

BIODEGRADATION

TEST SUBSTANCE

Identity: Perfluorooctylsulfonate, didecyldimethylammonium salt; may also be referred to as Fluoroalkyl ammonium derivative. [1-Decaminium, N-decyl-N,N-dimethyl-, salt with 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-1-octanesulfonic acid (1:1), CAS # 251099-16-8]

Remarks: The 3M production lot number was Lot 1. The test sample is L-14394 referred to by the test laboratory as P3025. The sample was labeled F-11615, Lot 1. The test sample is a mixture of the test substance in water (approximately 30-40% test substance, 60-70% water, and 0-5% of residual perfluorochemicals). All values reported relate to this mixture. The test sample appears to be a 2-phase dispersion (clear liquid with opaque solid) which rapidly separates after agitation. No calculations were made to adjust for the actual concentration of the test substance in the test sample.

METHOD:

Method: OECD Method 301D

Type: Aerobic

GLP: No

Year completed: 1997

Contact time: 28 days

Inoculum: Polyseed®- NX (with nitrification inhibitor), lot A306, Polybac Corp, Bethlehem, PA

RESULTS

COD: 125,000 mg O₂ / kg chemical

CBOD

Exposure period, Day	First Replicate CBOD Mean Value (mg/kg)	Second Replicate CBOD Mean Value (mg/kg)
5	<40,000	<40,000
10	<40,000	<40,000
20	<40,000	<40,000
28	55,039	<40,000

Remarks: Values reported are for the test sample. No calculations were made to adjust for the concentration of the test substance in the test sample.

CONCLUSIONS

This testing indicated that some portion of the test sample biodegraded in aerobic environments capable of supporting biodegradation. This study cannot rule out the possibility that conditions could be found that would allow for biodegradation of the fluorochemical component, nor could it exclude the possibility that a microorganism could eventually be enriched that could metabolically alter this chemical.

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

DATA QUALITY

Reliability: Klimisch ranking 2. This study lacks sufficient characterization of the test substance purity and analytical measurement of test concentrations. Because a mixture was tested, the testing cannot conclude what portions of the test sample were responsible for the apparent biodegradation values.

REFERENCES

This study was conducted by the 3M Company, Environmental Laboratory, St Paul, MN, Lab Request number P3025.

OTHER

Last changed: 5/24/00

3M ENVIRONMENTAL LABORATORY

**DETERMINATION OF THE 28-DAY CARBONACEOUS BIOCHEMICAL
OXYGEN DEMAND (CBOD) FOR P3025**

OECD Closed Bottle Test, #301D (Modified)

STUDY COMPLETED: March 28, 1997

FINAL REPORT COMPLETED: May 20, 1997

Prepared by:

Name: Susan A. Beach
Title: Advanced Environmental
Biologist
Laboratory: 3M Environmental
Laboratory
Address: Building 2-3E-09
935 Bush Avenue
St Paul, MN 55144

Env. Lab Request Number P3025

SUMMARY

In this study the aerobic, aquatic ready biodegradability of P3025 in general accordance with the "Closed Bottle Test", OECD Method No. 301 D was determined. The test substance was found to be not readily biodegradable, with removal values of <32% and 44% (duplicate) obtained after 28 days.

OBJECTIVE

The purpose of this study was to evaluate the ready biodegradability of P3025 after 5, 10, 20, and 28 days of exposure using a modified form of OECD Method 301 D.

MATERIALS AND METHODS

<u>Test Substance:</u>	P3025, date 10/7/96, Lot 1																
Appearance	Two-phase dispersion (clear liquid with opaque solid)																
Aqueous Solubility	Apparently not completely soluble in water at tested concentrations.																
Available Physicochemical Properties	<table><tr><td>Boiling Point:</td><td>100°C</td></tr><tr><td>Vapor Pressure:</td><td>ca. 18 mm Hg @ 20°C</td></tr><tr><td>Vapor Density:</td><td>< 1</td></tr><tr><td>Evaporation Rate:</td><td>< 1</td></tr><tr><td>Solubility in water:</td><td>Insoluble</td></tr><tr><td>Specific Gravity:</td><td>ca. 1.2</td></tr><tr><td>Percent Volatile:</td><td>60 - 70%</td></tr><tr><td>pH:</td><td>6 - 8</td></tr></table>	Boiling Point:	100°C	Vapor Pressure:	ca. 18 mm Hg @ 20°C	Vapor Density:	< 1	Evaporation Rate:	< 1	Solubility in water:	Insoluble	Specific Gravity:	ca. 1.2	Percent Volatile:	60 - 70%	pH:	6 - 8
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Vapor Pressure:	ca. 18 mm Hg @ 20°C																
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Percent Volatile:	60 - 70%																
pH:	6 - 8																
Date Received	10/16/96																
Storage	In the dark at ambient room temperature (22 ± 2°C)																
Test Solutions	Test solutions were prepared as Water-Accommodated Fractions (WAFs). Four stock solutions, each in duplicate, of approximately 6, 12, 25, and 50 mg/L were prepared in aerated mineral nutrient media. Each was capped and allowed to mix overnight, in the dark, at ambient room temperature. Test substance was apparently not completely miscible with water. A few white particulates were observed in each stock solution after mixing. The clear liquid portion was withdrawn for biodegradation testing.																
Reported values	The reported values were based on nominal concentrations in the test media at the beginning of the toxicity test.																

Test Organisms:

Type	Polyseed®-NX (with nitrification inhibitor)
Source	Polybac Corp., Bethlehem, PA
Lot number	A306
Preparation	Contents of one Polyseed®-NX capsule placed into 500 mL aerated mineral media. Solution stirred for sixty minutes on magnetic mixer. Solution kept stirring while aliquots withdrawn to inoculate test bottles.

Test Conditions:

Photoperiod	24-hours dark
Test Vessels	300 mL clear glass Wheaton BOD bottles with ground glass stoppers sealed with a water seal and capped.
Test Volume	300 mL
Loading	5 mL reconstituted inoculum added to each bottle except for media blanks which received none
No. Replicates	2 each for medium blank, seeded blank, reference standard and each test concentration. Only one replicate for toxicity check.
Mineral Medium	Prepared with Hach BOD Nutrient Buffer Pillows (catalogue # 14863-98) each dissolved in 19 liters Millipore Milli-Q water. Glass carboys aerated with laboratory air filtered through a 0.2 µm filter for approximately 2-hours. Carboy openings covered with new foam plugs and allowed to stand overnight in the dark at ambient room temperature.

Initial Stock Solution Loadings - nominal	First Replicate	Duplicate
	6.7 mg/L	5.6 mg/L
	12.3 mg/L	10.9 mg/L
	26.9 mg/L	24.3 mg/L
	51.6 mg/L	54.2 mg/L

Test Bottles	Each test BOD bottle was seeded and filled completely full with solution from the appropriate water-accommodated fraction.
Reference Standard	Glucose/glutamic acid mixture. Hach BOD standard solution ampules, cat. # 14865-10. Added 1.5 mL of solution to each reference standard BOD bottle.
Toxicity Check	Added 1.5 mL of Hach glucose/glutamic acid mixture to a seeded BOD bottle containing the test substance P3025 at the highest loading.

Test Temp., °C	21 - 22°C, continuously recorded.
Stock Solutions pH	Initial pH values ranged from 6.5 to 6.7.
Exposure Period	28-days
Measurements	Dissolved oxygen at 0, 5, 10, 20, and 28 days. Dissolved oxygen meter calibrated by means of Winkler titration of aerated Milli-Q water each day.
Date Test Initiated	2/28/97
Date Test Terminated	3/28/97
Procedure	The test generally followed the recommendations of the OECD Guidelines for Testing of Chemicals

CALCULATIONS

The reported values are based on nominal stock solution loadings at the beginning of the study. The concentrations tested and the corresponding response data derived from the duplicated test are used to calculate, if possible the mg of carbonaceous biochemical oxygen demand per kg of test substance (BOD). The percent biodegradability is then determined by dividing the CBOD by the Chemical Oxygen Demand (COD) and multiplying by one hundred.

RESULTS

Percent biodegradability values obtained after 5, 10, 20, and 28 days of exposure to P3025 are summarized in Table 1.

In duplicated measurements, the percent biodegradability of P3025 was reported as <32% and 44% of chemical oxygen demand.

ACCEPTABILITY CRITERIA

This biodegradation test meets quality criteria provided in test guidelines:

- 1) The glucose-glutamic acid reference standard 5-day C-BOD results were within the range normally seen in this laboratory. See Figure 1.
- 2) The oxygen depletion of the medium blanks was ≤ 0.5 mg/L after 5-days.
- 3) The oxygen depletion of the medium blanks was ≤ 1.5 after 28-days.
- 4) The C-BOD result for the toxicity check (highest tested concentration plus glucose-glutamic acid) was greater than the value found for glucose-glutamic acid alone. This indicated the test substance did not inhibit the microorganisms.
- 5) Only those data points where the corrected dissolved oxygen drop was > 2.0 mg/L and the final dissolved oxygen concentration in the bottle was > 0.5 mg/L were used to calculate C-BOD.

28-Day CBOD
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May 20, 1997

This test was conducted by Trudy K. Stiefenhofer as requested by Pam Wolf of 3M Specialty Chemicals Division.

REFERENCES

APHA, AWWA, WEF. 1995. Standard Methods for the Examination of Water and Wastewater. Method 5210 B, 5-Day BOD Test.

APHA, AWWA, WEF. 1995. Standard Methods for the Examination of Water and Wastewater. Method 4500-O C. Oxygen (Dissolved), Azide Modification.

OECD. 1993. 301 D Closed Bottle Test. Guidelines for Testing of Chemicals. Organisation for Economic Co-operation and Development, Paris, France.

OECD. 1981. Principles of Good Laboratory Practice, Annex 2, C(81) 30 (Final): 7-28. OECD Guidelines for Testing of Chemicals. Organisation for Economic Cooperation and Development, Paris, France.

Polybac. 1997. Polyseed®-NX, "The Original" CBOD Seed Inoculum. Polybac Corporation, Bethlehem, PA.

TABLE 1

SUMMARY OF READY BIODEGRADABILITY OF P3025

Exposure Period	CBOD, mg/Kg	Percent of COD*
5-Days	<40,000	<32
Duplicate	<40,000	<32
10-Days	<40,000	<32
Duplicate	<40,000	<32
20-Days	<40,000	<32
Duplicate	<40,000	<32
28-Days	55,039	44
Duplicate	<40,000	<32

*COD reported as 125,000 mg/kg

FIGURE 1

**CBOD GGA 5-day
Reference Standard**

