ANALYTICAL LABORATORY REPORT

ON THE

Determination of the Presence and Concentration of Potassium Perfluorooctanesulfonate (CAS Number: 2759-39-3) in the Serum and Liver of Sprague-Dawley® Rats Exposed to PFOS via Gavage

Laboratory Report No. <U2006>
Requester Project No. <3M TOX 6295.9>

Study Dates

Study Initiation: 26 May 1998
Study Completion: At Signature

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STATEMENT OF COMPLIANCE

Study Title: Analytical Laboratory Report on Presence and Concentration of Potassium Perfluorooctanesulfonate in Serum and Liver of Sprague-Dawley® Rats Exposed to PFOS via Gavage

Study Identification Number: FACT Tox-012

This study was conducted in compliance with Food and Drug Administration Good Laboratory Practice (GLP) Regulations for Nonclinical Laboratory Studies [Data Requirement(s): 21 CFR (Part 58)], with the exceptions in the bulleted list below. In addition, the present study has been audited retrospectively by an independent quality assurance unit. Audit procedures and findings for audits performed at the 3M Environmental Laboratory and at participating contract laboratories are housed with documentation pertinent to this study in archives at the 3M laboratory and will be retained for at least 10 years. The analytical portion completed at the 3M Environmental Lab was performed in accordance with 3M Environmental Technology and Safety Services Standard Operating Procedures.

Exceptions to GLP compliance:

- Storage containers for the reference substance, PFOS, were not labeled with name, CAS number, batch number, expiration date, or storage conditions.
- Details of the preparation, maximum storage time, and stability properties of the reference substance, PFOS, are unknown.
- The identity, strength, purity, and composition defining the test or control article was not determined at the time of the study; however, analyses to detail the characterization were ongoing at the time of this analytical study.
- Two separate study directors were assigned to the *in vivo* and the analytical portions of this study.
- From 15 September 1998 until 1 October 1998, the protocol was not sponsor approved and was during that time period not in compliance.
- Deviations were not consistently approved by the study director as required by GLP regulations.
- Data corrections were not always recorded as required by GLP regulations.
- Data were not always attributed to the individual responsible for recording the data.
- Records in electronic form do not meet the criteria set forth under 21 CFR(11).
- A finalized sample log-in /tracking system was not in place at the time of the study; however, draft documents did exist and were used to log in and track samples.

- Not all raw data were verified by the study director.
- No in-phase audits were conducted during the present study.
- At the time of the present study, personnel files did not always contain documentation of equipment and method training; however, personnel files have been updated, and this noncompliance has been resolved.

Signature of Study Director: Kristen Hart 27 Oct 99

Signature of Study Sponsor: Marvin T. Case 20 October 1999

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GLP STUDY
QUALITY ASSURANCE STATEMENT

Study Title: Analytical Laboratory Report on Presence and Concentration of Potassium Perfluorooctanesulfonate in Serum and Liver of Sprague-Dawley® Rats Exposed to PFOS via Gavage

Study ID Number: FACT Tox-012

This study has been inspected by the 3M Environmental Laboratory Quality Assurance Unit (QAU) as indicated in the following table. The findings were reported to the study director and management.

INSPECTION DATES		PHASE	DATE REPORTED TO	
From	*To*		*Management*	*Study Director*
9 August 1999	27 September 1999	Final Report	4 October 1999	4 October 1999

Kathleen M Stock 10-15-99

QAU Representative Date

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INTRODUCTION

Potassium Perfluorooctanesulfonate

CAS Number: 2795-39-3

Chemical Formula: $C_8F_{17}SO_3$-K+ Molecular Weight: 538

In the present study, groups of F0 rats were administered 0.1, 0.4, 1.6, or 3.2 mg PFOS kg/day in 0.5% Tween 80. (These doses correspond to concentrations of 0.02, 0.08, 0.32, and 0.64 mg/mL.) Groups of vehicle control F0 rats were administered only Tween 80. Male F0 animals were treated 42 days prior to mating and through the mating period; female F0 animals were administered PFOS daily 42 days prior to mating, through gestation, and up to 20 days following litter delivery. F1 male and female rats were exposed to the chemical *in utero* and during lactation. Following weaning at 21 days of age, selected F1 animals were treated during development and production of F2 animals. Various physical and biological parameters were monitored in F0, F1, and F2 animals.

In the analytical study reported here, liver and sera samples collected from the initial population of dosed animals (generation F0) and their offspring (generation F1) were analyzed for the presence of PFOS. (Analyses were performed to determine the presence of EtFOSE, PFOSA, POAA< PFOSEA, PFOSAA, and the monoester; however, these data were collected for informational purposes only, and were not reported.)

Liver samples were homogenized, and liver and sera samples were extracted by an ion-pairing extraction procedure. The extracts were quantitatively analyzed using high-pressure liquid chromatography/electrospray tandem mass spectrometry (HLPC/ESMSMS), and PFOS levels were evaluated against extracted standards. Analytical details are included in this report; further details are available in the study binder maintained by the 3M Fluor Analytical Chemistry Team (FACT).

Analyses assessing the toxicological effects of PFOS in the livers and sera of Sprague-Dawley rats were conducted in compliance with Good Laboratory Practice Regulations (21 CFR 58). Validated methods and standard operating procedures were followed during the preparation and analysis of the samples associated with this study.

SAMPLE RECEIPT

Tissue samples were received from Argus Research Laboratories sporadically, from August, 1998, through January, 1999. Samples received were packaged in dry ice. Specimens were registered with the 3M Environmental Laboratory and transferred to a freezer for storage. Sample receipt, identification, storage, and chain of custody protocols and data are located in the study folder for this report; the folder is located in the 3M archives.

Specimens analyzed at the 3M Environmental Laboratory will be maintained for a period of 10 years and will be stored at the laboratory at –20°C ± 10°C.

MATERIALS AND METHODS

Chemical Characterization

Table 1 presents information regarding characterization of the test, control, and reference substances used in the analytical portion of this study.

	ANALYSES PERFORMED AT THE 3M ENVIRONMENTAL LABORATORY
Reference Substance	Potassium perfluorooctanesulfonate
Source	3M Specialty Chemical Division
Preparation	Details unknown
Maximum Storage Time	Unknown
Storage Conditions	In a sealed container at room temperature, exposed to light
Chemical Lot Number	193
Physical Description and Identity Analyses	White powder
Purity	99.28%
Stability	Unknown
Control Substance	Rat liver and serum matrices
Source	Rabbit liver—Argus Research Laboratories, Inc. Rabbit serum—Sigma Chemical Company
Preparation	Unknown
Maximum Storage Time	Unknown
Storage Conditions	Frozen at –20°C
Chemical Lot Number	Liver:F00012 Serum: 17H9306
Physical Description and Identity Analyses	Rabbit liver and serum
Purity	Unknown
Stability	Unknown

Table 1. Characterization and Treatment of the Control, Reference, and Test Materials in the Study of the Presence and Concentration of Potassium Perfluorooctanesulfonate in Serum and Liver of Sprague-Dawley® Rats Exposed to PFOS via Gavage

	Analyses Performed at the 3M Environmental Laboratory
Test Material	Rat serum and liver
Source	Sprague-Dawley rats exposed to PFOS via gavage during the *in vivo* portion of 3M TOX 6295.9 at Argus Research Laboratories
Preparation	Liver: extraction per FACT M-1.0 (refer to Attachment F) Serum: extraction per FACT M-3.0 (refer to Attachment F)
Maximum Storage Time	1 week
Storage Conditions	Frozen at –20°C
Chemical Lot Number	NA
Physical Description and Identity Analyses	Rat liver and serum
Purity	NA
Stability	NA

Table 1. Characterization and Treatment of the Control, Reference, and Test Materials in the Study of the Presence and Concentration of Potassium Perfluorooctanesulfonate in Serum and Liver of Sprague-Dawley® Rats Exposed to PFOS via Gavage (continued)

Following analysis, remaining original specimens (test and control material) were stored frozen at –20°C and will be maintained for a period of 10 years.

Method Summaries

Following is a brief description of the methods used during this analytical study by the 3M Environmental Laboratory. Detailed descriptions of these methods are located in Attachment F.

Preparatory Methods:

- Method FACT-M-1.0: Extraction of Potassium Perfluorooctanesulfonate or Other Anionic Fluorochemical Surfactants from Liver for analysis Using HPLC/ESMS.

  In this method, an ion pairing reagent was added to the sample and the analyte ion pair was partitioned into MtBE. Four mLs of the MtBE extract was transferred to a centrifuge tube and put onto a nitrogen evaporator until dry. Each extract was reconstituted in 1.0 mL of methanol, then filtered through a 3 cc plastic syringe attached to a 0.2 µm nylon filter into glass autovials.

- Method FACT-M-3.0: Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical Compounds from Serum or other Fluid for Analysis Using HPLC-Electrospray/Mass Spectrometry.

  Sera samples were extracted using an ion-pairing extraction procedure. In summary, an ion pairing reagent was added to the sample and the analyte ion pair was partitioned into methyl-*tert*-butyl ether (MtBE). Four mL of the MtBE extract was removed and put onto a nitrogen evaporator until dry. Each extract was reconstituted in 1.0 mL of methanol and filtered through a 3-cc plastic syringe attached to a 0.2 µm nylon filter into glass autovials.

Analytical Methods

- Method FACT-M-2.0: Analysis of Fluorochemicals in Liver Extract Using HPLC-Electrospray/Mass Spectrometry

  The analysis was performed by monitoring a single ion characteristic of a particular fluorochemical, such as the perfluorooctanesulfonate (PFOS) anion, m/z = 499. Additionally, samples were analyzed using a tandem mass spectrometer to further verify the identity of a compound by detecting daughter ions of the selected parent ion.

- Method FACT M-4.0: Analysis of Potassium Perfluorooctanesulfonate or other Fluorochemical in Serum or other Fluid Using HPLC-Electrospray/Mass Spectrometry

  The analysis was performed by monitoring a single ion characteristic of a particular fluorochemical, such as the perfluorooctanesulfonate (PFOS) anion, m/z= 499. Additionally, samples were analyzed using a tandem mass spectrometer to further verify the identity of a compound by detecting daughter ions of the parent ion.

Confirmatory dose analyses were conducted following the end of the study, and the results of these analyses are presented in Attachment G.

Analytical Equipment

Following are typical analytical equipment settings for the procedures used in the present study. These settings vary somewhat during actual data collection. Exact settings during all phases of data collection are recorded and presented in the analysis section of the study binder for FACT Tox-12.

HPLC System: Hewlett-Packard Series 1100 Liquid Chromatograph

Column:	Keystone Betasil C18 Column 2X100 mm, 5µm particle size
Flow rate:	300 µL/min
Solvent A:	2.0 mM ammonium acetate
Solvent B:	Methanol
Solvent Gradient:	40% to 90% B in 8.5 minutes Hold at 90% B for 3.0 minutes Return to 40% B in 1.0 minute Hold at 40% B for 1.0 minute
Injection Volume:	10 µL
Run Time:	13.5 minutes

Electrospray Tandem Mass Spectrometer: Micromass Quattro II API Mass Spectrometer

Mass Lynx 3.1 Software

Cone Voltage:	30—60V
Collision Gas Energy:	40 eV
Mode:	Electrospray Negative
Source Block Temperature:	115°C
Desolvation Temperature:	250°C
Electrode:	Z-spray
Primary Ion/Daughter Ions:	499/80, 99, 130, 180, 230 amu

Protocol Deviations

There was one deviation to the protocol: The protocol called for the use of reference standard lot number 217; however, lot number 193 was used during the analytical portion of the study.

DATA SUMMARY, ANALYSES, AND RESULTS

Summary of Quality Control Analyses Results

- Calibration Check Standards: A mid-level, extracted matrix calibration check was analyzed at least every 10 samples to monitor instrument drift. Calibration check standards were compliant, and all calibration criteria were met (within ± 30%).
- Blanks: Four blanks were extracted concurrently with each batch of samples. Two extraction blanks were prepared with water as a surrogate matrix. Two additional samples of blank matrix were typically prepared with rabbit liver or sera and were used for extracted calibration curves. All blanks were nondetect for the compound(s) of interest.
- Duplicate matrix spike analyses including all target analytes were prepared and analyzed for one control animal. With a few exceptions, recoveries were within the acceptable range of 70-130%.

  Analysis of two liver matrix spike samples resulted in 51% and 61% recoveries of PFOS. These recoveries are below the lower boundary of the acceptable range; however, the second set of matrix spike samples were within the acceptable range. No action was taken to further characterize these matrix spikes.

  Analyses of both sets of sera matrix spikes resulted in PFOS recoveries greatly above the high limit of the acceptable range. Although 1 mL of sera was used for the analysis of PFOS levels in the sera of test animals, only a small amount of serum (500 µL) was available for the matrix spike preparation; this limited quantity of matrix may have been a source of error. As the extraction is scaled-down to accommodate the available matrix, the resulting extract is particularly susceptible to loss of solvent due to evaporation. These evaporative losses, which would not effect the PFOS concentration (PFOS has a low vapor pressure) in the extract, could result in a greater-than-expected PFOS concentration in the extract. When analyses of sera matrix spikes are performed with a full 1 mL of sera, high PFOS recoveries are unusual.
- Matrix Spikes: Matrix spikes and matrix spike duplicates were analyzed every 40 samples, with a minimum of two per batch during analytical analyses. The results of these analyses are located in the archived study binder.

Summary of Sample Results

- PFOS was detected in the livers of all control and exposed groups of F0 animals and F1 animals. PFOS was detected in the sera samples collected from all of the control and dosed F0 males and in sera samples collected from all dosed F0 females. The PFOS levels found in the samples from control animals were quite low—generally 100-less than levels detected in the low-dose group. PFOS concentration in exposed animals increased with increasing dose group, generally in a dose-related manner.

  PFOS levels in both sera and liver samples collected from female F0 animals were much less than levels detected in samples collected from the corresponding group of F0 males.

  PFOS concentrations measured in pooled liver samples collected from the exposed groups of F1 animals shortly after birth were roughly equal to or lower than concentrations measured in the corresponding groups of F0 females. No samples collected from F0 females that received 3.2 mg PFOS per kg/day were submitted for analysis. Table 1 summarizes results of the quantitative analyses performed on the F0 liver and sera samples examined.

Group	Dose (mg/kg/day)	Dose Concentration (mg/mL)	Average Concentration of PFOS in Serum (µg/mL)	Average Concentration of PFOS in Liver (µg/g)
0.0 mg/kg/day	0.0	0.0	Female: 0.0307 Male: 0.0244	Female: 0.171 Male: 0.665
0.1 mg/kg/day	0.1	0.02	Female: 5.28 Male: 10.5	Female: 14.8 Male: 84.9
0.4 mg/kg/day	0.4	0.08	Female: 18.9 Male: 45.4	Female: 58.0 Male: 176
1.6 mg/kg/day	1.6	0.32	Female: 82.0 Male: 152	Female: 184 Male: 323
3.2 mg/kg/day	3.2	0.64	Female: NR[a] Male: 273	Female: NR[a] Male: 1360

[a] Samples were not received.

Table 2. PFOS Concentrations in Serum and Liver Samples from F0 Male and Female Sprague-Dawley Rats

PFOS concentrations measured in pooled liver samples collected from the **control and exposed** groups of F1 animals shortly after birth were roughly the equal to or lower than concentrations measured in the corresponding groups of F0 females. No samples collected from F1 males or females that received 3.2 mg/kg/day were submitted for analysis. PFOS was detected in the liver of all other exposed groups of F1 animals. Table 2 summarizes results of the quantitative analyses performed on the F1 sera samples examined.

Group	Dose (mg/kg/day)	Dose Concentration (mg/mL)	Average Concentration of PFOS in Liver (µg/g)
0.0 mg/kg/day	0.0	0.0	0.0511
0.1 mg/kg/day	0.1	0.020	6.19
0.4 mg/kg/day	0.4	0.080	57.6
1.6 mg/kg/day	1.6	0.320	70.4
3.2 mg/kg/day	3.2	0.640	NR[a]

[a] Samples were not received.

Table 3. PFOS Concentrations in Liver Samples from F1 Male and Female Sprague-Dawley Rats

Paper and electronic copies of raw data and study reports generated during the present studies will be kept for at least a period of time as established by regulation and by the 3M Standard Operating Procedures. Paper and electronic copies of reports, protocols, and methods generated by the 3M Environmental Technology and Safety Services laboratory will be retained with the study folder for at least a period of time as established by regulation and by the 3M Standard Operating Procedures.

Statistical Methods

For data generated by the 3M Environmental Laboratory, means and standard deviations were calculated using Microsoft Excel®, and relative standard deviations were calculated manually.

Standard deviation is a measurement of analytical precision; thus it is used to gauge the precision of the analyses. Relative standard deviation presents a measure of the magnitude of the standard deviation.

$$\sqrt{\frac{n\Sigma x^2-(\Sigma x)^2}{n(n-1)}}$$

Means are calculated by adding individual entities and dividing the resultant sum by the number of individual entities. Standard deviations were calculated using the equation:

Relative standard deviations were calculated by division of the standard deviation by the mean with subsequent multiplication of the quotient by 100.

DATA QUALITY OBJECTIVES AND DATA INTEGRITY

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

No circumstances existed during the present study that would have affected the quality or integrity of the data. The following data quality objectives (DQOs) 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—PFOS in serum=1.75 ppb
- Limits of quantitation—Equal to the lowest acceptable standard in the calibration curve
- Duplicate frequency/acceptable precision—<30%
- Spike frequency/acceptable recoveries—70% to 130%
- Use of confirmatory methods—no confirmatory methods were used
- Demonstration of specificity—specificity was demonstrated by chromatographic retention time and mass spectral daughter ion characterization

STATEMENT OF CONCLUSION

Statement of Conclusion:

Under the conditions of the present studies, potassium perfluorooctanesulfonate was observed in the livers and sera of all groups of rats exposed to the chemical during the *in vivo* portion of the studies.

REFERENCES

None

ATTACHMENTS

- Attachment A: Report signature page
- Attachment B: Table of results of analysis of liver samples from F0 male and female Sprague-Dawley rats administered PFOS daily by gavage.
- Attachment C: Table of results of analysis of sera samples from F0 male and female Sprague-Dawley rats administered PFOS daily by gavage.
- Attachment D: Table of results of analysis of liver samples from F1 male and female Sprague-Dawley rats administered PFOS daily by gavage.
- Attachment E: In-life protocol and study protocol
- Attachment F: Preparatory and analytical methods
- Attachment G: Dose Analyses

ATTACHMENT A
REPORT SIGNATURE PAGE

Kristen J. Hansen, Ph.D., Study Director — Date: 10/27/99

Marvin T. Case, D.V.M., Ph.D., Study Sponsor — Date: 20 October 1999

Dale L. Bacon, Laboratory Director — Date: 10/27/99

ATTACHMENT B

Dose Group	Sample Number	Extracted Weight (g)	PPOS CALCULATED CONCENTRATION (mG/G)	Total Amount of PFOS (µg/g)	Mean PFOS (µg/g)	Relative Standard Deviation
Method Blank	H_2O Blank 1	N/A	<MDL	<MDL		
Method Blank	H_2O Blank 2	N/A	<MDL	<MDL	<MDL	<MDL
Matrix Blank	Rabbit Liver (Blank 1)	N/A	<MDL	<MDL		
Matrix Blank	Rabbit Liver (Blank 2)	N/A	<MDL	<MDL	<MDL	<MDL
QC — 100 ppb	8801F-MS	1.0169	60	50%		
	8801F-MSD	1.0169	72	60%	55%	18%
	8801M-MS	1.0525	100	87%		
	8801M-MSD	1.0525	93	81%	84%	7%
0.0 mg/kg/day	8801F	1.0169	674	0.674		
	8807F	1.0384	158	0.158		10.1[a]
	8808F	1.0177	165	0.165	0.171[a]	0.0172[a]
	8821F	1.0439	197	0.197		82.9
	8833F	1.0048	166	0.166	0.272	0.225
	8101M	1.0525	910	0.910		
	8103M	1.0407	600	0.600		
	8104M	1.0568	577	0.577		
	8107M	1.0104	593	0.593		21.0
	8108M	1.0321	643	0.643	0.665	0.139
0.1 mg/kg/day (0.02 mg/mL)	8838F	1.0374	17251	17.3		
	8840F	1.0168	14433	144		
	8842F	1.0489	15557	15.6		
	8864F	1.0608	13807	13.8		11.6
	8870F	1.0225	12784	12.8	14.8	1.71
	8138M	1.0617	80270	80.3		
	8140M	1.0509	76629	76.6		
	8142M	0.9961	92106	92.1		
	8145M	1.036	87938	87.9		7.39
	8146M	0.9788	87451	87.5	84.9	6.28

Table 4. Analyses of Liver Samples from F0 Sprague-Dawley Rats in the Study of the Presence and Concentration of Potassium Perfluorooctanesulfonate in Serum and Liver of Sprague-Dawley® Rats Exposed to PFOS via Gavage

ATTACHMENT B (CONTINUED)

DOSE GROUP	SAMPLE NUMBER	EXTRACTED WEIGHT (g)	PPOS CALCULATED CONCENTRATION (mg/g)	TOTAL AMOUNT OF PFOS (µg/g)	MEAN PFOS (µg/g)	RELATIVE STANDARD DEVIATION
0.4 mg/kg/day (0.08 mg/mL)	8890F	1.0346	61853	61.9		
	8893F	1.0247	62547	62.5		
	8895F	1.0154	49543	49.5		
	8902F	1.0039	64081	64.1		11.6
	8905F	1.0426	51922	51.9	58.0	6.73
	8172M	1.0234	137715	138		
	8174M	1.0067	189256	189		
	8175M	1.0471	183050	183		
	8176M	1.0335	173288	173		13.3
	8181M	1.0197	198173	198	176	23.4
1.6 mg/kg/day (0.32 mg/mL)	8919F	1.0756	200313	200		
	8921F	1.0544	212684	213		
	8926F	1.0219	282001	282		
	8934F	1.022	183576	184		48.0
	8937F	1.654	40958	41.0	184	88.3
	8209M	1.0016	374773	375		
	8213M	1.0211	325087	325		
	8219M	1.0421	279320	279		
	8221M	1.0153	335349	335		11.2
	8225M	1.0316	301145	301	323	36.1
3.2 mg/kg/day (0.64 mg/mL)	NR	NR	NR	NR		
	NR	NR	NR	NR		
	NR	NR	NR	NR		
	NR	NR	NR	NR		NR
	NR	NR	NR	NR	NR	NR
	8243M	1.0065	1271156	1271		
	8244M	1.0397	1116251	1116		
	8249M	1.0107	2077414	207 7		
	8250M	1.0522	1168784	1169		30.0
	8255M	0.9908	1149853	1150	1360	40.7

[a] Sample 8801F value was excluded from the calculations.

Table 4. Analyses of Liver Samples from F0 Sprague-Dawley Rats in the Study of the Presence and Concentration of Potassium Perfluorooctanesulfonate in Serum and Liver of Sprague-Dawley® Rats Exposed to PFOS via Gavage (continued)

ATTACHMENT C

Dose Group	Sample Number	PFOS Calculated Concentration (ng/g)	Total Amount of PFOS (µg/g)	Mean PFOS (µg/g)	Relative Standard Deviation (RSD)
Method Blank	H_2O Blank 1	<MDL			
Method Blank	H_2O Blank 2	<MDL	<MDL	<MDL	<MDL
Matrix Blank	Rabbit Liver (Blank 1)	<MDL	<MDL		
Matrix Blank	Rabbit Liver (Blank 2)	<MDL	<MDL	<MDL	<MDL
QC — 100 ppb	8801F-MS	60	50%		
	8801F-MSD	72	60%	55%	18%
	8801M-MS	100	84%		
	8801M-MSD	93	78%	81%	7%
0.0 mg/kg/day	8801F/M	369	0.369		
	8807F/M	51.5	0.0515		5.58[a]
	8808F/M	53.2	0.0532	0.0511[a]	0.00285[a]
	8821F/M	52.7	0.0527		124
	8833F/M	46.9	0.0469	0.115	0.142
0.1 mg/kg/day (0.02 mg/mL)	8838F/M	5872	5.87		
	8840F/M	5149	5.15		
	8842F/M	7390	7.39		
	8864F/M	6745	6.74		14.2
	8870F/M	5788	5.79	6.19	0.879
0.4 mg/kg/day (0.08 mg/mL)	8890F/M	62050	62.1		
	8893F/M	61745	61.7		
	8895F/M	47738	47.7		
	8902F/M	63057	63.1		11.7
	8905F/M	53492	53.5	57.6	6.72
1.6 mg/kg/day (0.32 mg/mL)	8919F/M	78376	78.4		
	8921F/M	58893	58.9		
	8926F/M	91956	92.0		
	8934F/M	65023	65.0		20.6
	8937F/M	57897	57.9	70.4	14.5

[a] Sample 8801F value not included in this calculation

Table 5. Analyses of Liver Samples from F1 Sprague-Dawley Rats in the Study of the Presence and Concentration of Potassium Perfluorooctanesulfonate in Serum and Liver of Sprague-Dawley® Rats Exposed to PFOS via Gavage

ATTACHMENT D

DOSE GROUP	SAMPLE NUMBER	PPOS REPORTED (mg/mL)	MEAN PFOS (µg/mL)	RELATIVE STANDARD DEVIATION (RSD)
Method Blank	H_2O Blank 1	<MDL		
Method Blank	H_2O Blank 2	<MDL	<MDL	<MDL
Matrix Blank	Rabbit Liver (Blank 1)	<MDL		
Matrix Blank	Rabbit Liver (Blank 2)	<MDL	<MDL	<MDL
QC — 100 ppb	8801F-MS	136%		
	8801F-MSD	133%	135%	1%
	8801M-MS	119%		
	8801M-MSD	121%	120%	1%
0.0 mg/kg/day	8801F	0.127		
	8807F	0.0229		30.9[a]
	8808F	0.0291	0.037[a]	0.00950[a]
	8821F	0.0445		87.6
	8833F	0.0265	0.0500	0.044
	8101M	0.0306		
	8103M	0.0311		
	8104M	0.0177		
	8107M	0.0213		2.48
	8108M	0.0214	0.0244	0.00605
0.1 mg/kg/day	8838F	5.14		
(0.02 mg/mL)	8840F	4.93		
	8842F	5.24		
	8864F	5.23		6.78
	8870F	5.89	5.28	0.358
	8138M	11.8		
	8140M	9.54		
	8142M	9.65		
	8145M	11.0		9.02
	8146M	10.4	10.5	0.946
0.4 mg/kg/day	8890F	20.9		
(0.08 mg/mL)	8893F	18.6		
	8895F	18.4		
	8902F	17.4		6.89
	8905F	19.1	18.9	1.30
	8172M	40.7		
	8174M	54.1		
	8175M	41.6		
	8176M	47.4		12.1
	8181M	43.3	45.4	5.49

Table 6. Analyses of Sera Samples from F0 Sprague-Dawley Rats in the Study of the Presence and Concentration of Potassium Perfluorooctanesulfonate in Serum and Liver of Sprague-Dawley® Rats Exposed to PFOS via Gavage

Dose Group	Sample Number	PPOS Reported (mg/mL)	Mean PFOS (µg/mL)	Relative Standard Deviation (RSD)
1.6 mg/kg/day (0.32 mg/mL)	8919F	75.3		
	8921F	79.2		
	8926F	113		
	8934F	74.4		21.4
	8937F	68.4	82.0	17.5
	8209M	144		
	8213M	151		
	8219M	148		
	8221M	165		5.20
	8225M	153	152	7.91
3.2 mg/kg/day (0.64 mg/mL)	NR	NR		
	NR	NR		
	NR	NR		
	NR	NR		NR
	NR	NR	NR	NR
	8243M	249		
	8244M	242		
	8249M	257		
	8250M	361		18.3
	8255M	255	273	49.8

[a] Sample 8801F value not included in this calculation

Table 6. Analyses of Sera Samples from F0 Sprague-Dawley Rats in the Study of the Presence and Concentration of Potassium Perfluorooctanesulfonate in Serum and Liver of Sprague-Dawley® Rats Exposed to PFOS via Gavage (continued)

ATTACHMENT E
IN-LIFE PROTOCOL AND ANALYTICAL STUDY PROTOCOL