

Determination of the Melting Point/Melting Range of
PFOS

MELTING POINT

TEST SUBSTANCE

Identity: Perfluorooctanesulfonate; may also be referred to as PFOS or FC-95. (1-Octanesulfonic acid, 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-, potassium salt, CAS # 2795-39-3)

Remarks: The test substance is a white powder. Sample was taken from 3M lot number 217. Sample was stored under ambient conditions prior to testing. Purity determined to be 90.49% by LC/MS, ¹H-HMR, ¹⁹F-NMR and elemental analyses techniques.

METHOD

Method: OECD 102

GLP: Yes

Year completed: 1998

Remarks: Study utilized a Büchi Melting Point B-540 instrument, calibrated and inspected just prior to use using anthraquinone and 1,8-naphthalimide.

RESULTS

Melting point value in °C: >400°C (No melting observed).

Decomposition (yes-temperature °C/ no /ambiguous): No

Sublimation (yes/no/ambiguous): No

Remarks: Measurements of the melting point / melting range were limited to ≤400°C, the maximum specification for the instrument used. Fine droplets were not observed to adhere uniformly or otherwise to the walls of the melting point tubes.

CONCLUSIONS

The melting point / melting range was not observed and therefore could not be determined.

Remarks: While no melting of the test substance was evident, discoloration of the test samples was observed. The white powder turned to a light brown and eventually black as temperatures rose.

Submitter: 3M Company, Environmental Laboratory, P.O. Box 33331, St. Paul, Minnesota, 55133

DATA QUALITY

Reliability: Klimisch ranking 1.

REFERENCES

Study conducted at the request of 3M Company by Wildlife International, Ltd. of Easton, Maryland.

OTHER

Last changed: 5/3/00

DETERMINATION OF THE
MELTING POINT/MELTING RANGE OF PFOS

WILDLIFE INTERNATIONAL LTD. PROJECT NUMBER: 454C-106

OECD Guideline for Testing of Chemicals, 102
Melting Point/Melting Range

AUTHORS:

Ronda L. Jacobs
Willard B. Nixon, Ph.D.

STUDY INITIATION DATE: December 18, 1998

STUDY COMPLETION DATE: February 24, 1999

Submitted to:

3M Corporation
Environmental Laboratory
P.O. Box 33331
St. Paul, Minnesota 55133



WILDLIFE INTERNATIONAL LTD.



8598 Commerce Drive
Easton, Maryland 21601
(410) 822-8600

- 2 -

GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

SPONSOR: 3M Corporation

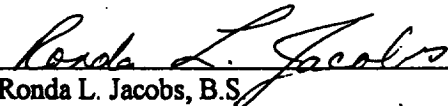
TITLE: Determination of the Melting Point/Melting Range of PFOS

WILDLIFE INTERNATIONAL LTD. PROJECT NUMBER: 454C-106

STUDY COMPLETION: February 24, 1999

This study was conducted in compliance with Good Laboratory Practice Standards as published by the U.S. Environmental Protection Agency in 40 CFR Part 792, 17 August 1989 and OECD Principles of Good Laboratory Practice, OCDE/GD (92) 32, Environment Monograph No. 45, Paris 1992.

STUDY DIRECTOR:



Ronda L. Jacobs, B.S.
Senior Chemist
Wildlife International Ltd.

24 Feb 99
DATE

- 3 -

QUALITY ASSURANCE STATEMENT

This study was examined for compliance with Good Laboratory Practice Standards as published by the U.S. Environmental Protection Agency in 40 CFR Part 792, 17 August 1989 and OECD Principles of Good Laboratory Practice, OCDE/GD (92) 32, Environment Monograph No. 45, Paris 1992. The dates of all inspections and audits and the dates that any findings were reported to the Study Director and Laboratory Management were as follows:

DATE REPORTED TO:

ACTIVITY:	DATE CONDUCTED:	STUDY DIRECTOR:	MANAGEMENT:
Melting Point	December 18, 1998	December 18, 1998	December 18, 1998
Draft Report and Data	December 23, 1998	December 23, 1998	December 23, 1998
Final Report	February 1, 1999	February 1, 1999	February 1, 1999


Linda R. Mitchell, B.S.
Manager, Quality Assurance

24 January 1999
DATE

- 4 -

REPORT APPROVAL

SPONSOR: 3M Corporation

TITLE: Determination of the Melting Point/Melting Range of PFOS

WILDLIFE INTERNATIONAL LTD. PROJECT NUMBER: 454C-106

STUDY DIRECTOR:

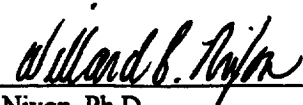


Ronda L. Jacobs
Senior Chemist



DATE

MANAGEMENT:



Willard B. Nixon, Ph.D.
Manager, Analytical Chemistry



DATE

TABLE OF CONTENTS

Title/Cover Page	1
Good Laboratory Practice Compliance Statement	2
Quality Assurance Statement	3
Report Approval	4
Table of Contents	5
Summary	6
Introduction	7
Purpose	7
Experimental Design	7
Materials and Methods	8
Test Substance	8
Test Procedure	8
Results	9
Melting Point and Melting Range	9
Conclusions	9
References	10

TABLE

Table 1 - Determination of Melting Point and Melting Range for Perfluorooctane Sulfonic Acid, Potassium Salt (PFOS)	11
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APPENDIX

Appendix I - Personnel Involved in the Study	12
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- 6 -

SUMMARY

SPONSOR:	3M Corporation
SPONSOR'S REPRESENTATIVE:	Ms. Susan A. Beach Environmental Laboratory P.O. Box 33331 St. Paul, Minnesota 55133
LOCATION OF STUDY, RAW DATA AND A COPY OF THE FINAL REPORT:	Wildlife International Ltd. Easton, Maryland 21601

WILDLIFE INTERNATIONAL LTD.	
PROJECT NUMBER:	454C-106
TEST SUBSTANCE:	Perfluorooctane Sulfonic Acid, Potassium Salt (PFOS)
STUDY:	Determination of the Melting Point/Melting Range of PFOS
TEST DATES:	Experimental Start - December 18, 1998 Experimental Termination - December 18, 1998

Product Chemistry Test	Results	Method
MELTING POINT:	None, $\geq 400^{\circ}\text{C}$, discoloration observed	OECD 102

- 7 -

INTRODUCTION

Wildlife International Ltd. attempted to determine the melting point and melting range of the test substance PFOS (perfluorooctane sulfonic acid, potassium salt). This study was conducted by Wildlife International Ltd. for 3M Corporation at the Wildlife International Ltd. analytical chemistry facility in Easton, Maryland. The study was performed following procedures in the OECD Guideline for Testing of Chemicals, 102, *Melting Point/Melting Range* (1). The experimental portion of this study was conducted on December 18, 1998. The original raw data generated by Wildlife International Ltd. and a copy of the final report are filed under Project Number 454C-106 in archives located on the Wildlife International Ltd. site.

PURPOSE

The purpose of this study was to determine the melting point and melting range of Perfluorooctane Sulfonic Acid, Potassium Salt (PFOS).

EXPERIMENTAL DESIGN

The melting point is defined as the temperature at which the phase transition from a solid to a liquid state at atmospheric pressure occurs. For some substances, the transition occurs over a range of temperatures defined as the melting range. Subsamples of the test substance were heated in a melting point tube in a temperature-controlled heating block. Measurements of the melting point/melting range were limited to $\leq 400^{\circ}\text{C}$ (approximately 673°K), the maximum specification for the instrumentation used. The temperature at which fine droplets adhere uniformly to the wall of the melting point tube would be accepted as the melting point. If a temperature range was noted between the formation of the fine droplets (beginning of melting) and transition to a liquid (end of melting), a melting range would be reported. A Büchi Melting Point B-540 instrument was used.

MATERIALS AND METHODS

Test Substance

The test substance was received from 3M Corporation on October 29, 1998 and was assigned Wildlife International Ltd. identification number 4675 upon receipt. The test substance, a white powder, was identified as: FC-95, Lot Number: 217, Expiration Date: 2008. The test substance was stored under ambient conditions.

Test Procedure

A Büchi Melting Point B-540 instrument with melting point tubes supplied by Büchi was used. The Büchi Melting Point B-540 instrument was calibrated and inspected just prior to use using anthraquinone and 1,8-naphthalimide.

The melting point and melting range determination attempts were performed as follows. Subsamples of the test substance were transferred to four melting point capillary tubes to a depth of approximately 3 mm. The tubes were tamped to pack the test substance. One subsample was subjected to a rapid temperature scan to determine an approximate melting point. An approximate melting point could not be determined. The test substance turned a light brown color and then blackened. Fine droplets were not observed. The instrument was then programmed to increment the temperature over a range of 300 to 400°C at a rate of 1°C/minute. The melting point and range determinations for the remaining three subsamples were attempted. At no time during the testing of Perfluorooctane Sulfonic Acid, Potassium Salt (PFOS) were fine droplets observed to adhere uniformly or otherwise to the walls of the melting point tubes. The three remaining subsamples reacted in a similar manner as the rapid temperature scan subsample. The white powder turned to a light brown. As the temperature increased, Perfluorooctane Sulfonic Acid, Potassium Salt (PFOS) darkened until it eventually turned black. When approaching 400°C, some of the test substance lost the blackened color and turned to a clear, crystal-like particulate. The remaining test substance stayed black. The barometric pressure was recorded at the time of the melting point/melting range determinations.

- 9 -

RESULTS

Melting Point and Melting Range

The results of the melting point and melting range determinations are presented in Table 1. Fine droplets were not observed to adhere uniformly or otherwise to the walls of the melting point tubes. Therefore, a melting point/melting range for Perfluorooctane Sulfonic Acid, Potassium Salt (PFOS) was not determined.

CONCLUSIONS

A product chemistry test was performed to determine the physical characteristics of the test substance Perfluorooctane Sulfonic Acid, Potassium Salt (PFOS). The melting point/melting range was not observed and therefore could not be determined.

- 10 -

REFERENCES

1. Organisation for Economic Cooperation and Development. 102. Guideline for Testing of Chemicals, 102: *Melting Point/Melting Range*.

- 11 -

Table 1

Determination of Melting Point and Melting Range for
Perfluorooctane Sulfonic Acid, Potassium Salt (PFOS)

Subsample ID	Subsample Amount (mm)	Instrument Gradient (°C/min)	Barometric Pressure (mm Hg)	Melting Point (°C and °K)	Melting Range (°C and °K)
Rep 1	3	1	770	None observed	None observed
Rep 2	3	1	770	None observed	None observed
Rep 3	3	1	770	None observed	None observed

- 12 -

APPENDIX I

Personnel Involved in the Study

The following key Wildlife International Ltd. personnel were involved in the conduct or management of this study:

1. Willard B. Nixon, Ph.D., Manager, Analytical Chemistry
2. Ronda L. Jacobs, Senior Chemist
3. Keri Leach, Technician

PROTOCOL

DETERMINATION OF THE MELTING POINT/MELTING RANGE OF PFOS

OECD Guideline for Testing of Chemicals, 103
Melting Point/Melting Range

3M Lab Request No. U2723

Submitted to

3M Corporation
Environmental Laboratory
P.O. Box 33331
St. Paul, Minnesota 55133



WILDLIFE INTERNATIONAL LTD.

8598 Commerce Drive
Easton, Maryland 21601
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December 2, 1998

DETERMINATION OF THE MELTING POINT/MELTING RANGE
OF PFOS

SPONSOR: 3M Corporation
Environmental Laboratory
P.O. Box 33331
St. Paul, Minnesota 55133

SPONSOR'S REPRESENTATIVE: Ms. Susan A. Beach

TESTING FACILITY: Wildlife International Ltd.
8598 Commerce Drive
Easton, Maryland 21601

STUDY DIRECTOR: Ronda Jacobs
Senior Chemist

LABORATORY MANAGEMENT: Willard B. Nixon, Ph.D.
Manager of Chemistry

FOR LABORATORY USE ONLY

Proposed Dates:	
Experimental Start Date: <u>18 Dec 98</u>	Experimental Termination Date: <u>18 Dec 98</u>
Project No.: <u>454C 106</u>	Int/Date: <u>By 18 Dec 98</u>
Test Substance No.: <u>4675</u>	Receipt Date: <u>29 Oct 98</u>

PROTOCOL APPROVAL

Ronda L. Jacobs
STUDY DIRECTOR

18 Dec 98
DATE

Willard B. Nixon
LABORATORY MANAGEMENT

12/18/98
DATE

Susan A. Beach
SPONSOR'S REPRESENTATIVE

12/7/98
DATE

INTRODUCTION

Wildlife International Ltd. will determine the melting point or melting range of PFOS (perfluorooctane sulfonic acid, potassium salt). The study will be conducted at the Wildlife International Ltd. analytical chemistry facility in Easton, Maryland. The study will be performed following procedures in OECD Guideline for Testing of Chemicals, 102, *Melting Point/Melting Range* (1). Raw data for all work performed at Wildlife International Ltd. and a copy of the final report will be filed by project number in archives located at Wildlife International Ltd. or at an alternative location to be specified in the final report.

PURPOSE

The purpose of this study is to determine the melting point or melting range of PFOS.

EXPERIMENTAL DESIGN

The melting point is defined as the temperature at which the phase transition from a solid to a liquid state at atmospheric pressure occurs. For some substances, the transition occurs over a range of temperatures defined as the melting range. Subsamples of the test substance will be heated in a melting point tube in a temperature-controlled heating block containing a melting point capillary. Measurement of melting points will be limited to $\leq 400^{\circ}\text{C}$ (approximately 673°K), the maximum specified for the instrumentation used. The temperature at which fine droplets adhere uniformly to the wall of the melting point tube will be accepted as the melting point. If a range of temperature is noted between the formation of the fine droplets (beginning of melting) and transition to a liquid (end of melting), a melting range will be reported.

MATERIALS AND METHODS

Test Substance

The Sponsor will be asked to provide the following information concerning the test substance, if available:

Chemical Name (or designation)	Composition (for multi-component mixtures)
Lot or Batch Number	Empirical formula(s)
Purity	Expiration Date

The attached form IDENTIFICATION OF TEST SUBSTANCE BY SPONSOR (Appendix I) is to be used to provide available information. The Sponsor is responsible for information on the purity and composition of the test substance, as well as all information related to the safe handling of the test

substance. The Sponsor also agrees to accept any unused test substance or test substance containers remaining at the end of the study.

Test Procedure

The melting point or range will be determined using a Büchi Melting Point B-540 instrument with melting point tubes supplied by Büchi. The instrument will be calibrated prior to use with a minimum of two reference standards. Subsamples of the test substance will be transferred to a minimum of three capillary tubes to a depth of approximately 3 mm. Tubes will be tamped to pack the test substance. Following an initial rapid scan(s) to determine an approximate melting point, the instrument will be programmed to increment the temperature over the melting point or range at a rate not to exceed 1°C/minute. The temperature at which fine droplets uniformly adhere to the tube wall will be accepted as the melting point. If the temperature increases significantly (approximately 2°C or more) between the formation of the fine droplets adhering to the tube walls until liquification, a melting range will be reported.

The melting point or range will be recorded in °C and reported both in °C and °K. The barometric pressure will be recorded at the time of measurement of the melting point or range.

Sample Handling and Safety

The Sponsor will identify any special handling or safety precautions to be used with the above referenced test substance. All normal precautions with respect to handling and storage will be taken.

Sample and Test Substance Retention

Upon completion of testing, portions of the test substance used as part of this study will be disposed of in accordance with federal, state and local regulations. Any unused portion of the test substance will be returned to the Sponsor.

RECORDS TO BE MAINTAINED

Records to be maintained for data generated by Wildlife International Ltd. will include, but not be limited to:

1. A copy of the signed protocol.
2. Identification and characterization of the test substance, if provided by the Sponsor.
3. Dates of initiation and completion of the study.

4. Dates of experimental start and termination.
5. Storage conditions of the test substance.
6. Test substance use log.
7. The instrument temperature program.
8. Individual and mean melting point measurements expressed in °C and °K.
9. Statistical calculations.
10. Test conditions.
11. A copy of the final report.

FINAL REPORT

A final report of the results of the study will be prepared by Wildlife International Ltd. The report will include, but not be limited to the following, when applicable:

1. Name and address of the facility performing the study.
2. Dates upon which the study was initiated and completed.
3. A statement of compliance signed by the Study Director addressing any exceptions to Good Laboratory Practice Standards
4. Purpose and procedure, as stated in the approved protocol, including all amendments and deviations to the protocol.
5. The test substance identification, including name, chemical abstract number or code number, purity, composition, empirical formula, molecular formula, manufacturer's lot/batch number, dissociation in water, method of analysis, or other information provided by the Sponsor.
6. Description of the test method or reference to the method used along with any modifications made.
7. Individual and mean values obtained for the melting point or range expressed in °C and °K.
8. Description of any problems experienced and how they were resolved.
9. A statement prepared by the Quality Assurance Unit listing the dates that study inspections and audits were made and any findings that were recorded.

CHANGING OF PROTOCOL

Planned changes to the protocol will be in the form of written amendments signed by the Study Director and the Sponsor. Amendments will be considered as part of the protocol and will be attached to the final protocol. Any other changes will be in the form of written deviations filed with the raw data. All changes to the protocol will be indicated in the final report.

GOOD LABORATORY PRACTICES

This study will be conducted according to the Good Laboratory Practices described in OECD (ISBN 92-84-12367-9) and EPA (40 CFR Parts 160 and/or 792). Each study conducted by Wildlife International Ltd. is routinely examined by the Wildlife International Ltd. Quality Assurance Unit for compliance with Good Laboratory Practices, Standard Operating Procedures and the specified protocol. A statement of compliance with Good Laboratory Practices will be prepared for all portions of the study conducted by Wildlife International Ltd. The Sponsor will be responsible for compliance with Good Laboratory Practices for procedures performed by other laboratories. Raw data for all work performed at Wildlife International Ltd. and a copy of the final report will be filed by project number in archives located on the Wildlife International Ltd. site or at an alternative location to be specified in the final report.

REFERENCES

1. **Organisation for Economic Cooperation and Development. 102. Guideline for Testing of Chemicals, *Melting Point/Melting Range*.**

APPENDIX I
IDENTIFICATION OF TEST SUBSTANCE BY SPONSOR

To be Completed by Sponsor

- I. Test Substance Identity (name to be used in the report): Perfluorooctane Sulfonic Acid, Potassium Salt (PFOS)
- Sample Code or Batch Number: Lot 217
- Purity (% Active Ingredient): 98.9% Expiration Date: 2008
- II. Test Substance Characterization
- Have the identity, strength, purity and composition or other characteristics which appropriately define the test substance been determined prior to the start of this study in accordance with GLP Standards? Yes ☐ No ☒
- III. Test Substance Storage Conditions
- Please indicate the recommended storage conditions at Wildlife International Ltd.
- Ambient room temperature.
- Has the stability of the test substance under these storage conditions been determined in accordance with GLP Standards? Yes ☐ No ☒
- Other pertinent stability information: _____
- IV. Toxicity Information:
- Mammalian: Rat LD50 251 mg/kg Mouse LD50 N/A
- Aquatic: Invertebrate Toxicity (EC/LC50) Fish Toxicity (LC50)
- Daphnia magna: 27 mg/L Rainbow trout: 11 mg/L
- Daphnia magna: 50 mg/L Fathead minnow: 38 mg/L
- Other Toxicity Information (including findings of chronic and subchronic tests):
- See MSDS.