

BIODEGRADATION

TEST SUBSTANCE

Identity: N-methylperfluorooctane sulfonamidoethanol; may also be referred to as N-MeFOSE Alcohol, FC-790, or FM-3925. (1-Octanesulfonamide, N-methyl-1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-N-(2-hydroxyethyl)-, CAS # 24448-09-7)

Remarks: Material is an off-white, waxy solid. Sample purity and lot number were not recorded.

METHOD

Method: COD/BOD study using 3M Environmental Laboratory standard protocols modeled after the Standard Methods for the Examination of Water and Wastewater, 14th Edition, American Public Health Association, 1976.

Type: Potassium dichromate reflux COD and Aerobic BOD

GLP: No

Year completed: 1979

Contact time (units): See Remarks

Inoculum: Not noted.

Remarks: The laboratory was not able to generate useable data for BOD due to insufficient oxygen depletion.

The COD was done in replicate.

RESULTS

COD: Analysis 1 = 170,000 mg/kg
Analysis 2 = 156,000 mg/kg
Average = 163,000 mg/kg

BOD: A twenty-day biochemical oxygen demand (BOD) test with suspended substrate concentrations up to 1000 mg/L showed insufficient oxygen depletion in excess of the blanks to allow the calculation of BOD values. This result indicates that N-MeFOSE alcohol is not likely to degrade in a non-acclimated aerobic biological treatment system.

Remarks: A 259 mg/L potassium hydrogen phthalate control, running concurrently with the same seed was found to have a BOD₅ of 231. The mean BOD for this control substrate at this concentration is 208 mg/L. This indicates that the seed was active and acceptable for BOD studies.

The fact that oxygen depletion rates during the first fourteen days in cultures containing N-MeFOSE alcohol closely followed those of the seeded controls suggests that the lack of degradation is not due to toxicity to heterotrophic microorganisms.

DATA QUALITY

Reliability: Klimisch ranking = 3. The purity of the test substance is not sufficiently characterized and the inoculum concentration is not described though the control indicates it was active. Study used individual weights that exceeded water solubility, thus the test substance was not solubilized and was not available to microbes.

REFERENCES

3M Technical Reports: "Biodegradation Studies on FM 3925", E.A. Reiner, Project 9970012600 Premanufacturing Notice FC-790, Report No. 016, January 8, 1979.

"Chemical Oxygen Demand of FM 3925", E.A. Reiner, Project 9970012600 Premanufacturing Notice FC-790, Report No. 017, January 8, 1979.

OTHER

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

Last changed: 5/17/00



Form 6747-11-A

TECHNICAL REPORT SUMMARY

Date
1/8/79

TO: TECHNICAL COMMUNICATIONS CENTER - 201-2CN

(Important - If report is printed on both sides of paper, send two copies to TCC.)

Division	Environmental Laboratory (EE & PC)	Dept. Number	0535
Project	Premanufacturing Notice FC-790	Project Number	9970012600
Report Title	Biodegradation Studies on FM 3925	Report Number	016
To	V. Pothapragada		
Author(s)	E. A. Reiner	Employee Number(s)	47816
Notebook Reference	None - See Environmental Lab Request #4197	No. of Pages Including Coversheet	
SECURITY ►	☐ Open (Company Confidential)	☐ Closed (Special Authorization)	3M CHEMICAL REGISTRY ►
			New Chemicals Reported ☐ Yes ☒ No

KEYWORDS:
(Select terms from 3M
Thesaurus. Suggest other
applicable terms.)

Biodegradation
EE & PC Div.
Fluorochemical
Degradation
BOD

CURRENT OBJECTIVE:

To evaluate the susceptibility of FM 3925 to
biodegradation.

REPORT ABSTRACT: (200-250 words) This abstract information is distributed by the Technical Communications Center to alert 3M'ers to Company R&D. It is Company confidential material.

A twenty-day biochemical oxygen demand (BOD) test with suspended substrate concentrations up to 1000 mg/l showed insufficient oxygen depletion in excess of the blanks to allow the calculation of BOD values. This result indicates that FM 3925 is not likely to degrade in a nonacclimated biological treatment system.

FM 3422 has previously been found to be resistant to biodegradation even in acclimated aerobic systems. Since it is chemically similar to FM 3422, FM 3925 is probably also resistant to biodegradation in acclimated microbial cultures.

Information Liaison
Initials: SLW

BIODEGRADATION STUDIES ON FM 3925

Introduction

Previous studies on FM 3422, ethyl fose alcohol ($C_8F_{17}SO_2N(C_2H_5)C_2H_4OH$), have shown that this compound is not measurably degraded by either acclimated or nonacclimated mixed microbial cultures (1,2). Therefore, it appeared unlikely that the FM 3925, methyl fose alcohol ($C_8F_{17}SO_2N(CH_3)C_2H_4OH$), because of its chemical similarity, would be readily degradable. The following work was conducted to check the susceptibility of FM 3925 to biodegradation.

Ultimate Biochemical Oxygen Demand

A twenty-day biochemical oxygen demand (BOD) was conducted on FM 3925 according to the standard Environmental Laboratory protocol (attached), except that FM 3925 was placed in the BOD bottles in solid form and that the bottles containing FM 3925 were continuously stirred for the entire incubation period. Stirring was done using a Hach Model 2173 with magnetic stirring bars. The seeded controls and reference compounds were not stirred.

The results of this study are shown in TABLE 1 and are plotted in Figure 1. No biodegradation of FM 3925 was observable by this method. The method requires that the dissolved oxygen depletion in a test culture exceed the oxygen depletion of the seeded control by at least 2 mg/l. This did not occur. Thus the BOD cannot be calculated. The BOD at each time is therefore considered to be less than 2000 mg of oxygen per kg of this compound.

A 259 mg/l potassium hydrogen phthalate control run concurrently with the same seed was found to have a BOD_5 of 231. The mean BOD for this control substrate at this concentration is 208 mg/l. This indicates that the seed was an active acceptable seed for BOD studies.

The fact that the test substrate did not degrade to a detectable level in this study suggests that these compounds would not degrade significantly in a nonacclimated waste treatment system.

Whether or not microbial populations can acclimate to degrade this compound cannot be determined from this study. However, since FM 3925 is structurally very similar to FM 3422, which has been shown not to degrade in acclimated cultures, we anticipate that FM 3925 will resist degradation by even acclimated microbial cultures.

The fact that oxygen depletion rates (Figure 1) during the first fourteen days in cultures containing FM 3925 closely followed those of the seeded controls suggests that lack of degradation is not due to toxicity to heterotrophic microorganisms.

Figure 1: Oxygen Concentration of Culture
Bottles During Ultimate BOD
Study of FM 3925

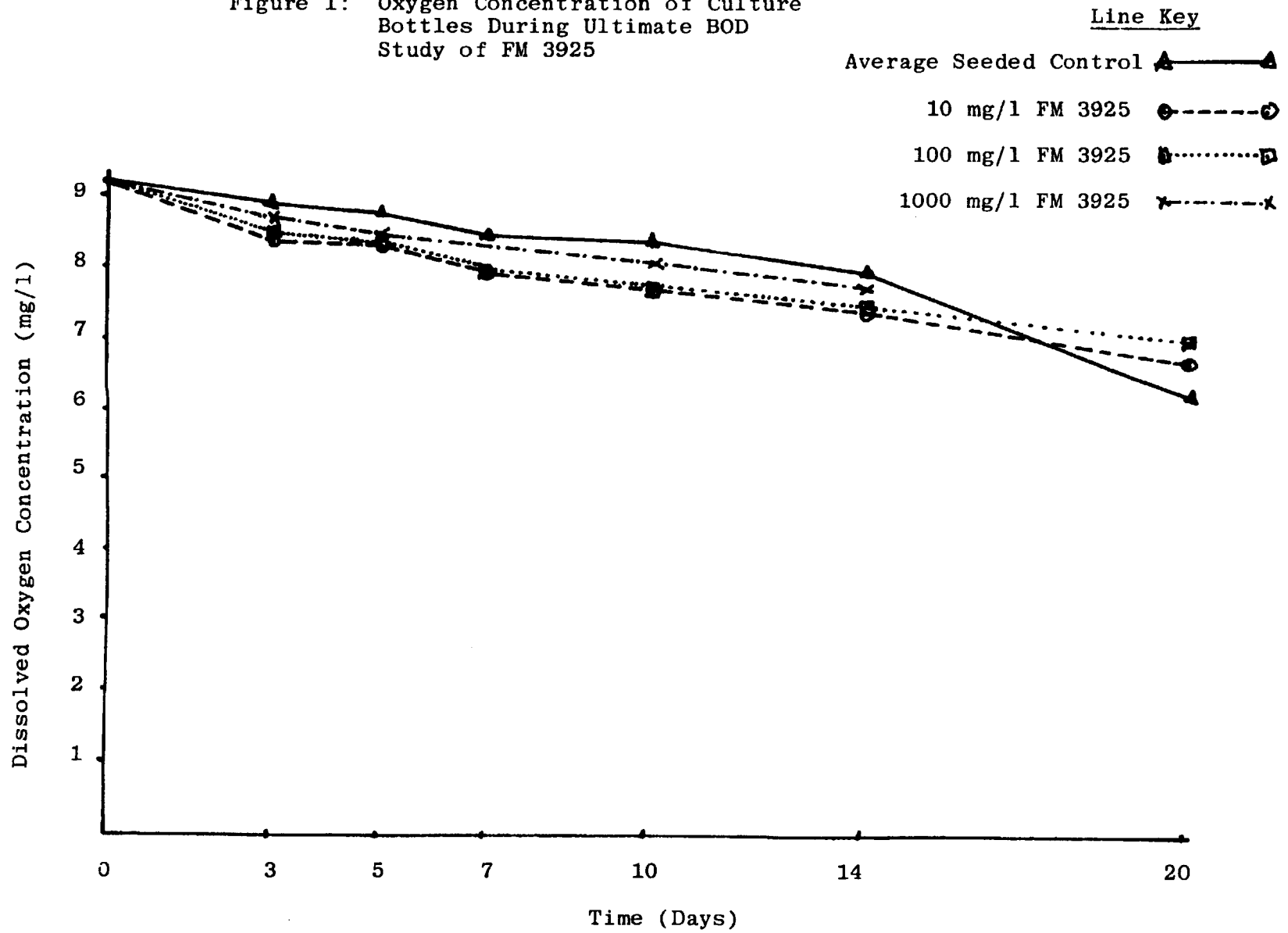


TABLE 1
ULTIMATE BOD DATA FOR FM 3925

<u>Description and Concentration</u>	<u>Dissolved O₂ Concentration</u>					
	<u>Day 3</u>	<u>Day 5</u>	<u>Day 7</u>	<u>Day 10</u>	<u>Day 14</u>	<u>Day 20</u>
Seeded Control #1	8.9	8.7	8.4	8.3	7.8	6.1
Seeded Control #2	8.9	8.9	8.6	8.5	7.9	6.3
Seeded Control #3	8.9	8.8	8.5	8.4	7.7	6.2
Seeded Control #4	8.9	8.9	8.6	8.5	8.1	6.1
Avg. Seeded Control	8.9	8.8	8.5	8.4	7.9	6.2
FM 3925:						
10 mg/l	8.4	8.3	7.9	7.7	7.4	6.7
32 mg/l	8.7	8.3	8.0	7.7	7.4	7.0
100 mg/l	8.5	8.3	7.7	7.7	7.5	7.0
560 mg/l	8.7	8.4	8.1	8.0	7.6	7.4
1000 mg/l	8.7	8.5	8.1	8.1	7.7	Lost
Potassium hydrogen phthalate:						
2.6 mg/l	6.5	-	-	-	-	-
4.3 mg/l	5.0	-	-	-	-	-
6.1 mg/l	3.3	-	-	-	-	-
7.8 mg/l	1.7	-	-	-	-	-

References:

- (1) Reiner, E. A., Biodegradation Studies of Fluorocarbons,
3M Technical Report 8/12/76.
- (2) Reiner, E. A., Biodegradation Studies of Fluorocarbons-II,
3M Technical Report 1/9/78.

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ULTIMATE BOD DATA SHEET
RESULTS EXPRESSED AS mg/Kg SAMPLE

DESCRIPTION AND CONCENTRATION	BOD ₃	BOD ₅	BOD ₇	BOD ₁₀	BOD ₁₄	BOD ₂₀
FM 3925 10 mg/l 32 100 560 1,000 INSUFFICIENT D.O. DEPLETIONS TO CONFIDENTLY CALCULATE ACTUAL BOD VALUES. ‡ LESS THAN VALUE ‡ ‡ ‡ ‡ ‡ <1800 NOTE: FM3925 WAS PLACED IN THE BOD BOTTLES IN THE SOLID FORM. THE BOTTLES WERE PLACED ON A CONTINUOUS STIRRING UNIT (HACH MODEL Z173) WITH MAGNETIC STIRRING BARS. THE SAMPLES WERE MIXED DURING THE ENTIRE INCUBATION PERIOD. G.O.D - mg/Kg 170,000 156,000 AVG. 163,000						

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Date **1/8/79**

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Division	Environmental Laboratory (EE & PC)		Dept. Number	0535
Project	Premanufacturing Notice FC-790		Project Number	9970012600
Report Title	Chemical Oxygen Demand of FM 3925		Report Number	017
To	V. Pothapragada			
Author(s)	E. A. Reiner		Employee Number(s)	47816
Notebook Reference	None - See Environmental Lab Request #4197		No. of Pages Including Coversheet	
SECURITY ►	☐ Open (Company Confidential)	☐ Closed (Special Authorization)	3M CHEMICAL REGISTRY ►	
			New Chemicals Reported ☐ Yes ☒ No	

KEYWORDS:
(Select terms from 3M
Thesaurus. Suggest other
applicable terms.)

Chemical Oxygen
Demand (COD)

Degradation

Fluorochemical

EE & PC-Div.

CURRENT OBJECTIVE:

To determine the extent to which FM 3925 is oxidized by refluxing in the presence of potassium dichromate under strongly acidic conditions.

REPORT ABSTRACT: (200-250 words) This abstract information is distributed by the Technical Communications Center to alert 3M'ers to Company R&D. It is Company confidential material.

The chemical oxygen demand (COD) of FM 3925 was determined according to the 3M Environmental Laboratory's standard protocol which is modeled after the Standard Methods procedure. The COD was determined to be 163,000 mg/kg. This is 35% of the total theoretical oxygen demand (assuming N is not oxidized) and 71% of the oxygen demand calculated assuming that only the nonfluorinated portion of the molecule is degraded.

Information Liaison
Initials: **SKW**

CHEMICAL OXYGEN DEMAND OF FM 3925

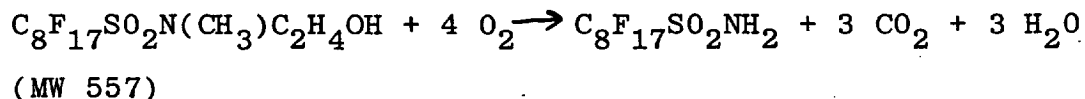
The chemical oxygen demand of FM 3925 was calculated according to the standard protocols of the Environmental Laboratory (attached). These protocols are modeled after the procedure described in Standard Methods (1).

The results of two replicate analyses are shown in TABLE 1.

TABLE 1
COD of FM 3925

Analysis 1	170,000 mg/kg
Analysis 2	156,000 mg/kg
Average	163,000 mg/kg

This COD is 35% of the total theoretical oxygen demand assuming complete oxidation to F^- , CO_2 , SO_2 , NH_3 , and H_2O . It is 71% of the oxygen demand calculated assuming that only the nonfluorinated portion of the molecule is degraded as in the following reaction:



Reference:

- (1) Standard Methods for the Examination of Water and Wastewater, 14th Edition, American Public Health Association, 1976.

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Attachment

3M ENVIRONMENTAL LABORATORY
CHEMICAL OXYGEN DEMAND PROTOCOL
(Used prior to January 4, 1979)

Chemical oxygen demand is determined by a modification of the method described in Standard Methods for the Examination of Water and Wastewater, American Public Health Association, p. 550-554, (1976).

Procedure Modifications

The reflux apparatus differs from that described in Standard Methods in that 500 ml round-bottomed boiling flasks with ground-glass 24/40 neck are used in place of comparable ground-glass Erlenmeyer flasks. In addition, electric heating mantles, which are capable of adequately boiling the contents of the reflux flasks, are used in place of a hot plate.

The silver sulfate catalyst is added directly to each COD flask prior to refluxing the sample-sulfuric, acid-dichromate mixture.

The ferrous ammonium sulfate titrant is standardized against the blank. It is assumed that no dichromate is reduced in the blank during the reflux procedure.

Sample Size and Reagent Quantity and Normalities

Sample size is 50 ml. The reagent quantities and normalities are those prescribed for this sample size in Table 508:1 of the 14th Edition of Standard Methods, except that Ag_2SO_4 is added in 0.12 g quantities and is not dissolved in the concentrated sulfuric acid reagent.

Procedure for Low COD Samples

The alternate Standard Methods COD procedure for low-COD samples is used when COD values of 50 mg/l or less have to be determined.

In this procedure the sample size and reagent quantities remain the same, but the normalities of the dichromate and ferrous ammonium sulfate solutions are reduced to 0.025 N and 0.01 N, respectively.

3M ENVIRONMENTAL LABORATORY
CHEMICAL OXYGEN DEMAND PROTOCOL

Chemical oxygen demand is determined according to the method described in Standard Methods for the Examination of Water and Wastewater, American Public Health Association, p. 550-554, (1976).

Procedure Modifications

The reflux apparatus differs from that described in Standard Methods in that 500 ml round-bottomed boiling flasks with ground-glass 24/40 neck are used in place of comparable ground-glass Erlenmeyer flasks. In addition, electric heating mantles, which are capable of adequately boiling the contents of the reflux flasks, are used in place of a hot plate.

Sample Size and Reagent Quantity and Normalities

Sample sizes of 50 ml are used with the reagent quantities and normalities given in Standard Methods, Table 508:1.

Procedure for Low COD Samples

The alternate Standard Methods COD procedure for low COD samples is used when COD values of 50 mg/l or less have to be determined. In this procedure the sample size and reagent quantities remain the same, but the normalities of the dichromate and ferrous ammonium sulfate solution are reduced to 0.025 N and 0.01 N, respectively.