smoothing Period: 1

Join Valley Threshold: 1.00
 Reduce Peak Calling: 2.00
 Active Baseline Minimum: 1.00

Draw Vertical Threshold: 3.00
 Disabled
 Shrinker Threshold: 10.00

Method Report

Page 4

Method file: C:\MSDCHEM\110599b.mdb
Last modified: Mon Nov 08 07:00:53 1999

Compound: 4: Surr (Ch4)

Printed: Mon Nov 08 07:00:54 1999

Compound Name: Surr (Ch4)
Internal Ref.: (None)
Quantify Trace: TIC
Conc. of Standards: Fixed
User RP Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 1.00
Chromatogram Mass Window: 1.000000

Retention Time (mins): 1.000, Enabled
Relative Retention Time: 1.000, 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: 70

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

Join valley threshold: 10.00
Reduce peak tailing: 10.00
Raise baseline maximum: 1.00

Draw vertical threshold: 10.00
Detect shoulders: 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

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 μ L 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 μ m

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 μ L/min. Back pressure: 65 bar

HPLC solvent gradient: 10min-10-95

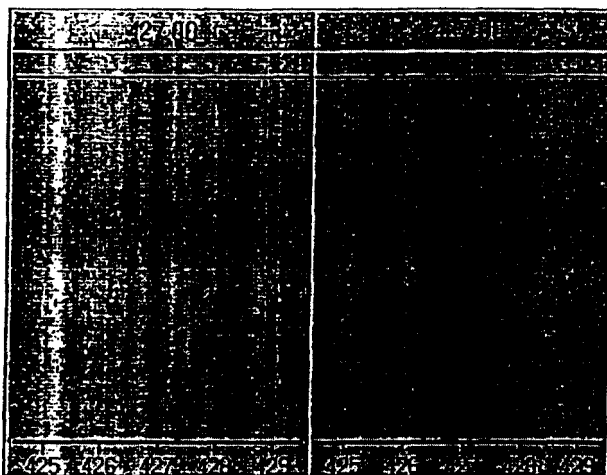
Time (min.)	%B
1) 0.00	10.0
2) 1.00	10.0
3) 5.50	95.0
4) 7.50	95.0
5) 8.00	10.0

Tuning Method Report

Page 1

Method: C:\MASSLYNX\SOUP020199.PRO\ACQUDB\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	

Scanning Method Report

Page 1

Method: C:\MSDCHEM\SOUP\020195.P\OACQUEM\10MCM-PP08-PC0A-PP0A-15
Last Modified: Fri Oct 15 10:33:03 1999

Printed: Fri Nov 01 15:31:30 1999

Solvent Delay (Mins) : 0.00

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

Inter Channel Delay (Secs) : 0.01

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Cell Energy (eV)	Cone (V)

1	411.00	169.00	0.30	30	15

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

Inter Channel Delay (Secs) : 0.01

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Cell Energy (eV)	Cone (V)

1	499.00	99.00	0.30	45	60

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

Inter Channel Delay (Secs) : 0.01

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Cell Energy (eV)	Cone (V)

1	498.00	78.00	0.30	45	60

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

Inter Channel Delay (Secs) : 0.01

Span (Daltons) : 0.00

Start Time (Mins) : 0.00

End Time (Mins) : 10.00

Repeats : 1

Channel	Parent	Daughter	Dwell (Secs)	Cell Energy (eV)	Cone (V)

1	427.00	80.00	0.30	40	60

Sample List: C:\MASSLYNX\Soup020199.pr0\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

FACT: DATA REVIEW SUMMARY FORM

Data Review Sign-Off

Date: 10/18/99
 Data Analyst: JAS
 FACT Study #: TOXNS
 Matrix/Timepoints: Day 1 & 4, Rat Liver, Control & dose, cat 10/18/99
 Sample List: 101599a
 Set-up Analysis Date/Analyst: 10/15/99 JAS (5)
 Instrument: Solup 020/99

Analyst Review Name / Date: JAS 10/18/99

Technical Review Name / Date: MEE 10/18/99 / h/h 10/18/99

Quality Assurance Review Name / Date: _____

Data Entry: LAC 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 (1) LAC 10/21/99
 Data Status: ETT

Analyte: PFOS (CH2) (1) (3)
 Acceptable Linear Range: See (5) 10/18/99
 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
 Data Status: Entered + Dilutions needed LAC 10/21/99

Analyte: PFOSA (CH3) (1) (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 LAC 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 # 101599.015) 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 rerun.
 (9) Re-run sample in 101599.043; suspect carry-over in results. h/h 10/18/99

FACT: DATA REVIEW SUMMARY FORM

Date: 10/17/99
 Data Analyst: JAS
 FACT Study #: Tox 145
 Matrix/Timepoints: Day 1 & 4, Red Liver, control & dose, ext 10/18/99
 Sample List: 105892
 Set-up Analysis
 Date/Analyst: 10/15/99 JAS
 Instrument: Sup 020199

Data Review Sign-Off

Analyst Review
 Name / Date: JAS 10/18/99

Technical Review
 Name / Date: Refer to page 1 of 2 UAC 10/21/99

Quality Assurance Review
 Name / Date: _____

Data Entry: UAC 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: JAS 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 # 10589025). A small peak that elutes just before the PFOA peak was noticed for most samples. The identity of the peak was not discovered. - JAS 10/18/99

(4) Area counts for the ISTD were good. All samples had deviations under 20% except the H2O blank which was at 92%. - JAS 10/18/99

(5) A second cal check separated by a H2O blank was used in the sample run in anticipation of carryover. - JAS 10/18/99.

(6) PFOA AREA COUNTS ARE VERY LOW. QUESTION NAME OF REPEATING ANALYSIS. MEE 10/18/99

(7) SECOND PEAK IN PFOA CHROMATOGRAM IS NOT A TARGET ANALYTE AND DOES NOT NEED TO BE QUANTITATED. MEE 10/18/99

Compound 1: PMA (CAL)

[illegible]

Quantity Compound Summary Report

QUALITY Rat Liver, day 1 & 4, control and dose- cont. 10/13/99

Sample Name: C:\MSDCHEM\chem\rat-liver\chem\10131990

Sample ID: 10131990

Method: C:\MSDCHEM\chem\rat-liver\chem\10131990

Last modified: Mon Oct 18 09:11:31 1999

Job Code:

Printed: Mon Oct 18 09:20:03 1999

Compound 31 9908 (CNS)

Sample	Sample Name	Type	Std Conc	RT	Area	Flag	Conc	Unit
1	101319901	Standard	6.13	7.315	27	db	0.00	
2	101319902	Standard	6.13	7.315	16	db	0.00	
3	101319903	Standard	6.13	7.315	11	db	0.00	
4	101319904	Standard	6.13	7.315	33	db	0.00	
5	101319905	Standard	6.13	7.315	53	db	0.00	
6	101319906	Standard	6.13	7.315	75	db	0.00	
7	101319907	Standard	6.13	7.315	95	db	0.00	
8	101319908	Standard	6.13	7.315	115	db	0.00	
9	101319909	Standard	6.13	7.315	135	db	0.00	
10	101319910	Standard	6.13	7.315	155	db	0.00	
11	101319911	Standard	6.13	7.315	175	db	0.00	
12	101319912	Standard	6.13	7.315	195	db	0.00	
13	101319913	Standard	6.13	7.315	215	db	0.00	
14	101319914	Standard	6.13	7.315	235	db	0.00	
15	101319915	Standard	6.13	7.315	255	db	0.00	
16	101319916	Standard	6.13	7.315	275	db	0.00	
17	101319917	Standard	6.13	7.315	295	db	0.00	
18	101319918	Standard	6.13	7.315	315	db	0.00	
19	101319919	Standard	6.13	7.315	335	db	0.00	
20	101319920	Standard	6.13	7.315	355	db	0.00	
21	101319921	Standard	6.13	7.315	375	db	0.00	
22	101319922	Standard	6.13	7.315	395	db	0.00	
23	101319923	Standard	6.13	7.315	415	db	0.00	
24	101319924	Standard	6.13	7.315	435	db	0.00	
25	101319925	Standard	6.13	7.315	455	db	0.00	
26	101319926	Standard	6.13	7.315	475	db	0.00	
27	101319927	Standard	6.13	7.315	495	db	0.00	
28	101319928	Standard	6.13	7.315	515	db	0.00	
29	101319929	Standard	6.13	7.315	535	db	0.00	
30	101319930	Standard	6.13	7.315	555	db	0.00	
31	101319931	Standard	6.13	7.315	575	db	0.00	
32	101319932	Standard	6.13	7.315	595	db	0.00	
33	101319933	Standard	6.13	7.315	615	db	0.00	
34	101319934	Standard	6.13	7.315	635	db	0.00	
35	101319935	Standard	6.13	7.315	655	db	0.00	
36	101319936	Standard	6.13	7.315	675	db	0.00	
37	101319937	Standard	6.13	7.315	695	db	0.00	
38	101319938	Standard	6.13	7.315	715	db	0.00	
39	101319939	Standard	6.13	7.315	735	db	0.00	
40	101319940	Standard	6.13	7.315	755	db	0.00	
41	101319941	Standard	6.13	7.315	775	db	0.00	
42	101319942	Standard	6.13	7.315	795	db	0.00	
43	101319943	Standard	6.13	7.315	815	db	0.00	
44	101319944	Standard	6.13	7.315	835	db	0.00	
45	101319945	Standard	6.13	7.315	855	db	0.00	
46	101319946	Standard	6.13	7.315	875	db	0.00	
47	101319947	Standard	6.13	7.315	895	db	0.00	
48	101319948	Standard	6.13	7.315	915	db	0.00	
49	101319949	Standard	6.13	7.315	935	db	0.00	
50	101319950	Standard	6.13	7.315	955	db	0.00	
51	101319951	Standard	6.13	7.315	975	db	0.00	
52	101319952	Standard	6.13	7.315	995	db	0.00	
53	101319953	Standard	6.13	7.315	1015	db	0.00	
54	101319954	Standard	6.13	7.315	1035	db	0.00	
55	101319955	Standard	6.13	7.315	1055	db	0.00	
56	101319956	Standard	6.13	7.315	1075	db	0.00	
57	101319957	Standard	6.13	7.315	1095	db	0.00	
58	101319958	Standard	6.13	7.315	1115	db	0.00	
59	101319959	Standard	6.13	7.315	1135	db	0.00	
60	101319960	Standard	6.13	7.315	1155	db	0.00	
61	101319961	Standard	6.13	7.315	1175	db	0.00	
62	101319962	Standard	6.13	7.315	1195	db	0.00	
63	101319963	Standard	6.13	7.315	1215	db	0.00	
64	101319964	Standard	6.13	7.315	1235	db	0.00	
65	101319965	Standard	6.13	7.315	1255	db	0.00	
66	101319966	Standard	6.13	7.315	1275	db	0.00	
67	101319967	Standard	6.13	7.315	1295	db	0.00	
68	101319968	Standard	6.13	7.315	1315	db	0.00	
69	101319969	Standard	6.13	7.315	1335	db	0.00	
70	101319970	Standard	6.13	7.315	1355	db	0.00	
71	101319971	Standard	6.13	7.315	1375	db	0.00	
72	101319972	Standard	6.13	7.315	1395	db	0.00	
73	101319973	Standard	6.13	7.315	1415	db	0.00	
74	101319974	Standard	6.13	7.315	1435	db	0.00	
75	101319975	Standard	6.13	7.315	1455	db	0.00	
76	101319976	Standard	6.13	7.315	1475	db	0.00	
77	101319977	Standard	6.13	7.315	1495	db	0.00	
78	101319978	Standard	6.13	7.315	1515	db	0.00	
79	101319979	Standard	6.13	7.315	1535	db	0.00	
80	101319980	Standard	6.13	7.315	1555	db	0.00	
81	101319981	Standard	6.13	7.315	1575	db	0.00	
82	101319982	Standard	6.13	7.315	1595	db	0.00	
83	101319983	Standard	6.13	7.315	1615	db	0.00	
84	101319984	Standard	6.13	7.315	1635	db	0.00	
85	101319985	Standard	6.13	7.315	1655	db	0.00	
86	101319986	Standard	6.13	7.315	1675	db	0.00	
87	101319987	Standard	6.13	7.315	1695	db	0.00	
88	101319988	Standard	6.13	7.315	1715	db	0.00	
89	101319989	Standard	6.13	7.315	1735	db	0.00	
90	101319990	Standard	6.13	7.315	1755	db	0.00	
91	101319991	Standard	6.13	7.315	1775	db	0.00	
92	101319992	Standard	6.13	7.315	1795	db	0.00	
93	101319993	Standard	6.13	7.315	1815	db	0.00	
94	101319994	Standard	6.13	7.315	1835	db	0.00	
95	101319995	Standard	6.13	7.315	1855	db	0.00	
96	101319996	Standard	6.13	7.315	1875	db	0.00	
97	101319997	Standard	6.13	7.315	1895	db	0.00	
98	101319998	Standard	6.13	7.315	1915	db	0.00	
99	101319999	Standard	6.13	7.315	1935	db	0.00	
100	101320000	Standard	6.13	7.315	1955	db	0.00	

Quantity Computed Summary Report

TOX145 - Pet Liver, day 1 & 6, Control and dose - sec. 10/13/79

Sample List: C:\MSALTR\comp-sec-prod\comp\day1015090

Last modified: Mon Oct 18 06:43:06 1979

Method: C:\MSALTR\comp-sec-prod\comp\day1015090

Last modified: Mon Oct 18 06:43:06 1979

Job Code:

Printed: Mon Oct 18 09:20:02 1979

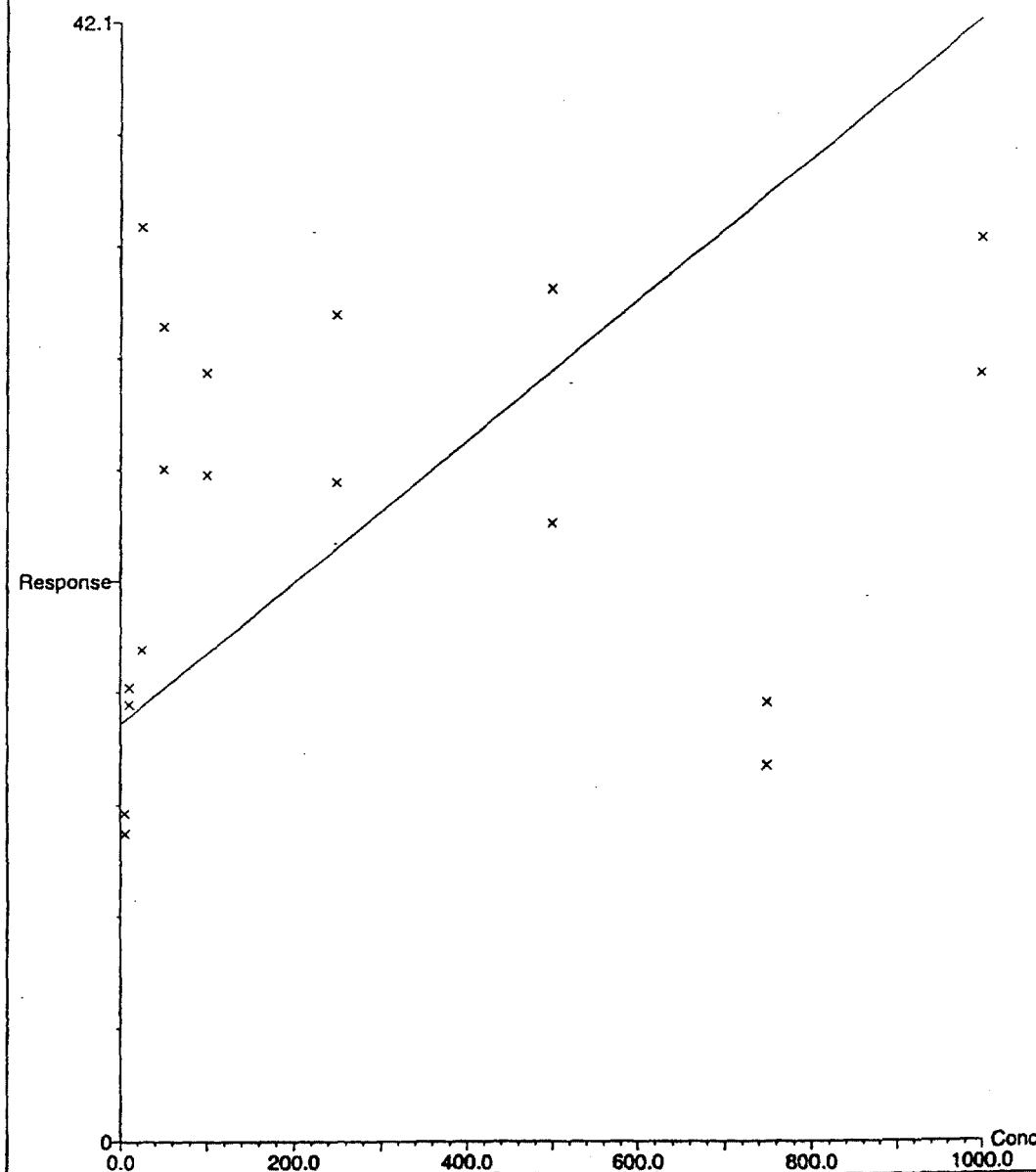
Compound 3: PROCA (CAL)									
		Sample Name	Type	Std Cond	SE	Area Flag	Comp. Unit		
1	101599001	MOON BLANK	Standard		7.810	177.00	0.00		
2	101599002	MOON BLANK	Standard		7.810	177.00	0.00		
3	101599003	MOON BLANK	Standard		7.810	177.00	0.00		
4	101599004	TOX145, REL10137-100ppb-11, SEC	Standard		7.796	145.00	0.00		
5	101599005	TOX145, REL10137-100ppb-11, SEC	Standard		7.796	145.00	0.00		
6	101599006	TOX145, REL10137-100ppb-11, SEC	Standard		7.796	145.00	0.00		
7	101599007	TOX145, REL10137-100ppb-11, SEC	Standard		7.796	145.00	0.00		
8	101599008	TOX145, REL10137-100ppb-11, SEC	Standard		7.796	145.00	0.00		
9	101599009	TOX145, REL10137-100ppb-11, SEC	Standard		7.796	145.00	0.00		
10	101599010	TOX145, REL10137-100ppb-11, SEC	Standard		7.814	145.00	0.00		
11	101599011	TOX145, REL10137-100ppb-11, SEC	Standard		7.814	145.00	0.00		
12	101599012	TOX145, REL10137-100ppb-11, SEC	Standard		7.807	145.00	0.00		
13	101599013	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
14	101599014	TOX145, REL10137-100ppb-11, SEC	Standard		7.808	145.00	0.00		
15	101599015	MOON BLANK	Standard		7.810	177.00	0.00		
16	101599016	TOX145, REL10137-100ppb-11, SEC	Standard		7.812	145.00	0.00		
17	101599017	TOX145, REL10137-100ppb-11, SEC	Standard		7.812	145.00	0.00		
18	101599018	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
19	101599019	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
20	101599020	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
21	101599021	MOON BLANK	Standard		7.810	177.00	0.00		
22	101599022	TOX145, REL10137-100ppb-11, SEC	Standard		7.812	145.00	0.00		
23	101599023	TOX145, REL10137-100ppb-11, SEC	Standard		7.812	145.00	0.00		
24	101599024	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
25	101599025	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
26	101599026	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
27	101599027	MOON BLANK	Standard		7.810	177.00	0.00		
28	101599028	TOX145, REL10137-100ppb-11, SEC	Standard		7.812	145.00	0.00		
29	101599029	TOX145, REL10137-100ppb-11, SEC	Standard		7.812	145.00	0.00		
30	101599030	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
31	101599031	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
32	101599032	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
33	101599033	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
34	101599034	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
35	101599035	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
36	101599036	MOON BLANK	Standard		7.810	177.00	0.00		
37	101599037	TOX145, REL10137-100ppb-11, SEC	Standard		7.812	145.00	0.00		
38	101599038	TOX145, REL10137-100ppb-11, SEC	Standard		7.812	145.00	0.00		
39	101599039	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
40	101599040	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
41	101599041	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
42	101599042	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
43	101599043	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
44	101599044	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
45	101599045	MOON BLANK	Standard		7.810	177.00	0.00		
46	101599046	TOX145, REL10137-100ppb-11, SEC	Standard		7.812	145.00	0.00		
47	101599047	TOX145, REL10137-100ppb-11, SEC	Standard		7.812	145.00	0.00		
48	101599048	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
49	101599049	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
50	101599050	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
51	101599051	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
52	101599052	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
53	101599053	MOON BLANK	Standard		7.810	177.00	0.00		
54	101599054	TOX145, REL10137-100ppb-11, SEC	Standard		7.812	145.00	0.00		
55	101599055	TOX145, REL10137-100ppb-11, SEC	Standard		7.812	145.00	0.00		
56	101599056	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
57	101599057	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
58	101599058	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
59	101599059	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
60	101599060	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
61	101599061	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
62	101599062	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
63	101599063	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		
64	101599064	TOX145, REL10137-100ppb-11, SEC	Standard		7.813	145.00	0.00		

Placed: Mon Oct 18 09:20:03 1991

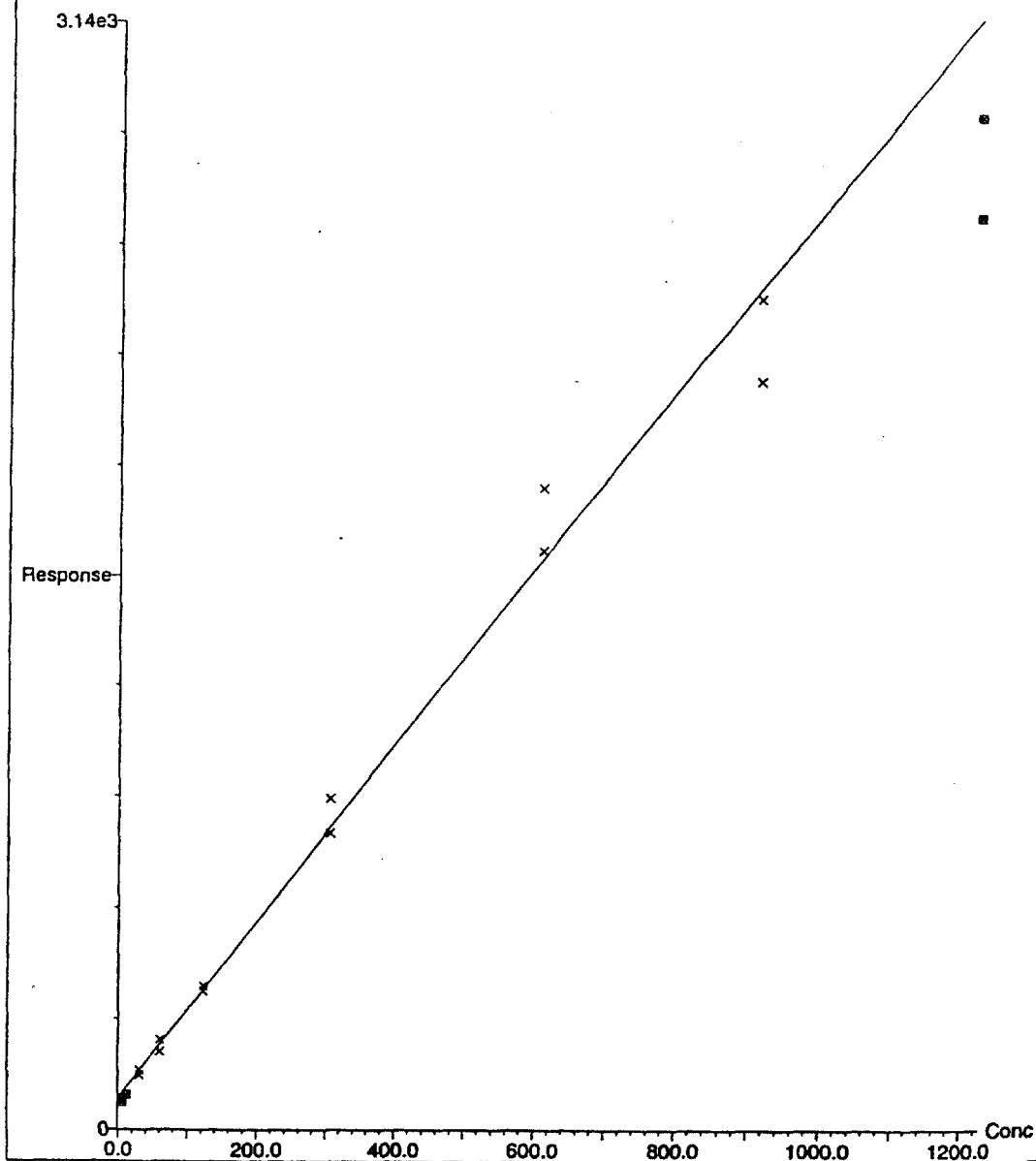
Compound 4: X8 (Cat.

[illegible]

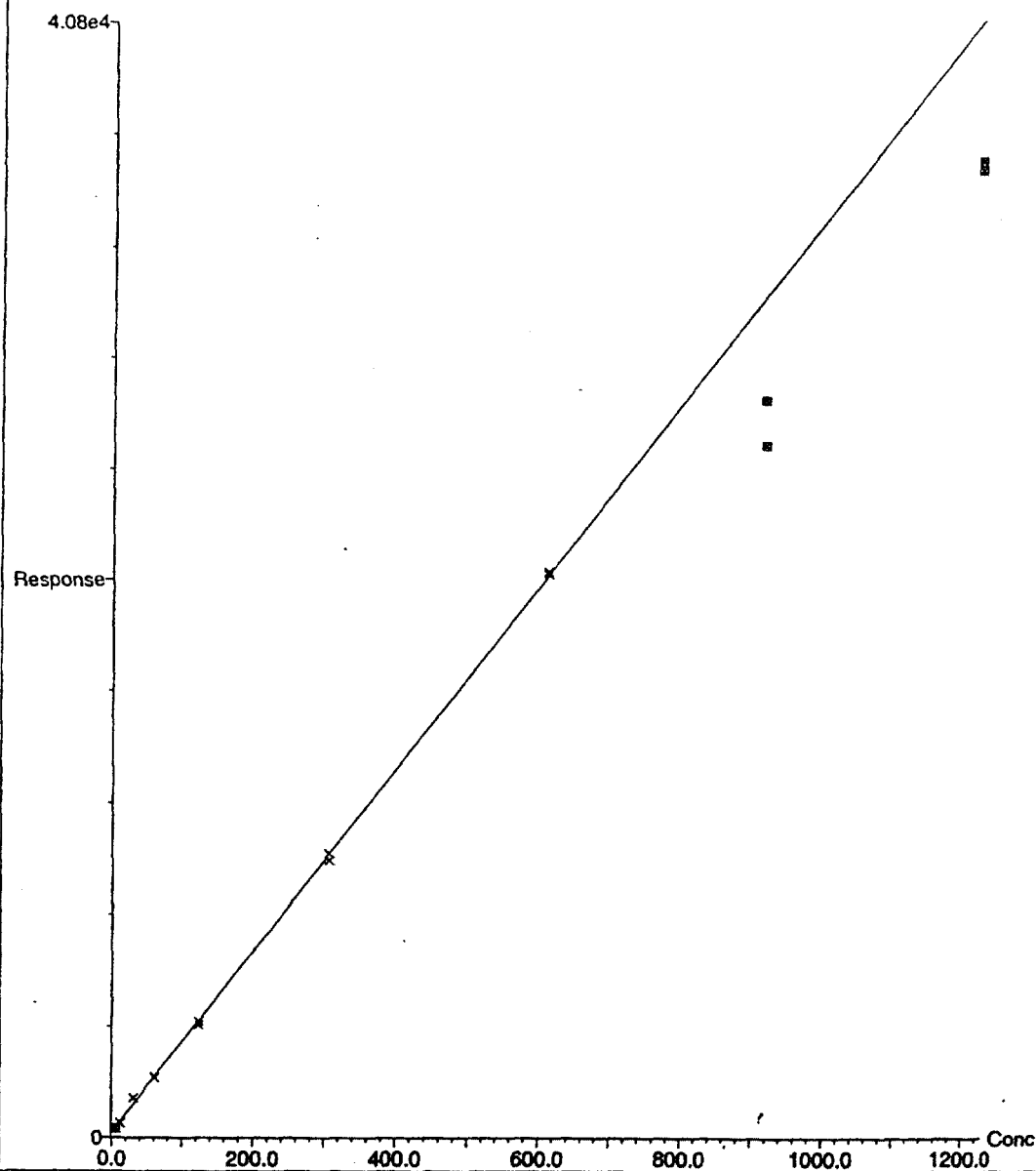
Compound 1 name: POAA (Ch1)
Coefficient of Determination: 89334.567901
Calibration curve: $0.0264689 \cdot x + 15.6723$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



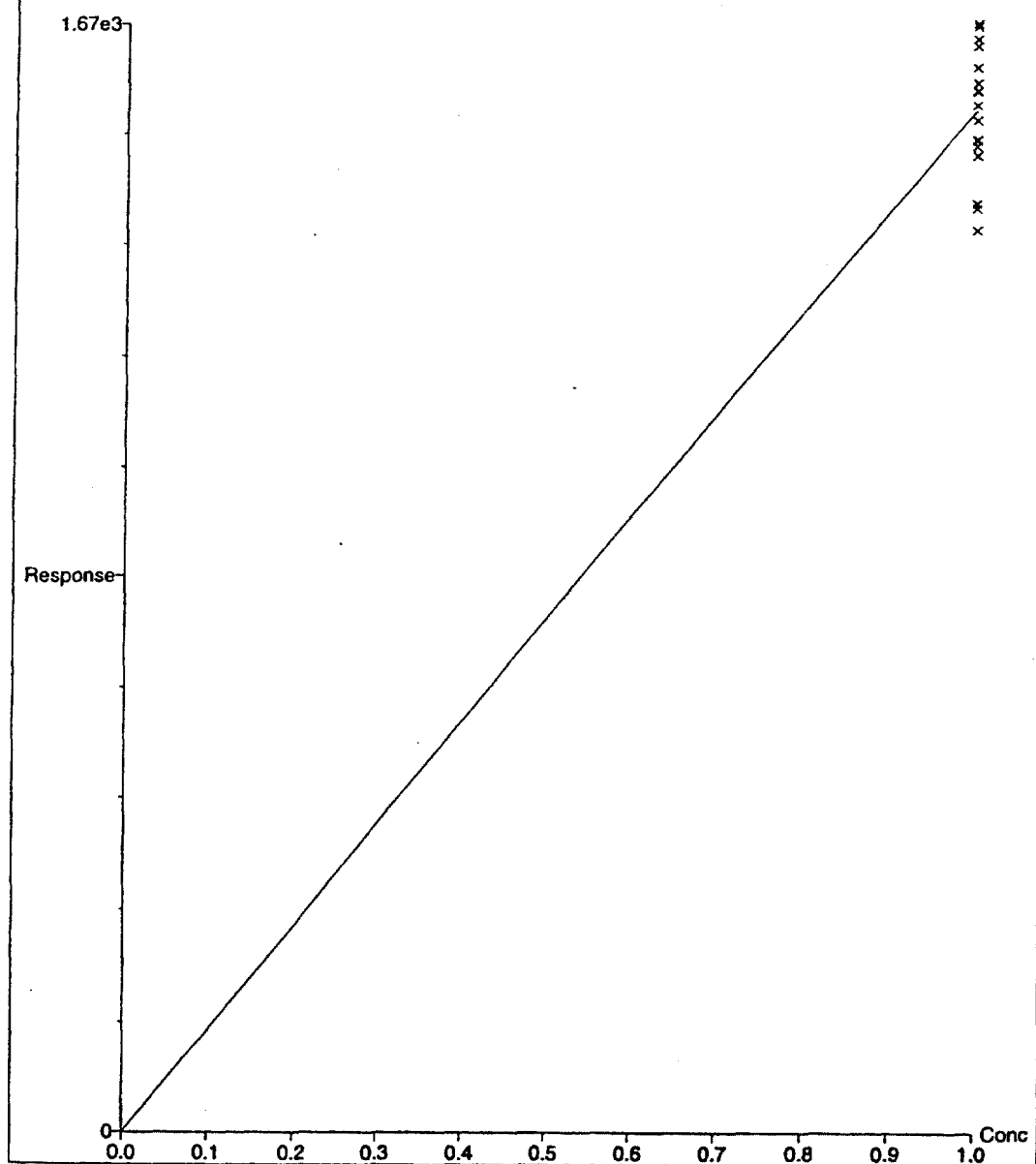
Compound 2 name: PFOS (Ch2)
Coefficient of Determination: 0.983941
Calibration curve: $2.48521 \cdot x + 89.0578$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound 3 name: PFOSA (Ch3)
Coefficient of Determination: 0.999594
Calibration curve: $33.0992 \cdot x + 198.731$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound 4 name: IS (Ch4)
Response Factor: 1542.30
Response type: External Std, Area
Curve type: RF



Method Report

Page 1

Method file: C:\MASSLYNX\scup-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

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): 7.119, 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: 100.00
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 1

Method file: C:\NASST\YH\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

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
RF Reference: (None)
Time Window (mins): 0.400

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: 66

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

Join valley threshold: 10.00
Reduce peak tailing: 100.00
Raise baseline maximum: 5.00

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 10.00

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:\MASSLIVE\comp-rates-pro\Method8\101559a.mdb
 Last modified: Mon Oct 18 06:43:21 1999

Compound: 3: PFOSA (CH₃)

Printed: Mon Oct 18 07:46:51 1999

Compound Name: PFOSA (CH₃)
 Internal Ref: (None)
 Quantify Trace: TIC
 Conc. of Standards: Conc. C
 User IR Value: 1.00000, Disabled
 User Peak Factor: 1.00000
 Integrate Window Factor: 2.000
 Chromatogram mass window: 1.000Da

Retention Time (mins): 7.797, Enabled
 Relative Retention Time: 0.000, Disabled
 RT Reference: (None)
 Time Window (mins): 0.675

Peak Selection: Nearest

Response Type: External
 Response: Area

Polynomial Type: Linear
 Polynomial Order: 1
 Fit Weights: 1/x
 Axis Transformation: None

Units:

Peak-to-peak amplitude: 80

Smoothing: Enabled

Window size (scans): 2

Number of smoothes: 1

Smoothing method:

Join valley threshold: 10.00

Reduce peak calling: 200.00

Reduce baseline minimum: 5.00

Draw vertical threshold: 90.00

Peak threshold: Enabled

Shoulder threshold: 10.00

1.50. Disabled
1.00. Disabled
2.00. Disabled
0.00. Disabled
0.00. Disabled

Relative height:
Relative height:
Relative area:
Absolute area:

Dew vertical threshold:	30.00
Direct radiation:	Disabled
Shade threshold:	30.00
Relative height:	1.30, Disabled
Relative area:	2.00, Disabled
Relative area:	2.00, Disabled
Absolute area:	8.00, Disabled

Method Report

Page 4

Method file: C:\MASS\TOL\smg-eres-problem\10121996.mdb

Last modified: Mon Oct 18 06:41:21 1999

Compound: 4: 15 (C8)

Printed: Mon Oct 18 07:45:51 1999

Compound Name: 15 (C8)
Internal Ref.: (None)
Quantity Trace: TIC
Comp. of Standards: P1.mn

User SP Value: 1.00000, Disabled
User Peak Factor: 1.00000
Intercept Window Factor: 2.000
Chromatogram Mass Window: 1.00000

Retention Time (min): 7.137, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: (None)
Time Window (min): 0.411

Peak Selection: None

Response Type: External
Response: Area

Polynomial Type: Linear
Number of Points: 10
File Reloading: 1/X
Axis Transformation: None

Units:

Peak-to-peak amplitude: 67

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

Join valley threshold: 10.00
Reduce peak tailing: 200.00
Noise threshold (mV): 5.00

Quattro II, #6414E

DATE: 10/15/99

ANALYST: jias

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? ☒ 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

Tuning Method Report

Page 1

Method: C:\MASSLYNX\SOUP-RATS.FRO\ACQDB\FPOS3

Printed: Fri Oct 15 11:42:32 1999



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
Gas Cell	4.1e-3	Ion Energy	2.0	
		Ion Energy Ramp	0.0	
		Lens 8	40	38
		Lens 9	0	-0
		Multiplier	650	

Scanning Method Report

Page 1

Method: C:\MSDLINK\ACQU-SAVE.PRO\ACQUOB\IONIN-PPOM-POAA-PPOMA-15
Last Modified: Fri Oct 15 11:25:03 1999
Printed: Fri Oct 15 11:42:48 1999

Solvent Delay (mins) : 0.00

Function : 1 MSK of 1 Mass Pair (MSF-)

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 MSK of 1 Mass Pair (MSF-)

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 MSK of 1 Mass Pair (MSF-)

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	495.00	78.00	0.30	45	60

Function : 4 MSK of 1 Mass Pair (MSF-)

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	423.00	80.00	0.30	40	60

Method Report

Page 1

Method File:
Last Modified:C:\MSASLYNK\SCUP-RATS.PRO\ACQUDE\10min-10-95
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 (ul/min)	200
Draw Position (mm)	0.00
Stop Time (mins)	9.00
Injection Volume(ul)	5.0
Vial Number	91

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	---	---

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

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	---	---

FACT: DATA REVIEW SUMMARY FORM

p/2

Date: 10-20-99
 Data Analyst: MMX
 FACT Study #: Tox 145
 Matrix/Timepoints: Pat 500-001 day 1, 4 Dilutions
 Sample List: 101999a
 Set-up Analysis Date/Analyst: MMX 10-19-99
 Instrument: Amelia 2000

Data Review Sign-Off

Analyst Review
 Name / Date: MMX 10-20-99

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

Quality Assurance Review
 Name / Date: _____

Data Entry: LAC 10/22/99

Comments / Justifications: ① excluded lowest two points to meet 30% deviation criteria MMX 10-20-99

② excluded highest two points to meet R² criteria. MMX 10-20-99

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 DILUTED DID NOT CONTAIN INTERNAL STANDARD. SAMPLES WERE QUANTIFIED USING AN EXTERNAL STANDARD CURVE. MEE 10/20/99.

Analyte: PFOS
 Acceptable Linear Range: 24.80 - 996.0 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: POAA (3)
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: _____
 Data Status: used to monitor levels of POAA not for quantitation.

LAC 10/22/99

$$p^2/2$$

Data Review Sign-Off

Comments / Justifications: _____

Analyte: _____

Acceptable Linear Range: _____

LOQ: _____ Extracted Blank: _____

R²: _____ Matrix Blank: _____

Calibration Checks: _____

MS/MSD: _____

Data Status: _____

1AC 10/21/99

LAC 10/22/98

Amila 002098: Queried and Printed by user 10/20/99

Compound 1: 1704

Accession Number	Protein Name	Type	Gen. Comp.	Accession Number
1 101599001	Protein X	Standard	97.50	1 101599001
2 101599002	Protein Y	Standard	97.50	2 101599002
3 101599003	Protein Z	Standard	97.50	3 101599003
4 101599004	Protein A	Standard	97.50	4 101599004
5 101599005	Protein B	Standard	97.50	5 101599005
6 101599006	Protein C	Standard	97.50	6 101599006
7 101599007	Protein D	Standard	97.50	7 101599007
8 101599008	Protein E	Standard	97.50	8 101599008
9 101599009	Protein F	Standard	97.50	9 101599009
10 101599010	Protein G	Standard	97.50	10 101599010
11 101599011	Protein H	Standard	97.50	11 101599011
12 101599012	Protein I	Standard	97.50	12 101599012
13 101599013	Protein J	Standard	97.50	13 101599013
14 101599014	Protein K	Standard	97.50	14 101599014
15 101599015	Protein L	Standard	97.50	15 101599015
16 101599016	Protein M	Standard	97.50	16 101599016
17 101599017	Protein N	Standard	97.50	17 101599017
18 101599018	Protein O	Standard	97.50	18 101599018
19 101599019	Protein P	Standard	97.50	19 101599019
20 101599020	Protein Q	Standard	97.50	20 101599020
21 101599021	Protein R	Standard	97.50	21 101599021
22 101599022	Protein S	Standard	97.50	22 101599022
23 101599023	Protein T	Standard	97.50	23 101599023
24 101599024	Protein U	Standard	97.50	24 101599024
25 101599025	Protein V	Standard	97.50	25 101599025
26 101599026	Protein W	Standard	97.50	26 101599026
27 101599027	Protein X	Standard	97.50	27 101599027
28 101599028	Protein Y	Standard	97.50	28 101599028
29 101599029	Protein Z	Standard	97.50	29 101599029
30 101599030	Protein A	Standard	97.50	30 101599030
31 101599031	Protein B	Standard	97.50	31 101599031
32 101599032	Protein C	Standard	97.50	32 101599032
33 101599033	Protein D	Standard	97.50	33 101599033
34 101599034	Protein E	Standard	97.50	34 101599034
35 101599035	Protein F	Standard	97.50	35 101599035
36 101599036	Protein G	Standard	97.50	36 101599036
37 101599037	Protein H	Standard	97.50	37 101599037
38 101599038	Protein I	Standard	97.50	38 101599038
39 101599039	Protein J	Standard	97.50	39 101599039
40 101599040	Protein K	Standard	97.50	40 101599040

Amillo 04080: Quantified and Printed by msh 10/20/98

Quantity Compound Summary Report
Ten 143: Net Wt: 1.6
Sample Label: 10199001-10199020
Last modified: Wed Oct 30 06:31:47 1999
Method: C:\VAL\VALTR\Amalis-resa-prov\Amalis\1019901
Last modified: Wed Oct 30 05:15:15 1999
Job Code:

Printed: Wed Oct 30 09:00:41 1999

Compound 3: PMAA

Sample	Sample/Lab	Type	RM Cond	RT	Area	Comp	Flag	Stdev
1 10199001	NetWt			7.821	14	0.00	Db	
2 10199002	NetWt			7.871	15	0.00	Db	
3 10199003	NetWt			7.848	29	0.21	Db	
4 10199004	NetWt			7.821	37	0.51	Db	
5 10199005	NetWt			7.803	146	4.61	Db	-1
6 10199006	NetWt			7.803	146	4.61	Db	-1
7 10199007	NetWt			7.803	146	4.61	Db	-1
8 10199008	NetWt			7.803	146	4.61	Db	-1
9 10199009	NetWt			7.803	146	4.61	Db	-1
10 10199010	NetWt			7.803	146	4.61	Db	-1
11 10199011	NetWt			7.803	146	4.61	Db	-1
12 10199012	NetWt			7.803	146	4.61	Db	-1
13 10199013	NetWt			7.803	146	4.61	Db	-1
14 10199014	NetWt			7.803	146	4.61	Db	-1
15 10199015	NetWt			7.803	146	4.61	Db	-1
16 10199016	NetWt			7.803	146	4.61	Db	-1
17 10199017	NetWt			7.803	146	4.61	Db	-1
18 10199018	NetWt			7.803	146	4.61	Db	-1
19 10199019	NetWt			7.803	146	4.61	Db	-1
20 10199020	NetWt			7.803	146	4.61	Db	-1
21 10199021	NetWt			7.803	146	4.61	Db	-1
22 10199022	NetWt			7.803	146	4.61	Db	-1
23 10199023	NetWt			7.803	146	4.61	Db	-1
24 10199024	NetWt			7.803	146	4.61	Db	-1
25 10199025	NetWt			7.803	146	4.61	Db	-1
26 10199026	NetWt			7.803	146	4.61	Db	-1
27 10199027	NetWt			7.803	146	4.61	Db	-1
28 10199028	NetWt			7.803	146	4.61	Db	-1
29 10199029	NetWt			7.803	146	4.61	Db	-1
30 10199030	NetWt			7.803	146	4.61	Db	-1
31 10199031	NetWt			7.803	146	4.61	Db	-1
32 10199032	NetWt			7.803	146	4.61	Db	-1
33 10199033	NetWt			7.803	146	4.61	Db	-1
34 10199034	NetWt			7.803	146	4.61	Db	-1
35 10199035	NetWt			7.803	146	4.61	Db	-1
36 10199036	NetWt			7.803	146	4.61	Db	-1
37 10199037	NetWt			7.803	146	4.61	Db	-1
38 10199038	NetWt			7.803	146	4.61	Db	-1
39 10199039	NetWt			7.803	146	4.61	Db	-1
40 10199040	NetWt			7.803	146	4.61	Db	-1
41 10199041	NetWt			7.803	146	4.61	Db	-1
42 10199042	NetWt			7.803	146	4.61	Db	-1
43 10199043	NetWt			7.803	146	4.61	Db	-1
44 10199044	NetWt			7.803	146	4.61	Db	-1
45 10199045	NetWt			7.803	146	4.61	Db	-1
46 10199046	NetWt			7.803	146	4.61	Db	-1
47 10199047	NetWt			7.803	146	4.61	Db	-1
48 10199048	NetWt			7.803	146	4.61	Db	-1
49 10199049	NetWt			7.803	146	4.61	Db	-1
50 10199050	NetWt			7.803	146	4.61	Db	-1

Quantity: Continued Summary Report
 Test 143: Rat Liver, Bilirubin, Day 1, 4
LIVER TTX 10-21-99 mdr RE
 Sample Date: 10-21-99
 Test Method: C:\VMS\DATA\liver-rat.m, ppo\comp\lab101999
 Test Date: Oct 20 08:11:41 1999
 Last Modified: C:\VMS\DATA\liver-rat.m, ppo\comp\lab101999
 Last Modified: Wed Oct 20 08:14:18 1999
 Job Code:

Printed: Wed Oct 20 08:00:41 1999

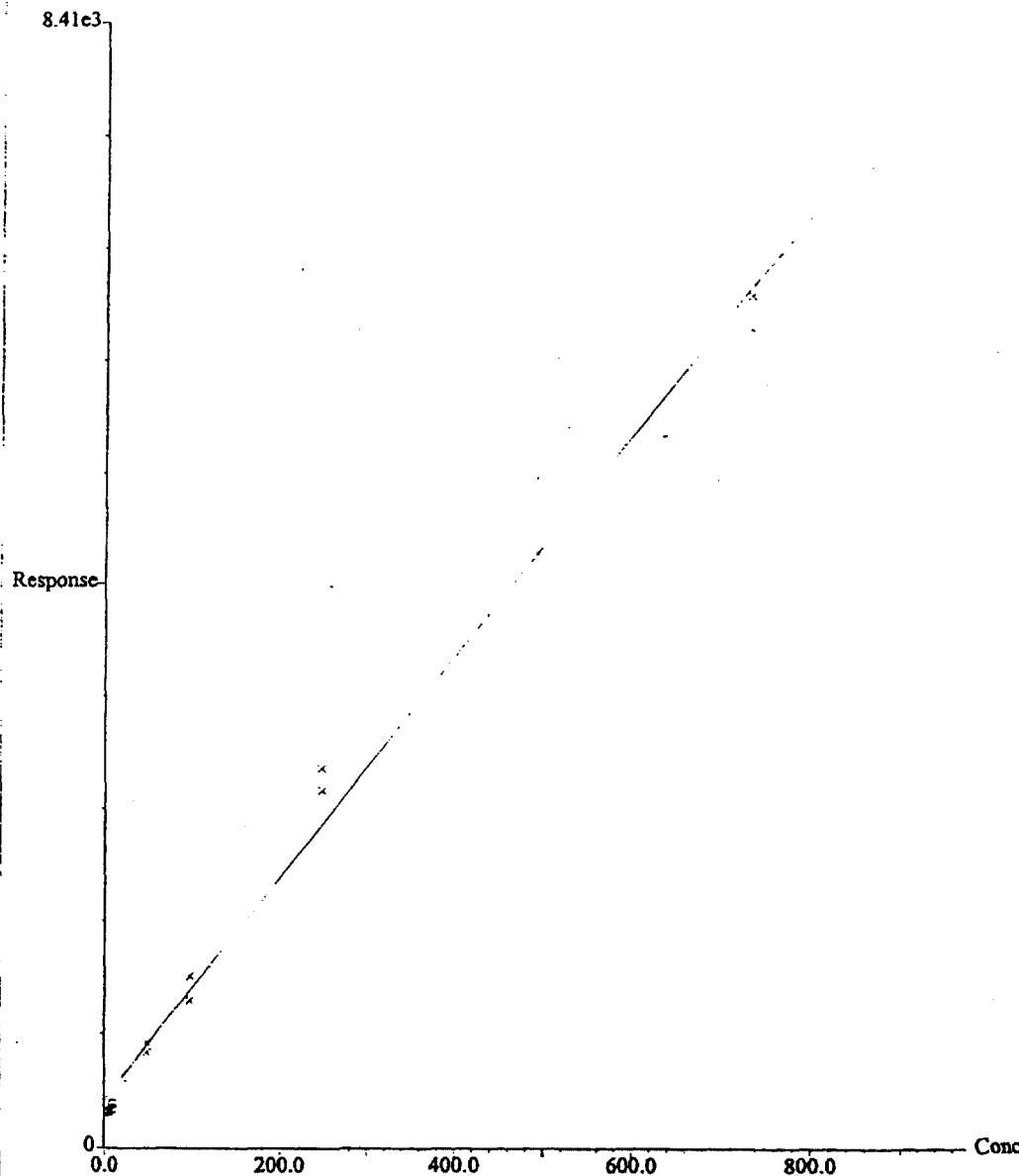
Compound 3: POAA

6 Mon	Complaint	Type	Ref Count	Area	Case	Flies Obs
1 10199001						
2 10199001						
3 10199002						
4 10199003						
5 10199004						
6 10199005						
7 10199006						
8 10199007						
9 10199008						
10 10199009						
11 10199010						
12 10199011						
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268 10199267					</	

Amelia 06200: Quantitated and Printed by mch 10/29/99

Amila 001280: Quantitated and Printed by amh 10/26/99

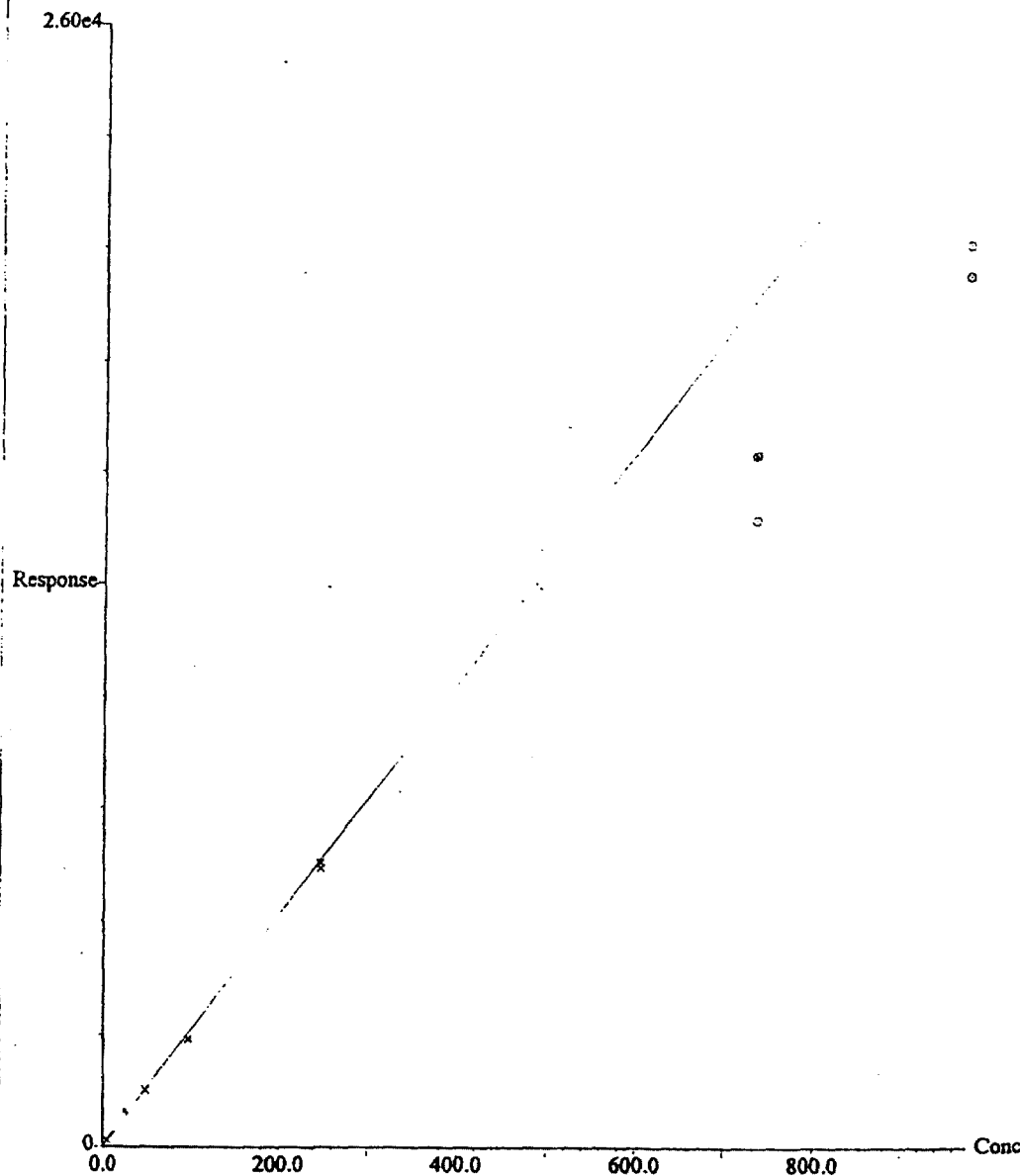
Compound 1 name: PFOS
Coefficient of Determination: 0.989844
Calibration curve: $8.25151 \cdot x + 352.987$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



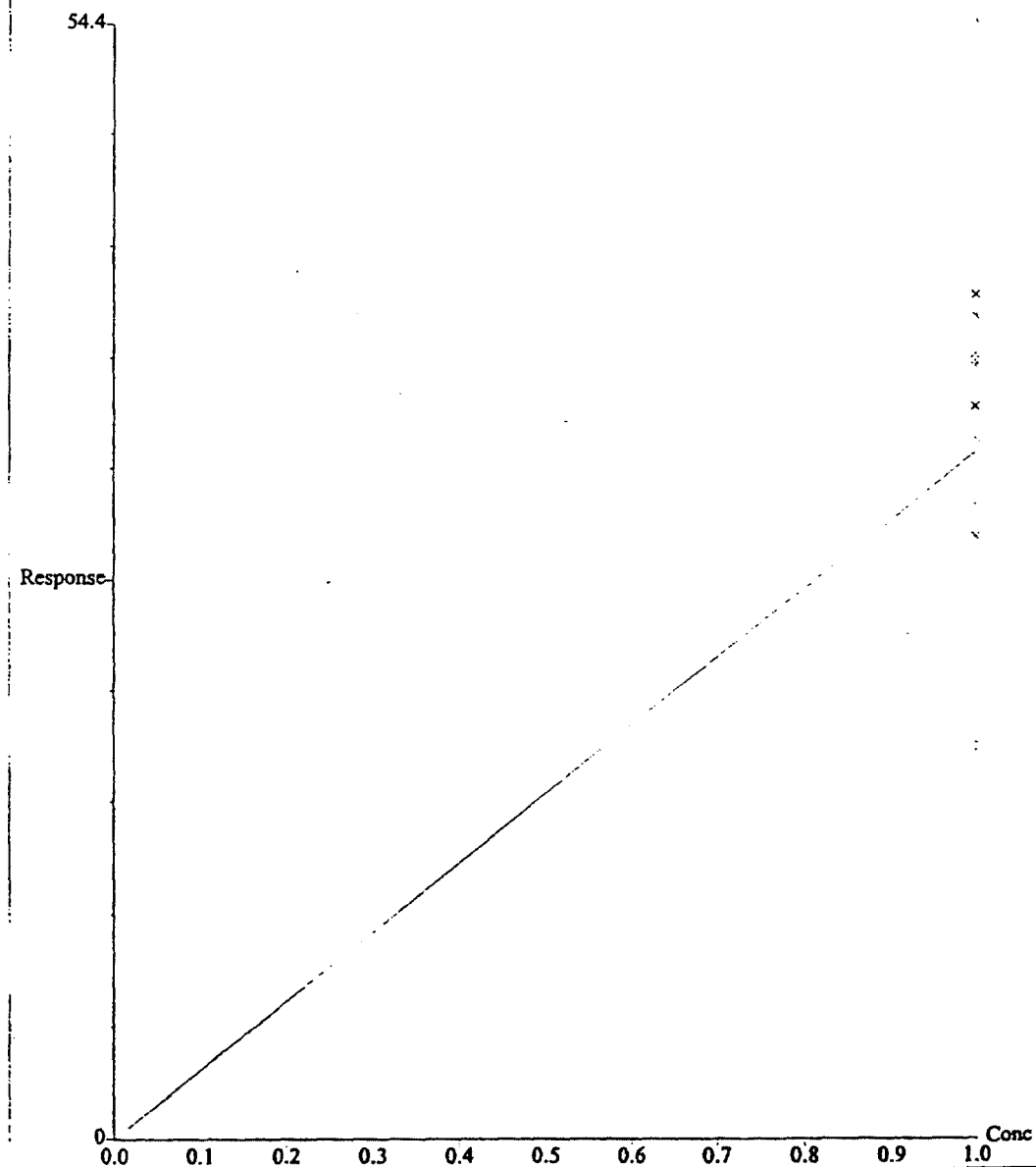
Amelia 062498: Quantitated and Printed by mmh 10/20/99

0 (RE) 10/21/99

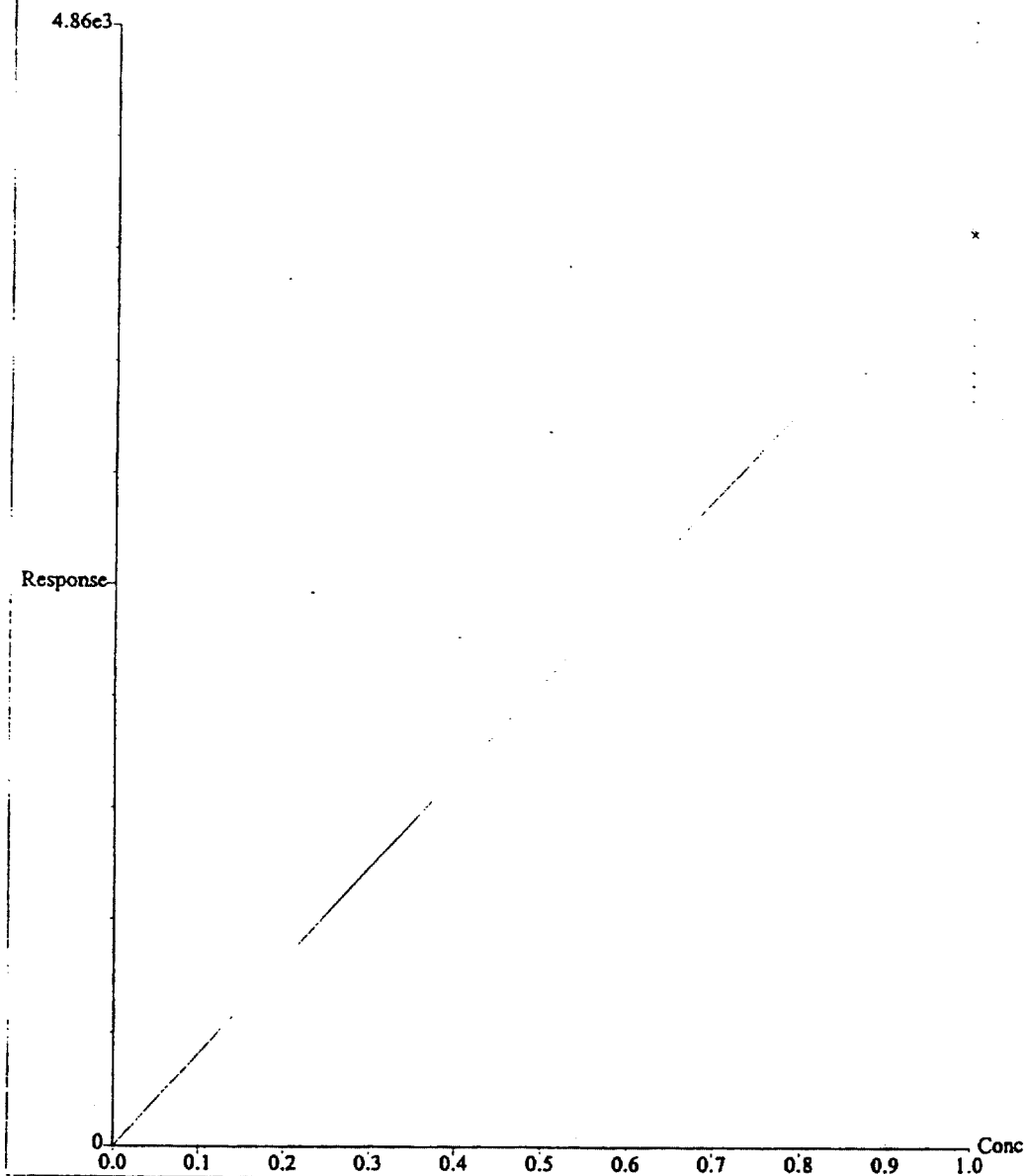
Compound 2 name: PFOSA
Coefficient of Determination: 0.997888
Calibration curve: $26.5524 * x + 23.6545$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound 3 name: POAA
Response Factor: 33.2811
Response type: External Std, Area
Curve type: RF



10/20/99
Compound 4 name: IS (Ch2)
Response Factor: 3976.31
Response type: External Std, Area
Curve type: RF



Draw vertical threshold:	99.40
Draw horizontal threshold:	99.40
Stroke threshold:	99.40
Response threshold:	99.40
Relative height:	1.50, Disabled
Absolute height:	9.00, Disabled
Relative area:	2.00, Disabled
Absolute area:	0.00, Disabled

Method Report

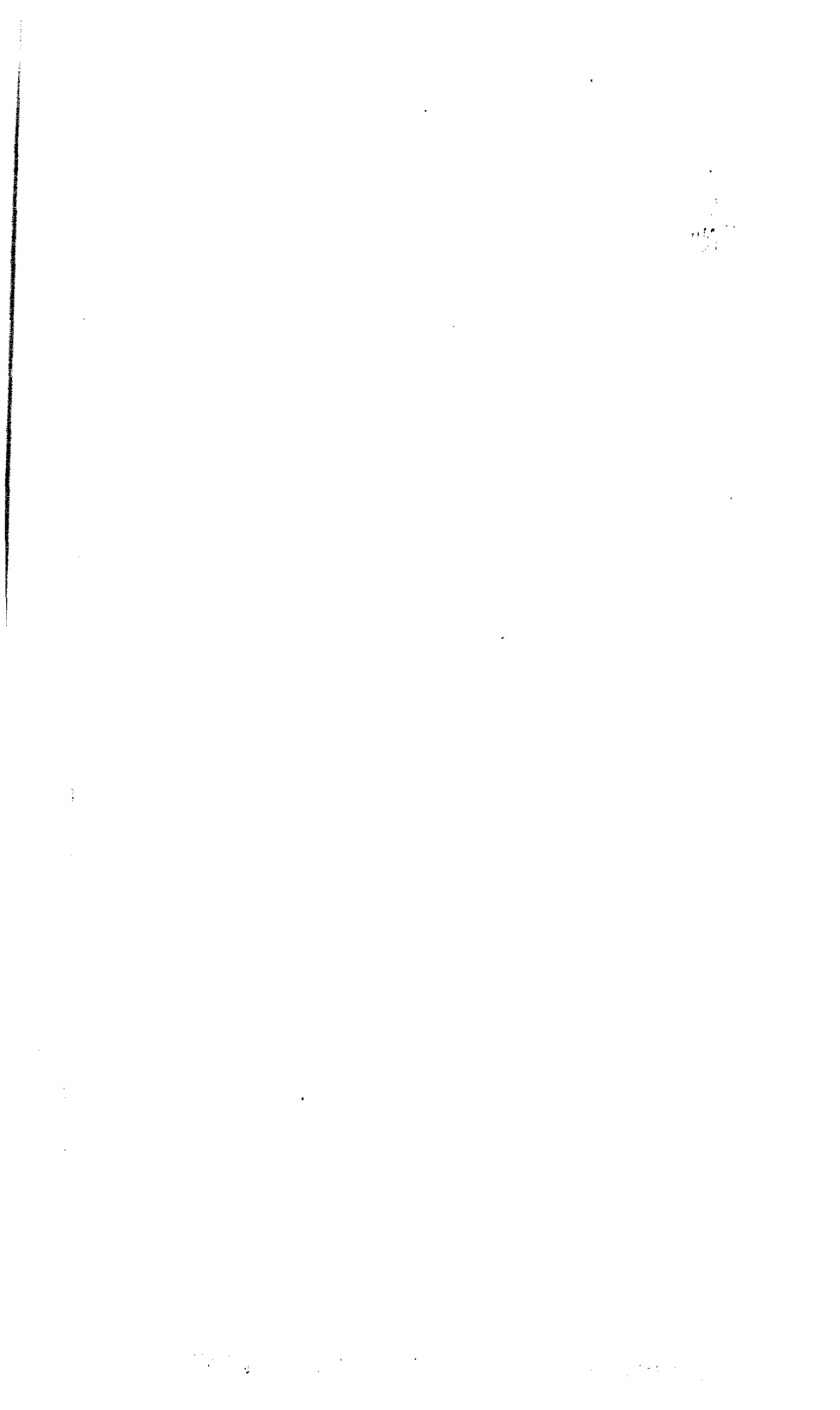
Method File: N:\msd11c\cals\PROV000001\01992c.mtd

Last Modified: Wed Oct 20 08:16:18 1990

Compound: 1: PROS

Printed: Thu Oct 21 15:13:17 1995

Compound Name:	PROS
Internal Ref.:	(None)
Quantity Used:	10 µl
Conc. of Standards:	Conc. A
Start RT Value:	1.00000, Disabled
Use as Reference:	1.00000
Integration Window Factor:	1.00000
Chromatogram Mass Window:	1.00000
Peak Location	
Retention Time (min):	7.167, Enabled
Relative Retention Time:	0.400, Disabled
RT Reference:	(None)
Time Window (mins):	0.330
Peak Selection:	Wearout
Response	
Response Type:	External
Response:	Area
Calibration Curve	
Polynomial Type:	Linear
Point of Origin:	Exclude
File Weighting:	1/X
Axis Transformation:	None
Concentration	
Units:	
Noise	
Peak-to-peak amplitude:	100
Smooth	
Smoothing:	Enabled
Window Size (Scans):	2
Smoothing Algorithm:	1
Smoothing Method:	
Baseline	
Join Valley Threshold:	10.00
Join Valley Setting:	20.00
Peak Detection Method:	3.00
Peak Separation	



Method Report
 Method file: M:\smallarea-rate\PROVATION\101999a.mdb
 Last modified: Wed Oct 20 06:15:18 1999
 Compound: 2: PFOA
 Printed: Thu Oct 21 15:13:10 1999

Compound Name: PFOA
 Internal Ref.: 1000001
 Quantity Traced: TIC
 Conc. of Standards: Conc. B
 User AF Value: 1.000000, Disabled
 User Peak Factor: 1.000000
 Interference Factor: 2.000
 Disabled Mass Window: 1.000000
 Peak Selection

Retention Time (min): 7.124, Enabled
 Retention Time (min): 0.000, Disabled
 Retention Time (min): 0.000, Disabled
 Time Window (min): 0.162
 Peak Selection: nearest

Response
 Response Type: External
 Response: Area

Calibration Curve

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

Dates:

Noise
 Peak Co-Peak Amplitude: 94
 Smooth

Smoothing: Enabled
 Window size (secs): 2
 Number of smoothing: 1
 Smoothing Method: Savitzky
 Savitzky

Join valley threshold: 10.00
 Reduce peak calling: 200.00
 Reduce peak calling: 5.00
 Peak Separation

Draw vertical threshold: 90.00
 Draw vertical threshold: Disabled
 Response Threshold: 90.00
 Response Threshold

Noise Weight: 1.00, Disabled
 Noise Weight: 2.00, Disabled
 Noise Weight: 2.00, Disabled
 Noise Weight: 0.00, Disabled

Method Report

Page 1

Method file: M:\amelie-katz.PRO\MethDB\101999a.mdb
Last modified: Wed Oct 20 08:16:18 1999

Compound: 1: POAA

Printed: Thu Oct 21 13:13:18 1999

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

User AF 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: Area
Calibration Curves

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

Units:
None

Peak-to-peak amplitude: 94
Smooth

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

Join valley threshold: 10.00
Reduce peak tailing: 200.00
Raise baseline maximum: 5.00
Peak Separation

Draw vertical threshold: 90.00
Detect shoulders: Disabled
Shoulder threshold: 10.00
Response Threshold

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

Method Report

Method file: N:\smaller-rats\PRO\Method\101999a.mdb

Last modified: Wed Oct 20 09:16:18 1999

Compound: 4: IS (CH2)

Printed: Thu Oct 21 13:13:18 1999

Compound Name: IS (CH2)
 Internal Ref.: (None)
 Quantify trace: TIC
 Conc. of standards: fixed

User AF Value: 1.000000, Disabled
 User Peak Factor: 1.000000
 Integrate window factor: 2.000
 Concentration mass window: 1.000000
Peak Location

Retention Time (min): 6.210, Enabled
 Relative Retention Time: 6.200, Disabled
 Peak Width (min): 0.406
 Time Window (min): 0.406

Peak Selection: Basecat

Response
 Response Type: External
 Response: Areas

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

Concentration

Units: None

Peak-to-peak amplitude: 24

Smoothing
 Smoothing: Enabled
 Number of points: 2
 Number of smooths: 1

Baseline
 Smoothing method: 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

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

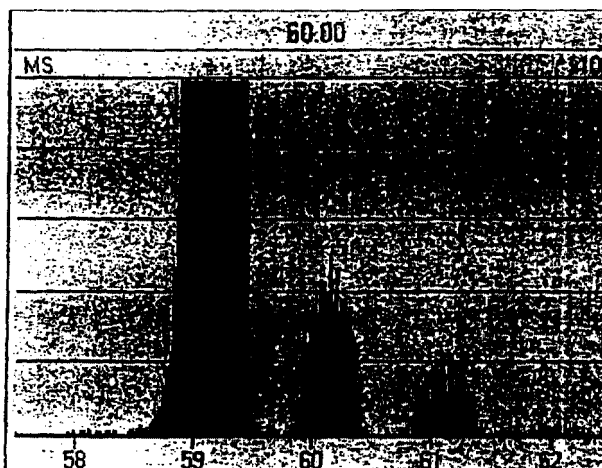
Time (min.)	%B
1) <u>0.00</u>	<u>10.0</u>
2) <u>1.00</u>	<u>10.0</u>
3) <u>5.50</u>	<u>95.0</u>
4) <u>7.50</u>	<u>95.0</u>
5) <u>8.00</u>	<u>10.0</u>

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	
Gas Cell	4.6e-3	HM Resolution	14.0	
		Collision	10	11
		Ion Energy	2.0	
		Ion Energy Ramp	0.0	
		Lens 8	40	39
		Lens 9	0	12
		Multiplier	650	

Scanning Method Report

Page 1

Method: C:\MSL\TIC\AMKLLA-RATE.PRO\ACQ\DS\1000V-PT08-POAA-12-PT08A

Last Modified: Mon Oct 11 06:22:17 1999

Printed: Tue Oct 19 16:22:46 1999

Solvent Delay (mins) : 0.00

Function : 1 100% 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 : 2 100% 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 : 3 100% 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	417.00	60.00	0.30	40	60

Function : 4 100% 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	76.00	0.40	45	60

Sample List: C:\MASSLYNX\amella-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

Sample List: C:\MASSLYNX\Kamella-raiz.PRO\SampleDB\101998a.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 82	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	---	---	---	---	---	---	---

FACT: DATA REVIEW SUMMARY FORM

Data Review Sign-Off

Date: 8/26/99
 Data Analyst: Ias
 FACT Study #: Tox 145 - MD/V
 Matrix/Timepoints: Relat Liver Curve - ed. 10/23/99 R44
 Sample List: 102599C
 Set-up Analysis Date/Analyst: 10/25/99 Ias
 Instrument: Analisa 062498

Analyst Review Name / Date: Ias 10/26/99

Technical Review Name / Date: MFE 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: PFOS (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 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. The 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, 971 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 count of IS looked consistent. The H₂O Bk had a deviation of 58%. It is not known why the deviation of the H₂O Bk is so high. - Ias 10/26/99

FACT: DATA REVIEW SUMMARY FORM

Data Review Sign-Off

Date: 10/26/99
 Data Analyst: JAS
 FACT Study #: Tox 145 - A0/V
 Matrix/Timepoints: Pellet Liver/urine - col. 10/22/99 P461
 Sample List: 102599C
 Set-up Analysis Date/Analyst: 10/25/99 JAS
 Instrument: Amelia 062998

Analyst Review Name / Date: JAS 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 LAC 10/29/99

Comments / Justifications: _____

Analyte: PFOSA C444
 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

④ The 6.13 and 12.30 ppb points were dropped so that the H₂O & Methanol blanks would be less than the LOQ. Methanol blanks for PFOSA were fairly high - JAS 10/26/99
 ⑤ POAA EXTRACTION EFFICIENCY APPEARS TO BE LOW BASED ON TUBE AREA COUNTS. MEE 10/27/99.

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: _____

LAC 10/29/99

Amelia 002480: Questionnaire was printed by jae 10/26/99

Quantity Compound Summary Report

TCM145-Tabble Liver Curve- det: 10/22/99 NW
 Sample List: C:\MSDCHEM\exam145\040499.FM0\Sample01\102390
 Last modified: Tue Oct 26 09:24:20 1999
 Method: C:\MSDCHEM\exam145\040499.FM0\MSDCHEM\102390
 Last modified: Tue Oct 26 09:24:02 1999
 Job Code:

Printed: Tue Oct 26 09:43:02 1999

Compound 1: PMA (CM)

Sample	Sample List	Type	Std Conc	RT	Area	Conc. Flag	Conv
1 10239031	MOCH Blank						
2 10239032	TCM145-MBL1023-100ppm-1, 10/22/99 NW	Standard	6.13	6.976	100	233.08 ng	
3 10239033	TCM145-MBL1023-Liver Blank-1, 10/22/99 NW			6.972	48	45.08 ng	
4 10239034	TCM145-MBL1023-Liver Blank-1, 10/22/99 NW	Standard	6.13	6.966	51	35.89 ng	871
5 10239035	TCM145-MBL1023-100ppm-1, 10/22/99 NW	Standard	12.26	6.973	42	21.77 ng	771
6 10239036	TCM145-MBL1023-100ppm-1, 10/22/99 NW	Standard	24.52	6.982	36	76.40 ng	-150
7 10239037	TCM145-MBL1023-100ppm-1, 10/22/99 NW	Standard	49.04	6.987	34	87.83 ng	-29
8 10239038	TCM145-MBL1023-100ppm-1, 10/22/99 NW	Standard	137.00	6.923	59	654.83 ng	48
9 10239039	TCM145-MBL1023-100ppm-1, 10/22/99 NW	Standard	107.00	6.920	151	664.57 ng	
10 10239040	TCM145-MBL1023-100ppm-1, 10/22/99 NW	Standard	613.00	6.921	200	664.57 ng	
11 10239041	TCM145-MBL1023-100ppm-1, 10/22/99 NW	Standard	325.00	6.918	363	897.16 ng	-2
12 10239042	TCM145-MBL1023-100ppm-1, 10/22/99 NW	Standard	1237.00	6.924	350	1238.10 ng	1

Amble 002190: Quantified and printed by Leo 10/26/99

Page 3

FOIA b 7 - Exemption 1, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, 21, 22, 23, 24, 25, 26, 27, 28, 29, 30, 31, 32, 33, 34, 35, 36, 37, 38, 39, 40, 41, 42, 43, 44, 45, 46, 47, 48, 49, 50, 51, 52, 53, 54, 55, 56, 57, 58, 59, 60, 61, 62, 63, 64, 65, 66, 67, 68, 69, 70, 71, 72, 73, 74, 75, 76, 77, 78, 79, 80, 81, 82, 83, 84, 85, 86, 87, 88, 89, 90, 91, 92, 93, 94, 95, 96, 97, 98, 99, 100, 101, 102, 103, 104, 105, 106, 107, 108, 109, 110, 111, 112, 113, 114, 115, 116, 117, 118, 119, 120, 121, 122, 123, 124, 125, 126, 127, 128, 129, 130, 131, 132, 133, 134, 135, 136, 137, 138, 139, 140, 141, 142, 143, 144, 145, 146, 147, 148, 149, 150, 151, 152, 153, 154, 155, 156, 157, 158, 159, 160, 161, 162, 163, 164, 165, 166, 167, 168, 169, 170, 171, 172, 173, 174, 175, 176, 177, 178, 179, 180, 181, 182, 183, 184, 185, 186, 187, 188, 189, 190, 191, 192, 193, 194, 195, 196, 197, 198, 199, 200, 201, 202, 203, 204, 205, 206, 207, 208, 209, 210, 211, 212, 213, 214, 215, 216, 217, 218, 219, 220, 221, 222, 223, 224, 225, 226, 227, 228, 229, 230, 231, 232, 233, 234, 235, 236, 237, 238, 239, 240, 241, 242, 243, 244, 245, 246, 247, 248, 249, 250, 251, 252, 253, 254, 255, 256, 257, 258, 259, 260, 261, 262, 263, 264, 265, 266, 267, 268, 269, 270, 271, 272, 273, 274, 275, 276, 277, 278, 279, 280, 281, 282, 283, 284, 285, 286, 287, 288, 289, 290, 291, 292, 293, 294, 295, 296, 297, 298, 299, 300, 301, 302, 303, 304, 305, 306, 307, 308, 309, 310, 311, 312, 313, 314, 315, 316, 317, 318, 319, 320, 321, 322, 323, 324, 325, 326, 327, 328, 329, 330, 331, 332, 333, 334, 335, 336, 337, 338, 339, 340, 341, 342, 343, 344, 345, 346, 347, 348, 349, 350, 351, 352, 353, 354, 355, 356, 357, 358, 359, 360, 361, 362, 363, 364, 365, 366, 367, 368, 369, 370, 371, 372, 373, 374, 375, 376, 377, 378, 379, 380, 381, 382, 383, 384, 385, 386, 387, 388, 389, 390, 391, 392, 393, 394, 395, 396, 397, 398, 399, 400, 401, 402, 403, 404, 405, 406, 407, 408, 409, 410, 411, 412, 413, 414, 415, 416, 417, 418, 419, 420, 421, 422, 423, 424, 425, 426, 427, 428, 429, 430, 431, 432, 433, 434, 435, 436, 437, 438, 439, 440, 441, 442, 443, 444, 445, 446, 447, 448, 449, 450, 451, 452, 453, 454, 455, 456, 457, 458, 459, 460, 461, 462, 463, 464, 465, 466, 467, 468, 469, 470, 471, 472, 473, 474, 475, 476, 477, 478, 479, 480, 481, 482, 483, 484, 485, 486, 487, 488, 489, 490, 491, 492, 493, 494, 495, 496, 497, 498, 499, 500, 501, 502, 503, 504, 505, 506, 507, 508, 509, 510, 511, 512, 513, 514, 515, 516, 517, 518, 519, 520, 521, 522, 523, 524, 525, 526, 527, 528, 529, 530, 531, 532, 533, 534, 535, 536, 537, 538, 539, 540, 541, 542, 543, 544, 545, 546, 547, 548, 549, 550, 551, 552, 553, 554, 555, 556, 557, 558, 559, 560, 561, 562, 563, 564, 565, 566, 567, 568, 569, 570, 571, 572, 573, 574, 575, 576, 577, 578, 579, 580, 581, 582, 583, 584, 585, 586, 587, 588, 589, 590, 591, 592, 593, 594, 595, 596, 597, 598, 599, 600, 601, 602, 603, 604, 605, 606, 607, 608, 609, 610, 611, 612, 613, 614, 615, 616, 617, 618, 619, 620, 621, 622, 623, 624, 625, 626, 627, 628, 629, 630, 631, 632, 633, 634, 635, 636, 637, 638, 639, 640, 641, 642, 643, 644, 645, 646, 647, 648, 649, 650, 651, 652, 653, 654, 655, 656, 657, 658, 659, 660, 661, 662, 663, 664, 665, 666, 667, 668, 669, 670, 671, 672, 673, 674, 675, 676, 677, 678, 679, 680, 681, 682, 683, 684, 685, 686, 687, 688, 689, 690, 691, 692, 693, 694, 695, 696, 697, 698, 699, 700, 701, 702, 703, 704, 705, 706, 707, 708, 709, 710, 711, 712, 713, 714, 715, 716, 717, 718, 719, 720, 721, 722, 723, 724, 725, 726, 727, 728, 729, 730, 731, 732, 733, 734, 735, 736, 737, 738, 739, 740, 741, 742, 743, 744, 745, 746, 747, 748, 749, 750, 751, 752, 753, 754, 755, 756, 757, 758, 759, 760, 761, 762, 763, 764, 765, 766, 767, 768, 769, 770, 771, 772, 773, 774, 775, 776, 777, 778, 779, 780, 781, 782, 783, 784, 785, 786, 787, 788, 789, 790, 791, 792, 793, 794, 795, 796, 797, 798, 799, 800, 801, 802, 803, 804, 805, 806, 807, 808, 809, 810, 811, 812, 813, 814, 815, 816, 817, 818, 819, 820, 821, 822, 823, 824, 825, 826, 827, 828, 829, 830, 831, 832, 833, 834, 835, 836, 837, 838, 839, 840, 841, 842

C:\Users\JPC\Local Settings\Temp\Temp1\20120909

Last modified: Tue Dec 28 09:34:39 1999
 Method: C:\WINDOWS\smal1n062496.FPO\WinChn\10359.PC

Last modified: Tue Dec 16 09:34:42 1998
Job Code:

Printed: Tue Oct 26 05:43:52 1998

Compound 2: PFOs (Ch2)

	Sample/Slide Test	Type	Std. Count	RT	Area	Comp. Phase	Notes
1	HE08 Blank			7.148	45	0.00 bsk	
2	1023590035			7.146	33	0.00 bsk	
3	1023590037			7.144	32	0.00 bsk	
4	1023590038			7.142	31	0.00 bsk	
5	1023590039			7.140	30	0.00 bsk	
6	1023590040			7.138	29	0.00 bsk	
7	1023590041			7.136	28	0.00 bsk	
8	1023590042			7.134	27	0.00 bsk	
9	1023590043			7.132	26	0.00 bsk	
10	1023590044			7.130	25	0.00 bsk	
11	1023590045			7.128	24	0.00 bsk	
12	1023590046			7.126	23	0.00 bsk	
13	1023590047			7.124	22	0.00 bsk	
14	1023590048			7.122	21	0.00 bsk	
15	1023590049			7.120	20	0.00 bsk	
16	1023590050			7.118	19	0.00 bsk	
17	1023590051			7.116	18	0.00 bsk	
18	1023590052			7.114	17	0.00 bsk	
19	1023590053			7.112	16	0.00 bsk	
20	1023590054			7.110	15	0.00 bsk	
21	1023590055			7.108	14	0.00 bsk	
22	1023590056			7.106	13	0.00 bsk	
23	1023590057			7.104	12	0.00 bsk	
24	1023590058			7.102	11	0.00 bsk	
25	1023590059			7.100	10	0.00 bsk	
26	1023590060			7.098	9	0.00 bsk	
27	1023590061			7.096	8	0.00 bsk	
28	1023590062			7.094	7	0.00 bsk	
29	1023590063			7.092	6	0.00 bsk	
30	1023590064			7.090	5	0.00 bsk	
31	1023590065			7.088	4	0.00 bsk	
32	1023590066			7.086	3	0.00 bsk	
33	1023590067			7.084	2	0.00 bsk	
34	1023590068			7.082	1	0.00 bsk	
35	1023590069			7.080	0	0.00 bsk	
36	1023590070			7.078	0	0.00 bsk	
37	1023590071			7.076	0	0.00 bsk	
38	1023590072			7.074	0	0.00 bsk	
39	1023590073			7.072	0	0.00 bsk	
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43	1023590077			7.064	0	0.00 bsk	
44	1023590078			7.062	0	0.00 bsk	
45	1023590079			7.060	0	0.00 bsk	
46	1023590080			7.058	0	0.00 bsk	
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48	1023590082			7.054	0	0.00 bsk	
49	1023590083			7.052	0	0.00 bsk	
50	1023590084			7.050	0	0.00 bsk	
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52	1023590086			7.046	0	0.00 bsk	
53	1023590087			7.044	0	0.00 bsk	
54	1023590088			7.042	0	0.00 bsk	
55	1023590089			7.040	0	0.00 bsk	
56	1023590090			7.038	0	0.00 bsk	
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67	1023590101			7.016	0	0.00 bsk	
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77	1023590111			6.996	0	0.00 bsk	
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79	1023590113			6.992	0	0.00 bsk	
80	1023590114			6.990	0	0.00 bsk	
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182	1023590216			6.786	0	0.00 bsk	
183	1023590217			6.784	0	0.00 bsk	
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185	1023590219			6.780	0	0.00 bsk	
186	1023590220			6.778	0	0.00 bsk	
187	1023590221			6.776	0	0.00 bsk	
188	1023590222			6.774	0	0.00 bsk	
189							

Quantify Compound Summary Report

TUM145-Rabbit Liver Curve- Oct. 10/22/99 Raw

Sample List: C:\MSASLTPQ\msaslab2400_PMO\SampleData\102399c

Last modified: Tue Oct 26 09:24:39 1999

Lab: C:\MSASLTPQ\msaslab2400_PMO\LabData\102399c

Last modified: Tue Oct 26 09:24:42 1999

Job Code:

Printed: Tue Oct 26 09:43:52 1999

Compound 3: 18 [C3]

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Amelia 042189: Quantitized and printed by Lee 10/26/98

Quality Compound Summary Report

T0115-Blank Liver Sample- Oct. 10/22/99 Raw

Sample List: C:\msd1200\msd1200\MSD1200\MSD1200\102398a

Last modified: Tue Oct 26 09:34:39 1999

Method: C:\msd1200\msd1200\MSD1200\MSD1200\102398a

Job Code: C:\msd1200\msd1200\MSD1200\MSD1200\102398a

Job Code:

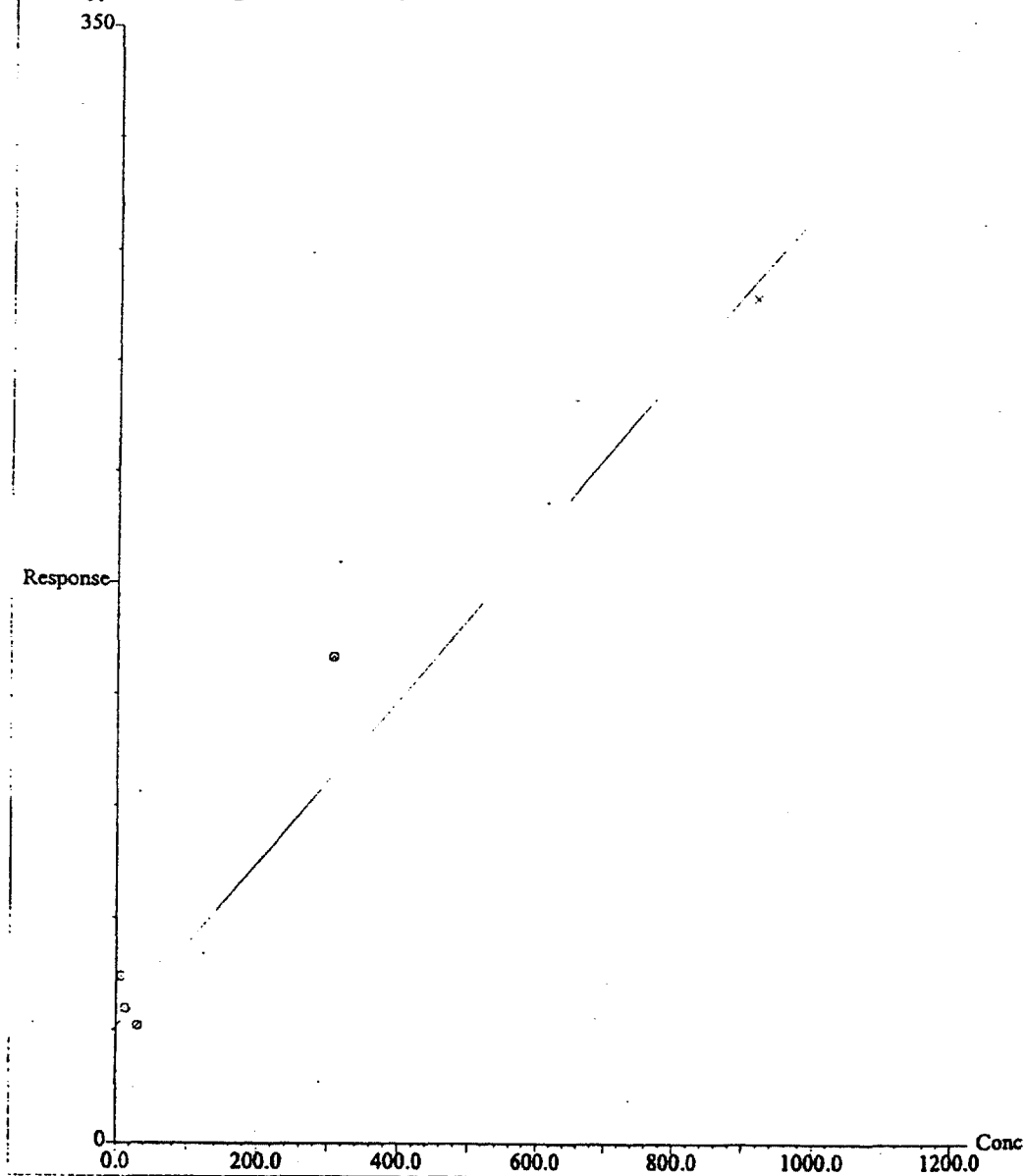
Printed: Tue Oct 26 09:43:33 1999

Compound 4: PTDA (Ch4)

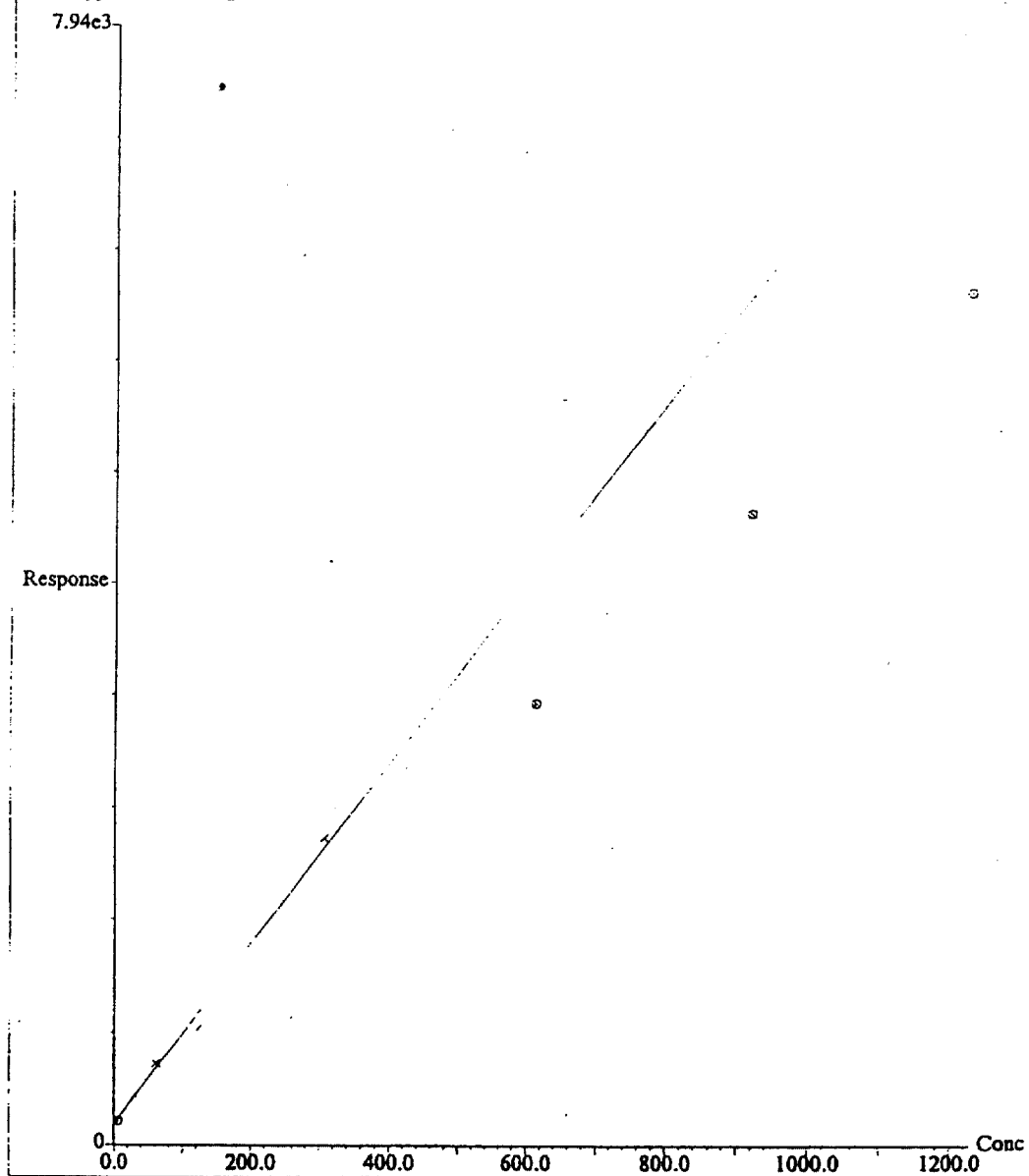
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2 102399036	Blank			7.774	350	15.55	bb	
3 102399037	T0115-MSD1200-102398a-1, 10/22/99 Raw			7.774	268	15.55	bb	
4 102399038	T0115-MSD1200-102398a-1, 10/22/99 Raw			7.774	204	7.44	bb	
5 102399039	T0115-MSD1200-102398a-1, 10/22/99 Raw			7.774	242	9.40	bb	
6 102399040	T0115-MSD1200-102398a-1, 10/22/99 Raw			7.774	342	15.55	bb	
7 102399041	T0115-MSD1200-102398a-1, 10/22/99 Raw			7.774	444	22.22	bb	
8 102399042	T0115-MSD1200-102398a-1, 10/22/99 Raw			7.774	1233	65.73	bb	
9 102399043	T0115-MSD1200-102398a-1, 10/22/99 Raw			7.774	1239	105.80	bb	
10 102399044	T0115-MSD1200-102398a-1, 10/22/99 Raw			7.774	3751	322.47	bb	
11 102399045	T0115-MSD1200-102398a-1, 10/22/99 Raw			7.774	12449	664.82	bb	
12 102399046	T0115-MSD1200-102398a-1, 10/22/99 Raw			7.774	22627	1291.35	bb	
13 102399047	T0115-MSD1200-102398a-1, 10/22/99 Raw			7.774	22627	1291.35	bb	

Amis 02299: Quantized and printed by Jan 10/26/99

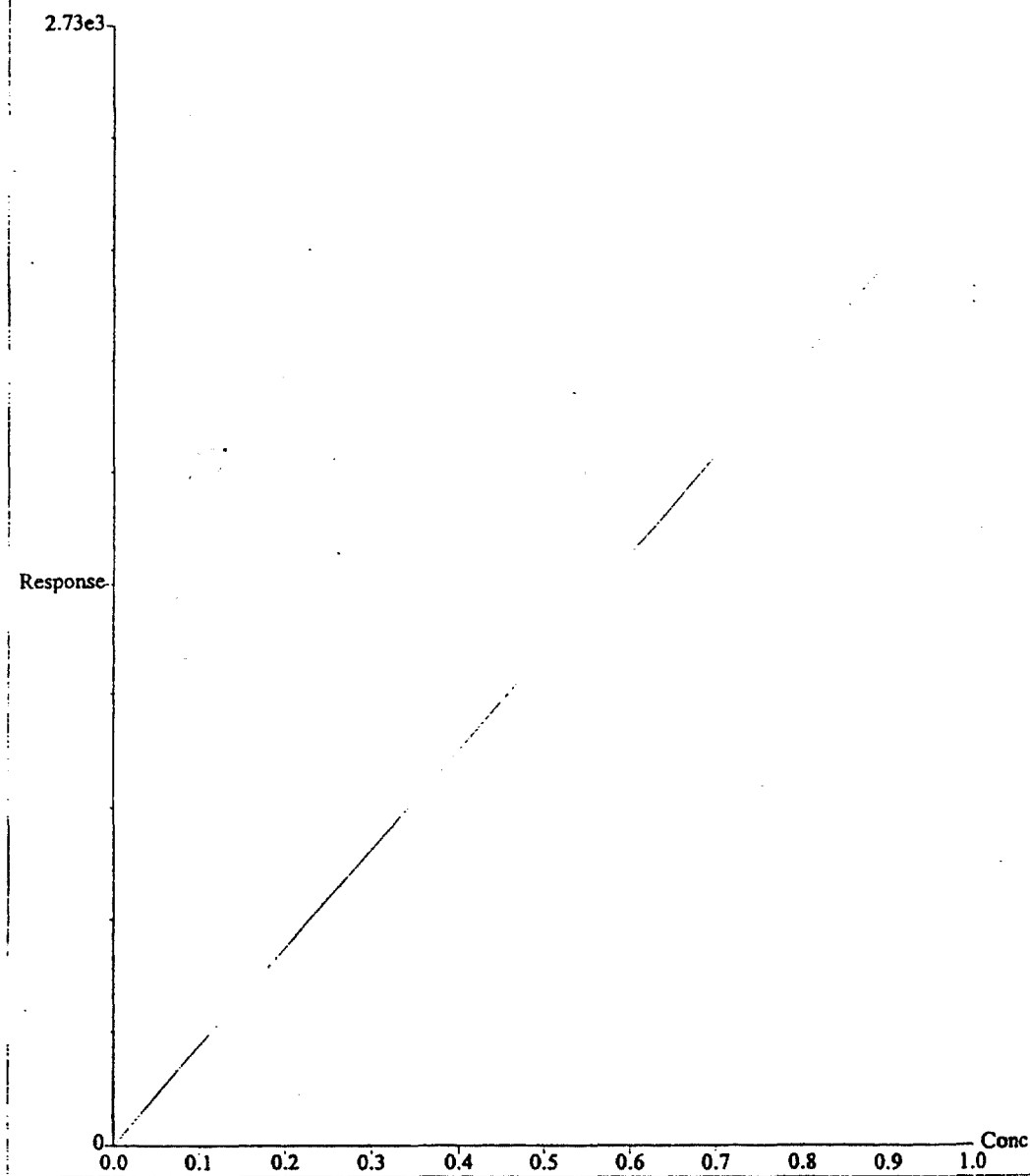
Compound 1 name: POAA (Ch1)
Coefficient of Determination: 0.996986
Calibration curve: $0.253166 * x + 36.3287$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



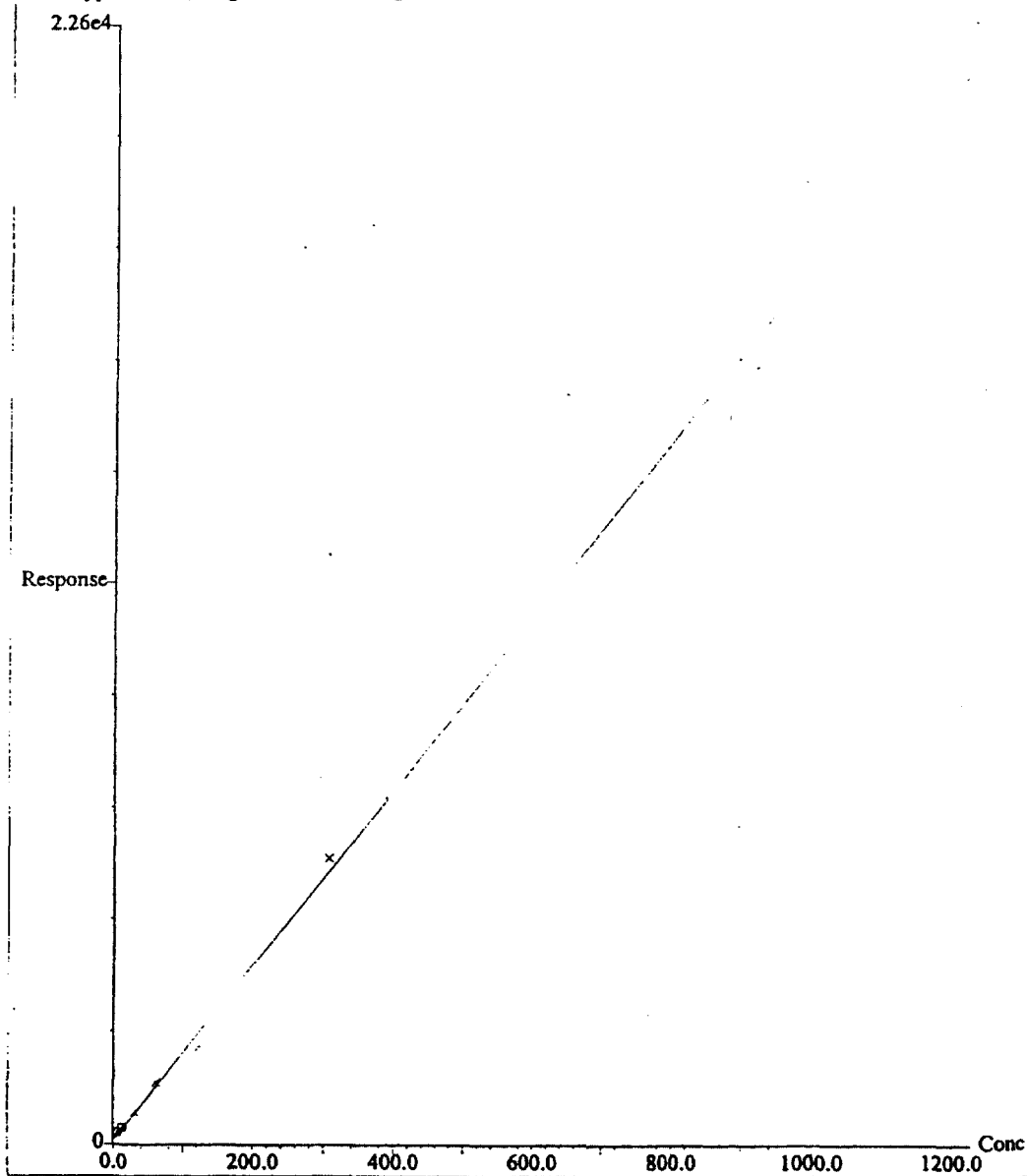
Compound 2 name: PFOS (Ch2)
Coefficient of Determination: 0.992922
Calibration curve: $6.34685 \cdot x + 149.692$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound 3 name: IS (Ch3)
Response Factor: 2378.96
Response type: External Std, Area
Curve type: RF



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



Method Report

Method file: C:\MS31\TM\msl\14082496.FPO\Method\101559c.mdb

Last modified: Tue Oct 26 09:34:42 1999

Compound: 1: POAA (CN1)

Printed: Tue Oct 26 09:44:34 1999

Compound Name: POAA (CN1)

Internal Ref.: (None)

Quantity Traced: TIC

Conc. of Standards: Conc. B

User RT Value: 1.000000, Disabled

User RT Range: 1.000000

Integration Window Factor: 2.000

Chromatogram data window: 1.00000a

Peak Location

Retention Time (min): 6.499, Enabled

RT Reference: 6.499, Disabled

RT Difference: (None)

Time Window (min): 0.252

Peak Selection: Nearest

Response

Response Type: External

Response: Areas

Calibration Curve

Polynomial Type: Linear

Point of Origin: Exclude

Fit Weighting: 1/N

Axis Transformation: None

Concentration

Units:

Noise

Peak-to-peak amplitude: 91

Smooth

Smoothing: Enabled

Window size (scan): 2

Number of smooths: 1

Smoothing method: Baseline

Join valley threshold: 20.00
Reduce peak tailing: 200.00
Base baseline maximum: 5.00
Peak separation
Draw vertical threshold: 50.00
Detect shoulders: Disabled
Shoulder threshold: 10.00
Response threshold
Relative height: 1.50, Disabled
Absolute height: 0.50, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

Method Report

Page 2

Method file: C:\MSDCHEM\msd1062498.PRO\method\10159C.mdb
Last modified: Tue Oct 26 09:36:42 1999
Compound: 2: PTOS (CH2)
Printed: Tue Oct 26 09:44:34 1999

Compound Name:	PTOS (CH2)
Internal Ref.:	(None)
Quantity Traced:	TIC
Comp. of Standards:	Comp. A
User RF Value:	1.00000, Disabled
User Peak Factor:	1.000000
Integration Window Factor:	2.000
Chromatogram Mass Window:	1.00000e
Peak Selection:	
Retention Time (min):	7.109, Enabled
Relative Retention Time:	0.000, Disabled
RT Reference:	(None)
Time Window (min):	0.727
Peak Selection:	Nearest
Response	
Response Type:	External
Response:	Area
Calibration Curves	
Polynomial Type:	Linear
Point of Origin:	Exclude
RT Reference:	2
Axis Transformation:	None
Concentration	
Units:	
Noise	
Peak-to-peak amplitude:	96
Smooth	
Smoothing:	Enabled
Window size (scan):	2
Number of smooths:	1
Smoothing method:	Baseline
Baseline	
Join valley threshold:	30.00
Reduce peak calling:	200.00
Reduce baseline maximum:	5.00
Peak Detection	
Dew vertical threshold:	90.00
Detect shoulders:	Disabled
Shoulder threshold:	20.00
Shoulder threshold:	20.00
Relative height:	1.50, Disabled
Absolute height:	0.00, Disabled
Relative area:	2.00, Disabled
Absolute area:	0.00, Disabled

Method Report

Page 3

Method file: C:\MSDCHEM\Amelia062498.PRO\MethD8\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 RT Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram mass window: 1.00000e
Peak Location

Retention Time (mins): 4.924, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: [None]
Time Window (mins): 0.252

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: 74
Smooth

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

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

Method Report

Method file: C:\MSDCHEM\msdch01\0041008.PRO\METHODS\102396C.MXD

Last modified: Tue Oct 26 09:34:42 1999

Compound: 6: PFOXA (C6H)

Printed: Tue Oct 26 09:44:34 1999

Compound Name: PFOXA (C6H)
 Molecular Weight: 238.07
 Quantify Trace: TIC
 Conc. of Standards: Conc. 8

User AF Values: 1.000000, Disabled
 User Peak Factor: 1.000000
 Internal Standard: 2.000000
 Chromatogram mass window: 1.00000m

Peak Location

Retention Time (min): 7.851, Enabled
 Relative Retention Time: 0.000, Disabled
 Retention Time (min): 7.851
 Time Window (min): 0.311

Peak Selection:

Response: Nearest
 Response Type: External
 Response: Areas

Calibration Curves

Polynomial Type: Linear
 Fitting Origin: Exclude
 Peak Selection: Nearest
 Axis Transformation: None

Units:

Units: None
 Peak-to-peak amplitude: 310
 Smooth

Smoothing:

Smoothing: Enabled
 Number of points: 3
 Smoothing method: Baseline

Join valley threshold: 30.00
Peak separation: 30.00
Peak baseline minimum: 5.00
Peak separation: 30.00
Draw vertical threshold: 30.00
Peak separation: 30.00
Shoulder threshold: 30.00
Response threshold: 30.00
Relative height: 1.50, Disabled
Relative height: 1.50, Disabled
Relative area: 2.00, Disabled
Absolute area: 8.00, Disabled

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? ☒ 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>

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
Analysar 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	

Scanning Method Report

Page 1

Method: C:\MSLTH\AMLLA-RATE.PRO\ACQDS\IONEN-PT08-POAA-18-PT08A
Last Modified: Mon Oct 11 09:22:17 1999
Printed: Mon Oct 23 10:13:30 1999

Solvent Delay (mins) : 0.00

Function : 1 MSW 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 : 3

Channel	Parent	Daughter	Dwell (Secs)	Coll Energy (eV)	Cone (V)

1	413.00	169.00	0.30	30	15

Function : 2 MSW 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 MSW 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 MSW 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	496.00	76.00	0.40	45	60

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

Masslynx - Sample List

Page 1

Sample List: C:\MASSLYNX\amella-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

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

FACT: DATA REVIEW SUMMARY FORM

Date: 1/29/99
 Data Analyst: JAS
 FACT Study #: T2145
 Matrix/Timepoints: Twice 80-sec Confirmation - Oct 10/29/99
 Sample List: 102899a
 Set-up Analysis Date/Analyst: 10/29/99 MEE/EAS
 Instrument: Sup 020199

Data Review Sign-Off

Analyst Review Name / Date: JAS 10/29/99

Technical Review Name / Date: MEE 10/29/99 / 16h Nov-4-99

Quality Assurance Review Name / Date: _____

Data Entry: DATA NOT Reported (X) GML 11/19/99

Comments / Justifications: _____

Analyte: POAA (CH1) (3) (5)
 Acceptable Linear Range: 1.00 ppb → 251 ppb
 LOQ: 1.00 ppb Extraction Blank: NA
 R²: 0.996938 Matrix Blank: NA
 Calibration Checks: OK - Range from 13% → 4%
 MS/MSD: (X) DATA NOT Reported GML 11/9/99
 Data Status: _____

Analyte: PFOS (CH2) (3) (5)
 Acceptable Linear Range: 1.0 ppb → 252 ppb
 LOQ: 1.0 ppb Extracted Blank: NA
 R²: 0.999345 Matrix Blank: NA
 Calibration Checks: OK - Range from 6 → 4%
 MS/MSD: (X) 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: (X) 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 502 ppb and 752 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, 5.00 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 calculation. Further analysis will occur after reextraction of the samples. GML 11/9/99

FACT: DATA REVIEW SUMMARY FORM

Date: 10/29/99
 Data Analyst: JAS
 FACT Study #: T02145
 Matrix/Timepoints: T02145 - dose confirmation - ext 11/2/99
 Sample List: 102899a
 Set-up Analysis Date/Analyst: 10/28/99 MEE/JAS
 Instrument: Samp 020199

Data Review Sign-Off

Analyst Review
 Name / Date: _____

Technical Review
 Name / Date: _____

Quality Assurance Review
 Name / Date: _____

Data Entry: _____

Comments / Justifications: _____

Analyte: I6 (C44)
 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 - JAS 11/2/99

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 - JAS 11/2/99

Quantify Compound Summary Report
 TOX145-tween 80 dose confirmation- PFOS/POOA/POAA ext. 10/28/99 lss

Page 1

Sample List: C:\MS6LTXE\comp020199.prn\SampleDB\102899a
 Last modified: Fri Oct 29 07:02:56 1999
 Method: C:\MS6LTXE\comp020199.prn\MethodB\102899a
 Last modified: Fri Oct 29 06:53:35 1999
 Job Code:

Printed: Fri Oct 29 07:37:25 1999

Compound 1: POAA (Ch1)

#	Name	Sample/lot Test	Type	Std Conc	RY	Area	Flags	Conc.	%Dev
1	102899001	MeOH Blk							
2	102899002	MeOH Blk							
3	102899003	PCMX-99086-43-1, 1ppb- 9/10/99 RMW	Standard	1.00	7.043	17	MM	0.05	-15
4	102899004	PCMX-99086-43-2, 5ppb- 9/10/99 RMW	Standard	5.02	7.107	30	bb	3.69	-26
5	102899005	PCMX-99086-43-3, 10ppb- 9/10/99 RMW	Standard	10.00	7.085	87	MM	10.39	4
6	102899006	PCMX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.10	7.087	193	bb	24.79	-1
7	102899007	PCMX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	50.20	7.092	364	bb	48.04	-4
8	102899008	PCMX-99086-43-6, 100ppb- 9/10/99 RMW	Standard	100.00	7.085	743	bb	99.65	-0
9	102899009	PCMX-99086-43-7, 250ppb- 9/10/99 RMW	Standard	251.00	7.110	1780	bb	240.63	-4
10	102899010	PCMX-99086-43-8, 500ppb- 9/10/99 RMW	Standard	502.00	7.101	3005	bbx	407.36	-19
11	102899011	PCMX-99086-43-9, 750ppb- 9/10/99 RMW	Standard	752.00	7.111	3991	bbx	561.42	-24
12	102899012	MeOH Blk							
13	102899013	TOX145-PFOS/POAA in Tween-dose-2ppb- 10/28/99 IAS							
14	102899014	TOX145-PFOS/POAA in Tween-mo-4ppb- 10/28/99 IAS							
15	102899015	PCMX-99086-43-4, 25ppb- 9/10/99 RMW	QC	25.10	7.129	219	MM	28.36	13
16	102899016	MeOH Blk							
17	102899017	TOX145-PFOA in Tween-dose-200ppb- 10/28/99 IAS							
18	102899018	TOX145-PFOA in Tween-mo-300ppb- 10/28/99 IAS							
19	102899019	PCMX-99086-43-7, 250ppb- 9/10/99 RMW	QC	251.00	7.132	1775	bb	240.01	-4
20	102899020	MeOH Blk							
21	102899021	PCMX-99086-43-1, 1ppb- 9/10/99 RMW	Standard	1.00	7.128	19	MM	1.10	10
22	102899022	PCMX-99086-43-2, 5ppb- 9/10/99 RMW	Standard	5.02	7.126	46	bb	4.76	-5
23	102899023	PCMX-99086-43-3, 10ppb- 9/10/99 RMW	Standard	10.00	7.130	95	MM	11.48	15
24	102899024	PCMX-99086-43-4, 25ppb- 9/10/99 RMW	Standard	25.10	7.157	212	bb	27.42	9
25	102899025	PCMX-99086-43-5, 50ppb- 9/10/99 RMW	Standard	50.20	7.125	422	bb	56.00	12
26	102899026	PCMX-99086-43-6, 100ppb- 9/10/99 RMW	Standard	100.00	7.130	819	bb	109.94	10
27	102899027	PCMX-99086-43-7, 250ppb- 9/10/99 RMW	Standard	251.00	7.133	1618	bb	245.90	-2
28	102899028	PCMX-99086-43-8, 500ppb- 9/10/99 RMW	Standard	502.00	7.128	3158	bbx	428.05	-15
29	102899029	PCMX-99086-43-9, 750ppb- 9/10/99 RMW	Standard	752.00	7.129	4081	bbx	551.60	-26

Quantity Compared Summary Report
 TUL15-7000 80 Check Confirmation- PFC6/PFC6A/POA use: 10/26/99 Las

Sample List: C:\msdmt\TUL15\7000\20199\pfc6\pfc6a\102699a
 Last modified: Fri Oct 29 07:12:15 1999
 Sample Name: TUL15-7000 80 Check Confirmation- PFC6/PFC6A/POA use: 10/26/99 Las
 Job Code: PFC6-7000 80 Check Confirmation- PFC6/PFC6A/POA use: 10/26/99 Las

Printed: Fri Oct 29 07:12:15 1999

Compared 21 PFC6 (CHK)

Q	Name	Sample/lot Name	Type	Std Conc	SP	Area	Flags	Conc	Qty
1	102699001	NaOH BLK			7.245	2376	55 MB	0.14	8
2	102699002	NaOH BLK			7.245	2376	55 MB	0.14	8
3	102699003	PFC6-7000-43-1, 10ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21
4	102699004	PFC6-7000-43-2, 20ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21
5	102699005	PFC6-7000-43-3, 30ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21
6	102699006	PFC6-7000-43-4, 50ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21
7	102699007	PFC6-7000-43-5, 50ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21
8	102699008	PFC6-7000-43-6, 100ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21
9	102699009	PFC6-7000-43-7, 200ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21
10	102699010	PFC6-7000-43-8, 200ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21
11	102699011	PFC6-7000-43-9, 750ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21
12	102699012	NaOH BLK			7.245	2376	55 MB	0.14	8
13	102699013	TUL15-7000 80 Check Confirmation- PFC6/PFC6A/POA use: 10/26/99 Las			7.245	2376	55 MB	0.14	8
14	102699014	TUL15-7000 80 Check Confirmation- PFC6/PFC6A/POA use: 10/26/99 Las			7.245	2376	55 MB	0.14	8
15	102699015	NaOH BLK			7.245	2376	55 MB	0.14	8
16	102699016	NaOH BLK			7.245	2376	55 MB	0.14	8
17	102699017	TUL15-7000 80 Check Confirmation- PFC6/PFC6A/POA use: 10/26/99 Las			7.245	2376	55 MB	0.14	8
18	102699018	TUL15-7000 80 Check Confirmation- PFC6/PFC6A/POA use: 10/26/99 Las			7.245	2376	55 MB	0.14	8
19	102699019	NaOH BLK			7.245	2376	55 MB	0.14	8
20	102699020	NaOH BLK			7.245	2376	55 MB	0.14	8
21	102699021	PFC6-7000-43-1, 10ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21
22	102699022	PFC6-7000-43-2, 20ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21
23	102699023	PFC6-7000-43-3, 30ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21
24	102699024	PFC6-7000-43-4, 50ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21
25	102699025	PFC6-7000-43-5, 50ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21
26	102699026	PFC6-7000-43-6, 100ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21
27	102699027	PFC6-7000-43-7, 200ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21
28	102699028	PFC6-7000-43-8, 200ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21
29	102699029	PFC6-7000-43-9, 750ppb- 9/10/99 MW	Standard	1.01	7.245	109 MB	443 MB	0.79	-21

Comp 020159: Quantitated and printed by LSC on 10/23/10

Quantity Compound Summary Report
Y01151-train 84 dose confirmation- PF04/PFOA/PFOA Wt. 16/10/99 Lbs

Sample List: C:\MSDEV\X\comp20159\pfoa\sample\101899a
Last modified: Fri Oct 29 07:03:56 1999
Created: 10/29/99 07:03:56
Last modified: Fri Oct 29 06:52:25 1999
Job Code:

Printed: Fri Oct 29 07:37:25 1999

Compound 3: PFOA (C43)

# Name	Sample/lot	Type	Ref. Conc.	PF	Age	Comp.	Wt.
1 10289901	Sample R1	Standard	1.01	7.402	33	0.00	0.00
2 10289902	Sample R1	Standard	1.01	7.402	33	0.00	0.00
3 10289903	PCIL-91086-43-1, 10ppb- 9/10/99 New	Standard	5.03	7.766	763 MCK	0.00	-100
4 10289904	PCIL-91086-43-2, 10ppb- 9/10/99 New	Standard	10.10	7.766	2519 MCK	0.00	-100
5 10289905	PCIL-91086-43-3, 10ppb- 9/10/99 New	Standard	15.15	7.766	1642 MCK	2.11	-79
6 10289906	PCIL-91086-43-4, 10ppb- 9/10/99 New	Standard	20.20	7.766	1042 MCK	3.11	-79
7 10289907	PCIL-91086-43-5, 10ppb- 9/10/99 New	Standard	25.25	7.771	13708 MCK	39.00	-22
8 10289908	PCIL-91086-43-6, 10ppb- 9/10/99 New	Standard	30.30	7.765	43375 MCK	96.04	-4
9 10289909	PCIL-91086-43-7, 10ppb- 9/10/99 New	Standard	35.35	7.811	105764 MCK	249.16	-1
10 10289910	PCIL-91086-43-8, 10ppb- 9/10/99 New	Standard	40.40	7.818	205809 MCK	491.80	-2
11 10289911	PCIL-91086-43-9, 10ppb- 9/10/99 New	Standard	45.45	7.821	305816 MCK	735.16	-4
12 10289912	Sample R1	Standard	736.80	7.823	2111 MCK	0.00	-4
13 10289913	TOX15-PFOA/PFOA in PFOA-dose-10ppb- 10/28/99 LBS	OC	25.10	7.623	4283732 MCK	10459.75	
14 10289914	TOX15-PFOA/PFOA in PFOA-dose-10/28/99 LBS	OC	25.10	7.623	431522 MCK	10518.37	
15 10289915	PCIL-91086-43-2, 21ppb- 9/10/99 New	Standard	25.10	7.623	82116 MCK	143.25	479
16 10289916	PCIL-91086-43-3, 21ppb- 9/10/99 New	Standard	25.10	7.623	14416 MCK	24.75	
17 10289917	TOX15-PFOA in PFOA-dose-20ppb- 10/28/99 LBS	OC	251.00	7.623	432155 MCK	18455.25	
18 10289918	TOX15-PFOA in PFOA-dose-20ppb- 10/28/99 LBS	OC	251.00	7.623	4271593 MCK	10421.03	
19 10289919	Sample R1	Standard	1.01	7.623	359270 MCK	379.83	31
20 10289920	PCIL-91086-43-1, 10ppb- 9/10/99 New	Standard	5.03	7.623	14416 MCK	27.09	011
21 10289921	PCIL-91086-43-2, 10ppb- 9/10/99 New	Standard	10.10	7.623	82116 MCK	10.78	311
22 10289922	PCIL-91086-43-3, 10ppb- 9/10/99 New	Standard	15.15	7.623	8260 MCK	13.46	31
23 10289923	PCIL-91086-43-4, 10ppb- 9/10/99 New	Standard	20.20	7.623	16534 MCK	31.29	35
24 10289924	PCIL-91086-43-5, 10ppb- 9/10/99 New	Standard	25.25	7.623	25954 MCK	54.26	8
25 10289925	PCIL-91086-43-6, 10ppb- 9/10/99 New	Standard	30.30	7.623	25954 MCK	54.26	8
26 10289926	PCIL-91086-43-7, 10ppb- 9/10/99 New	Standard	35.35	7.610	220816 MCK	316.40	14
27 10289927	PCIL-91086-43-8, 10ppb- 9/10/99 New	Standard	40.40	7.610	220816 MCK	316.40	14
28 10289928	PCIL-91086-43-9, 10ppb- 9/10/99 New	Standard	45.45	7.610	217905 MCK	523.17	4
29 10289929	PCIL-91086-43-9, 10ppb- 9/10/99 New	Standard	736.80	7.808	304590 MCK	715.21	-2

Comp 820191: Quantitated and printed by LSC on 10/22/99

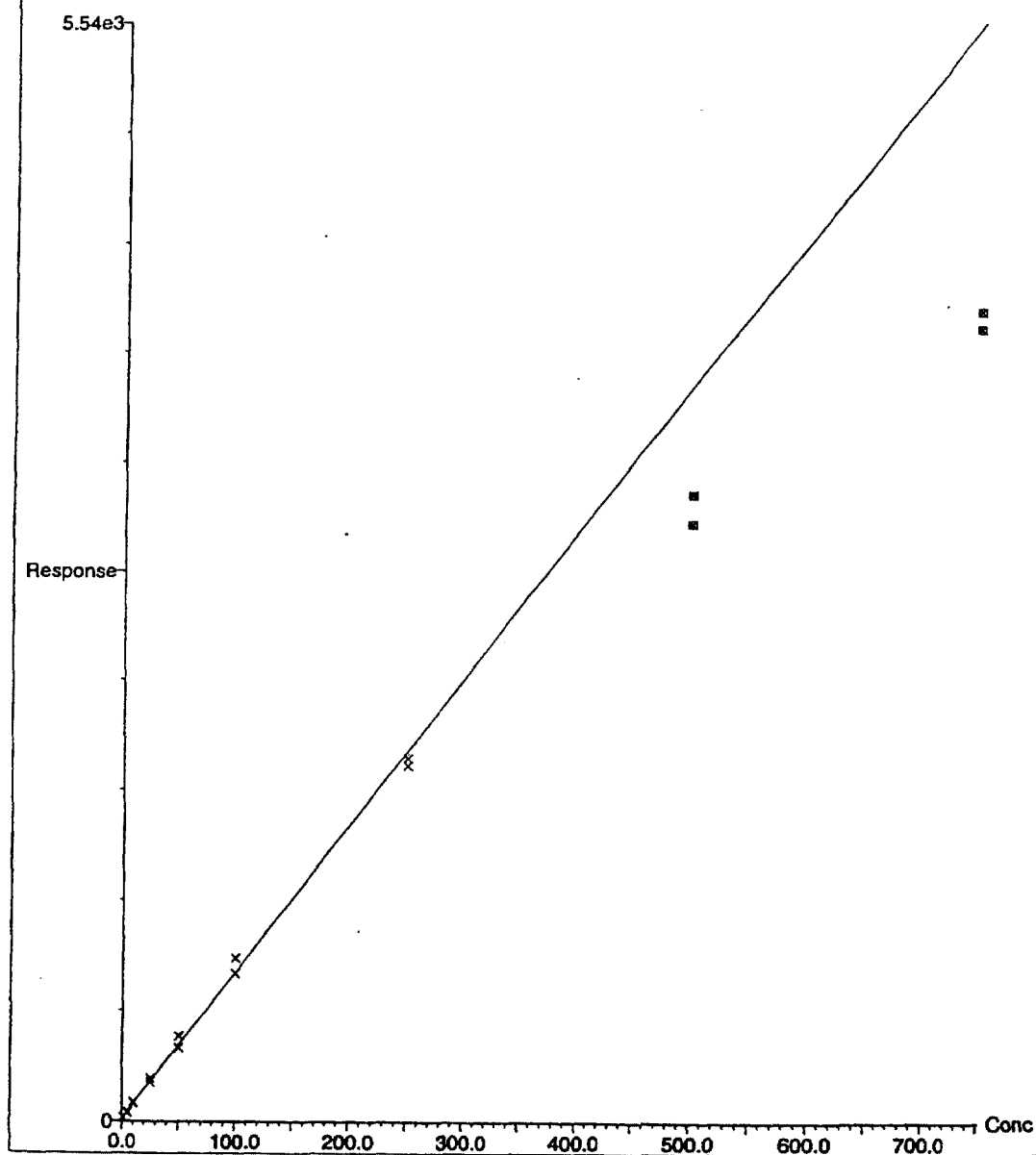
QUALITY Compound Summary Report
 T00145-t000 20 dose confirmation- PPOH/PP0H/PP0A cont. 10/20/99 100
 Sample List: C:\MSD8\T001\temp201099.mn\temp100\1020099a
 Last modified: Fri Oct 29 07:01:06 1999
 Method: C:\MSD8\T001\temp201099.mn\method\1020099a
 Last modified: Fri Oct 29 06:53:55 1999
 Job Code:
 Printed: Fri Oct 29 07:37:25 1999

Compound 4: 20 (Ch4)

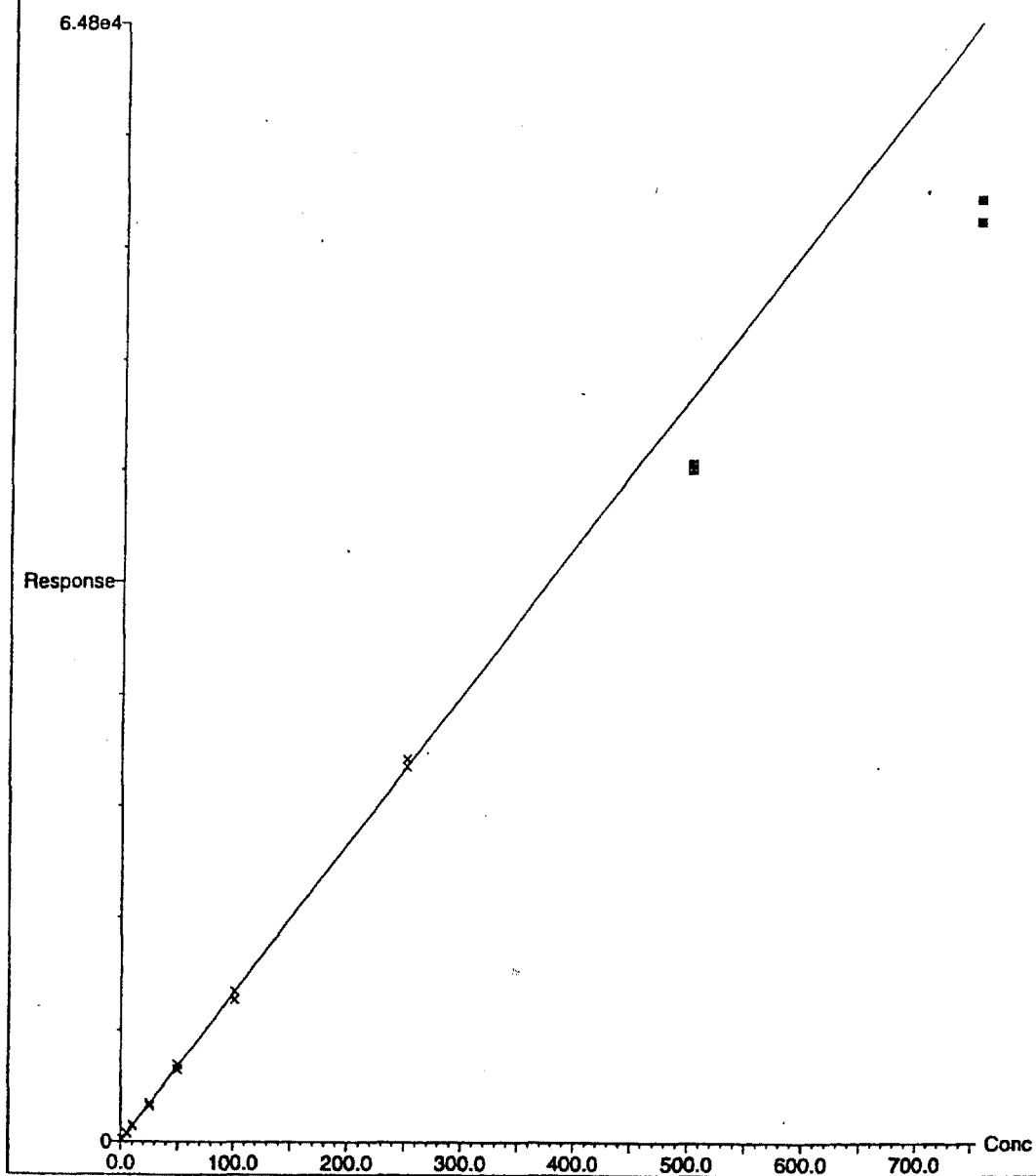
S Name	Samplelist Name	Type	Std Conc	RT	Area	Flags	Conc. Value
1 102199001	Mock Mix			7.061	16		0.00 -100
2 102199002	PCUL-99006-43-1, 10ppb- 9/10/99 MW	Standard	1.00	7.063	4165	20	1.26 26
3 102199003	PCUL-99006-43-2, 10ppb- 9/10/99 MW	Standard	1.00	7.101	4466	20	1.40 40
4 102199004	PCUL-99006-43-3, 10ppb- 9/10/99 MW	Standard	1.00	7.181	4666	20	1.40 40
5 102199005	PCUL-99006-43-4, 10ppb- 9/10/99 MW	Standard	1.00	7.183	4814	20	1.45 45
6 102199006	PCUL-99006-43-5, 10ppb- 9/10/99 MW	Standard	1.00	7.184	4814	20	1.45 45
7 102199007	PCUL-99006-43-6, 10ppb- 9/10/99 MW	Standard	1.00	7.182	4172	20	1.26 26
8 102199008	PCUL-99006-43-7, 10ppb- 9/10/99 MW	Standard	1.00	7.185	2078	20	0.87 13
9 102199009	PCUL-99006-43-8, 10ppb- 9/10/99 MW	Standard	1.00	7.183	2479	20	0.75 25
10 102199010	PCUL-99006-43-9, 10ppb- 9/10/99 MW	Standard	1.00	7.124	2203	20	0.67 31
11 102199011	Mock Mix			7.124	3463	20	1.05 5
12 102199012	T00145-PP0H/PP0A 10 T000-mn-20ppb- 10/20/99 TALS			7.129	3158	20	1.07 7
13 102199013	PCUL-99006-43-4, 10ppb- 9/10/99 MW	QC	1.00	7.124	3185	20	0.96 -4
14 102199014	Mock Mix			7.128	3479	20	1.33 31
15 102199015	T00145-PP0H/PP0A 10 T000-mn-30ppb- 10/20/99 TALS			7.124	3545	20	1.07 7
16 102199016	PCUL-99006-43-7, 10ppb- 9/10/99 MW	QC	1.00	7.127	2482	20	0.81 -19
17 102199017	Mock Mix			7.145	3119	20	0.84 -4
18 102199018	PCUL-99006-43-1, 10ppb- 9/10/99 MW	Standard	1.00	7.145	3219	20	0.96 -4
19 102199019	PCUL-99006-43-2, 10ppb- 9/10/99 MW	Standard	1.00	7.145	2979	20	0.98 -10
20 102199020	PCUL-99006-43-3, 10ppb- 9/10/99 MW	Standard	1.00	7.153	2937	20	0.89 -11
21 102199021	PCUL-99006-43-4, 10ppb- 9/10/99 MW	Standard	1.00	7.141	2987	20	0.88 -12
22 102199022	PCUL-99006-43-5, 10ppb- 9/10/99 MW	Standard	1.00	7.145	2985	20	0.76 -22
23 102199023	PCUL-99006-43-6, 10ppb- 9/10/99 MW	Standard	1.00	7.145	2441	20	0.74 -26
24 102199024	PCUL-99006-43-7, 10ppb- 9/10/99 MW	Standard	1.00	7.121	2112	20	0.64 -36

Comp 030139: Quantitized and printed by LAS on 10/20/99

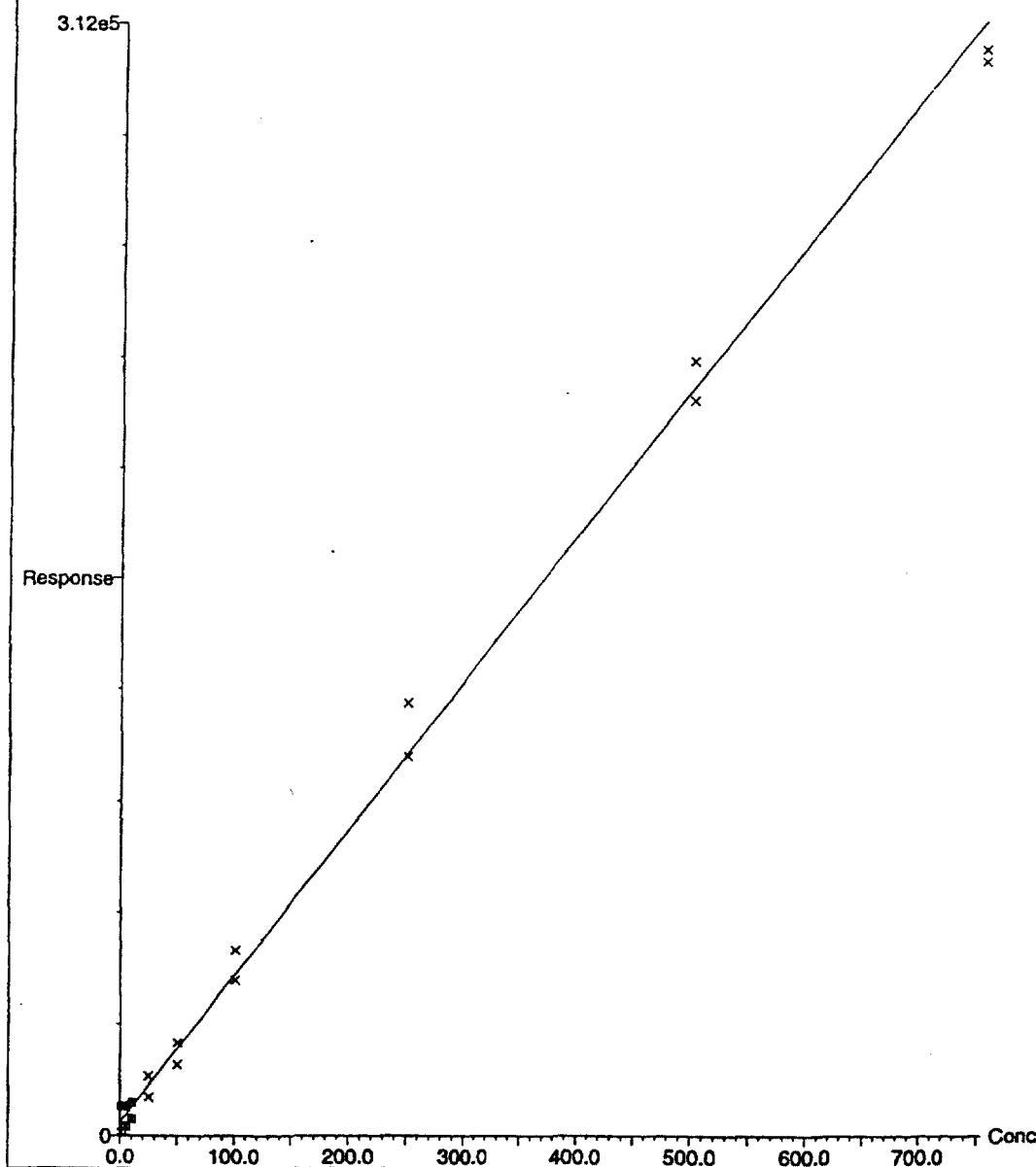
Compound 1 name: POAA (Ch1)
Coefficient of Determination: 0.996938
Calibration curve: $7.35186 \times 10^{-5} x + 10.5814$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



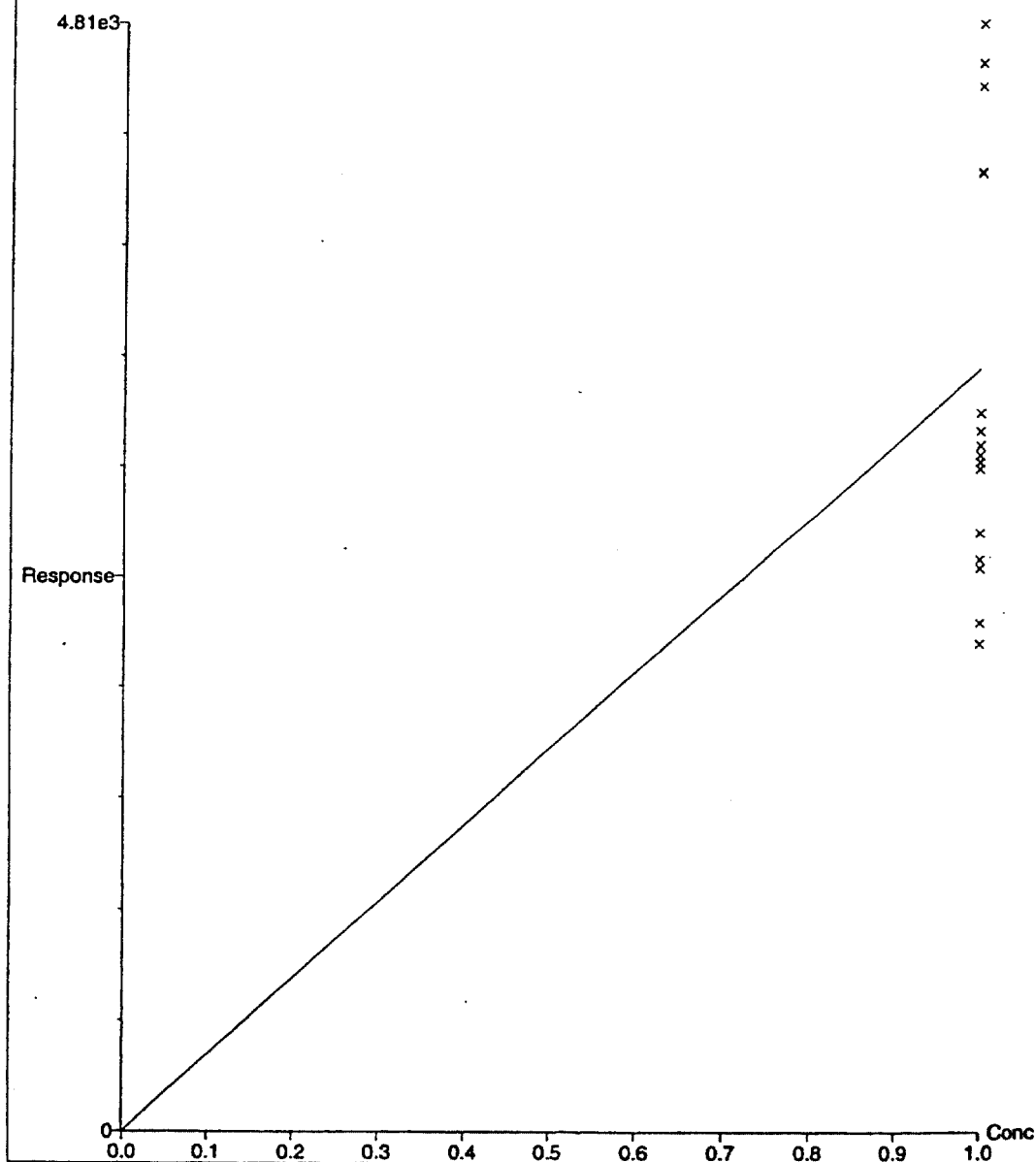
Compound 2 name: PFOS (Ch2)
Coefficient of Determination: 0.999345
Calibration curve: $85.8285 \cdot x + 40.9705$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound 3 name: PFOSA (Ch3)
Coefficient of Determination: 0.995986
Calibration curve: $409.188 \cdot x + 3750.14$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound 4 name: IS (Ch4)
Response Factor: 3310.11
Response type: External Std, Area
Curve type: RF



Method Report

Method File: C:\MSDCHEM\temp021999\prelim\chem0011021999a.mdb
 Last modified: Fri Oct 29 09:31:35 1999

Compound: 1: PCNA (GL)

Printed: Fri Oct 29 07:38:25 1999

Compound Name: PCNA (GL)
 Internal Ref.: [None]
 Quantify Trace: TIC
 Conc. of Standards: Conc. A
 User NF Value: 1.00000, Disabled
 User Peak Factor: 1.00000
 Integrate Window Factor: 2.000
 Chromatogram Mass Window: 1.00000

Retention Time (min): 7.152, Enabled
 Relative Retention Time: 0.000, Disabled
 RT Reference: [None]
 Time Window (min): 0.350

Peak Selection: nearest

Response Type: External
 Response: Area

Polynomial Type: Linear
 Point of Origin: Baseline
 Fit Method: Least Squares
 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
 Noise baseline maximum: 5.00

Draw vertical threshold: 90.00
 Detect shoulders: Disabled
 Shoulder threshold: 20.00

1.00, Disabled
0.00, Disabled
2.00, Disabled
0.00, Disabled

Relative height:
Relative width:
Relative area:
Absolute area:

Method Report
 Method file: C:\MSDCHEM\comp20199.gro\MethodM1020999.mdb
 Last modified: Fri Oct 29 06:31:25 1999
 Compound: 2: PPOS (C32)
 Printed: Fri Oct 29 07:38:25 1999

Compound Name: PPOS (C32)
 Internal Ref.: [None]
 Quantity Traced: TIC
 Conc. of Standards: Conc. N
 User RF Value: 1.00000, Disabled
 User Peak Factor: 1.00000
 Integrate Window Factor: 2.000
 Chromatogram Mass Window: 1.00000e

Retention Time (min): 7.335, Enabled
 Relative Retention Time: 0.000, Disabled
 Peak Width (min): 0.000, Disabled
 Time Window (min): 0.334

Peak Selection: Nearest

Response Type: External
 Response: Area

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

Units:

Peak-to-peak amplitude: 88

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

Join valley threshold: 30.00
 Reduce peak tailing: 200.00
 Make baseline minimum: 3.00

Draw vertical threshold: 90.00
 Draw horizontal threshold: Enabled
 Shoulder threshold: 30.00

2.50, Disabled
0.00, Disabled
2.00, Disabled
0.00, Disabled

Relative height:
Absolute height:
Relative area:
Absolute area:

Method Report

Method file: C:\MSDCHEM\comp20199.meth
 Last modified: Fri Oct 29 06:55:15 1999

Compound: 3: PFOEA (CH3)

Printed: Fri Oct 29 07:38:25 1999

Compound Name: PFOEA (CH3)
 Internal Ref.: (None)
 Conc. of Standard: Conc. C

User RF Value: 1.00000, Disabled
 User Peak Factor: 1.00000
 Concentration factor: 1.00000
 Chromatogram mass window: 1.00000

Retention Time (min): 9.051, Enabled
 Retention Time (min): 9.051, Disabled
 RT Reference: (None)
 Time Window (min): 0.245

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: 147

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

3-sigma valley threshold: 30.00
 Reduce peak tailing: 20.00
 Raise baseline maximum: 5.00

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

Relative height:
Relative height:
Relative area:
Absolute area:

1.50, Disabled
0.50, Disabled
2.50, Disabled
0.50, Disabled

Method Report

Page 4

Method file: C:\MASSL\YK\coups020199.pro\MethDa\102899a.mdb
Last modified: Fri Oct 29 06:53:35 1999

Compound: 4: 15 (Ch4)

Printed: Fri Oct 29 07:38:23 1999

Compound Name: 15 (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: 1.000
Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.199, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: (None)
Time Window (mins): 0.250

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: 100.00
Raise baseline maximum: 5.00

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

Relative height: 1.00, Disabled
Absolute height: 0.00, Disabled
Relative area: 2.00, Disabled
Absolute area: 0.00, Disabled

Quattro II, #6414E

DATE: 10-28-99

ANALYST: jas

STUDY NUMBER: TOX145 MATRIX: Tween samples DESCRIPTION: dose confirmation

FACT Method:

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

Mobile Phase: (A) 2.0 mM $\text{NH}_4\text{CH}_3\text{CO}_2$ (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 μ m

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 μ L/min. Back pressure: 65 bar

HPLC solvent gradient: 10min-10-95

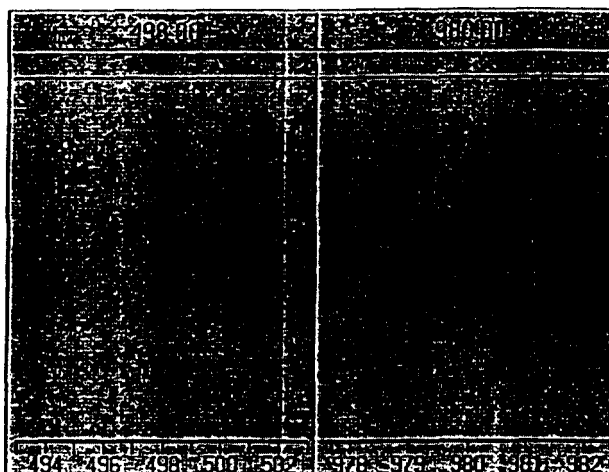
Time (min.)	%B
1) 0.00	10.0
2) 1.00	10.0
3) 5.50	95.0
4) 7.50	95.0
5) 8.00	10.0

Tuning Method Report

Page 1

Method: C:\MASSLYNX\SOUP020199.PRO\ACQUDE\PFOS3

Printed: Thu Oct 28 15:21:34 1999



MS

MS

SOURCE (ESP-)	Set	Rdbk	MS1	Set	Rdbk
Capillary	1.60	1.59	LM Resolution	14.0	
Cone	60	-60	HM Resolution	14.0	
Extractor	5	-7	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	249	Multiplier	650	

Pressures	Rdbk	MS2	Set	Rdbk
Analyser Vacuum	6.1e-5	LM Resolution	14.0	
		HM Resolution	14.0	
		Collision	60	28
Gas Cell	4.1e-3	Ion Energy	2.0	
		Ion Energy Ramp	0.0	
		Lens 8	40	38
		Lens 9	0	28
		Multiplier	650	

Method Report

Page 1

Method File:
Last Modified:C:\MASSLYNX\SOFT\20199.PRO\ACQ\10min-10-95
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

Scanning Method Report

Page 1

Method: C:\MSALYTH\SCW920199.PRO\ACQDS\10MIN-PROD-FOAA-PROD-12
Last Modified: Fri Oct 15 11:35:03 1999
Printed: Thu Oct 20 13:44:10 1999

Solvent Delay (mins) : 0.00
Function : 1 MMN of 1 Mass Pair (MSP-)
Inter Channel Delay (Secs) : 0.01
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 (MSP-)
Inter Channel Delay (Secs) : 0.01
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 (MSP-)
Inter Channel Delay (Secs) : 0.01
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 (MSP-)
Inter Channel Delay (Secs) : 0.01
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

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

Masslynx - Sample List

Sample List: C:\MASSLYNX\sup020199.pr\SampleDB\102899a.SPL

Printed: Thu Oct 28 13:44:09 1999

	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	--	--	--	--

11

FACT: DATA REVIEW SUMMARY FORM

Date: 11/08/99
 Data Analyst: IAS
 FACT Study #: Tox 145
 Matrix/Timepoints: Reflex - control; dose day 29 - est 11/8/99
 Sample List: 110599a
 Set-up Analysis Date/Analyst: IAS 11/25/99
 Instrument: Amelia 062498

Data Review Sign-Off

Analyst Review
 Name / Date: IAS 11/08/99

Technical Review
 Name / Date: MEE 11/8/99 / 11/8/99

Quality Assurance Review
 Name / Date: _____

Data Entry: DATA Reported GML 11/8/99

Comments / Justifications:

Analyte: POAA (CH1) ①
 Acceptable Linear Range: 61.30ppb → 1227ppb
 LOQ: 61.30ppb Extraction Blank: less than LOQ
 R²: 0.989198 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

① The low level points (6.13ppb, 12.20ppb, 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

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

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

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

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

FACT: DATA REVIEW SUMMARY FORM

Date: 11/08/99
 Data Analyst: JAS
 FACT Study #: Tox145
 Matrix/Timepoints: Rat Liver - control / dose day 29 - end 11/1/99
 Sample List: 110599a
 Set-up Analysis
 Date/Analyst: JAS 11/05/99
 Instrument: Andia 062498

Data Review Sign-Off

Analyst Review
 Name / Date: JAS 11/08/99

Technical Review
 Name / Date: Refer to page 1 IAC 11/09/99

Quality Assurance Review
 Name / Date: _____

Data Entry: refer to page 1 IAC 11/09/99

Comments / Justifications: _____

Analyte: PFOSA (CH₄)
 Acceptable Linear Range: 30.70 ppb → 1027 ppb
 LOQ: 30.70 ppb Extraction Blank: less than LOQ
 R²: 0.989845 Matrix Blank: less than LOQ
 Calibration Check: OK - Range from 15% → 26%
 MS/MSD: N/A 1466, 1376 (High)
 Data Status: OK - GUL 11/08/99 Data Reported 60346 OK

① Low level points 6.3 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: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: JAS 11/08/99
 R²: _____ Matrix Blank: _____
 Calibration Check: _____
 MS/MSD: _____
 Data Status: _____

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

ORE GUL 11/08/99

GUL 11/08/99

Printed: Mon May 08 09:10:10 1990

100% (100%)

[illegible]

Amelia 02288; Quantitated and reviewed by LAG on 11/8/99

Amelia 00400: Querrelated and reviewed by lsc on 11/6/99

Quantity Compound Summary Report

TOK145-ret Liver. Day79 dose and control - act. 11/4/99 Rm

Sample List: C:\MSA2\PRO\mali\0103486 PRO\Sample.mali\110399a

Last modified: Mon Nov 08 08:34:18 1999

Method: F:\mali\Backup\comp\mali\ret\pre\MSA2\010399a

Last created: Mon Nov 08 08:30:37 1999

Job Code:

Printed: Mon Nov 08 08:18:10 1999

Compound 2: PROD (O2)

# Name	Sample List	Type	Std Conc	RT	Area	Conc.	Flag	Nov	Mod	Comment
1 11039901	NEOM Blank			7.236	77	0.00	hd			
2 11039902	NEOM Blank			7.237	28	0.00	hd			
3 11039903	NEOM Blank			7.242	35	0.00	hd			
4 11039904	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.214	93	0.00	hd			
5 11039905	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	6.16	7.213	149	0.00	hd			-100
6 11039906	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	12.30	7.183	145	0.00	hd			-100
7 11039907	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	12.30	7.183	145	0.00	hd			-100
8 11039908	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	30.70	7.185	508	25.94	hd			-15
9 11039909	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	61.40	7.184	846	61.71	hd			-1
10 11039910	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	123.80	7.183	1339	124.39	hd			9
11 11039911	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	617.00	7.188	4205	639.59	hd			14
12 11039912	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	617.00	7.188	4205	639.59	hd			14
13 11039913	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	921.00	7.184	8133	825.08	hd			-10
14 11039914	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	1228.08	7.163	9947	1017.66	hd			-17
15 11039915	NEOM Blank			7.189	32	0.00	hd			
16 11039916	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.186	215	715.93	hd			
17 11039917	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.186	215	715.93	hd			
18 11039918	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.164	7.164	201	0.00	hd		
19 11039919	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.166	196	0.00	hd			
20 11039920	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.165	197	0.00	hd			
21 11039921	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.165	238	221.00	hd			-22
22 11039922	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.181	148	0.00	hd			
23 11039923	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.188	148	0.00	hd			
24 11039924	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.186	145	0.00	hd			
25 11039925	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.182	9453	10112.21	hd			
26 11039926	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.182	18926	11802.79	hd			
27 11039927	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.171	18926	11802.79	hd			
28 11039928	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.167	2676	233.78	hd			-17
29 11039929	NEOM Blank			7.166	72	0.00	hd			
30 11039930	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.167	10765	11889.34	hd			
31 11039931	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.167	10765	11889.34	hd			
32 11039932	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.167	2816	268.48	hd			-13
33 11039933	NEOM Blank			7.172	95	0.00	hd			
34 11039934	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.183	136	0.00	hd			
35 11039935	TOK145-NEL1049-WB1-1, NW- Box 99-211			7.164	281	2.00	hd			-57
36 11039936	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	6.16	7.166	335	5.93	hd			-19
37 11039937	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	12.30	7.166	345	29.87	hd			-3
38 11039938	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	20.70	7.160	879	64.46	hd			6
39 11039939	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	41.40	7.160	1398	127.48	hd			1
40 11039940	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	82.80	7.160	2796	255.96	hd			17
41 11039941	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	165.60	7.163	6459	651.32	hd			6
42 11039942	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	331.20	7.167	8248	864.33	hd			-9
43 11039943	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	662.40	7.165	16512	1677.24	hd			-12
44 11039944	TOK145-NEL1049-WB1-1, NW- Box 99-211	Standard	1228.08	7.162	10512	1077.24	hd			-12

Amile 002100: Quantified and reviewed by Jan on 11/6/99

Qualify Compound Summary Report

TDX15-rat liver, Days dose and control- sat. 11/4/99 new

Sample List: C:\MSSET\TDX15\456899.RMO\Sample\08\110598a

Batch: 110598a

Method: F:\admin\backlog\sepr\amalia-rat\pva\backlog\110598a

Last modified: Mon Nov 08 08:20:27 1999

Job Code:

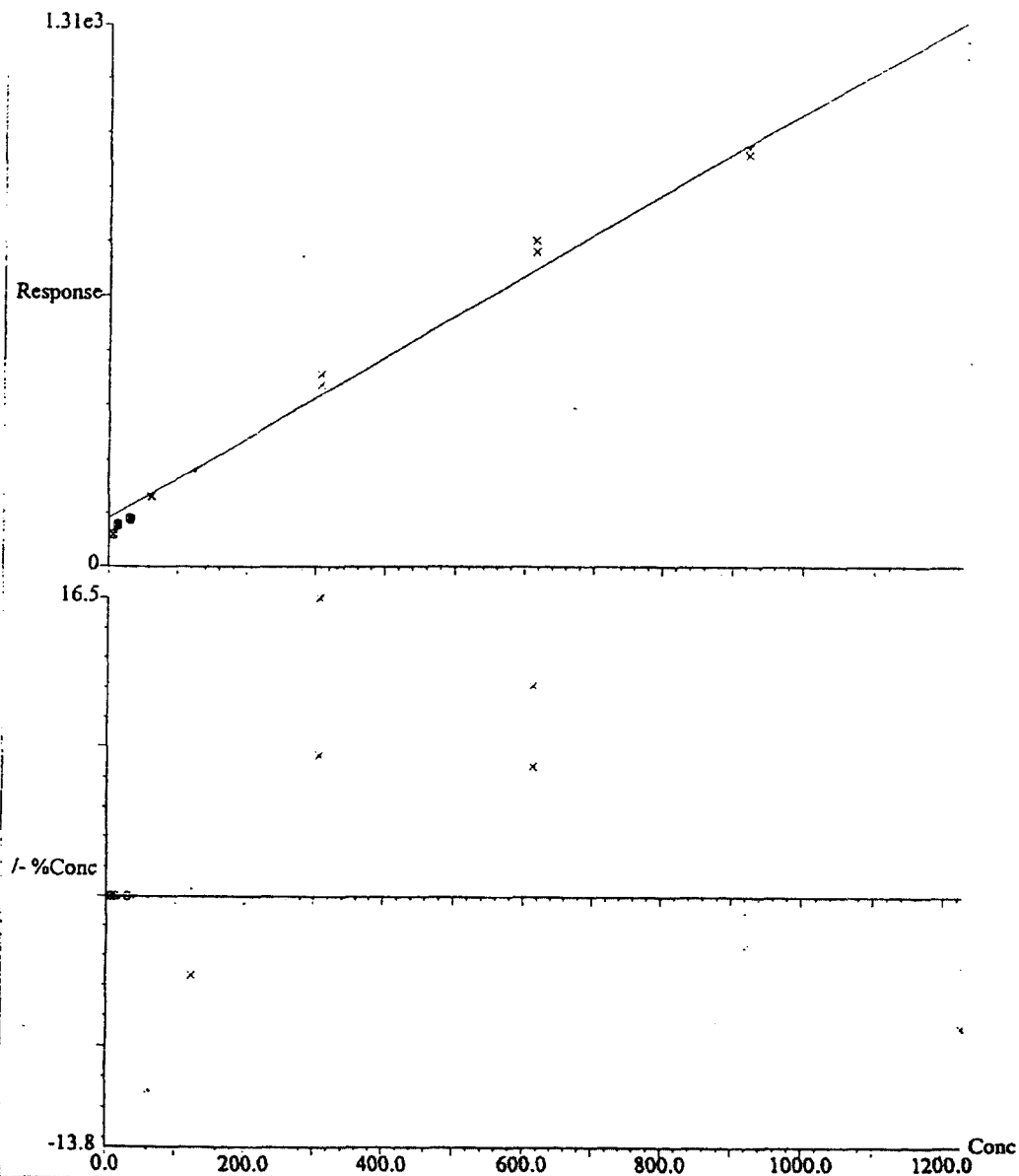
Printed: Mon Nov 08 08:16:10 1999

Compound 4: PFOLX (CM4)

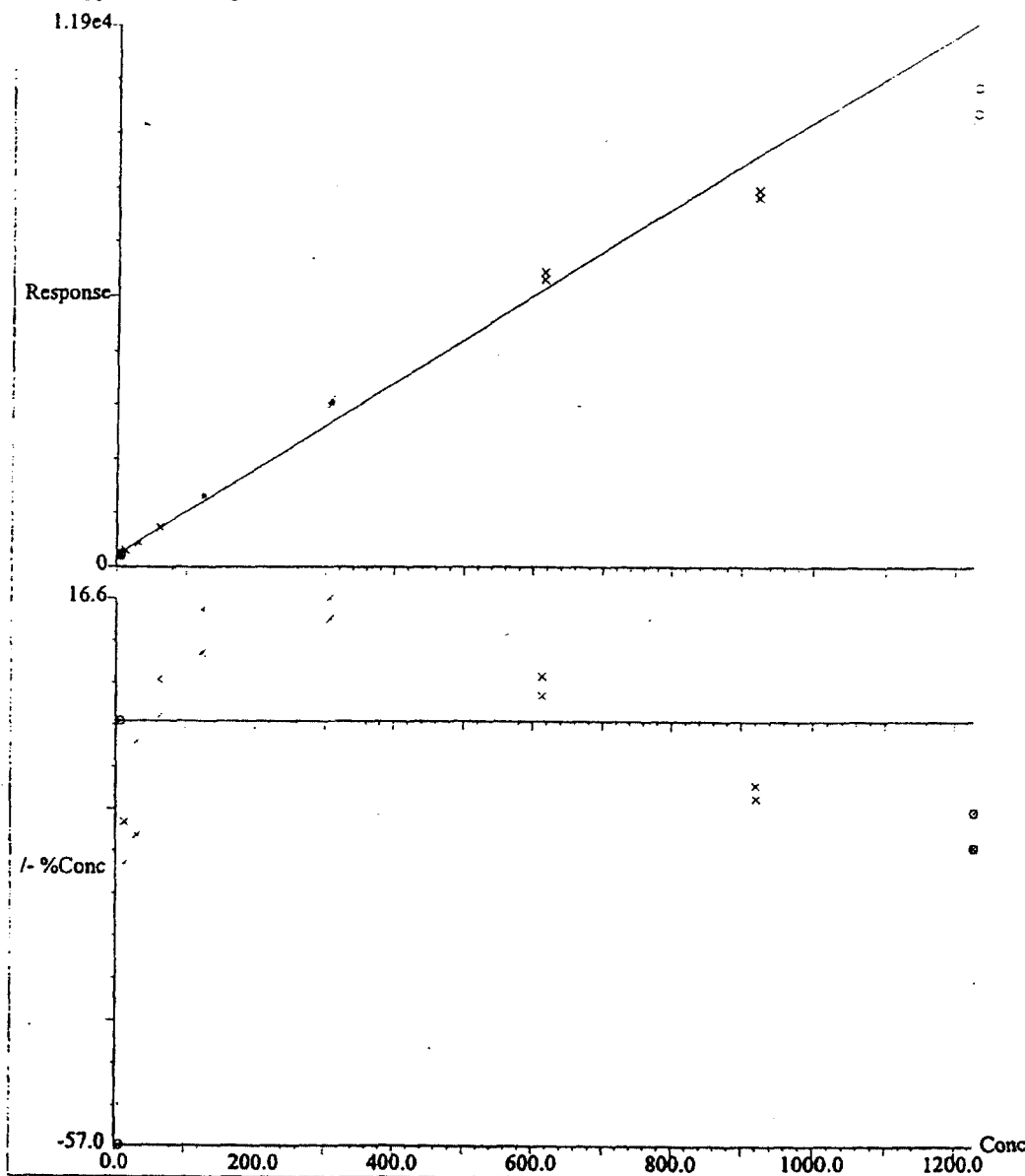
#	Name	Sample/lot Test	Type	Std Conc	RT	Area	Conc	Flag	Over	Real Comment
1	110599001	MON Blank			7.794	321	11.54 M			
2	110599002	MON Blank			7.776	225	7.16 M			
3	110599003	MON Blank			7.724	45	0.00 M			
4	110599004	MON Blank			7.724	154	1.44 M			
5	110599005	TDX15-RAT11049-0811-1, NW- Box 99-211			7.750	196	1.44 M			
6	110599006	TDX15-RAT11049-0811-1, NW- Box 99-211			7.726	281	3.79 M			
7	110599007	TDX15-RAT11049-0811-1, NW- Box 99-211	Standard	6.13	7.742	281	6.67 M			-38
8	110599008	TDX15-RAT11049-0811-1, NW- Box 99-211	Standard	12.30	7.737	273	25.50 M			-17
9	110599009	TDX15-RAT11049-0811-1, NW- Box 99-211	Standard	20.00	7.737	123	1.44 M			
10	110599010	TDX15-RAT11049-0811-1, NW- Box 99-211	Standard	123.00	7.748	212	32.13 M			
11	110599011	TDX15-RAT11049-0811-1, NW- Box 99-211	Standard	307.00	7.757	4347	340.04 M			17
12	110599012	TDX15-RAT11049-0811-1, NW- Box 99-211	Standard	613.00	7.770	12081	682.71 M			13
13	110599013	TDX15-RAT11049-0811-1, NW- Box 99-211	Standard	1227.00	7.733	13566	890.30 M			-3
14	110599014	TDX15-RAT11049-0811-1, NW- Box 99-211	Standard	1227.00	7.778	20740	1110.52 M			-4
15	110599015	MON Blank			7.754	49	0.00 M			
16	110599016	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211			7.750	6202	382.04 M			
17	110599017	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211			7.750	5802	338.88 M			
18	110599018	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211			7.772	176	2.12 M			
19	110599019	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211			7.772	144	0.74 M			
20	110599020	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211			7.774	176	2.12 M			
21	110599021	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211	QC	307.00	7.748	6332	370.74 M			21
22	110599022	MON Blank			7.799	47	0.00 M			
23	110599023	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211			7.778	134	0.18 M			
24	110599024	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211			7.778	169	0.74 M			
25	110599025	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211			7.769	169	0.74 M			
26	110599026	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211			7.774	3532	196.81 M			
27	110599027	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211			7.781	1571	108.57 M			
28	110599028	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211			7.772	4001	228.00 M			
29	110599029	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211	QC	307.00	7.772	2652	146.00 M			-24
30	110599030	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211			7.774	2652	146.00 M			
31	110599031	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211			7.777	3573	315.13 M			
32	110599032	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211	QC	307.00	7.753	6243	354.01 M			15
33	110599033	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211			7.753	124	0.18 M			
34	110599034	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211			7.781	138	0.41 M			
35	110599035	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211			7.780	141	0.57 M			
36	110599036	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211	Standard	6.13	7.769	181	2.87 M			-53
37	110599037	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211	Standard	12.30	7.757	181	9.37 M			-24
38	110599038	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211	Standard	20.00	7.757	529	2.37 M			-3
39	110599039	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211	Standard	41.30	7.770	1172	40.21 M			
40	110599040	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211	Standard	123.00	7.754	2299	125.54 M			2
41	110599041	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211	Standard	307.00	7.772	3742	324.98 M			6
42	110599042	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211	Standard	613.00	7.767	1184	463.16 M			6
43	110599043	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211	Standard	1227.00	7.767	1184	463.16 M			6
44	110599044	TDX15-RATL 9803240-30ppm-MD, 11/4/99 Box 99-211	Standard	1227.00	7.750	20714	1132.13 M			-3

Amelin 062488: Quantitated and reviewed by IAC on 11/9/99

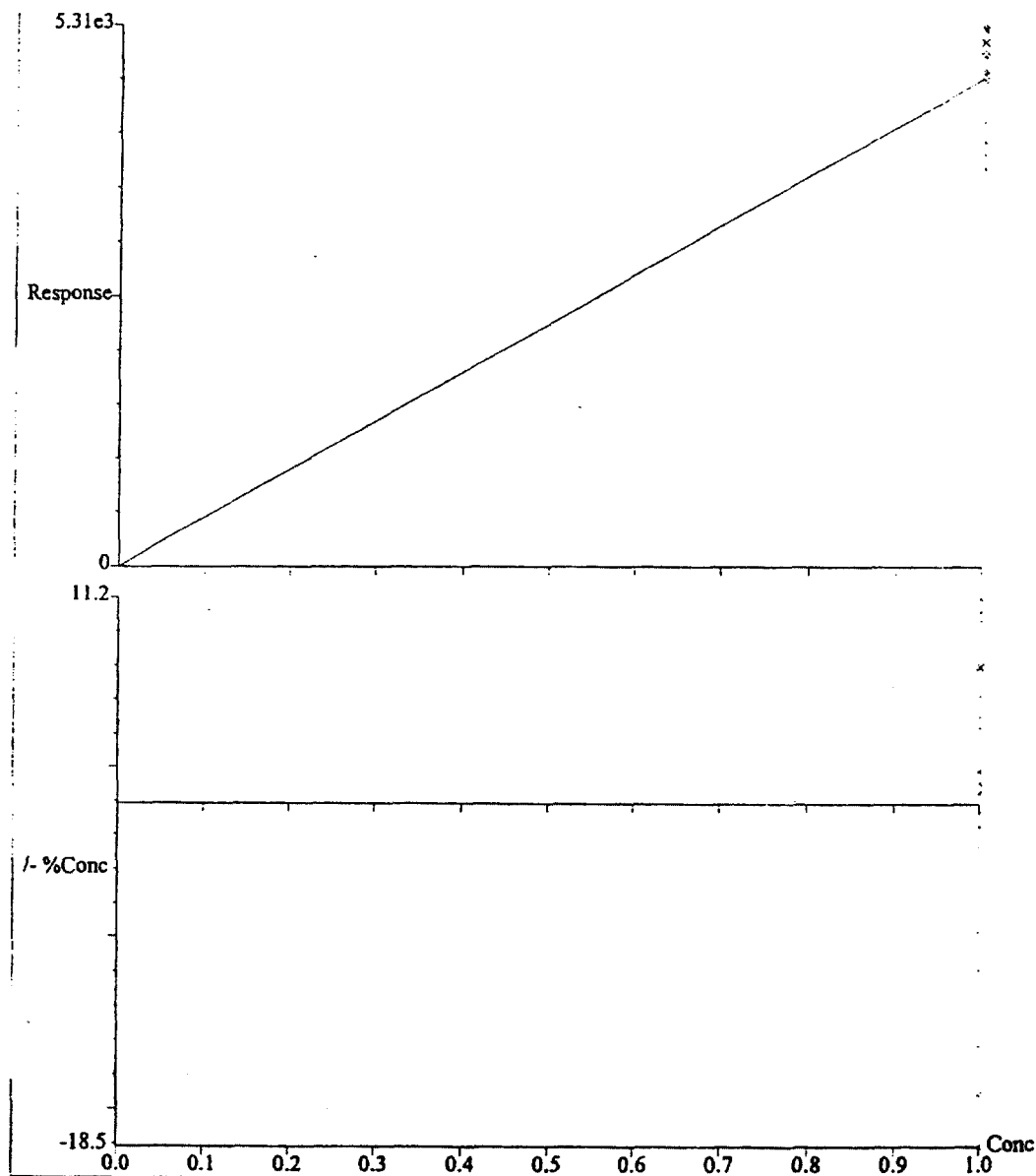
Compound 1 name: POAA (Chl)
Coefficient of Determination: 0.989198
Calibration curve: $0.973462 * x + 119.913$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound 2 name: PFOS (Ch2)
Coefficient of Determination: 0.982749
Calibration curve: $9.51640 \cdot x + 260.801$
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



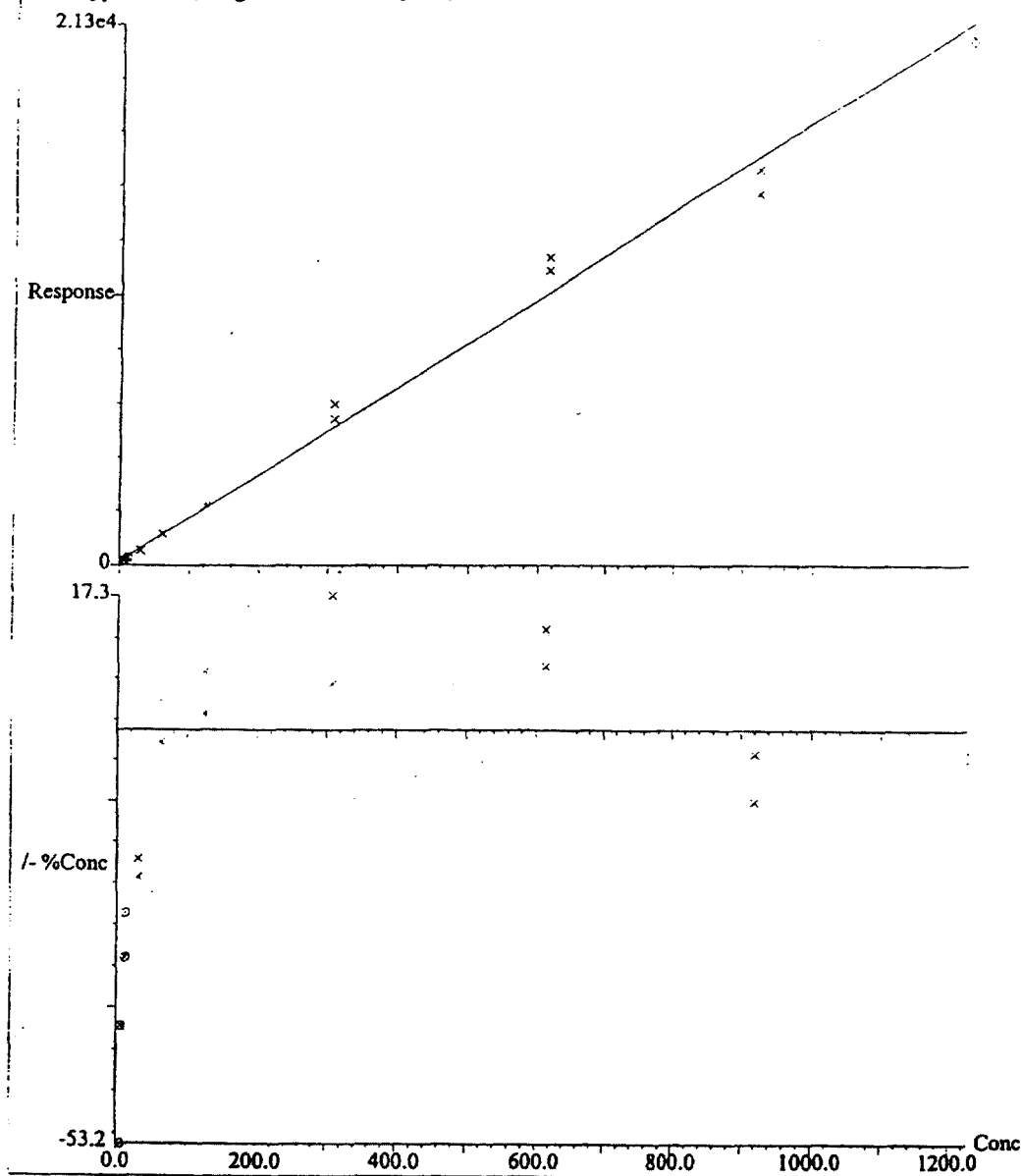
Compound 4 name: PFOSA (Ch4)

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



Method Report

Page 1

Method file: F:\amelia backup sep99\amelia-rats.pro\MethDB\110399a.mdb
Last modified: Mon Nov 08 08:20:27 1999C

Compound: 1: PGAA (Ch1)

Printed: Mon Nov 08 08:36:38 1999

Compound Name: PGAA (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: 1.000000
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: None
Noise

Peak-to-peak amplitude: 530
Smooth

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

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

Draw vertical threshold: 50.00
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

Join valley threshold:	30.00
Reduce peak falling:	200.00
Auto baseline minimum:	5.00
Peak separation	
Draw vertical threshold:	90.00
Detect shoulders:	Disabled
Shoulder threshold:	10.00
Asymptote threshold	
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: F:\msd\bin\backup\msd\msd\11-0399-mdb
 Last modified: Mon Nov 08 08:20:27 1999
 Compound: 2: PTOS (CH2)
 Printed: Mon Nov 08 08:56:38 1999

Compound Name: PTOS (CH2)
 Internal Ref.: [None]
 Quantity Trace: TIC
 Conc. of Standards: Conc. B

User RF Value: 1.000000, Disabled
 User Peak Factor: 1.000000
 Integration Factor: 2.000000
 Chromatogram Mass Window: 1.000000e

Peak Location
 Retention Time (mins): 1.149, Enabled
 Relative Retention Time: 0.000, Disabled
 RT Reference: [None]
 Time Window (mins): 0.129

Peak Selection: Nearest
 Response: Nearest

Response Type: External
 Response: Areas

Calibration Curves
 Polynomial Type: Linear
 Point at Origin: Enabled
 TIC Weighting: 1/X
 Data Transformation: None
 Concentration

Units: Molar

Peak-to-peak amplitude: 56

Smoothing: 56

Smoothing: Enabled

Window size (across): 2

Number of smooths: 1

Smoothing method: Baseline

Method Report

Method file: F:\analysis backup\analysis-rts.pro\Method\110599a.mtd
 Last modified: Mon Nov 08 08:20:27 1999

Compound: 3: Surr (Ch3)

Printed: Mon Nov 08 08:35:38 1999

Compound Name: Surr (Ch3)
 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: 1.00000e
 Peak Location

Retention Time (min): 7.033, Enabled
 Relative Retention Time: 0.000, Disabled
 RT Reference: [None]
 Time Window (min): 0.310

Peak Selection: Nearest
 Response

Response Type: External
 Response: Areas

Calibration Curves

Polynomial Type: Linear
 Point of Origin: Exclude
 Fit Selection: 1
 Axis Transformation: None

Units:

Units: None

Peak-to-peak amplitude: 90

Smoothing

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

Baselines

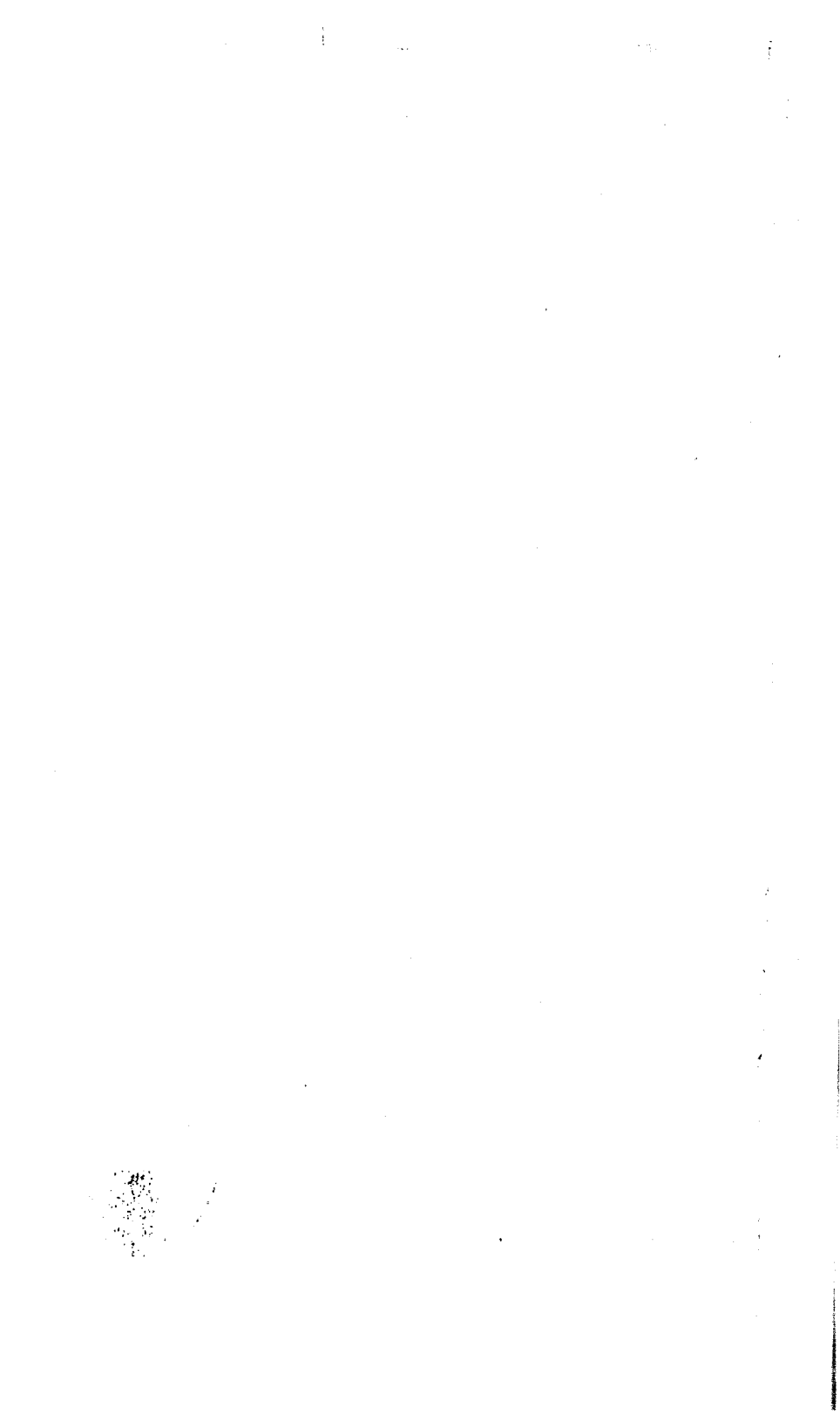
Join valley threshold: 30.00
 Reduce peak tailing: 200.00
 Raise baseline maximum: 5.00

Peak Separation

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

Response Thresholds

Relative Height: 1.50, Disabled
 Absolute Height: 0.00, Disabled
 Relative Area: 2.00, Disabled
 Absolute Area: 0.00, Disabled



Join valley threshold:	30.00
Reduce peak calling:	200.00
Reduce peak minimum:	1.00
Peak separation	
Draw vertical threshold:	50.00
Detect anomalies:	Disabled
Smooth threshold:	20.00
Response threshold	
Relative height:	1.30, Disabled
Absolute height:	0.00, Disabled
Relative area:	0.00, Disabled
Absolute area:	0.00, Disabled

Method Report

Method file: F:\small6 backup\supply\small6-rs6a.pro\MethodM\110399a.mtd
Last modified: Mon Nov 08 08:20:27 1998

Compound: 4: PTDA (CN4)
Printed: Mon Nov 08 08:36:38 1999

Compound Name: PTDA (CN4)
Internal Ref.: (None)
Quantity Traced: TIC
Conc. of Standards: Conc. C

User Rf Value: 1.000000, Disabled
User Peak Factor: 1.000000
Integrate Window Factor: 2.000
Chromatogram Area Window: 1.000000e

Peak Location
Retention Time (min): 7.770, Enabled
Relative Retention Time: 0.000, Disabled
RT Reference: (None)
Time Window (min): 0.286

Peak Selection: Nearest
Response: (None)

Response Type: External
Response: Areas

Calibration Curves
Polynomial Type: Linear
Point of Origin: Disabled
TIC Weighting: 1/X
Axis Transformation: None
Concentration: (None)

Units: (None)
Noise: (None)

Peak-to-peak amplitude: 82
Slope: (None)

Smoothing: Enabled
Window size (scans): 2
Range of samples: 1
Smoothing method: Baseline

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? ☒ 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

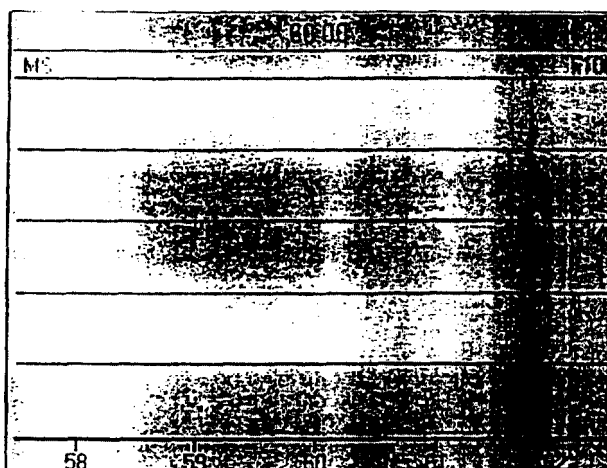
Time (min.)	%B
1) <u>0.00</u>	<u>10.0</u>
2) <u>1.00</u>	<u>10.0</u>
3) <u>5.50</u>	<u>95.0</u>
4) <u>7.50</u>	<u>95.0</u>
5) <u>8.00</u>	<u>10.0</u>

Tuning Method Report

Page 1

Method: C:\MASSLYNX\AMELIA062498.PRO\ACQUDE\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
Analysar Vacuum	1.2e-4	LM Resolution	14.0	
		HM Resolution	14.0	
		Collision	10	12
Gas Cell	4.8e-3	Ion Energy	2.0	
		Ion Energy Ramp	0.0	
		Lens 8	40	40
		Lens 9	0	10
		Multiplier	650	

Scanning Method Report

Page 1

Method: C:\HASL\TQC\MELIAQ42488.PRG\ACQSR\IONCH-PP08-POAA-18-PP08A
Last Modified: Mon Oct 11 07:22:17 1999
Printed: Fri Nov 03 09:24:14 1999

Solvent Delay (Mins) : 0.00

Function : 1 MW 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)
1 413.75 169.00 0.30 30 15

Function : 2 MW 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)
1 499.30 99.00 0.30 45 60

Function : 3 MW 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)
1 417.11 80.00 0.30 40 60

Function : 4 MW 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)
1 499.71 78.00 0.40 45 60

Method Report

Page 1

Method File:
Last Modified:

C:\MASSLYNX\AMELIA062498.PRO\ACQUDE\10min-10-95
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

Masslynx - Sample List

Page 1

Sample List: C:\MASSLYNX\amalia062498.PRO\SampleDB\110599a.SPL
Printed: Fri Nov 05 09:25:34 1999

Page Position 1

	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

Masslynx - Sample List

Page 2

Sample List: C:\MASSLYNX\data\062498.PRO\SampleDB\110599a.SPL

Page Position: (2, 1)

Printed: Fri Nov 05 09:25:34 1999

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	---	---	---	---	---	---	---

FACT: DATA REVIEW SUMMARY FORM

Date: 11/11/99
 Data Analyst: JAS
 FACT Study #: Tox 145
 Matrix/Timepoints: Pet Liver, Day 29 dose deletions - cat 11/11/99
 Sample List: 11099A
 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 / kln 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 → 1227 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. kln Nov-15-99

LAC 11/15/99

FACT: DATA REVIEW SUMMARY FORM

Date: 11/11/99
 Data Analyst: JAS
 FACT Study #: Tox145
 Matrix/Timepoints: Retliner, Day 29 dose dilutions - out 11/11/99
 Sample List: 111099a
 Set-up Analysis Date/Analyst: JAS 11/10/99
 Instrument: Samp 023199

Data Review Sign-Off

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

Technical Review Name / Date: Refer to page 1 of 2 UC 11/15/99

Quality Assurance Review Name / Date: _____

Data Entry: LAC 11/15/99

Comments / Justifications: _____

Analyte: Surr (Func 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 11/11/99

Analyte: _____
 Acceptable Linear Range: _____
 LOQ: _____ Extracted Blank: _____
 R²: _____ Matrix Blank: _____
 Calibration Checks: _____
 MS/MSD: JAS 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. - JAS 11/11/99

UC 11/15/99

Quantity Compound Primary Report

Tufts-Treat Liver, day 39 data-dilutions- enr. 11/4/99 Nov

Sample List: C:\MSDCHEM\Temp26189.prn(c:\msdchem\111099a

Last modified: Thu Nov 11 07:01:30 1999

Method: C:\MSDCHEM\Temp26189.prn(c:\msdchem\111099a

Last modified: Thu Nov 11 07:01:22 1999

Job Code:

Printed: Thu Nov 11 07:26:25 1999

Compound 1: POAA (Purity 1)

S Name	Sample/Inj Time	Type	Std Conc	RT	Area	Area Flag	Conc. Ubr
1 111099001	Mock Blank						
2 111099002	Mock Blank						
3 111099003	Mock Blank						
4 111099004	TOX15-MBL1049-MBL1-1, Nov	Standard	7.125	7.125	93 Mb	0.00	0.00
5 111099005	TOX15-MBL1049-MBL1-2, Nov	Standard	6.33	7.166	197 Mb	0.00	5
6 111099006	TOX15-MBL1049-MBL1-3, Nov	Standard	12.33	7.093	197 Mb	0.00	100
7 111099007	TOX15-MBL1049-MBL1-4, Nov	Standard	16.70	7.083	197 Mb	0.00	100
8 111099008	TOX15-MBL1049-MBL1-5, Nov	Standard	16.70	7.083	197 Mb	0.00	100
9 111099009	TOX15-MBL1049-MBL1-6, Nov	Standard	16.70	7.083	197 Mb	0.00	100
10 111099010	TOX15-MBL1049-MBL1-7, Nov	Standard	16.70	7.083	197 Mb	0.00	100
11 111099011	TOX15-MBL1049-MBL1-8, Nov	Standard	16.70	7.083	197 Mb	0.00	100
12 111099012	TOX15-MBL1049-MBL1-9, Nov	Standard	16.70	7.083	197 Mb	0.00	100
13 111099013	TOX15-MBL1049-MBL1-10, Nov	Standard	16.70	7.083	197 Mb	0.00	100
14 111099014	TOX15-MBL1049-MBL1-11, Nov	Standard	16.70	7.083	197 Mb	0.00	100
15 111099015	Mock Blank						
16 111099016	TOX15-MBL1049-MBL1-12, Nov	Standard	16.70	7.083	197 Mb	0.00	100
17 111099017	TOX15-MBL1049-MBL1-13, Nov	Standard	16.70	7.083	197 Mb	0.00	100
18 111099018	TOX15-MBL1049-MBL1-14, Nov	Standard	16.70	7.083	197 Mb	0.00	100
19 111099019	TOX15-MBL1049-MBL1-15, Nov	Standard	16.70	7.083	197 Mb	0.00	100
20 111099020	TOX15-MBL1049-MBL1-16, Nov	Standard	16.70	7.083	197 Mb	0.00	100
21 111099021	TOX15-MBL1049-MBL1-17, Nov	Standard	16.70	7.083	197 Mb	0.00	100
22 111099022	Mock Blank						
23 111099023	TOX15-MBL1049-MBL1-18, Nov	Standard	16.70	7.083	197 Mb	0.00	100
24 111099024	TOX15-MBL1049-MBL1-19, Nov	Standard	16.70	7.083	197 Mb	0.00	100
25 111099025	TOX15-MBL1049-MBL1-20, Nov	Standard	16.70	7.083	197 Mb	0.00	100
26 111099026	TOX15-MBL1049-MBL1-21, Nov	Standard	16.70	7.083	197 Mb	0.00	100
27 111099027	TOX15-MBL1049-MBL1-22, Nov	Standard	16.70	7.083	197 Mb	0.00	100
28 111099028	TOX15-MBL1049-MBL1-23, Nov	Standard	16.70	7.083	197 Mb	0.00	100
29 111099029	TOX15-MBL1049-MBL1-24, Nov	Standard	16.70	7.083	197 Mb	0.00	100
30 111099030	TOX15-MBL1049-MBL1-25, Nov	Standard	16.70	7.083	197 Mb	0.00	100
31 111099031	TOX15-MBL1049-MBL1-26, Nov	Standard	16.70	7.083	197 Mb	0.00	100
32 111099032	TOX15-MBL1049-MBL1-27, Nov	Standard	16.70	7.083	197 Mb	0.00	100
33 111099033	TOX15-MBL1049-MBL1-28, Nov	Standard	16.70	7.083	197 Mb	0.00	100

QC

307.00

274.09

-11

GROUP 070199: Quantitated and printed by IAS on 11/11/99

Quantity Compound Summary Report
 TOX145-see liver, day 29 dose dilution- cont. 11/4/99 MW
 Sample List: C:\MSDEV\TOX\comp29189.gpr\samplelist111099a
 Last modified: Thu Nov 11 07:10:24 1999
 Method: C:\MSDEV\TOX\comp29189.gpr\method111099a
 Last modified: Thu Nov 11 07:10:23 1999
 Job Code:
 Printed: Thu Nov 11 07:26:35 1999

Compound 3: PFOS (Peak 3)

S Name	Sample List	Type	Std Conc	RT	Area	Flags	Chen. Value
1 111099001	MeOH Blank			7.264	16		0.00
2 111099002	MeOH Blank			7.264	16		0.00
3 111099003	MeOH Blank			7.269	16		0.00
4 111099004	TOX145-RBL1049-WB13-2, MW	Standard	6.14	7.263	113		0.00
5 111099005	TOX145-RBL1049-WB1313-2, MW	Standard	6.14	7.264	213		1.84
6 111099006	TOX145-RBL1049-6ppb-2, MW	Standard	12.10	7.263	213		1.84
7 111099007	TOX145-RBL1049-10ppb-2, MW	Standard	30.76	7.263	313		32.40
8 111099008	TOX145-RBL1049-60ppb-2, MW	Standard	61.40	7.264	510		32.40
9 111099009	TOX145-RBL1049-120ppb-2, MW	Standard	121.00	7.269	510		63.16
10 111099010	TOX145-RBL1049-100ppb-2, MW	Standard	121.00	7.269	510		117.75
11 111099011	TOX145-RBL1049-100ppb-2, MW	Standard	314.00	7.263	713		51.40
12 111099012	TOX145-RBL1049-100ppb-2, MW	Standard	314.00	7.263	713		51.40
13 111099013	TOX145-RBL1049-100ppb-2, MW	Standard	931.00	7.269	1129		994.02
14 111099014	TOX145-RBL1049-120ppb-2, MW	Standard	1238.00	7.269	1129		1180.39
15 111099015	MeOH Blank			7.264	27		0.00
16 111099016	TOX145-RT, 9803259/M/Day 29/Samp/Rep-D106, 11/4/99			7.264	27		0.00
17 111099017	TOX145-RT, 9803259/M/Day 29/Samp/Rep-D106, 11/4/99			7.264	27		0.00
18 111099018	TOX145-RT, 9803259/M/Day 29/Samp/Rep-D106, 11/4/99			7.269	27		0.00
19 111099019	TOX145-RT, 9803259/M/Day 29/Samp/Rep-D106, 11/4/99			7.263	269		321.25
20 111099020	TOX145-RT, 9803259/M/Day 29/Samp/Rep-D106, 11/4/99			7.263	317		344.40
21 111099021	TOX145-RBL1049-100ppb-2, MW	QC	302.00	7.267	302		235.12
22 111099022	TOX145-RBL1049-100ppb-2, MW			7.264	302		235.12
23 111099023	TOX145-RBL1049-WB13-2, MW			7.264	13		0.00
24 111099024	TOX145-RBL1049-WB1313-2, MW			7.264	103		0.00
25 111099025	TOX145-RBL1049-6ppb-2, MW	Standard	6.14	7.245	103		0.00
26 111099026	TOX145-RBL1049-10ppb-2, MW	Standard	12.10	7.245	213		1.84
27 111099027	TOX145-RBL1049-10ppb-2, MW	Standard	30.76	7.244	313		32.40
28 111099028	TOX145-RBL1049-60ppb-2, MW	Standard	61.40	7.244	549		31.93
29 111099029	TOX145-RBL1049-120ppb-2, MW	Standard	121.00	7.251	833		97.48
30 111099030	TOX145-RBL1049-120ppb-2, MW	Standard	121.00	7.249	833		104.17
31 111099031	TOX145-RBL1049-100ppb-2, MW	Standard	314.00	7.249	1371		51.40
32 111099032	TOX145-RBL1049-100ppb-2, MW	Standard	314.00	7.251	1371		51.40
33 111099033	TOX145-RBL1049-100ppb-2, MW	Standard	931.00	7.251	4373		995.95
34 111099034	TOX145-RBL1049-100ppb-2, MW	Standard	931.00	7.251	4373		995.95
35 111099035	TOX145-RBL1049-120ppb-2, MW	Standard	1238.00	7.254	1376		1132.17

COMP 02019: Quantities and Printed by L&S on 11/11/99

Quantity Compound Summary Report

TOL145-RT Liver, day 29 dose 411000000 - cont. 11/4/99 MW

Sample List: C:\MSD\TOL145\day29\day29.mpl\comp\all11099a

Last modified: 11/04/99 13:59

Sample Name: C:\MSD\TOL145\day29\day29.mpl\comp\all11099a

Last modified: 11/04/99 13:59

Job Order: 11/04/99 13:59

Printed: Thu Nov 11 07:26:13 1999

Compound 3: PFODA (Peak 3)

Q	Name	Sample Name	Type	Std Comp	RT	Area	Flags	Comp	MW
1	11109001	MeOH Blank			7.848	276	MC	5.43	
2	11109002	MeOH Blank			7.857	107	MC	0.29	
3	11109003	MeOH Blank			7.858	55	MC	0.00	
4	11109004	MeOH Blank			7.859	322	MC	0.26	
5	11109005	TOL145-MRL11049-0011-2, MW	Standard	6.13	7.853	284	MC	5.85	-5
6	11109006	TOL145-MRL11049-0011-2, MW	Standard	12.30	7.837	1432	MC	11.42	1
7	11109007	TOL145-MRL11049-0011-2, MW	Standard	12.30	7.837	1432	MC	11.42	1
8	11109008	TOL145-MRL11049-0011-2, MW	Standard	61.30	7.811	2997	MC	62.98	3
9	11109009	TOL145-MRL11049-0011-2, MW	Standard	61.30	7.811	2997	MC	62.98	3
10	11109010	TOL145-MRL11049-0011-2, MW	Standard	123.00	7.825	3852	MC	118.26	-4
11	11109011	TOL145-MRL11049-0011-2, MW	Standard	123.00	7.825	3852	MC	118.26	-4
12	11109012	TOL145-MRL11049-0011-2, MW	Standard	307.00	7.835	10248	MC	321.02	5
13	11109013	TOL145-MRL11049-0011-2, MW	Standard	307.00	7.835	10248	MC	321.02	5
14	11109014	TOL145-MRL11049-0011-2, MW	Standard	923.00	7.813	23348	MC	921.34	8
15	11109015	TOL145-MRL11049-0011-2, MW	Standard	923.00	7.813	23348	MC	921.34	8
16	11109016	TOL145-MRL11049-0011-2, MW	Standard	1237.00	7.835	34711	MC	1134.40	-9
17	11109017	MeOH Blank			7.848	44	MC	0.00	
18	11109018	TOL145-RT, 9802351/M/Day29/Day29-D104, 11/4/99			7.837	48	MC	0.00	
19	11109019	TOL145-RT, 9802351/M/Day29/Day29-D104, 11/4/99			7.844	72	MC	0.00	
20	11109020	TOL145-RT, 9802351/M/Day29/Day29-D104, 11/4/99			7.847	45	MC	0.00	
21	11109021	TOL145-RT, 9802351/M/Day29/Day29-D104, 11/4/99	QC	107.00	7.852	70	MC	0.00	-13
22	11109022	MeOH Blank			7.836	8319	MC	243.43	
23	11109023	MeOH Blank			7.836	41	MC	0.00	
24	11109024	TOL145-MRL11049-0011-2, MW	Standard	6.13	7.830	80	MC	0.00	
25	11109025	TOL145-MRL11049-0011-2, MW	Standard	6.13	7.828	274	MC	5.34	-10
26	11109026	TOL145-MRL11049-0011-2, MW	Standard	12.30	7.812	1485	MC	12.19	-2
27	11109027	TOL145-MRL11049-0011-2, MW	Standard	12.30	7.812	1485	MC	12.19	-2
28	11109028	TOL145-MRL11049-0011-2, MW	Standard	41.30	7.818	2109	MC	41.15	3
29	11109029	TOL145-MRL11049-0011-2, MW	Standard	41.30	7.818	2109	MC	41.15	3
30	11109030	TOL145-MRL11049-0011-2, MW	Standard	123.00	7.825	3948	MC	121.29	-1
31	11109031	TOL145-MRL11049-0011-2, MW	Standard	123.00	7.825	3948	MC	121.29	-1
32	11109032	TOL145-MRL11049-0011-2, MW	Standard	307.00	7.830	10281	MC	320.82	5
33	11109033	TOL145-MRL11049-0011-2, MW	Standard	307.00	7.830	10281	MC	320.82	5
34	11109034	TOL145-MRL11049-0011-2, MW	Standard	923.00	7.813	23121	MC	919.31	-8
35	11109035	TOL145-MRL11049-0011-2, MW	Standard	923.00	7.813	23121	MC	919.31	-8
36	11109036	TOL145-MRL11049-0011-2, MW	Standard	1237.00	7.832	34204	MC	1146.94	-7

Page 02019: Quantitated and printed by IAS on 11/11/99

8000 82019: Quantities and prices by lot on 11/11/98

Quantity Compound Summary Report
 TOL45-Fret 1LWRF, day 29 dose 45LWRFmg- wt: 11/4/99 mm

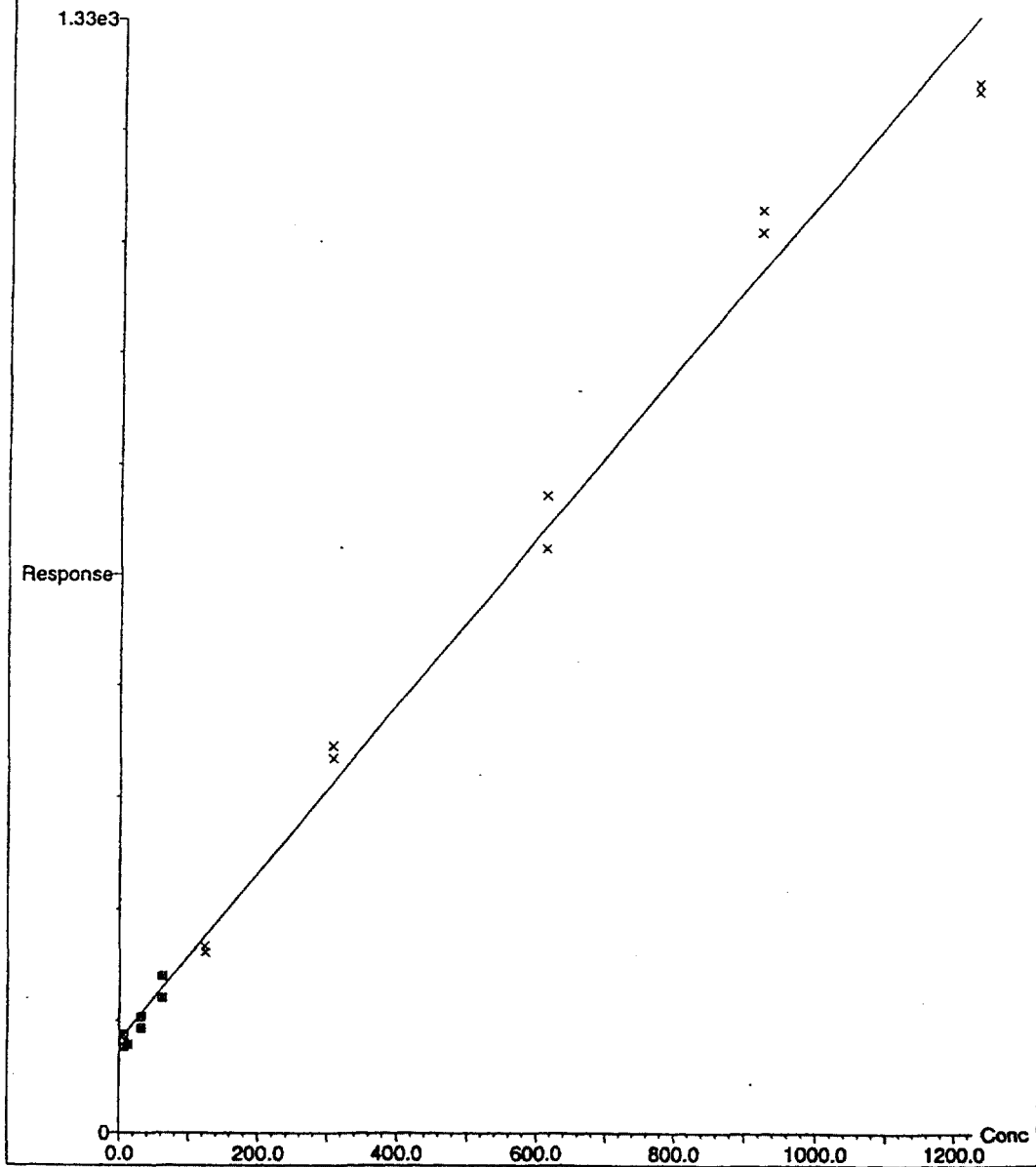
Sample List: C:\MSD\B\T\N\seep20819.prn\SampleList\11109906
 Date Modified: Thu Nov 11 07:08:32 1999
 Job Name: 11109906
 Last Modified: Thu Nov 11 07:08:32 1999
 Job Code:

Printed: Thu Nov 11 07:10:35 1999

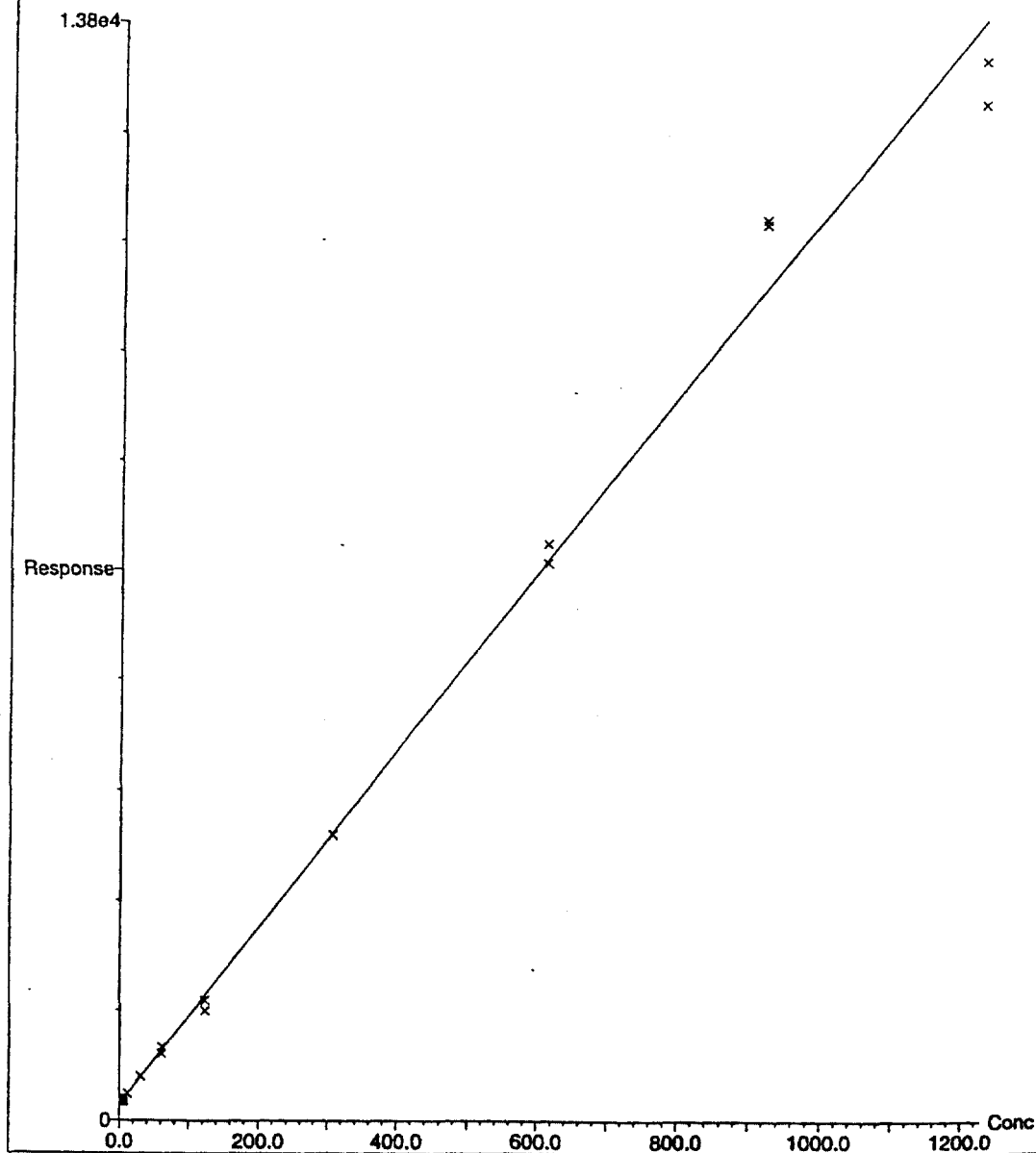
Compound 4: Refr (Phase 4)

Sample	Sample Name	Type	Std Conc	RF	Area	Peak	Chrom. Name
1	111099001	Neon Blank					
2	111099002	Neon Blank					
3	111099003	Neon Blank					
4	111099004	TOL45-BL1049-10ppb-2, mm	Standard	7.120	1012	bb	0.78 -21
5	111099005	TOL45-BL1049-10ppb-2, mm	Standard	7.100	4657	bb	0.41 -11
6	111099006	TOL45-BL1049-10ppb-2, mm	Standard	7.101	4632	bb	0.49 -1
7	111099007	TOL45-BL1049-10ppb-2, mm	Standard	7.110	4618	bb	0.49 -1
8	111099008	TOL45-BL1049-10ppb-2, mm	Standard	7.093	4618	bb	0.49 -1
9	111099009	TOL45-BL1049-10ppb-2, mm	Standard	7.093	4618	bb	0.49 -1
10	111099010	TOL45-BL1049-10ppb-2, mm	Standard	7.085	4642	bb	0.46 -4
11	111099011	TOL45-BL1049-10ppb-2, mm	Standard	7.100	4618	bb	0.49 -5
12	111099012	TOL45-BL1049-10ppb-2, mm	Standard	7.084	4650	bb	1.00 -8
13	111099013	TOL45-BL1049-10ppb-2, mm	Standard	7.084	4650	bb	1.00 -8
14	111099014	TOL45-BL1049-10ppb-2, mm	Standard	7.084	4650	bb	0.49 -5
15	111099015	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
16	111099016	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
17	111099017	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
18	111099018	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
19	111099019	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
20	111099020	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
21	111099021	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
22	111099022	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
23	111099023	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
24	111099024	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
25	111099025	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
26	111099026	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
27	111099027	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
28	111099028	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
29	111099029	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
30	111099030	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
31	111099031	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
32	111099032	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5
33	111099033	TOL45-BL1049-10ppb-2, mm	Standard	7.087	4650	bb	0.49 -5

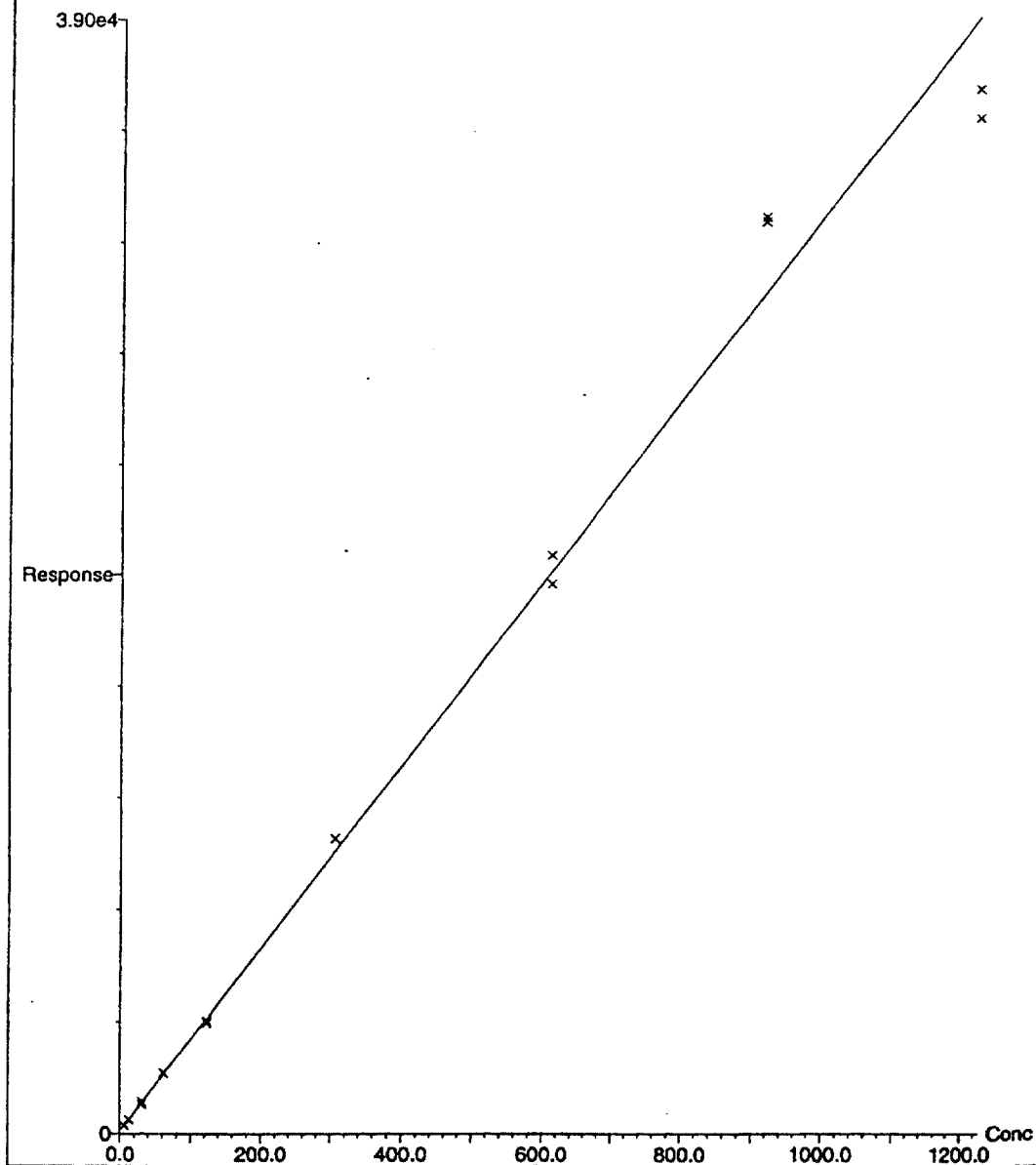
Compound 1 name: POAA (Func 1)
Coefficient of Determination: 0.980656
Calibration curve: $0.993942 \cdot x + 109.996$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



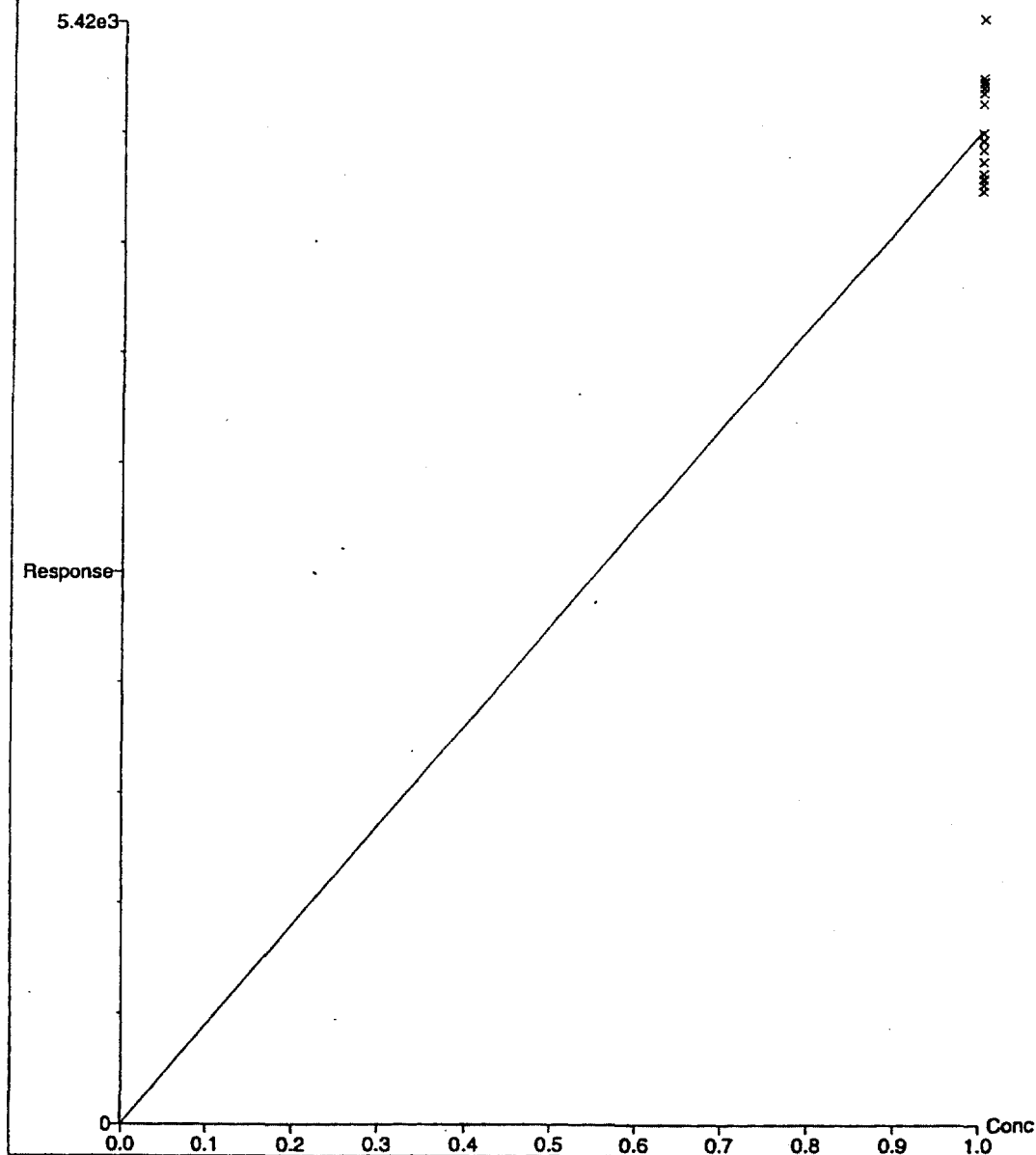
Compound 2 name: PFOS (Func 2)
Coefficient of Determination: 0.991927
Calibration curve: $11.1011 \cdot x + 194.545$
Response type: External Std, Area
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



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



Compound 4 name: Surr (Func 4)
Response Factor: 4875.54
Response type: External Std, Area
Curve type: RF



Relative height:
Relative area:
Relative area:
Absolute area:

1.50. Disabled
2.50. Disabled
3.00. Disabled
6.00. Disabled

Method Report

Page 1

Method File: C:\MSDCHEM\chemp20199_protect\chem\111099a.mdb
Last Modified: Thu Nov 11 07:09:22 1999

Compound: 1: POLA (Punc 1)
Printed: Thu Nov 11 07:34:44 1999

Compound Name: POLA (Punc 1)
Internal Ref: (none)
Quantity Traced: TIC
Conc. of Standards: Conc. A

User BP Values: 1.00000 Disabled
User Peak Factor: 1.00000
Integer Window Factor: 2.000
Chromatogram Mass Window: 1.00000

Detection Time (mins): 7.067, Enabled
Retentive Retention Time: 0.000, Disabled
Time Window (mins): 0.000

Peak Selection: Nearest

Response Type: Internal Area

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

Units:

Peak-to-peak amplitude: 22

Smoothing: Disabled
Window size (secs): 2
Number of points: 1
Smoothing method:

20% valley threshold: 10.00
20% peak threshold: 10.00
Noise baseline minimum: 5.00

Down vertical threshold: 00.00
Up vertical threshold: 00.00
Shoulder threshold: 10.00

Relative height:
Relative width:
Relative area:
Absolute area:

1.50, Disabled
0.50, Disabled
0.50, Disabled
0.50, Disabled
0.50, Disabled

Method Report

Method File: C:\MSDCHEM\mp020191-pro\msd00\111099a.mdb
 Last modified: Thu Nov 11 07:06:32 1999

Compound: 2: PROD (Punc 2)

Printed: Thu Nov 11 07:31:44 1999

Compound Name: PROD (Punc 2)
 Internal Ref.: [None]
 Quantity Traced: TIC
 Conc. of Standard: Conc. B

User RF Value: 1.00000, Disabled
 User Peak Factor: 1.00000
 Integrate Window Factor: 3.000
 Chromatogram Mass Window: 1.000Da

Retention Time (min): 3.38, Disabled
 Relative Retention Time: 0.000, Disabled
 RT Reference: [None]
 Time Window (min): 0.175

Peak Selection:

None

Response Type: Integrated
 Response: Area

Polynomial Type: Linear
 Number of Points: 10
 Fit Weights: 1/2
 Axis Transformation: None

Units:

Peak-to-peak amplitude:

98

Smoothing: Enabled

Window size (scans):

3

Number of smoothers:

1

Smoothing method:

Join valley threshold:

10.00

Reduce peak widths:

20.00

Auto baseline minimum:

5.00

Draw vertical threshold:

50.00

Reject baseline:

Disabled

Reject threshold:

10.00

Method Report

Method file: C:\MASSIVE\temp\20195.prp\Method\111095c.mdb
 Last modified: Thu Nov 11 07:08:22 1999

Compound: 4, Butt (Punc 4)

Printed: Thu Nov 11 07:18:44 1999

Compound Name: Butt (Punc 4)
 Internal Ref.: [None]
 Quantity Traces: TIC
 Conc. of Standards: Fixed
 User ID Value: 1.00000, Disabled
 User Peak Factor: 1.00000
 Integrate window factor: 2.000
 Chromatogram mass window: 1.0000Da

Retention Time (mins): 7.161, Enabled
 Relative Retention Time: 6.000, Disabled
 RT Reference: [None]
 Time Window (mins): 0.250

Peak Selection: Nearest

Response Type: External
 Response: Area

Polynomial Type: Linear
 Peak Smoothing: None
 Fit Weights: 1/X
 Axis Transformation: None

Units:

Peak-to-peak amplitude: 92

Smoothing: Enabled
 Window size (scan): 2
 Number of smoothers: 1
 Smoothing method:

Join valley threshold: 35.00
 Remove peak calling: 200.00
 Active baseline minimum: 2.00

Draw vertical threshold: 90.00
 Detect smoothers: Disabled
 Smoother threshold: 35.00

Relative height:
Relative width:
Relative area:
Absolute area:

1.00, Disabled
1.00, Disabled
2.00, Disabled
8.00, Disabled

Quattro II, #6414E

DATE: 11-10-99

ANALYST: jias

STUDY NUMBER: TOX145 MATRIX: rat liver DESCRIPTION: dilutions-day29

FACT Method: ETS-8-7.0

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

Mobile Phase: (A) 2.0 mM $\text{NH}_4\text{CH}_3\text{CO}_2$ (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 μ m

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 μ L/min. Back pressure: 65 bar

HPLC solvent gradient: 10min-10-95

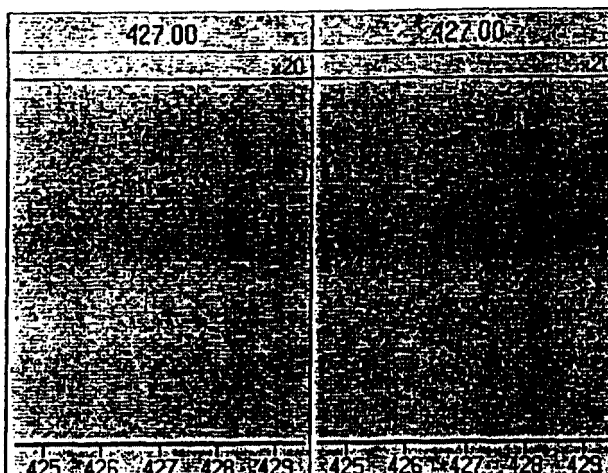
Time (min.)	%B
1) 0.00	10.0
2) 1.00	10.0
3) 5.50	95.0
4) 7.50	95.0
5) 8.00	10.0

Tuning Method Report

Page 1

Method: C:\MASSLYNX\SOUP020199.PRO\ACQUDE\PFOS3

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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	

Method Report

Page 1

Method File: C:\MSL\LYNK\SOUP020199.PRO\ACQ008\10min-10-95
Last Modified: Wednesday, November 10, 1999 10:43:07
Printed: Wednesday, November 10, 1999 10:49:20

MP1100 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

MP1100 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

MP1100 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

MP1100 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

Sample List: C:\MASSLYNX\Soup020199.pr\SampleDB\111099a.SPL
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	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-RBL11049-800ppb-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-RBL11049-800ppb-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

Masslynx - Sample List

Page 2

Sample List: C:\MASSLYNX\sup020199.pro\SampleDB\111099a.SPL
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	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	--	--	--	--