AR226-0028
(5)

3M ENVIRONMENTAL LABORATORY

METHOD

EXTRACTION OF POTASSIUM PERFLUOROOCTANESULFONATE OR OTHER FLUOROCHEMICAL COMPOUNDS FROM URINE FOR ANALYSIS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY/MASS SPECTROMETRY

Method Number: ETS-8-96.0

Adoption Date: 7-28-99

Revision Date: NA

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Approved By:

Laboratory Manager	Date 7/28/99
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Technical Reviewer	Date 7/27/99

1.0 SCOPE AND APPLICATION

1.1 **Scope:** This method is for the extraction of potassium perfluorooctanesulfonate (PFOS) or other fluorochemical compounds from urine.

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

1.3 **Matrices:** Human, rat, and monkey urine or other fluids as designated in the validation report.

2.0 SUMMARY OF METHOD

2.1 This method describes the procedure for extracting potassium perfluorooctanesulfonate (PFOS) or other fluorochemicals from urine, or other fluids, using an ion pairing reagent and methyl-*tert*-butyl ether (MtBE). In this method, eight fluorochemicals are extracted: PFOS, PFOSA, PFOSAA, EtFOSE-OH, M556, M570, perfluorooctanoate (POAA), and surrogate standard (see *3.0* ***Definitions***). An ion pairing reagent is added to two ml of sample and the analyte ion pair is partitioned into MtBE. The MtBE extract is removed and put onto a nitrogen evaporator until dry. Each extract is reconstituted in 0.5 ml of methanol, then filtered through a 0.2 µm nylon filter attached to 3 cc plastic syringe into glass autovials.

2.2 These sample extracts are analyzed following method ETS-8-97.0 or other appropriate method.

3.0 DEFINITIONS

3.1 PFOS: perfluorooctanesulfonate (anion of potassium salt) $C_8F_{17}SO_3^-$

3.2 PFOSA: perfluorooctane sulfonylamide $C_8F_{17}SO_2NH_2$

3.3 PFOSAA: perfluorooctane sulfonylamido (ethyl)acetate $C_8F_{17}SO_2N(CH_2CH_3)CH_2CO_2^-$

3.4 EtFOSE-OH: 2(N-ethylperfluorooctane sulfonamido)-ethyl alcohol $C_8F_{17}SO_2N(CH_2CH_3)CH_2CH_2OH$

3.5 M556: $C_8F_{17}SO_2N(H)(CH_2COOH)$

3.6 M570: $C_8F_{17}SO_2N(CH_3)CH_2COOH$

3.7 POAA: perfluorooctanoate $C_7F_{15}COO^-$

3.8 Surrogate standard THPFOS: 1H-1H-2H-2H perfluorooctane sulfonic acid, used as an internal standard in this method.

4.0 WARNINGS AND CAUTIONS

4.1 Health and safety warnings

4.1.1 Use universal precautions, especially laboratory coats, goggles, and gloves when handling animal tissue, which may contain pathogens.

5.0 INTERFERENCES

5.1 At this time, it is unknown how the extraction method is affected by potential interferences that may be present such as conjugated fluorochemicals (eg. Glucuronides). Conjugates may become deconjugated during extraction or analysis resulting in a high bias for reported results of target analytes.

6.0 EQUIPMENT

6.1 The following equipment is used while performing this method. Equivalent equipment is acceptable.

6.1.1 Vortex mixer, VWR, Vortex Genie 2

6.1.2 Centrifuge, Mistral 1000 or IEC

6.1.3 Shaker, Eberbach or VWR

6.1.4 Nitrogen evaporator, Organomation

6.1.5 Balance (± 0.100 g)

7.0 SUPPLIES AND MATERIALS

7.1 Gloves

7.2 Eppendorf or disposable pipettes

7.3 Nalgene bottles, capable of holding 250 ml and 1 L

7.4 Volumetric flasks, glass, type A

7.5 I-CHEM vials, glass, 40 ml glass

7.6 Centrifuge tubes, polypropylene, 15 ml

7.7 Labels

7.8 Oxford Dispenser – 3.0 to 10.0 ml

7.9 Syringes, capable of measuring 2.5 µL to 50 µL

7.10 Graduated pipettes

7.11 Syringes, disposable plastic, 3 cc

7.12 Syringe filters, nylon, 0.2 µm, 25 mm

7.13 Timer

7.14 Crimp cap autovials and caps

7.15 Crimpers

Note: Prior to using glassware and bottles, rinse 3 times with methanol and 3 times with Milli-Q™ water. Rinse glass syringes a minimum of 9 times with methanol, 3 rinses from 3 separate vials.

8.0 REAGENTS AND STANDARDS

8.1 Type I reagent grade water, Milli-Q™ or equivalent; all water used in this method should be Milli-Q™ water and may be provided by a Milli-Q TOC Plus™ system

8.2 Sodium hydroxide (NaOH), J.T Baker or equivalent

8.3 Tetrabutylammonium hydrogen sulfate(TBA), Kodak or equivalent

8.4 Sodium carbonate (Na_2CO_3), J.T. Baker or equivalent

8.5 Sodium bicarbonate ($NaHCO_3$), J.T. Baker or equivalent

8.6 Methyl-T-Butyl Ether, Omnisolv, glass distilled or HPLC grade

8.7 Methanol, Omnisolv, glass distilled or HPLC grade

8.8 Urine frozen from supplier

8.9 Fluorochemical standards

8.9.1 PFOS (3M Specialty Chemical Division), molecular weight = 538

8.9.2 PFOSA (3M Specialty Chemical Division), molecular weight = 499

8.9.3 PFOSAA (3M Specialty Chemical Division), molecular weight = 585

8.9.4 EtFOSE-OH (3M Specialty Chemical Division), molecular weight = 570

8.9.5 M556 (3M Specialty Chemical Division), molecular weight = 557

8.9.6 M570 (3M Specialty Chemical Division), molecular weight = 571

8.9.7 POAA (3M Specialty Chemical Division), molecular weight = 452

8.9.8 THPFOS (1-H,1-H, 2-H, 2-H $C_8F_{13}SO_3H$) molecular weight = 428

8.9.9 Other fluorochemicals, as appropriate

8.10 Reagent preparation

NOTE: When preparing larger volumes than listed in reagent, standard, or surrogate preparation, adjust accordingly.

8.10.1 10 N sodium hydroxide (NaOH): Weigh approximately 200 g NaOH. Pour into a 1000 ml beaker containing 500 ml Milli-Q™ water, mix until all solids are dissolved. Store in a 1 L Nalgene bottle.

8.10.2 1 N sodium hydroxide (NaOH): Dilute 10 N NaOH 1:10. Measure 10 ml of 10 N NaOH solution into a 100 ml volumetric flask and dilute to volume using Milli-Q™ water. Store in a 125 ml Nalgene bottle.

8.10.3 0.5 M tetrabutylammonium hydrogen sulfate (TBA): Weigh approximately 169 g of TBA into a 1 L volumetric containing 500 ml Milli-Q™ water. Adjust to pH 10 using approximately 44 to 54 ml of 10 N NaOH (While adding the last ml of NaOH, add slowly because the pH changes abruptly). Dilute to volume with Milli-Q™ water. Store in a 1 L Nalgene bottle.

8.10.3.1 TBA requires a check prior to each use to ensure pH = 10.0. Adjust as needed using 1 N NaOH solution.

8.10.4 0.25 M sodium carbonate/sodium bicarbonate buffer ($Na_2CO_3/NaHCO_3$): Weigh approximately 26.5 g of sodium carbonate (Na_2CO_3) and 21.0 g of sodium bicarbonate ($NaHCO_3$) into a 1 L volumetric flask and bring to volume with Milli-Q™ water. Store in a 1 L Nalgene bottle.

8.11 Standards preparation

8.11.1 Prepare PFOS standards for the standard curve.

8.11.2 Prepare other fluorochemical standards, as appropriate. Multicomponent fluorochemical standards are acceptable (for example, one working standard solution containing 1.00 ppm PFOS, 1.02 ppm PFOSA, 0.987 ppm PFOSAA, and 1.10 ppm EtFOSE-OH.)

8.11.3 Weigh approximately 100 mg of PFOS into a 100 ml volumetric flask and record the actual weight in the Standard Logbook.

8.11.4 Bring to volume with methanol for a stock standard of approximately 1000 ppm (μg/ml).

8.11.5 Dilute the stock solution with methanol for a working standard 1 solution of approximately 50 ppm.

8.11.6 Dilute working standard 1 with methanol for a working standard 2 solution of approx. 5.0 ppm.

8.11.7 Dilute working standard 1 with methanol for a working standard 3 solution of approx. 0.50 ppm.

8.12 Surrogate stock standard preparation

8.12.1 Weigh approximately 50-60 mg of surrogate standard 1-H,1-H, 2-H, 2-H, $C_8F_{13}SO_3H$ into a 50 ml volumetric flask and record the actual weight.

8.12.2 Bring to volume with methanol for a surrogate stock of approximately 1000-1200 ppm.

8.12.3 Prepare a surrogate working standard. Transfer approximately 1 ml of surrogate stock to a 10 ml volumetric flask and bring to volume with methanol for a working standard of 100-120 ppm. Record the actual volume transferred in the Standard Logbook.

9.0 Sample Handling

9.1 All samples are received frozen and must be kept frozen until the extraction is performed.

9.2 Allow samples to thaw to room temperature prior to extraction.

10.0 Quality Control

10.1 Solvent Blanks, Method blanks and matrix blanks

10.1.1 An aliquot of 2.0 ml methanol is used as a solvent blank.

10.1.2 Extract two 2.0 ml aliquots of Milli-Q™ water following this procedure and use as method blanks.

10.1.3 Extract two 2.0 ml aliquots of the urine following this procedure and use as matrix blanks. See **11.1.4**.

10.2 Matrix spikes

10.2.1 Prepare and analyze matrix spike and matrix spike duplicate samples to determine the accuracy of the extraction.

10.2.2 Prepare each spike using a sample chosen by the analyst, usually the control matrix received with each sample set.

10.2.3 Expected concentrations should fall in the mid-range of the initial calibration curve. Additional spikes may be included and may fall in the low-range of the initial calibration curve.

10.2.4 Prepare one matrix spike and matrix spike duplicate per 40 samples, with a minimum of 2 matrix spikes per batch.

10.3 Continuing calibration verifications

10.3.1 Prepare continuing calibration verification samples to ensure the accuracy of the initial calibration curve.

10.3.2 Prepare, at a minimum, one continuing calibration verification per group of 10 samples. For example, if a sample set = 34, four checks are prepared and extracted.

10.3.3 Prepare each continuing calibration verification from the same matrix used to prepare the initial curve.

10.3.4 The expected concentrations will fall within the mid-range of the initial calibration curve. Additional spikes may be included that fall in the low-range of the initial calibration curve. This is necessary if the analyst must quantitate using only the low end of the calibration curve (for example, 5 ppb – 100 ppb, rather than 5 ppb – 1000 ppb).

11.0 CALIBRATION AND STANDARDIZATION

11.1 Prepare matrix calibration standards

11.1.1 Transfer 2.0 ml of urine to a 15 ml centrifuge tube.

11.1.2 Record each sample volume on the extraction sheet.

11.1.3 While preparing a total of twenty-two aliquots in 15 ml centrifuge tubes, mix or shake between aliquots.

11.1.4 Two 2.0 ml aliquots serve as matrix blanks.

11.1.5 Typically use the standard concentrations and spiking amounts listed in Table 1, at the end of this section, to spike, in duplicate, two standard curves, for a total of twenty standards, two matrix blanks, and two method blanks.

11.1.6 Refer to validation report **FACT-TOX-131, W2067,** which lists the working ranges and the Linear Calibration Range (LCR) for calibration curves.

11.1.7 Use Attachment D as an aid in calculating the concentrations of the working standards. See Section **13.0** to calculate actual concentrations of PFOS in calibration standards.

11.2 To each standard, blank, or continuing check, add appropriate amount of surrogate working standard for the concentration to fall within the calibration curve range 10 ppb - 1500 ppb.

11.3 Extract spiked matrix standards following **12.6-12.16** of this method. Use these standards to establish each initial curve on the mass spectrometer.

Table 1 Approximate spiking amounts for standards and spikes Using 2.0 ml of matrix			
Working standard (approx. conc.)	µL	Approx. final conc. of analyte in matrix	Approx. final conc. Of analyte in solvent
-	-	Blank	Blank
0.500 ppm	5	1.25 ppb	5.00 ppb
0.500 ppm	10	2.50 ppb	10.0 ppb
0.500 ppm	25	6.00 ppb	25.0 ppb
5.00 ppm	5	12.5 ppb	50.0 ppb
5.00 ppm	10	25.0 ppb	100 ppb
5.00 ppm	25	62.5 ppb	250 ppb
50.0 ppm	5	125 ppb	500 ppb
50.0 ppm	7.5	200 ppb	750 ppb
50.0 ppm	10	250 ppb	1000 ppb
50.0 ppm	15	375 ppb	1500 ppb

12.0 PROCEDURE

12.1 Obtain frozen samples and allow to thaw at room temperature or in a lukewarm waterbath.

12.2 Vortex mix for 15 seconds, then transfer 2.0 ml or other appropriate volume to a 15 ml polypropylene centrifuge tube.

12.3 Return unused samples to freezer after extraction amounts have been removed.

12.4 Record the initial volume on the sample weight/volume worksheet. . See **Attachment D**. The original weight/volume worksheet is included in the study binder.

12.5 Label the tube with the study number, sample ID, date and analyst initials. See attached worksheet for documenting the remaining steps.

12.6 Spike all samples, including blanks and standards, ready for extraction with surrogate standard as described in **11.2**.

12.7 Spike each matrix with the appropriate amount of standard as described in **11.1**, or **Table 1** in that section, for the calibration curve standards. Also prepare matrix spikes and continuing calibration standards.

12.8 Vortex mix the standard curve samples, matrix spike samples, and continuing calibration samples for 15 seconds.

12.9 Check to ensure the 0.5 M TBA reagent is at pH 10. If not, adjust accordingly.

12.10 To each sample, add 1 ml 0.5 M TBA and 2 ml of 0.25M sodium carbonate/sodium bicarbonate buffer.

12.11 Using an Oxford Dispenser, add 5 ml methyl-*tert*-butyl ether.

12.12 Cap each sample and put on the shaker at a setting of 300 rpm, for 20 minutes.

12.13 Centrifuge for 20 to 25 minutes at a setting of 3500 rpm, or until layers are separated.

12.14 Label a fresh 15 ml centrifuge tube with the same information as in **12.5**.

12.15 Remove 4.0 ml of the organic layer to this clean 15 ml centrifuge tube.

12.16 Put each sample on the analytical nitrogen evaporator until dry, approximately 30 to 60 minutes.

12.11 Add 0.5 ml of methanol to each centrifuge tube using a graduated pipette. If excessive residue is present, add the methanol and allow the extract to sit for 30 minutes prior to vortexing.

12.17 Vortex mix for 30 seconds.

12.18 Label the autovial with the study number, animal number and gender, sample timepoint, matrix, final solvent, extraction date, fluorochemical components, extraction type, vial file archive number, and analyst(s) performing the extraction.

12.19 Attach a 0.2 µm nylon mesh filter to a 3 cc syringe and transfer the sample to this syringe. Filter into a 1.5 ml glass autovial or low-volume autovial when necessary.

12.20 Cap and store extracts at room temperature or refrigerated at approximately 4 °C until analysis.

12.21 Complete the extraction worksheet, attached to this document, and tape in the study notebook or include in study binder, as appropriate.

13.0 Data Analysis and Calculations

13.1 Calculations

13.1.1 Calculate actual concentrations of PFOS, or other applicable fluorochemical, in calibration standards using the following equation:

$$\frac{\text{ml of standard} \times \text{concentration of standard (µg /ml)}}{\text{ml of standard} + \text{ml of surrogate standard} + \text{initial matrix volume (ml)}} =$$

Final Concentration (µg/ml) of PFOS in matrix

14.0 Method Performance

14.1 The method detection limit (MDL) is analyte and matrix specific. Refer to MDL report for specific MDL and limit of quantitation (LOQ) values (see **Attachment B**).

14.2 The following quality control samples are extracted with each batch of samples to evaluate the quality of the extraction and analysis.

14.2.1 Method blanks and matrix blanks.

14.2.2 Matrix spike and matrix spike duplicate samples to determine accuracy and precision of the extraction.

14.2.3 Continuing calibration check samples to determine the continued accuracy of the initial calibration curve.

14.3 Refer to section 14 of ETS-8-97.0 for method performance criteria.

15.0 Pollution Prevention and Waste Management

15.1 Human and monkey sample waste is disposed in infectious biohazard waste containers, all other sample waste is disposed in noninfectious biohazard waste containers. Flammable solvent waste is disposed in high BTU containers. Used glass pipette waste is disposed in broken glass containers located in the laboratory.

16.0 Records

16.1 Complete the extraction worksheet attached to this method, and tape in the study notebook or include in the 3-ring study binder, as appropriate.

17.0 Tables, Diagrams, Flowcharts, and Validation Date

17.1 Attachment A, Extraction worksheet

17.2 Attachment B, MDL/LOQ values and summary

17.3 Attachment C, Calibration standard concentration worksheet

17.4 Attachment D, Sample weight/volume worksheet

18.0 References

18.1 The validation report associated with this method is **FACT-TOX-131, W2067.**

19.0 Affected Documents

19.1 ETS-8-97.0, "Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemical Compounds in Urine Extracts Using HPLC-Electrospray Mass Spectrometry/Mass Spectrometry"

20.0 Revisions

Revision Number	Reason For Revision	Revision Date

Extraction Worksheet ETS-8-96.0

Study #_____ Matrix_____ Box #_____ Wk/Day_____ DateSpiked/Analyst	Surrogate Std Approx. ppm Actual ppm #_____	FC-Mix approx. 0.5 ppm actual ppm #_____	FC-Mix approx. 5 ppm actual ppm #_____	FC-Mix approx. 50 ppm actual ppm #_____	Comments
CCV					
MS					
MSD					
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	

Blank	Std #	amount = ml
Urine Extraction Method	:	**Date & Initials**
Vortex 15 sec.		
Pipette Matrix , spike with appropriate surrogate or FC-Mix	Volume	ml
Pipette 1 ml of 0.5 M TBA, pH 10. pH = _____	Std. #_____	
Pipette 2 ml of 0.25 Na_2CO_3/0.25M $NaHCO_3$ buffer	Std. #_____	
Dispense 5 ml of methyl-t-butyl ether	TN-A-_____	
Shake 20 min.	Shaker speed:	
Centrifuge 20-25 min.	Centrifuge speed:	
Remove a 4 ml aliquot of organic layer		
Put on Nitrogen Evaporator to dryness	Temperature:	
Add methanol Volume_____ ml	TN-A-_____	
Vortex 30 sec.		
Filter using a 3cc B-D syringe with a 0.2µm filter into an autosample vial		

Cont. Cal. Verifications used same matrix as for std curve.

MDL/LOQ values for human urine

Compound	MDL (ppb)	LOQ (ppb)	Linear Calibration Range (LCR)
PFOS	4.6	14.7	15 ppb - 1500 ppb (in final MeOH extract)
POAA	22.9	72.9	50 ppb – 1500 ppb (in final MeOH extract)
PFOSA	n/d	n/d	25 ppb - 1000 ppb (in final MeOH extract)
PFOSAA	n/d	n/d	25 ppb - 1000 ppb (in final MeOH extract)
EtFOSE-OH	n/d	n/d	25 ppb - 1000 ppb (in final MeOH extract)
M556	n/d	n/d	25 ppb - 1000 ppb (in final MeOH extract)
M570	n/d	n/d	25 ppb - 1000 ppb (in final MeOH extract)

n/d = not determined

NOTE: to calculate MDL, LOQ, and LCR values in ug/ml of urine divide the above values by 4.

MDL/LOQ values in rat and monkey urine were not statistically determined. Two curves in each of these matrices were extracted and analyzed with the human urine curves to determine equivalence. Responses in the rat and monkey were similar to the human responses, therefore, their MDL and LOQ are assumed to be similar to the values determined for human urine.

If a suitable amount of clean, control matrix is available, samples will be evaluated versus a curve extracted from urine originating from the same species as the specimens.

Please see LOQ Summary and MDL study in **FACT-TOX-131, W2067** for further information.

Compound: PFOS

Human Urine	Prepared range of standards (ppb) (ng/ml)	LCR from curve (ppb) (ng/ml)	% Recovery Range	RSD Range
Full Range	n/d	n/d	n/d	n/d
Low Curve	n/d	n/d	n/d	n/d
High curve	n/d	n/d	n/d	n/d
1/X	2.5ppb – 1500 ppb	15 ppb – 1500 ppb	75-116	+/- 30%

Compound: POAA

Human Urine	Prepared range of standards (ppb) (ng/ml)	LCR from curve (ppb) (ng/ml)	% Recovery Range	RSD Range
Full Range	n/d	n/d	n/d	n/d
Low Curve	n/d	n/d	n/d	n/d
High curve	n/d	n/d	n/d	n/d
1/X, quadratic	2.5ppb – 1500 ppb	50 ppb – 1500 ppb	86-105	+/- 30%

Ion Pair Standard Curves – Urine

Prep date(s): **Standard number:**
Analyte(s): **Equipment number:**
Sample matrix: **Final solvent and TN:**
Blank fluid/identifier:
Method/revision: **Box Number:**
Target analyte(s):
FC mix std approx. 0.500 ppm:
FC mix std approx. 5.00 ppm:
FC mix std approx. 50.0 ppm:
Surrogate std approx. 100 ppm:

Actual concentrations of standards in the FC mix

PFOS Std conc ug/ml	PFOSA Std conc ug/ml	PFOSAA Std conc ug/ml	EtFOSE Std conc ug/ml	POAA Std conc ug/ml	M556 Std conc ug/ml	M570 Std conc ug/ml	All Am't spiked ml	All Final vol ml
0.500	0.501	0.500	0.501	0.499	0.500	0.501	0.005	2.0075
0.500	0.501	0.500	0.501	0.499	0.500	0.501	0.010	2.0125
0.500	0.501	0.500	0.501	0.499	0.500	0.501	0.025	2.0275
5.00	5.01	5.00	5.01	4.99	5.00	5.01	0.005	2.0075
5.00	5.01	5.00	5.01	4.99	5.00	5.01	0.010	2.0125
5.00	5.01	5.00	5.01	4.99	5.00	5.01	0.025	2.0275
50.0	50.1	50.0	50.1	49.9	50.0	50.1	0.005	2.0075
50.0	50.1	50.0	50.1	49.9	50.0	50.1	0.0075	2.0100
50.0	50.1	50.0	50.1	49.9	50.0	50.1	0.010	2.0125
50.0	50.1	50.0	50.1	49.9	50.0	50.1	0.015	2.0130

Calculated concentrations of standards in the sample matrix

PFOS Final conc ng/ml	PFOSA Final conc ng/ml	PFOSAA Final conc ng/ml	EtFOSE Final conc ng/ml	POAA Final conc ng/ml	M556 Final conc ng/ml	M570 Final conc ng/ml	Surrogate Std conc ng/ml	All Am't spiked ml
1.25	1.25	1.25	1.25	1.24	1.25	1.25	100	0.0025
2.48	2.49	2.48	2.49	2.48	2.48	2.49		
6.17	6.18	6.17	6.18	6.15	6.17	6.18	**Surrogate** Final conc ng/ml	
12.5	12.5	12.5	12.5	12.4	12.5	12.5		
24.8	24.9	24.8	24.9	24.8	24.8	24.9		
61.7	61.8	61.7	61.8	61.5	61.7	61.8	125	
125	125	125	125	124	125	125		
187	187	187	187	186	187	187		
248	249	248	249	248	248	249		
372	372	372	372	371	372	372		

Calculated concentrations of standards in methanol extract (0.5 ml final volume)

PFOS Final conc ng/ml	PFOSA Final conc ng/ml	PFOSAA Final conc ng/ml	EtFOSE Final conc ng/ml	POAA Final conc ng/ml	M556 Final conc ng/ml	M570 Final conc ng/ml	Surrogate Std conc ng/ml	All Am't spiked ml
5.00	5.01	5.00	5.01	4.99	5.00	5.01	100	0.0025
10.0	10.0	10.0	10.0	10.0	10.0	10.0		
25.0	25.1	25.0	25.1	25.0	25.0	25.1	**Surrogate** Final conc ng/ml	
50.0	50.1	50.0	50.1	49.9	50.0	50.1		
100	100	100	100	100	100	100		
250	251	250	251	250	250	251	500	
500	501	500	501	499	500	501		
750	752	750	752	749	750	752		
1000	1002	1000	1002	998	1000	1002		
1500	1503	1500	1503	1497	1500	1503		

Prep Date(s):
Analyst(s):
Sample Matrix:
Method/Revision:
Target Analyte(s):

Study Number:
Equipment Number:
Final Solvent & TN Number:
Matrix Blank/Identifier:
Box:

Sample ID	Initial Wt./Vol. g/mL/L			Comments

Summary of method:

Notes: