

Study Title

Characterization Study of PFOA (lot #332),
Primary Standard – Test Control Reference #TCR-99030-030
PHASE: SOLUBILITY DETERMINATION

Data Requirement

40 CFR 160.105(b)

Author

Mark E. Ellefson

Phase Completion Date

Date of Signing

Performing Laboratory

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Project Identification

3M Environmental Laboratory Study # FACT-TCR-006 (LIMS #E00-1763)
Centre Analytical Laboratories Study # 023-034

Total Number of Pages

29

GLP COMPLIANCE STATEMENT

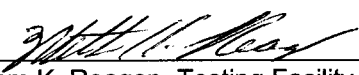
Study Title: Characterization Study of PFOA (lot #332), Primary Standard – Test Control
Reference #TCR-99030-030, PHASE: SOLUBILITY DETERMINATION

Study Identification Number: FACT-TCR-006, Centre Analytical Laboratories Study # 023-034

This phase of the study was conducted in compliance with Environmental Protection Agency (EPA) Good Laboratory Practice (GLP) Standards 40 CFR 160 with the exceptions in the bulleted list below.

- None


Mark E. Ellefson, Principal Analytical Investigator
03/30/01
Date


William K. Reagen, Testing Facility Management
03/30/01
Date

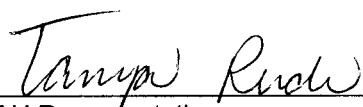
QUALITY ASSURANCE STATEMENT

Study Title: Characterization Study of Ammonium Perfluorooctanoate (PFOA), Primary Standard # TCR-99030-030, PHASE: SOLUBILITY DETERMINATION

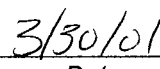
Study Identification Number: FACT-TCR-006, Centre Analytical Laboratories Study # 023-034

This phase of the 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 laboratory management.

Inspection Dates	Phase	Date Reported to	
		Management	Study Director
7/17/00	Solubility Sample Preparation	7/17/00	7/17/00
7/17/00	In Phase Sample Dilutions	7/17/00	7/17/00
1/15 – 1/16/01	Data Audit	01/19/01	01/19/01
01/31/01-02/02/01	Draft Report	2/02/01	2/02/01



QAU Representative



Date

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STUDY INFORMATION

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Experimental Dates

Sample Analysis Initiation: 17 July 2000
Sample Analysis Completion: 12 September 2000

Archives

Reserve samples of the reference standards will be maintained by the 3M Environmental Laboratory. All study data for this phase and a copy of the phase report are archived by the 3M Environmental Laboratory.

SUMMARY

Solubility of PFOA in water is a significant parameter because the spatial and temporal movement (mobility) of a substance through the environment is largely determined by its solubility in water. Water soluble substances readily gain access to humans and other living organisms; furthermore the knowledge of the solubility in water is a prerequisite for testing biological degradation, bioaccumulation, and other tests. Solubility of PFOA in methanol and acetone is a significant parameter for general laboratory knowledge in the preparation and analysis of PFOA. The approximate solubility of PFOA was determined by performing a solubility screen test (ETS-8-170.1). PFOA was determined to be soluble at concentrations greater than 10% (mass/volume) in water, methanol and acetone at 23 °C. According to the screen test, if the test substance is determined to be soluble at concentrations greater than 10% (mass/volume) then no additional testing is required.

Results are presented in table 1:

Table 1. Summary Table of Solubility of PFOA TCR-99030-030 in Water, Methanol and Acetone.

SOLVENT	PFOA SOLUBILITY (MASS/VOLUME)
Water	> 10%
Methanol	> 10 %
Acetone	> 10%

The solubility of PFOA TCR-99030-030 was determined to be greater than 10% (mass/volume) in water, methanol and acetone at 23 °C.

PURPOSE

The purpose of this phase of the study was to determine the solubility of PFOA in water, methanol, and acetone as part of the characterization of this test substance.

TEST SUBSTANCE

PFOA, Test Control Reference #TCR-99030-030

Table 2. Characterization of the Test Substance

TEST SUBSTANCE	PFOA
Source	3M Specialty Materials, Bldg. 236-1B-10
Expiration Date	01-01-2010
Storage Conditions	Frozen
3M Laboratory Identification Number	TCR-99030-030
Physical Description	White Powder
Purity	* 95.0%

**The purity of the PFOA used in this study was determined to be 95.0%. The same lot/source of compound was used throughout the entire study.*

TEST SYSTEM

Table 3. Description of Test Systems Used in this Study

	WATER	METHANOL	ACETONE
Source	Millipore System	B & J Brand	EM Science
Expiration Date	NA	2005	2005
Storage Conditions	Ambient	Ambient, Flammable Cabinet	Ambient, Flammable Cabinet
3M Laboratory Identification Number	NA	TN-A: 4466	TN-A: 4616
Purity/Grade	ASTM Type I	HPLC Grade	HPLC Grade

METHOD SUMMARIES

The solubility determination of PFOA TCR-99030-030 in water, methanol, and acetone was performed according to United States Environmental Protection Agency OPPTS 830.7840, "Water Solubility: Column Elution Method; Shake Flask Method" and OECD 105, "Water Solubility" Guidelines as implemented by 3M Laboratory ETS-8-170.1 "Solubility Screen Test: Approximate Solubility Determination of a Test Substance in Various Solvents".

The Solubility Screen Test Method was used to determine the solubility range of PFOA TCR-99030-030 in water, methanol, and acetone. Incremental amounts of water, methanol, or

acetone were added to PFOA neat material and a qualitative determination of the solubility was made. The screen test indicated the solubility of PFOA to be greater than 10% (mass/volume) in water, methanol, and acetone at 23 °C. A detailed description of the Solubility Screen Test method used in this study is located in Attachment A.

PREPARATORY METHODS

- **ETS-8-170.1 "Solubility Screen Test: Approximate Solubility Determination of a Test Substance in Various Solvents."** This method is a pre-requisite to the shake flask and column elution methods and provides an estimate of the solubility of the test substance in the solvent of choice. If the test substance is determined to be soluble at concentrations greater than 10% (mass/volume) then no additional testing is required. Approximately 10 mg PFOA was weighed into a glass vial, and 0.1 mL of the appropriate solvent was added. Following solvent addition, the vial was shaken, sonicated, and observed for particulates. The solubility screen test estimated the solubility of PFOA to be greater than 10% (mass/volume) in water, methanol and acetone at 23 °C. The solubility screening information was used to determine that no additional solubility testing was necessary.

Table 4. Summary of Sample Preparation.

STEP	SAMPLE PREPARATION	DATE(S) PERFORMED
1	10 mg test substance is weighed into tared 2.5 mL – 4.0 mL glass screw-capped vial.	9/08/00
2	0.1 mL of test solvent (water, methanol or acetone) is added to glass screw-capped vial.	9/08/00
3	Following the addition of test solvent, the vial is capped, shaken vigorously (or vortexed), and the mixture is sonicated. Record mixing and sonication times on the worksheet.	9/08/00
4	Following sonication, the sample is checked visually for undissolved particles of the test substance. If all of the test substance is dissolved, the solubility is determined to be greater than 10% (mass/volume) and no additional solubility testing is required. If a portion of the test substance remains undissolved, additional dilutions are performed using the appropriate solvent.	9/08/00

PROTOCOL DEVIATIONS

There were no deviations from the original protocol for this phase.

DATA SUMMARY, ANALYSES, AND RESULTS

ANALYTICAL RESULTS

- **Specificity (according to OPPTS 830.7840):** The solubility determination as stated in the EPA Guidelines follows. This method should only be applied to:
 - Pure substance. This study shows a purity of 95.0%
 - Substances that are stable in water. Hydrolytic studies have been conducted at the 3M Environmental Laboratory according to the United States Environmental Protection Agency Guidelines, OPPTS 830.2110 "Hydrolysis as a Function of pH." Refer to 3M Environmental Laboratory Report No. W1878 (currently in submittal) for study conclusions and results.

DATA SUMMARY

Table 5. Summary Table of Solubility of PFOA TCR-99030-030 in Water, Methanol and Acetone.

SOLVENT	PFOA SOLUBILITY (MASS/VOLUME)
Water	> 10%
Methanol	> 10 %
Acetone	> 10%

The solubility of PFOA TCR-99030-030 in water, methanol and acetone is greater than 10% (mass/volume) at 23 °C.

Attachment B contains solubility screening test sample preparation worksheets.

STATISTICAL METHODS AND CALCULATIONS

No statistical methods or calculations were required. The data was reported as the solubility of PFOA being greater than 10% (mass/volume) at 23 °C in water, methanol, and acetone as determined by the initial screening method.

STATEMENT OF CONCLUSION

Under the conditions of the present study, the solubility of PFOA TCR# 99030-030 in ASTM Type I water, methanol and acetone is greater than 10% (mass/volume) at 23 °C.

REFERENCES

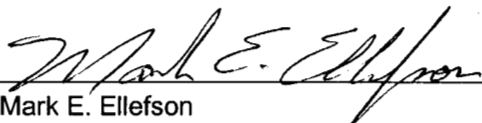
1. Fate, Transport and Transformation Test Guidelines Office of Prevention, Pesticides and Toxic Substances (OPPTS) 830.7840 Water Solubility: Column Elution Method; Shake Flask Method. EPA 712-C-96-041, August 1996.
2. OECD 105: Water Solubility. Adopted 27 July, 1995.

LIST OF ATTACHMENTS

- Attachment A: Solubility Screening Method ETS-8-170.1 "Solubility Screen Test: Approximate Solubility Determination of a Test Substance in Various Solvents".
- Attachment B: Solubility Screening Test Sample Preparation Worksheets

SIGNATURE PAGE

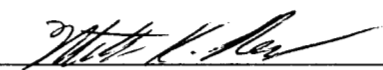
We certify that this report is a true and complete representation of the data for this phase of the study:



Mark E. Ellefson
Principal Analytical Investigator

03/30/01

Date



William K. Reagan
Laboratory Manager

03/30/01

Date

Attachment A: Solubility Screening Method

3M ENVIRONMENTAL LABORATORY

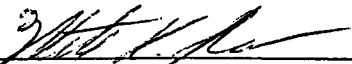
SOLUBILITY SCREEN TEST: APPROXIMATE SOLUBILITY DETERMINATION OF A TEST SUBSTANCE IN VARIOUS SOLVENTS

Method Number: ETS-8-170.1

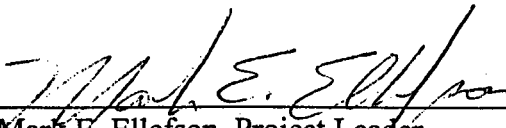
Adoption Date: 09/08/00

Effective Date: 03/14/01

Approved By:


William K. Reagen, Laboratory Manager

03/14/01
Date


Mark E. Ellefson, Project Leader

03/14/01
Date

1.0 SCOPE AND APPLICATION

- 1.1 Purpose.** According to methods set forth by the United States Environmental Protection Agency (US EPA) and the Organization for the Economic Cooperation and Development (OECD) a preliminary study, which will be outlined in this method, serves as a prerequisite to performing solubility testing via the Column Elution Method and Shake Flask Methods. The EPA and OECD solubility testing guidelines state that if the preliminary solubility determination test indicates a solubility of $> 10^{-2}$ g/L (10 ppm), the Shake Flask Method (ETS-8-172.0) is to be used. If preliminary testing indicates a solubility of $< 10^{-2}$ g/L (10 ppm) the Column Elution Method (ETS-8-171.0) will be used to determine the solubility in the solvent of interest. Additionally, due to the tendency of certain compounds to be highly soluble in particular solvents, no further solubility determination will be necessary for compounds having solubility greater than 10% (mass/volume).

Exact Copy of Original ^{1.2} Compatible Analytes. Test substance and degradation products for solubility testing.

ETS-8-170.1

Page 1 of 9

- 1.3 Acceptable Matrices.** Aqueous (e.g. Milli-Q water, 0.01M CaCl_2), Acetone, Methanol, or other solvent(s) of interest.

2.0 SUMMARY OF METHOD

- 2.1 Preliminary Solubility Determination in solvent.** Weigh approximately 10 mg of test substance into a 2.5 mL-4 mL glass screw-capped vial. Add test solvent in varying increments to the vial. After each solvent addition, vortex mix for approximately 15 sec., and sonicate 2-5 minutes. Make observations of the solution and visually confirm whether or not particles are present; it may be necessary to allow the solution to sit for up to 5 minutes. Add test solvent in a stepwise fashion up to 2 mL. If the substance fully dissolves after the first solvent addition of 100 μL , the test substance shall be considered highly soluble, and no further testing is required. If there are still undissolved particles in the vial, weigh 10 mg of test substance into a 100 mL glass stoppered volumetric flask or graduated cylinder. Add test solvent until visual confirmation of the substance dissolution has been achieved. Record all observations on a standardized preparation sheet or logbook. Once 100 mL solvent has been reached, the approximate concentration is at 100 $\mu\text{g/mL}$. If the solubility limit has not yet been achieved, weigh approximately 10 mg of test substance and transfer to a 1 L graduated cylinder/volumetric flask (concentration is approximately 10 ppm). The container should be sonicated and allowed to sit overnight to allow for maximal dissolution. If the undissolved particles are still observed after sitting > 12 hours, the column elution method will be utilized (ETS-8-171.0). If the test substance dissolves in 1 L or less of solvent, the shake flask method (ETS-8-172.0) will be used to determine the solubility.

3.0 DEFINITIONS

- 3.1 Test substance.** A liquid or solid material for which the relative solubility in a specified solvent will be determined.
- 3.2 Test solvent.** The matrix to which the test substance of interest is introduced. The test solvent may include but is not limited to aqueous matrices, including ASTM Type I Water and various salt matrices (e.g. 0.01M CaCl_2 (aq)); and organic solvent matrices, including methanol, and acetone.

4.0 WARNINGS AND CAUTIONS

4.1 Health and Safety Warnings:

- 4.1.1** Wear the proper lab attire for all parts of these procedures. Wear gloves and eye protection at all times.
- 4.1.2** Handle all solvents in a hood for all parts of the described sample preparation procedure.
- 4.1.3** For potential hazards of each chemical used, refer to material safety data sheets, packing materials, and 3M Environmental Laboratory's Chemical Hazard Review.
- 4.1.4** No mouth pipetting is allowed.

4.2 Cautions:

- 4.2.1 Glassware in which standards are prepared should be triple rinsed with acetone and methanol to reduce the possibility of accidental contamination.

5.0 INTERFERENCES

- 5.1 Impurities may significantly affect the solubility of the test substance. The purity of the test substance should be known and documented prior to starting the screening procedure.

6.0 EQUIPMENT

- 6.1 Analytical balance sensitive to 0.1 mg.
6.2 Vortex mixer.
6.3 Sonicating device.

7.0 SUPPLIES AND MATERIALS

- 7.1 Disposable glass graduated pipettes, 1 mL to 100 mL.
7.2 Disposable glass Pasteur pipettes and rubber bulbs.
7.3 Glass beakers, various sizes.
7.4 2.5 mL-4 mL glass screw-top vial.
7.5 Glass volumetric flasks, 10 mL to 1000 mL.
7.6 10 µL-1000 µL Pipetteman™ manual pipettor and plastic pipette tips, or equivalent.

8.0 REAGENTS AND STANDARDS

- 8.1 **Methanol** (MeOH), HPLC/SPEC/GC grade from EM Science, or equivalent.
8.2 **Acetone**, HPLC/SPEC/GC grade from EM Science or equivalent.
8.3 **Water**, ASTM Type I, or equivalent.
8.4 **Calcium Chloride Dihydrate**, Approximately 99% or better, from Sigma or equivalent.
8.5 **0.01 M CaCl₂**, A 0.01 M CaCl₂ stock solution is prepared by weighing 1.5 g CaCl₂ Dihydrate in a weigh boat and transferring to a 1 L volumetric flask and diluting to the mark with Milli-Q™ water.
8.6 **Test substance of known purity.**

9.0 SAMPLE HANDLING

- 9.1 Record times of initial preparation and dilution on a sample preparation sheet or logbook.
9.2 Once the preliminary testing has been completed and the appropriate information is extracted, the screen test samples should be disposed into the proper waste stream.

10.0 QUALITY CONTROL

10.1 Not applicable.

11.0 CALIBRATION AND STANDARDIZATION

11.1 The compounds of interest must be of known characterization according to laboratory specifications.

11.2 All equipment used, such as the analytical balance, should be calibrated daily prior to use.

12.0 PROCEDURES

12.1 Solubility screen.

12.1.1 Weigh $10 \text{ mg} \pm 1 \text{ mg}$ of test substance into a 4 mL glass screw-top vial or equivalent. Record the weight (refer to attachment A for an example of a standardized prep sheet).

12.1.2 Add solvent (e.g. water, methanol, acetone) according to the table below to the test substance.

<i>4 mL vial or equivalent</i>	Solvent addition 1	Solvent addition 2	Solvent addition 3	Solvent addition 4
Total volume solvent added (mL)...	0.1	0.5	1	2
Approximate Solubility ($\mu\text{g/mL}$)...	100000	200000	10000	5000

12.1.3 Following addition of solvent, vortex mix approximately 15 seconds, sonicate 2-5 minutes. Visually check for undissolved particles. If undissolved test substance remains, continue adding solvent according to the above chart. It may be necessary to allow the solution to settle for up to 5 minutes before making observations. Record observations on the standardized prep sheet.

12.1.4 If all particles dissolve, then estimate the approximate solubility and document the concentration. Because the concentration is $>10 \mu\text{g/mL}$, the shake flask method (ETS-8-172.0) will be used to determine the accurate solubility point. However, if the test substance dissolves after the *first* solvent addition of $100 \mu\text{L}$ the solubility is determined to be $>10\%$ (mass/volume). The test substance shall be regarded as "highly soluble," and no further testing is required.

12.1.5 If the test substance did not dissolve in the 2 mL of solvent, weigh $10 \text{ mg} \pm 1 \text{ mg}$ of test substance into a 100 mL glass volumetric flask or equivalent. Record the weight (refer to attachment A for an example of a standardized prep sheet).

12.1.6 Add solvent according to the table below to the test substance to deliver the appropriate amount of solvent. As described in 12.1.3, vortex mix, sonicate, and allow the test solution to settle. Record all observations and procedures on the preparation sheet.

<i>100 mL volumetric flask or equivalent</i>	Solvent addition 1	Solvent addition 2	Solvent addition 3
Total volume solvent added (mL)...	10	50	100
Approximate Solubility (µg/mL)...	1000	200	100

12.1.7 If the solubility point is reached at or prior to the complete addition of 100 mL, the concentration estimation at which the substance is soluble should be documented and shall be used as a starting point for the shake flask method (ETS-8-172.0).

12.1.8 If the 100 mL solvent level has been reached without complete dissolution of test substance, weigh out 10 mg ± 1 mg test substance and transfer to a 1 L volumetric flask, graduated cylinder, or equivalent. Dilute to 1 L with test solvent. The approximate concentration of test substance is at 10 µg/mL. The flask should be allowed to sit 12-24 hours to allow for maximal dissolution. If the undissolved particles are still observed, the column elution method will be utilized (ETS-8-171.0). If no visible particulates are observed, the shake flask method (ETS-8-172.0) shall be used to determine the solubility.

13.0 DATA ANALYSIS AND CALCULATIONS

13.1 The solubility determination in this method is qualitative/semi-quantitative.

13.2 The point to which the substance dissolves in solvent is confirmed visually. The solubility point is therefore a qualitative determination.

13.3 The concentration estimation is semi-quantitative in that the approximate concentration is calculated by the following equation:

$$c = a/b$$

Where:

c= the semi-quantitative concentration µg/mL,

a= amount of substance weighed out (µg), and

b= the approximate amount of solvent added (mL).

14.0 METHOD PERFORMANCE

14.1 Limitation of data. The accuracy to which the solubility is determined is subject to a large margin of error due to the way in which the solvent is added to the solute. Since large and varying increments of solvent are being added to the solute, this error margin must be considered when reporting the concentration estimation.

14.2 The data obtained through this study is an estimation only and should be treated as a qualitative estimation of the solubility of test substance in a given solvent.

- 14.3 For an accurate measurement of the substance solubility concentration, the shake flask method, or column elution method will be used.

15.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

- 15.1 Dispose of sample waste by placing in high or low BTU containers as appropriate. Use broken glass containers to dispose of glass pipettes.

16.0 RECORDS

- 16.1 Sign and date all observations and calculations.

17.0 ATTACHMENTS

- 17.1 **Attachment A**-Example of a Standardized Sample Preparation Sheet: Solubility Screen Prep Sheet.

18.0 REFERENCES

- 18.1 Organization for Economic Cooperation and Development. OECD Guideline for Testing of Chemicals. Water Solubility -OECD Guideline 105: pp. 1-7, Adopted 1995
- 18.2 United States Environmental Protection Agency. OPPTS 830.7860 Water Solubility (Generator Column Method). Prevention, Pesticides and Toxic Substances: Fate, Transport and Transformation Test Guidelines. EPA 712-C-96-042: pp. 1-17, 1996
- 18.3 United States Environmental Protection Agency. OPPTS 830.7840 Water Solubility: Column Elution Method; Shake Flask Method. Prevention, Pesticides and Toxic Substances: Fate, Transport and Transformation Test Guidelines. EPA 712-C-96-041: pp. 1-12, 1996
- 18.4 3M Environmental Laboratory Method ETS-8-171.0, "Column Elution Method: Solubility Determination of Test Substance in Various Solvents."
- 18.5 3M Environmental Laboratory Method ETS-8-172.0, "Shake Flask Method: Solubility Determination of Test Substance in Various Solvents."

19.0 AFFECTED DOCUMENTS

- 19.1 ETS-8-171.0, "Column Elution Method: Solubility Determination of Test Substance in Various Solvents."
- 19.2 ETS-8-172.0, "Shake Flask Method: Solubility Determination of Test Substance in Various Solvents."

20.0 REVISIONS

<u>Revision Number</u>	<u>Reason For Revision</u>	<u>Revision Date</u>
1	It was desirable to make the method more universally applicable by removing references to specific test substances.	03/13/01

Attachment A: Example Preparation Worksheet, page 1 of 2

3M Environmental Laboratory SOLUBILITY SCREEN

GLP Study Number: _____ Sample preparation worksheet

Test Substance: _____ ID#: _____ Date: _____

Source: _____

Solvent: _____ ID#: _____

Source: _____

Step

1 Weigh approximately 10 mg $\pm$ 1 mg of test substance into a 2.5 mL-4 mL glass screw-top vial.

Weight of test substance: _____ mg

Balance ID: _____

Date/Initials: _____

2nd replicate optional: _____ mg

Thermometer ID: _____

2 Add test solvent according to the table below. Following each addition of solvent, shake vigorously/vortex, and sonicate the mixture and visually check for undissolved particles.

Solubility data		volume added (mL)/ approx conc. after addition (ug/mL)	Sample Prep	OBSERVATIONS/NOTES
Analyst _____	Date/Time _____ / _____	Step 2-1	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes -continue to step 2-2 NO - The solution is at 10%, and is considered "infinitely soluble." No further solubility testing is required.
	Total volume solvent added (mL) 0.1			
	Approximate Solubility (ug/mL) 100,000			
Analyst _____	Date/Time _____ / _____	Step 2-2	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL) 0.5			
	Approximate Solubility (ug/mL) 20,000			
Analyst _____	Date/Time _____ / _____	Step 2-3	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL) 1			
	Approximate Solubility (ug/mL) 10,000			
Analyst _____	Date/Time _____ / _____	Step 2-4	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL) 2			
	Approximate Solubility (ug/mL) 5,000			

Conclusions:

Did the substance dissolve in 2 mL or less of solvent?

Yes -Approximate concentration at which the test substance is soluble in solvent: _____ ug/ml

The solubility of the test substance is to be determined via the shake flask method.

or **No** Continue to page two for further testing.

Notes/ additional comments:

Attachment A Cont.: Example Preparation Worksheet, page 2 of 2

3M Environmental Laboratory SOLUBILITY SCREEN

GLP Study Number: _____ Sample preparation worksheet

Test Substance: _____ ID#: _____ Date: _____
Source: _____

Solvent: _____ ID#: _____
Source: _____

Step (continued from page 1)

***Did the test substance dissolve in 2 mL? Yes/No If YES, the test is complete, and there is no need to continue with the screen test. If NO, continue to step 3 to determine the approximate solubility of the substance.

3 Weigh approximately 10 mg $\pm$ 1 mg of test substance into a 100 mL graduated stoppered cylinder/volumetric flask.

Weight of test substance: _____ mg Balance ID: _____ Date/Initials: _____
2nd replicate optional: _____ mg Thermometer ID: _____

4 Add test solvent according to the table below. Following each addition of solvent, shake vigorously/vortex, and sonicate the mixture and visually check for undissolved particles.

Solubility data		volume added (mL)/ approx conc. after addition (ug/mL)	Sample Prep	OBSERVATIONS/NOTES
Analyst _____	Date/Time _____ / _____	Step 4-1	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)... 10			
	Approximate Solubility (ug/mL)... 1,000			
Analyst _____	Date/Time _____ / _____	Step 4-2	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)... 50			
	Approximate Solubility (ug/mL)... 200			
Analyst _____	Date/Time _____ / _____	Step 4-3	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)... 100			
	Approximate Solubility (ug/mL)... 100			

5 Weigh approximately 10 mg $\pm$ 0.5 mg of test substance into a 1 L graduated stoppered cylinder/volumetric flask/or equivalent.

Weight of test substance: _____ mg Balance ID: _____ Date/Initials: _____

Analyst _____	Date/Time _____ / _____	Step 5-1	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)... 1000			
	Approximate Solubility (ug/mL)... 10			

Conclusions:

Did the substance dissolve in 1000 mL or less of solvent?

Yes -Approximate concentration at which the test substance is soluble in solvent: _____ ug/ml

The solubility of the test substance is to be determined via the shake flask method.

or No -The solubility of the test substance is to be determined via the column elution method.

Notes/ additional comments:

Attachment B: Solubility Screening Test Sample Preparation
Worksheets

3M Environmental Laboratory SOLUBILITY SCREEN

GLP Study Number: TCR-006

Sample preparation worksheet

Test Substance: PFOA

ID#: TCR-99030-30

Date: 8 Sept 00 cnc

Source: 3M Internal

Solvent: Water

ID#: N/A

Source: Milli-Q, 18.2 MΩ

Step

1 Weigh approximately 10 mg $\pm$ 0.5 mg of test substance into a 2.5 mL-4 mL glass screw-top vial.

Weight of test substance: 10.33 mg

Balance ID: 4917

Date/Initials: 8 Sept 00 cnc

2nd replicate optional: _____ mg

Thermometer ID: Therm 04

2 Add test solvent according to the table below. Following each addition of solvent, shake vigorously/vortex, and sonicate the mixture and visually check for undissolved particles.

Solubility data		volume added (mL)/ approx conc. after addition (ug/mL)	Sample Prep	OBSERVATIONS/NOTES
Analyst cnc	Date/Time 8 Sept 00 1:54	Step 2-1 Total volume solvent added (mL)... 0.1 Approximate Solubility (ug/mL)... 100,000	Flask vortex-mixed? Yes/No _____ Flask sonicated? Yes/No _____ Flask allowed to settle? Yes/No Time 15 min	Is there solute still present? Yes-continue to step 2-2 NO The solution is at 10%, and is considered "infinitely soluble." No further solubility testing is required.
Analyst	Date/Time	Step 2-2 Total volume solvent added (mL)... 0.5 Approximate Solubility (ug/mL)... 20,000	Flask vortex-mixed? Yes/No _____ Flask sonicated? Yes/No _____ Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
Analyst	Date/Time	Step 2-3 Total volume solvent added (mL)... 1 Approximate Solubility (ug/mL)... 10,000	Flask vortex-mixed? Yes/No _____ Flask sonicated? Yes/No _____ Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
Analyst	Date/Time	Step 2-4 Total volume solvent added (mL)... 2 Approximate Solubility (ug/mL)... 5,000	Flask vortex-mixed? Yes/No _____ Flask sonicated? Yes/No _____ Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No

Conclusions:

Did the substance dissolve in 2 mL or less of solvent?

Yes - Approximate concentration at which the test substance is soluble in solvent: $> 100 \mu\text{g/mL}$

The solubility of the test substance is to be determined via the shake flask method.

or No Continue to page two for further testing.

Notes/ additional comments:

① 1:54 pm @ cnc
8 Sept 00

② Late entry by KLT: I Did Vortex on 9/22/00
8 Sept 00 1:17 PM

9/22/00 KLT

KLT 9/22/00

Initial
Date
Exact Copy of Original

3M Environmental Laboratory SOLUBILITY SCREEN

GLP Study Number: TCR-006

Sample preparation worksheet

Test Substance: PFOA

ID#: TCR-99030-30

Date: _____

Source: 3M Internal

Solvent: Water

ID#: _____

Source: _____

Step (continued from page 1)

***Did the test substance dissolve in 2 mL? Yes/No If YES, the test is complete, and there is no need to continue with the screen test. If NO, continue to step 3 to determine the approximate solubility of the substance.

3 Weigh approximately 10 mg $\pm$ 0.5 mg of test substance into a 100 mL graduated stoppered cylinder/volumetric flask.

Weight of test substance: _____ mg

Balance ID: _____

Date/Initials: _____

2nd replicate optional: _____ mg

Thermometer ID: _____

4 Add test solvent according to the table below. Following each addition of solvent, shake vigorously/vortex, and sonicate the mixture and visually check for undissolved particles.

Solubility data		volume added (mL) approx conc. after addition (ug/mL)	Sample Prep	OBSERVATIONS/NOTES
Analyst _____	Date/Time _____ / _____	Step 4-1	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)... 10			
	Approximate Solubility (ug/mL)... 1,000			
Analyst _____	Date/Time _____ / _____	Step 4-2	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)... 50			
	Approximate Solubility (ug/mL)... 200			
Analyst _____	Date/Time _____ / _____	Step 4-3	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)... 100			
	Approximate Solubility (ug/mL)... 100			

5 Weigh approximately 10 mg $\pm$ 0.5 mg of test substance into a 1 L graduated stoppered cylinder/volumetric flask/or equivalent.

Weight of test substance: _____ mg

Balance ID: _____

Date/Initials: _____

Analyst _____	Date/Time _____ / _____	Step 5-1	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)... 1000			
	Approximate Solubility (ug/mL)... 10			

Conclusions:

Did the substance dissolve in 1000 mL or less of solvent?

Yes -Approximate concentration at which the test substance is soluble in solvent: _____ ug/ml

The solubility of the test substance is to be determined via the shake flask method.

or No -The solubility of the test substance is to be determined via the column elution method.

Notes/additional comments:

9/12/00 KLT
No further
testing req

3M Environmental Laboratory SOLUBILITY SCREEN

GLP Study Number: TCR-006

Sample preparation worksheet

Test Substance: PFOA

ID#: TCR-99030-30

Date: 8 Sept 00 CMC

Solvent: Methanol

Source: 3M Internal

ID#: TNA 4466

Source: ~~MTI~~ B & J Brand

Step

1 Weigh approximately 10 mg $\pm$ 0.5 mg of test substance into a 2.5 mL-4 mL glass screw-top vial.

Weight of test substance: 10-33 mg

Balance ID: 4917

Date/Initials: 8 Sept 00 CMC

2nd replicate optional: _____ mg

Thermometer ID: Thermoch

2 Add test solvent according to the table below. Following each addition of solvent, shake vigorously/vortex, and sonicate the mixture and visually check for undissolved particles.

Solubility data		volume added (mL)/ approx conc. after addition (ug/mL)	Sample Prep	OBSERVATIONS/NOTES
Analyst CMC	Date/Time 8 Sept 00 11:55 am	Step 2-1	Flask vortex-mixed? Yes/No Room Temp 23 °C Flask sonicated? Yes/No Time 5 min Flask allowed to settle? Yes/No Time 15 min	Is there solute still present? Yes-continue to step 2-2 NO The solution is at 10%, and is considered "infinitely soluble." No further solubility testing is required.
	Total volume solvent added (mL)...	0.1		
	Approximate Solubility (ug/mL)...	100,000		
Analyst	Date/Time / /	Step 2-2	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)...	0.5		
	Approximate Solubility (ug/mL)...	20,000		
Analyst	Date/Time / /	Step 2-3	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)...	1		
	Approximate Solubility (ug/mL)...	10,000		
Analyst	Date/Time / /	Step 2-4	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)...	2		
	Approximate Solubility (ug/mL)...	5,000		

Conclusions:

Did the substance dissolve in 2 mL or less of solvent?

Yes -Approximate concentration at which the test substance is soluble in solvent: 2107 ug/ml

The solubility of the test substance is to be determined via the shake flask method.

or No Continue to page two for further testing.

Notes/ additional comments:

① Flask was vortex-mixed on 08 Sept, 2000. The choice "yes" was initially inadvertently not circled. The choice was circled by KLT on 0122-01 as a late entry. KLT 02/07/01

3M ETS Laboratory

9-12-00 KLT

Initial

Date

Exact Copy of Original

3M Environmental Laboratory SOLUBILITY SCREEN

GLP Study Number: TCR-006

Sample preparation worksheet

Test Substance: PFOA

ID#: TCR-99030-30

Date:

Source: 3M Internal

Solvent: Methanol

ID#:

Source:

Step (continued from page 1)

***Did the test substance dissolve in 2 mL? Yes/No YES, the test is complete, and there is no need to continue with the screen test. If NO, continue to step 3 to determine the approximate solubility of the substance.

3 Weigh approximately 10 mg $\pm$ 0.5 mg of test substance into a 100 mL graduated stoppered cylinder/volumetric flask.

Weight of test substance: _____ mg

Balance ID: _____

Date/Initials: _____

2nd replicate optional: _____ mg

Thermometer ID: _____

4 Add test solvent according to the table below. Following each addition of solvent, shake vigorously/vortex, and sonicate the mixture and visually check for undissolved particles.

Solubility data		volume added (mL)/ approx conc. after addition (ug/mL)	Sample Prep	OBSERVATIONS/NOTES
Analyst _____	Date/Time _____ / _____	Step 4-1	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)...	10		
	Approximate Solubility (ug/mL)...	1,000		
Analyst _____	Date/Time _____ / _____	Step 4-2	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)...	50		
	Approximate Solubility (ug/mL)...	200		
Analyst _____	Date/Time _____ / _____	Step 4-3	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)...	100		
	Approximate Solubility (ug/mL)...	100		

5 Weigh approximately 10 mg $\pm$ 0.5 mg of test substance into a 1 L graduated stoppered cylinder/volumetric flask/or equivalent.

Weight of test substance: _____ mg

Balance ID: _____

Date/Initials: _____

Analyst _____	Date/Time _____ / _____	Step 5-1	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)...	1000		
	Approximate Solubility (ug/mL)...	10		

Conclusions:

Did the substance dissolve in 1000 mL or less of solvent?

Yes -Approximate concentration at which the test substance is soluble in solvent: _____ ug/ml

The solubility of the test substance is to be determined via the shake flask method.

or No -The solubility of the test substance is to be determined via the column elution method.

Notes/ additional comments:

9/12/00 HLT

3M Environmental Laboratory SOLUBILITY SCREEN

GLP Study Number: TCR-006

Sample preparation worksheet

Test Substance: PFOA

ID#: TCR-99030-30

Date: 8 Sept 00 CMC

Source: 3M Internal

Solvent: Acetone

ID#: TNA 4666

Source: EMS Science

Step

1 Weigh approximately 10 mg $\pm$ 0.5 mg of test substance into a 2.5 mL-4 mL glass screw-top vial.

Weight of test substance: 10.17 mg

Balance ID: 4917

Date/Initials: 8 Sept 00 CMC

2nd replicate optional: — mg

Thermometer ID: Thermo

2 Add test solvent according to the table below. Following each addition of solvent, shake vigorously/vortex, and sonicate the mixture and visually check for undissolved particles.

Solubility data		volume added (mL)/ approx conc. after addition (ug/mL)	Sample Prep	OBSERVATIONS/NOTES
Analyst Cix	Date/Time 8 Sept 00 11:55 pm	Step 2-1	Flask vortex-mixed? Yes/No Room Temp 23 °C Flask sonicated? Yes/No Time 5 min Flask allowed to settle? Yes/No Time 15 min	Is there solute still present? Yes-continue to step 2-2 NO- The solution is at 10%, and is considered "infinitely soluble." No further solubility testing is required.
Analyst	Date/Time / /	Step 2-2	Flask vortex-mixed? Yes/No Room Temp °C Flask sonicated? Yes/No Time min Flask allowed to settle? Yes/No Time min	Is there solute still present? Yes/No
Analyst	Date/Time / /	Step 2-3	Flask vortex-mixed? Yes/No Room Temp °C Flask sonicated? Yes/No Time min Flask allowed to settle? Yes/No Time min	Is there solute still present? Yes/No
Analyst	Date/Time / /	Step 2-4	Flask vortex-mixed? Yes/No Room Temp °C Flask sonicated? Yes/No Time min Flask allowed to settle? Yes/No Time min	Is there solute still present? Yes/No

Conclusions:

Did the substance dissolve in 2 mL or less of solvent?

Yes -Approximate concentration at which the test substance is soluble in solvent: 7109 ug/ml

The solubility of the test substance is to be determined via the shake flask method.

or No Continue to page two for further testing.

Notes/ additional comments:

① Flask was vortex-mixed on 08 Sept. 00 by CMC, but was inadvertently not circled on the prep sheet. A 3M ETS Laboratory late entry correction was made by KCT on 01-22-01 KCT 02/07/01

Initial

Date

3M Environmental Laboratory SOLUBILITY SCREEN

GLP Study Number: TCR-006

Sample preparation worksheet

Test Substance: PFOA

ID#: TCR-99030-30

Date: _____

Source: 3M Internal

Solvent: Acetone

ID#: _____

Source: _____

Step (continued from page 1)

***Did the test substance dissolve in 2 mL? Yes/No If YES, the test is complete, and there is no need to continue with the screen test. If NO, continue to step 3 to determine the approximate solubility of the substance.

3 Weigh approximately 10 mg $\pm$ 0.5 mg of test substance into a 100 mL graduated stoppered cylinder/volumetric flask.

Weight of test substance: _____ mg

Balance ID: _____

Date/Initials: _____

2nd replicate optional: _____ mg

Thermometer ID: _____

4 Add test solvent according to the table below. Following each addition of solvent, shake vigorously/vortex, and sonicate the mixture and visually check for undissolved particles.

Solubility data		volume added (mL)/ approx conc. after addition (ug/mL)	Sample Prep	OBSERVATIONS/NOTES
Analyst _____	Date/Time _____ / _____	Step 4-1	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)... 10			
	Approximate Solubility (ug/mL)... 1,000			
Analyst _____	Date/Time _____ / _____	Step 4-2	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)... 50			
	Approximate Solubility (ug/mL)... 200			
Analyst _____	Date/Time _____ / _____	Step 4-3	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)... 100			
	Approximate Solubility (ug/mL)... 100			

5 Weigh approximately 10 mg $\pm$ 0.5 mg of test substance into a 1 L graduated stoppered cylinder/volumetric flask/or equivalent.

Weight of test substance: _____ mg

Balance ID: _____

Date/Initials: _____

Analyst _____	Date/Time _____ / _____	Step 5-1	Flask vortex-mixed? Yes/No Room Temp _____ °C Flask sonicated? Yes/No Time _____ min Flask allowed to settle? Yes/No Time _____ min	Is there solute still present? Yes/No
	Total volume solvent added (mL)... 1000			
	Approximate Solubility (ug/mL)... 10			

Conclusions:

Did the substance dissolve in 1000 mL or less of solvent?

Yes -Approximate concentration at which the test substance is soluble in solvent: _____ ug/mL

The solubility of the test substance is to be determined via the shake flask method.

or No -The solubility of the test substance is to be determined via the column elution method.

Notes/ additional comments:

NO FURTHER
TESTING REQUIRED
KLT 9-12-06

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3M Environmental Laboratory
Report No. FACT-TCR-006