Study Title

Solubility of Perfluoro-1-octanesulfonyl Fluoride (POSF) in Water

Data Requirement

40 CFR 160.105(b)

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Project Identification

3M Environmental Laboratory Study # LIMS E01-0859

Total Number of Pages

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STUDY INFORMATION

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Experimental Dates

Sample Analysis Initiation: 24 May 2001
Sample Analysis Completion: 01 Jun 2001

SUMMARY

Solubility of Perfluoro-1-octanesulfonyl fluoride (POSF) 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. The approximate solubility of POSF was determined by performing a solubility screen test (ETS-8-170.1). POSF was determined to be soluble at concentrations less than 294 ηg/mL in water at 22-24°C. Results are presented in table 1:

Table 1. Summary Table of Solubility of POSF TN-A-2825 in Water.

SOLVENT	POSF SOLUBILITY
Water	< 294 ηg/mL

The solubility of POSF was determined to be less than 294 ηg/mL in water at 22-24°C.

PURPOSE

The purpose of this study was to determine the solubility of POSF in water.

TEST SUBSTANCE

Perfluoro-1-octanesulfonyl fluoride (POSF), TN-A-2825

Table 2. Characterization of the Test Substance

TEST SUBSTANCE	POSF
Source	Aldrich
Expiration Date	01-01-2010
Storage Conditions	Ambient
3M Laboratory Identification Number	TN-A-2825
Physical Description	Clear liquid, density = 1.824g/mL
Purity	98%

TEST SYSTEM

Table 3. Description of test systems used in this study

	WATER
Source	Millipore System
Expiration Date	NA
Storage Conditions	Ambient
3M Laboratory Identification Number	NA
Purity/Grade	ASTM Type I

METHOD SUMMARIES

The solubility determination of POSF TN-A-2825 in water was performed according to 3M Laboratory Method 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 POSF TN-A-2825 in water. Incremental amounts of water were added to POSF neat material and a qualitative determination of the solubility was made. The screen test indicated the solubility of POSF to be less than 294 ηg/mL in water at 22-24˚C. A detailed description of the Solubility Screen Test method used in this study is located in Attachment A.

PREPARATORY METHODS

- **ETS-8-170.0 "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. Microliter amounts of sample were added to varying amounts of water. Following sample addition, the volumetric flasks were shaken and observed for POSF droplets on the bottom. The solubility screen test estimated the solubility of POSF to be less than 294 ηg/mL in water at 22-24˚C.

Table 4. Summary of Sample Preparation.

STEP	SAMPLE PREPARATION	DATE(S) PERFORMED
1	10 uL test substance is added to 4.203L (4.33mg/L) of water in a 4 L volumetric flask.	5/24/01
2	The volumetric flask is sealed, shaken and then allowed to sit over night for 24 hours.	5/24/01
3	The volumetric flask is visually checked for the test substance, which would be in the form of oily droplets on the bottom.	5/25/01
4	This same test was done with 5uL in 6.205L (1.47ug/mL) and 1uL in 6.205L (294ng/mL) in a 6 L volumetric flask. The 294ng/mL solution was allowed to sit for 5 days. The sample was still visible in the flask after 5 days.	5/25/01 to 6/01/01

DATA SUMMARY

Table 5. Summary Table of Solubility of POSF TN-A-2825 in Water.

SOLVENT	POSF SOLUBILITY
Water	< 294 ηg/mL

The solubility of POSF, TN-A-2825 in water is less than 294 ηg/mL at 22-24°C.

STATISTICAL METHODS AND CALCULATIONS

No statistical methods or calculations were required. The data was reported as the solubility of POSF being less than 294 ηg/mL at 22-24°C in water as determined by the initial screening method.

STATEMENT OF CONCLUSION

Under the conditions of the present study, the solubility of POSF TN-A-2825 in ASTM Type I water is less than 294 ηg/mL at 22-24 °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 27July, 1995.

LIST OF ATTACHMENTS

- Attachment A: Solubility Screening Method ETS-8-170 "Solubility Screen Test: Approximate Solubility Determination of a Test Substance in Various Solvents", including the 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

06/12/01
Date

William K. Reagen
Laboratory Manager

06/12/01
Date

ATTACHMENT A

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 — Date: 03/14/01

Mark E. Ellefson, Project Leader — Date: 03/14/01

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).

1.2 **Compatible Analytes.** Test substance and degradation products for solubility testing.

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 µg/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 $CaCl_2$,** A 0.01 M $CaCl_2$ stock solution is prepared by weighing 1.5 g $CaCl_2$ 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 mg ± 1 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.

4 mL vial or equivalent	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 (µg/mL)…	100000	20000	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 µg/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 µL 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 mg ± 1 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.

100 mL volumetric flask or equivalent	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

Revision Number	Reason For Revision	Revision Date
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 ± 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 ____/____ Total volume solvent added (mL) Approximate Solubility (ug/mL)	**Step 2-1** 0.1 100,000	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.
Analyst _____ Date/Time ____/____ Total volume solvent added (mL) Approximate Solubility (ug/mL)	**Step 2-2** 0.5 20,000	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 ____/____ Total volume solvent added (mL) Approximate Solubility (ug/mL)	**Step 2-3** 1 10,000	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 ____/____ Total volume solvent added (mL) Approximate Solubility (ug/mL)	**Step 2-4** 2 5,000	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: _____ 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 ± 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	Is there solute still present? Yes/No
Total volume solvent added (mL)...	10	Flask sonicated? Yes/No Time_____min	
Approximate Solubility (ug/mL)...	1,000	Flask allowed to settle? Yes/No Time_____min	
Analyst _____ Date/Time_____/_____	**Step 4-2**	Flask vortex-mixed? Yes/No Room Temp_____°C	Is there solute still present? Yes/No
Total volume solvent added (mL)...	50	Flask sonicated? Yes/No Time_____min	
Approximate Solubility (ug/mL)...	200	Flask allowed to settle? Yes/No Time_____min	
Analyst _____ Date/Time_____/_____	**Step 4-3**	Flask vortex-mixed? Yes/No Room Temp_____°C	Is there solute still present? Yes/No
Total volume solvent added (mL)...	100	Flask sonicated? Yes/No Time_____min	
Approximate Solubility (ug/mL)...	100	Flask allowed to settle? Yes/No Time_____min	

5 *Weigh approximately 10 mg ± 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	Is there solute still present? Yes/No
Total volume solvent added (mL)...	1000	Flask sonicated? Yes/No Time_____min	
Approximate Solubility (ug/mL)...	10	Flask allowed to settle? Yes/No Time_____min	

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: