BIODEGRADATION (BOD/COD)

TEST SUBSTANCE

Identity: A mixture containing perfluorooctanesulfonate, which may also be referred to as PFOS, FC-95, or as a component of FC-600. (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 3M production lot number was not noted. The test sample is FC-600. Current information indicates it is a mixture of 1.0% PFOS, 81.20% water, 12.00% diethylene glycol butyl ether, 1.00% sodium octyl sulfate, 2.00% propane Sultone foamer, 1.00% sodium decyl sulfate, 0.85% xanthan gum, 0.1% N-(3-chloroallyl) hexaminium chloride, 0.80% starch, and 0.05 % tolyltriazole.

The following is an abbreviated summary of multiple biodegradation studies on FC-600. These studies apply to a mixture with incompletely characterized concentration of impurities. Data does not accurately reflect degradation potential of the fluorochemical component of the test sample.

METHOD:

Methods: OECD 301E and BOD/COD/TOC (summary listing only)
Type: Aerobic
GLP: No
Year completed: 1975, 1983, 1984, 1988
Contact time: 20 days (BOD) and 28 days (OECD 301E)
Inoculum: 1984 study used a 50/50 mixture of soil extract and secondary effluent. Sandy loam soils obtained from Ramsey and Washington counties in Minnesota. The secondary effluent sample was supernatant of activated sludge mixed liquor taken from the Metro Wastewater Treatment Plant, St. Paul, MN. Inoculum for the other studies was not noted.

RESULTS

Chemical Oxygen Demand:		467,000 mg/L*
Biochemical Oxygen Demand (BOD):		
	5-Day:	50,000 mg/L*
	11-Day:	255,000 mg/L*
	14-Day:	240,000 mg/L*
	20-Day:	237,000 mg/L*

* Data was available as a summary list only.

Total Organic Carbon (calculated for the product): 0.097 g/g

Total Organic Carbon Removal: 90 – 91% in 28 days

Dissolved Organic Carbon Removal: 79.9 – 94% in 28 days**
** Range over 3 separate OECD 301E studies

Remarks: Testing was conducted on the mixture as described in the Test Substance Remarks field. The value reported applies to that mixture and not the fluorochemical proportion alone.

CONCLUSIONS

No reliable conclusions can be derived from this study. The indication is that the majority of the product components are readily biodegradable.

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

DATA QUALITY

Reliability: Klimisch ranking 3. The inoculum is not properly characterized in most studies. The testing was conducted on a mixture with chemicals having varying degrees of biodegradability, leading to potential misinterpretation of the results. The sample purity was not properly characterized and the study lacks analytical confirmation of the amount of fluorochemical proportion in the solutions.

REFERENCES

Testing was conducted by the 3M Company, 3M Environmental Lab, St. Paul, MN, Lab Request Numbers 2874 and 8483.

3M Environment Assessment inquiry #82326, E. A. Reiner, 3M Environmental Laboratory, St. Paul, MN, April 21, 1983

3M Technical Report "OECD Biodegradation Testing on FC-600 and FC-203." E. A. Reiner, Project 9972410100, 3M Germany, Report Number 001, April 18, 1983

3M Technical Report "Modified OECD Screening Test on FC-203, FC-203A, and FC-600." E. A. Reiner, Project 9970010100, International, Report Number 003, July 9, 1984.

OTHER

Last changed: 6/28/00

Form 10820-5-PWO
cc: LAB REQUEST BOOK – 42-5W

3M Scotch 800 Paper

PRODUCT ECOLOGICAL ASSESSMENT
REQUEST FOR LABORATORY WORK

Ref: ENVIR. ASSESS. INO. # _____ DIVISION

LAB REQUEST NO. 2814

PRODUCT: _____

REQUESTER	LABORATORY ARD 9/18
Name: Deb Brown Phone No. 5953	Date Received Sample: 8/20
Request Date: 8/15 75	Analyst: Peterson / Mohamed
Project No. 75-6256 Prob. No. 68	Date Completed:
Code No: 3307	Hours Spent:
Sample Date: — Date Needed:	Y 75 B 21 P 28 + Bioassay
Description:	Please Call Requester If Delays Are Encountered.
Purpose of Tests	

PHYSICAL & CHEMICAL PROPERTIES			
Heat of Combustion Cal/G (BTU/Lb.)			FC 6.00
Density			
Ash			
Other			
WATER POLLUTION			
pH			
COD		X	167,000
BOD_5		X	50,000
BOD, Ultimate	11 days	X	255,000
TOC	19 days		210,000
GC	20 days		237,000
Oxygen Uptake			
Dehydrogenase Activity (TTC)			
Other			
BIOASSAY			
Specify Organism and Method:			
Blue Gill, Static		—	
Fathead Static		—	
LEACHATE FROM LEACHING TEST			
pH			
COD			
BOD_5			
BOD, Ultimate			
TOC			
GC			
Bioassay			
Other			
OTHER			
Bioaccumulation			
Persistence			
Degradation Products			

White - Return to Requester Canary - Environmental Engrg. Office File Pink - Environmental Engrg. Lab Goldenrod - Return to Requester

FORM 10620-8-PWD

LR#

File: Page No. 8483

ENVIRONMENTAL ENGR. LAB – WORK SHEET

SAMPLE DESCRIPTION

Date: 12-27-82
Analyst: W. G. Steele
Hrs.:

OECD SCREENING TEST

Preparation of Test Solutions.

Reference Material Used:

Sodium benzoate ($C_7H_5NaO_2$) F.W. = 144.11
Laboratory Grade (formerly designated U.S.P. Equivalent in chemical purity to U.S.P. specifications.)
Fisher Scientific Company – LOT 783798

30.3 mg $C_7H_5NaO_2$ diluted to 1.0 L with nutrient solution, (THEORETICAL DOC = 20 mg/L).

FC-600 (ANTWERP)

527 mg FC-600 diluted to 2.0 L with nutrient solution

FC-203 (ANTWERP)

153 mg FC-203 diluted to 2.0 L with nutrient solution.

Reviewed by:

FORM 10620-8-PWO

LR #

File: Page No. 8483

ENVIRONMENTAL ENGR. LAB – WORK SHEET

SAMPLE DESCRIPTION OECD MODIFIED SCREENING TEST.

Date: 4/05/83
Analyst: W.G. Schmid
Hrs.:

DOC LOSS FROM STERILE CONTROLS

TEST SOLUTION	DOC CONCENTRATION (mg/L) INITIAL	DAY 28	% REMAINING
FC-203 (DUP. DOC ANALYSIS)	23.0/24.4	23.4/23.1	98.1 %
FC-600 (DUP. DOC ANALYSIS)	22.6/23.9	23.3/23.8	101.3 %

handled identically to samples

Reviewed by:

3M/Environmental Lab

OECD Modified Screening Test – START DATE → FILE LR# 8483

SEC. W. G. Schmid DATE 10-27-82 SHEET NO.

ANALYSIS FORM 1038A

		A	B	C	D	E	F	G	H	J	K	L	M	N	O	P	R
	Initial analyses		C-1	C-2	R-1	R-2	203-1	203-2	600-1	600-2	203 (STERILE)	600 (STERILE)					
1	Day 0	0.7	0.8	19.1	19.8	21.6	21.4	22.2	22.7								
2		0.2	1.0	20.0	20.0	22.5	23.3	21.0	22.0								
3			1.4				21.6		21.7								
4																	
5																	
6																	
7	Day 27	0.5	1.1	0.0	1.9	5.5	4.7	5.4	3.5								
8		0.3	0.3	0.1	0.5	4.7	4.4	5.6	2.7								
9																	
10																	
11																	
12																	
13	Day 28	-0.6	0.5	0.4	-0.1	5.9	7.9	6.1	2.5		20.3	20.6					
14		-0.6	0.1	0.3	0.5	5.2	7.4	4.4	1.7								
15			0.1			6.1		5.1									
16																	
17																	
18	TOC ANALYSES USING TC MODE WITH NITROGEN SPARGE OF ACIDIFIED SAMPLES.		ANALYZED 3/25/83 THRU 3/29/83 W. G. Schmid														
19																	
20																	
21																	
22																	
23	Initial – Day-0	1.1	1.3	20.3	19.7	22.5	23.0	23.8	24.0		23.0	22.6					
24	" (dup)	0.9	0.8	21.1	20.0	22.2	23.3	24.5	23.7		24.4	23.9					
25	Day-7	0.3	-0.4	1.4	2.3	14.1	13.8	13.5	12.4								
26	" (dup)	1.1	1.8	2.2	2.0	14.0	12.8	13.0	13.1								
27	Day-14	0.8	0.4	1.4	0.7	9.1	9.5	8.6	7.9								
28	" (dup)	0.1	0.1	1.4	1.4	9.0	11.3	9.0	7.8								
29	Day-21	0.7	0.1	1.5	0.4	6.8	8.6	6.2	4.6								
30	" (dup.)	0.3	0.6	2.0	0.6	7.5	7.3	6.9	5.3								
31	Day-27	0.1	0.7	0.7	1.4	5.7	4.4	4.3	3.3								
32	(dup.)	0.5	0.8	1.1	0.5	5.3	4.9	5.0	2.9								
33	Day-28	-0.1	1.0	1.6	1.4	5.2	15.0	4.9	2.4		23.4	23.3					
34	(dup.)	0.1	0.8	1.5	1.5	5.1	15.4	5.3	2.1		29.1	23.8					
35																	

VALUES USED FOR REPORTING

W.G. Schmid 4/05/83

OECD SCREENING TEST

3M ENVIRONMENTAL LABORATORY

FILE LR # 8483

~~DRG~~ W.A. Schaal

START DATE 10-27-82 SHEET

ANALYSIS FORM

		A C-1	B C-2	C R-1	D R-2	E 203-1	F 203-2	G 600-1	H 600-2	J 203-S	K 600-S	L
1	~~Initial~~ Weight (g)	706.9	715.5	713.8	709.0	720.7	712.6	708.3	693.8	713.7	715.5	
2	10-27-82											
3												
4	Weight After Sampling	678.9	684.5	689.5	685.3	697.6	686.6	674.0	673.1	690.9	691.9	
5	~~11-10-82~~ 11-03-82											
6	W.A. Schaal											
7												
8	Weight After Sampling	651.4	655.4	668.9	660.3	669.9	662.8	643.9	648.2	666.1	666.5	
9	11-10-82 W.A. Schaal											
10												
11												
12	Weight After Sampling	627.0	627.7	641.6	634.1	645.7	638.3	617.0	621.7	640.6	639.5	
13	11-17-82 W.A. Schaal											
14												
15	Weight After Sampling	603.9	597.3	618.8	609.7	614.5	618.7	594.5	598.4	612.6	613.4	
16	~~11-22-82~~ W.A. Schaal											
17	11/23/82											
18	W.A.S. 11/23/82											
19	Weight After Sampling											
20	~~11-24-82~~	Samples Tossed										
21	11/24/82 W.A. Schaal											
22												
23												
24	Final pH	6.3	6.5	7.1	7.0	6.3	6.1	6.0	6.4	5.4	5.5	
25												
26												
27												
28												
29												
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5. ANNEX

SAMPLE FORM SHEET FOR THE MODIFIED OECD SCREENING TEST

Exp. no.:
Date of start of test:
Test / Standard material:
Theoretical test conc.: mg DOC/l
Inoculum:
Carbon analyser:

A: Control[2]:

	TOC*	DOC**
	mg/l	
Stock solution of the test material (1000 mg/l, dilution .../1000 ml of nutrient solution)		

B: Carbon determinations:

Sodium Benzoate - Reference Compound

Culture medium	Flask no.	Analyses	Theor. conc. mg/l	DOC - concentrations after x days mg/l: 0 (C_0)	~7	~14	~21	~27	28
Mineral nutrient solution with test material and with inoculum	1	a_1	/	19.1				0.0	0.4
		a_2	/	20.0				0.1	0.3
		$m_1 = \frac{a_1 + a_2}{2}$ (C_t)		19.55				0.05	0.35
	2	b_1	/	19.8				1.9	0.1
		b_2	/	20.0				0.5	0.5
		$m_2 = \frac{b_1 + b_2}{2}$ (C_t)		19.9				1.2	0.2
Mineral nutrient solution without test material but with inoculum	Blank	c_1	/						
		c_2	/						
		$m_3 = \frac{c_1 + c_2}{2}$ (C_{b1})	/	0.82				0.55	0.1

} See FC-600 sheet.

* Disagreement between DOC and TOC values points towards insufficient solubility of the test material.

** All DOC values determined after membrane filtration.

12

"Ready Biodegradability:
Modified OECD Screening Test"

C: Evaluation of raw data:

Sodium Benzoate — Reference Compound

DOC - concentrations minus blanks	Flask no.		% DOC removal after x days: 7	14	21	27	28
	1	$D_1 = \left[1 - \frac{m_{1_t} - m_{3_t}}{m_1 - m_3}\right] \cdot 100$				[illegible] (102.7)	98.1
	2	$D_2 = \left[1 - \frac{m_{2_t} - m_{3_t}}{m_2 - m_3}\right] \cdot 100$				96.6	98.4
	mean	$D = \frac{D_1 + D_2}{2}$ for day x				100	98

5. ANNEX

SAMPLE FORM SHEET FOR THE MODIFIED OECD SCREENING TEST

Exp. no.:
Date of start of test:
Test / Standard material:
Theoretical test conc.: mg DOC/l
Inoculum:
Carbon analyser:

A: Controls:

	TOC*	DOC**
Stock solution of the test material (1000 mg/l, dilution .../1000 ml of nutrient solution)	mg/l	

B: Carbon determinations:

FC-203

Culture medium	Flask no.	Analyses	Theor. conc. mg/l	DOC - concentrations after x days mg/l: 0 (C_0)	~7	~14	~21	~27	28
Mineral nutrient solution with test material and with inoculum	1	a_1	/	21.6				5.5	5.9 5.2
		a_2	/	22.5				4.7	6.1
		$m_1 = \frac{a_1 + a_2}{2}$ (C_t)		22.05				5.10	5.73
	2	b_1	/	21.4 23.3				4.7	7.9 7.4
		b_2	/	21.6				4.4	7.4
		$m_2 = \frac{b_1 + b_2}{2}$ (C_t)		22.1				4.55	7.65
Mineral nutrient solution without test material but with inoculum	Blank	c_1	/						
		c_2	/						
		$m_3 = \frac{c_1 + c_2}{2}$ (C_{b1})	/	0.82				0.55	-0.1

See FC-60[illegible] data sheet

* Disagreement between DOC and TOC values points towards insufficient solubility of the test material.

** All DOC values determined after membrane filtration.

"Ready Biodegradability:
Modified OECD Screening Test"

C: Evaluation of raw data:

FC-203

DOC - concentrations minus blanks	Flask no.		% DOC removal after x days 7	14	21	27	28
	1	$D_1 = \left[1 - \frac{m_{1t} - m_{3t}}{m_1 - m_3}\right] \cdot 100$				78.6	72.5
	2	$D_2 = \left[1 - \frac{m_{2t} - m_{3t}}{m_2 - m_3}\right] \cdot 100$				81.2	63.6
	mean	$D = \frac{D_1 + D_2}{2}$ for day x				80	68

5. ANNEX

SAMPLE FORM SHEET FOR THE MODIFIED OECD SCREENING TEST

Exp. no.:
Date of start of test:
Test - standard material:
Theoretical test conc.: mg DOC/l
Inoculum:
Carbon analyser:

A: Controls:

	TOC*	DOC**
	mg/l	
Stock solution of the test material (1000 mg/l, dilution .../1000 ml of nutrient solution)		

B: Carbon determinations:

FC-600

Culture medium	Flask no.	Analyses	Theor. conc. mg/l	DOC - concentrations after x days mg/l: 0 (C_0)	~7	~14	~21	~27	28
Mineral nutrient solution with test material and with inoculum	1	a_1	/	22.2				5.4	4.4
		a_2	/	21.0				5.6	5.1
		$m_1 = \frac{a_1 + a_2}{2}$ (C_t)		21.60				5.5	5.2
	2	b_1	/	22.7 / 22.0				3.5	2.5
		b_2	/	21.7				2.7	1.7
		$m_2 = \frac{b_1 + b_2}{2}$ (C_t)		22.13				3.1	2.1
Mineral nutrient solution without test material but with inoculum	Blank	Bk-1 c_1	/	0.7 / 0.2				0.5 / 0.3	- 0.6 - 0.6
		Bk-2 c_2	/	0.8 / 1.0 / 1.4				1.1 / 0.3	0.5 / 0.1 / 0.1
		$m_3 = \frac{c_1 + c_2}{2}$ (C_{b1})	/	0.82				0.55	- 0.10 (neg.)

* Disagreement between DOC and TOC values points towards insufficient solubility of the test material.

** All DOC values determined after membrane filtration.

avg of 5 results from 2 blanks

"Ready Biodegradability:
Modified OECD Screening Test"

C: Evaluation of raw data:

FC-600

	Flask no.		% DOC removal after x days 7	14	21	27	28
DOC - concentrations minus blanks	1	$D_1 = \left[1 - \frac{m_{1t} - m_{3t}}{m_1 - m_3}\right] \cdot 100$				76.2	74.5
	2	$D_2 = \left[1 - \frac{m_{2t} - m_{3t}}{m_2 - m_3}\right] \cdot 100$				88.2	89.2
	mean	$D = \frac{D_1 + D_2}{2}$ For day x				82	82

60/

Form 6747-11-E

TECHNICAL REPORT SUMMARY

TO: TECHNICAL COMMUNICATIONS CENTER – 201-2C-12

(Important – If report is printed on both sides of paper, send two copies to TCC.) Guidelines on reverse side.

Division: 3M Environmental Laboratory (EE & PC) St. Paul Minnesota	Dept. Number: 0535
Project: 3M Germany	Project Number: 9972410100
Report Title: OECD Biodegradation Testing on FC-600 and FC-203	Report Number: 60
To: K. F. Schroeder, 3M Neuss, Germany	Period Covered or Date: 4/18/83
Author(s): E. A. Reiner, Environmental Laboratory - 21-2W-05	Employee Number(s): 47816
Notebook Reference: None - See 3M Environmental Lab Request 8483	No. of Pages Including Coversheet: 15

SECURITY ▶ ☐ Open Report & Summary ☐ Closed Report–Open Summary

3M CHEMICAL REGISTRY ▶ ☐ Check box if new chemicals are reported. Use Chemical Registry Form 6092 to report all new substances.

KEYWORDS:
Lab Code

Other Keywords

CURRENT OBJECTIVE:

1. To measure the biodegradability of two "Light Water" products using the internationally recognized Modified OECD Screening Test.

2. To compare results with those previously obtained using the same test method.

REPORT ABSTRACT: This abstract information is distributed by the Technical Communications Center to alert 3M'ers to Company R&D.

This study used the Modified OECD Screening Test with dissolved organic carbon (DOC) analysis to measure the biodegradability of "Light Water" Brand AFFF products: FC-203 and FC-600. In 28 days, the DOC levels of FC-600 degraded by 80% and 92% for an average of 86% in two parallel tests. In 27 days the DOC of FC-203 degraded by 77% and 81% for an average of 79%. The FC-203 result differed from a similar test done in 1980 in which 93% DOC degradation occurred within 21 days. In the present study parallel sterile controls proved that the DOC loss was not due to chemical or physical processes such as adsorption, volatilization, or precipitation of the organic compounds.

Information Liaison Initials:

Introduction

The 3M Environmental Laboratory undertook this study of the biodegradability of "Light Water" products, FC-600 and FC-203, because of concern expressed by 3M Germany about inconsistencies in past biodegradation test results.

Modified OECD Screening Tests conducted in 1980 by the 3M Environmental Laboratory showed that three "Light Water" products were highly biodegradable. FC-203A, FC-206, and FC-3017 (FC-206 F) all were shown to have 90% of their dissolved organic carbon removed in the OECD Screening Test. These results, however, contradict those of H. Hellmann and D. Muller[1] using a similar yet not identical test method, the TOC Degradation Test[2]. In their study FC-206 and FC-3017 were found to have total organic carbon reductions of only 6% and 8%, respectively, in 21 days. These low biodegradation results of Hellmann, however, were also contradicted by his own work. His BOD_5/COD ratios for FC-3017 and FC-206 were 0.5 and 0.4, respectively. This showed that 50% and 40% of the theoretical oxygen demand was expressed within only 5 days. These levels of biodegradation are much greater than the 8% and 6% TOC removals which he observed in the even longer (21 day) TOC degradation test. This is clearly an inexplicable discrepancy in his work.

Hellmann's TOC degradation results are also contradicted by BOD_{20}/COD measurements made by the 3M Environmental Laboratory and other independent laboratories on 3M AFFF products. Such results typically show BOD_{20} values to range between 60 and 90% of the COD. For example, a collection of such data on 3 U.S. military specification AFFF products made by 3M is shown in Table 1.

TABLE 1

BOD, CARBONACEOUS BOD, AND COD DATA ON MIL-SPEC AFFF PRODUCT

Lab	BOD_{20} (mg/kg)	C-BOD (mg/kg)	COD (mg/kg)	$\frac{BOD_{20}}{COD}$	$\frac{C\text{-}BOD_{20}}{COD}$
		FC-203C Lot 501			
3M	580,000	600,000	730,000 740,000	.79	.81
Pace	800,000*	730,000*	810,000 730,000		
Serco	440,000	440,000	737,000	.60	.60
Capsule	570,000	620,000	950,000	.6	.65
Galbraith	616,000	671,000	776,450	.79	.86
S & W Seed					
Galbraith Knoxville	670,000	641,000	776,450	.86	.82
Average	575,000	594,000	782,000	.73	.75
S. D.	85,000	90,000	80,000	.12	.12

* Calculated BOD's from different dilutions were very inconsistent. Pace agreed to repeat this work but inadvertently lost their repeated test results as well. We did not request a third testing nor did we include these values in the averages.

		FC-206C Lot 502			
3M	290,000	290,000	380,000 360,000	0.78	0.78
Pace	350,000	370,000	450,000	0.78	0.82
Serco	240,000	260,000	370,000	0.65	0.70
Capsule	260,000	320,000	410,000	0.63	0.78
Galbraith	416,500	411,000	407,250	1.02	1.01
S & W Seed					
Galbraith Knoxville	395,500	374,000	407,250	.97	.92
Average	325,000	338,000	396,000	.81	.83
S. D.	73,000	57,00	33,000	.16	.11
		FC-780B Lot 501			
3M	240,000	230,000	320,000 330,000	.74	.71
Pace	250,000	250,000	350,000	.71	.71
Serco	210,000	220,000	287,000	.73	.77
Capsule	240,000	230,000	360,000	.67	.64
Average	235,000	232,000	329,000	.71	.71
S. D.	17,000	13,000	28,000	.03	.05

Methods and Materials

1. Procedure

Testing was performed in accordance with the OECD Guideline for testing of chemicals adopted May 12, 1981.

2. Chemicals

The reference material used was laboratory grade sodium benzoate ($C_7H_5NaO_2$) F.W. = 144.11. A 30.3 mg sample of this compound was diluted to 1.0 liters with the nutrient solution described by the method to yield a theoretical DOC of 20 mg/l. The "Light Water" samples used were supplied by G. Piessens, E.T.S.L. and consisted of FC-203 and FC-600. Lot numbers were not provided. Nutrient solutions of the "Light Water" products with DOC's of approximately 20 mg/l (based on calculations from the formulation) were prepared by bringing both 527 mg of FC-600 and 153 mg of FC-203 to 2.0 liters with nutrient solution.

3. Inoculum

The inoculum was a 50/50 mixture of the soil and secondary effluent inocula each prepared in accordance with the OECD procedure. The soil used was fresh loamy sand garden soil obtained from White Bear Lake, Minnesota, and the water for the soil extraction was chlorine-free well water. The secondary effluent sample was the supernatant from an activated sludge aeration basin at the St. Paul Metro Wastewater Treatment Plant. The combined inoculum was passed through Whatman® 54 filter paper prior to usage.

4. Sterile Controls

Replicate vessels containing 400 mg/l $HgCl_2$ served as sterile controls for both test materials and the reference compound. The sterile controls were handled in the same way as the viable cultures.

5. Instrument

The organic carbon analyzer used was a Dohrmann DC-52A. Its sensitivity limit is approximately 2 mg of carbon per liter.

6. Sample Handling and Storage

The sample preservation method used was that prescribed by the TOC manufacturer. The method involved adding 1 drop of concentrated HCl per 10 ml of filtered sample. This brought the samples to less than pH 2. The samples were then stored under refrigeration in vials with aluminum foil-lined caps. Dohrmann literature indicates that the method can stabilize potassium hydrogen phthalate, a readily biodegradable material, for several months.

Due to problems with the sensitivity of the TOC analyzer, only half of the samples were analyzed within a month after sampling. Following 4-5 months of storage, all of the samples were reanalyzed. Comparison of the TOC analyses done within 1 month with those done after 4-5 months of storage showed that in only one stored sample had the TOC changed significantly. This sample was discarded.

Results and Discussion

Table 2 summarizes the results of this study. Appendices 1 and 2 contain the actual data sheets and plots of the degradation as a function of time. Table 2 shows that the sodium benzoate reference compound was nearly completely degraded within 14 days. Degradation of both FC-600 and FC-203 continued throughout the course of the 28-day test period without plateauing. At 28 days the average TOC removal for FC-600 was 86% and at 27 days the average TOC removal for FC-203 was 79%. The test procedure recommends that chemicals giving a result greater than 70% loss of DOC (within 28 days) should be regarded as readily biodegradable. These 3M formulated AFFF products are thus even more completely degraded than is necessary for a chemical to be considered readily biodegradable.

TABLE 2

Biodegradation (% DOC removal) of Sodium Benzoate, FC-203 and FC-206

Product	% Degradation at Day: 7	14	21	27	28
Sodium Benzoate	94.4	94.7	93.2	93.5	94.5
Sodium Benzoate Duplicate	92.3	96.3	99.6	97.7	100.0
FC-203	37.4	59.2	68.4	76.7	78.0
FC-203 Duplicate	43.0	54.6	66.0	81.3	*
FC-600	46	63.5	73.5	82.1	79.9
FC-600 Duplicate	47.2	67.1	80.2	88.7	92.1

*Sample discarded due to probable contamination.

The sterile control data in Table 3 provide further substantial evidence that soluble TOC loss from the test samples is not due to physical modes such as adsorption, volatilization, or precipitation. These samples were handled identically to the test vessels, except that they contained $HgCl_2$ to prevent microbial growth. At the end of 28-day test period, these controls contained all of the initial DOC.

TABLE 3

Percent of Initial DOC in Sterile Controls at 28 days

Product	% DOC Remaining
FC-203	98.1
FC-600	101.3

Conclusion

The present study demonstrates that Light Water products FC-203 and FC-600 are "readily biodegradable" according to the recommended 70% DOC removal criteria of the Modified OECD Screening Test. FC-203 and FC-206 had average losses of DOC of 79% and 86%, respectively.

References

1. Dr. H. Hellmann and D. Muller, "Environmental Impact of Fire Extinguishing Foam Compounds (Umweltgefahrdung durch Feuerlosch-Schaummittell)." The study was carried out by the West German Institute of Hydrology (Bundesanstalt fur Gewasserkunde), April 18, 1979.

2. D. Muller and T. Tittizer, "The TOC Degradation Test - A Process to Test Organic Materials for Complete Biodegradation (Der TOC - Abbautest - ein Verfahren zur Prufung von Organbischen Substanzen auf Vollstandigen Biologischen Abbau)." Developed at the Bundesanstalt for Gewasserkunde, Koblenz, Z. F. Waser-und Abwasser-Forschung (1979).

3. Organization for Economic Cooperation and Development, Guidelines For Testing of Chemicals, Section 301 E, Ready Biodegradability: Modified OECD Screening Test, May 12, 1981.

4. Dohrman DC-52A Operating Manual, 4th Ed., 1978.

Appendix 1

5. ANNEX

SAMPLE FORM SHEET FOR THE MODIFIED OECD SCREENING TEST

Exp. no.: LR # 8483
Date of start of test: 10-27-82
Standard material: SODIUM BENZOATE
Theoretical test conc.: 20 mg DOC/l
Inoculum: SECONDARY / soil
Carbon analyser: DOHRMANN DC-53A

A: Controls:

	TOC*	DOC**
	mg/l	
Stock solution of the test material (1000 mg/l, dilution .../1000 ml of nutrient solution)		

B: Carbon determinations:

SODIUM BENZOATE

Culture medium	Flask no.	Analyses	Theor. conc. mg/l	DOC - concentrations after x days mg/l 0 (C_0)	7	14	21	27	28
Mineral nutrient solution with test material and with inoculum	1	a_1	/	20.3	1.4	1.4	1.5	0.7	1.6
		a_2	/	21.1	2.2	1.4	2.0	1.1	1.5
		$m_1 = \frac{a_1 + a_2}{2}$ (C_t)		20.7	1.8	1.4	1.75	1.8	1.55
	2	b_1	/	19.7	2.3	0.7	0.4	1.4	1.4
		b_2	/	20.0	2.0	1.4	0.6	0.5	1.5
		$m_2 = \frac{b_1 + b_2}{2}$ (C_t)		19.85	2.15	1.05	0.5	0.95	0.45
Mineral nutrient solution without test material but with inoculum	Blank #1	c_1	/	1.1	0.3	0.8	0.7	0.1	-0.1
		c_2	/	0.9	1.1	0.1	0.3	0.5	0.1
		$m_3 = \frac{c_1 + c_2}{2}$ (C_{b1})	/	1.0	0.7	0.45	0.5	0.3	0.0
Mineral nutrient solution without test material but with inoculum	Blank #2	c_1	/	1.3	-0.4	0.4	0.1	0.7	1.0
		c_2	/	0.8	1.8	0.1	0.6	0.8	0.8
		$m_3 = \frac{c_1 + c_2}{2}$ (C_{b1})	/	1.05	0.7	0.25	0.35	0.75	0.9

* Disagreement between DOC and TOC values points towards insufficient solubility of the test material.
** All DOC values determined after membrane filtration.

	DOC - AFTER x DAYS 0	7	14	21	27	28
AVERAGE OF BLANKS #1 AND #2	1.02	0.7	0.35	0.42	0.52	0.45

W.G. Schul 11.05-83

"Ready Biodegradability:
Modified OECD Screening Test"

LR# 8483

C: Evaluation of raw data:

SODIUM BENZOATE

	Flask no.		% D.O.C removal after x days				
			7	14	21	27	28
DOC - concentrations minus blanks	1	$D_1 = \left[1 - \frac{m_{1_t} - m_{3_t}}{m_1 - m_3}\right] \cdot 100$	94.4	94.7	93.2	93.5	94.4
	2	$D_2 = \left[1 - \frac{m_{2_t} - m_{3_t}}{m_2 - m_3}\right] \cdot 100$	92.3	96.3	99.6	97.7	100.0
	mean	$D = \frac{D_1 + D_2}{2}$ for day x	93	96	96	96	97

W.A. Schuh
4-05-93

"Ready Biodegradability:
Modified OECD Screening Test"

301 E

5. ANNEX

SAMPLE FORM SHEET FOR THE MODIFIED OECD SCREENING TEST

Exp. no.: LR# 8483
Date of start of test: 10-27-82
~~Test~~ · Standard material: SODIUM BENZOATE
Theoretical test conc.: mg DOC/l
Inoculum: SECONDARY EFF. / SOIL
Carbon analyser: DOHRMANN DC-52A

A: Controls:

	TOC*	DOC**
	mg/l	
Stock solution of the test material (1000 mg/l, dilution .../1000 ml of nutrient solution)		

B: Carbon determinations:

TEST MATERIAL: FC-203

Culture medium	Flask no.	Analyses	Theor. conc. mg/l	DOC - concentrations after x days mg/l: 0 (C_0)	7	14	21	27	28
Mineral nutrient solution with test material and with inoculum	1	a_1	/	22.5	14.1	9.1	6.8	5.7	5.2
		a_2	/	22.2	14.0	9.0	7.5	5.3	5.1
		$m_1 = \frac{a_1 + a_2}{2}$ (C_t)		22.35	14.05	9.05	7.15	5.5	5.15
	2	b_1	/	23.0	13.8	9.5	8.6	4.4	15.0
		b_2	/	23.3	12.8	11.3	7.3	4.9	15.4
		$m_2 = \frac{b_1 + b_2}{2}$ (C_t)		23.15	13.3	10.4	7.95	4.65	15.2
Mineral nutrient solution without test material but with inoculum	Blank #1	c_1	/						
		c_2	/						
		$m_3 = \frac{c_1 + c_2}{2}$ (C_{b1})	/						
Mineral nutrient solution without test material but with inoculum	Blank #2	c_1	/						
		c_2	/						
		$m_3 = \frac{c_1 + c_2}{2}$ (C_{b1})	/						

NOT USED — POSSIBLE CONTAMINATION OF SAMPLE PRIOR TO ANALYSIS

* Disagreement between DOC and TOC values points towards insufficient solubility of the test material.
** All DOC values determined after membrane filtration.

→ SEE SODIUM BENZOATE PAGE FOR BLANK DATA.

Wade A. Schi
4-05-83

"Ready Biodegradability:
Modified OECD Screening Test"

L.R # 6483

C: Evaluation of raw data:

FC-203

	Flask no.		% DOC removal after x days 7	14	21	27	28
DOC - concentrations minus blanks	1	$D_1 = \left[1 - \frac{m_{1_t} - m_{3_t}}{m_1 - m_3}\right] \cdot 100$	37.4	59.2	68.4	76.7	78.0
	2	$D_2 = \left[1 - \frac{m_{2_t} - m_{3_t}}{m_2 - m_3}\right] \cdot 100$	43.0	54.6	66.0	81.3	★
	mean	$D = \frac{D_1 + D_2}{2}$ for day x	40	57	67	79	78 (1)

★ NO DATA – RESULTS APPEAR TO SHOW SAMPLE CONTAMINATION PRIOR TO DOC ANALYSES

(1) VALUE BASED ON ONE TEST SOLUTION RESULT.

W.A. Schuld
4-05-83

"Ready Biodegradability:
Modified OECD Screening Test"

301 E

5. ANNEX

SAMPLE FORM SHEET FOR THE MODIFIED OECD SCREENING TEST

Exp. no.: LR # 8483
Date of start of test: 10-27-82
~~Test~~ Standard material: SODIUM BENZOATE
Theoretical test conc.: mg DOC/l
Inoculum: SECONDARY EFF. /SOIL
Carbon analyser: DC-52A (DOHRMANN)

A: Controls:

	TOC*	DOC**
	mg/l	
Stock solution of the test material (1000 mg/l, dilution .../1000 ml of nutrient solution)		

B: Carbon determinations:

TEST MATERIAL: FC-600

Culture medium	Flask no.	Analyses	Theor. conc. mg/l	DOC - concentrations after x days mg/l					
				0 (C_0)	7	14	21	27	28
Mineral nutrient solution with test material and with inoculum	1	a_1	/	23.8	13.5	8.6	6.2	4.3	4.9
		a_2	/	24.5	13.0	9.0	6.9	5.0	5.3
		$a_3 = \frac{a_1 + a_2}{2}$ (C_t)		24.15	13.25	8.8	6.55	4.65	5.1
	2	b_1	/	24.0	12.4	7.9	4.6	3.3	2.4
		b_2	/	23.7	13.1	7.8	5.3	2.9	2.1
		$b_3 = \frac{b_1 + b_2}{2}$ (C_t)		23.85	12.75	7.85	4.95	3.1	2.25
Mineral nutrient solution without test material but with inoculum	Blank #1	c_1	/						
		c_2	/						
		$c_3 = \frac{c_1 + c_2}{2}$ (C_{b1})	/						
Mineral nutrient solution without test material but with inoculum	Blank #2	d_1	/						
		d_2	/						
		$d_3 = \frac{d_1 + d_2}{2}$ (C_{b1})	/						

* Disagreement between DOC and TOC values points towards insufficient solubility of the test material.
** All DOC values determined after membrane filtration.

SEE SODIUM BENZOATE PAGE FOR BLANK DATA.

W. G. Schul
4-05-83

"Ready Biodegradability:
Modified OECD Screening Test"

LR# 8483

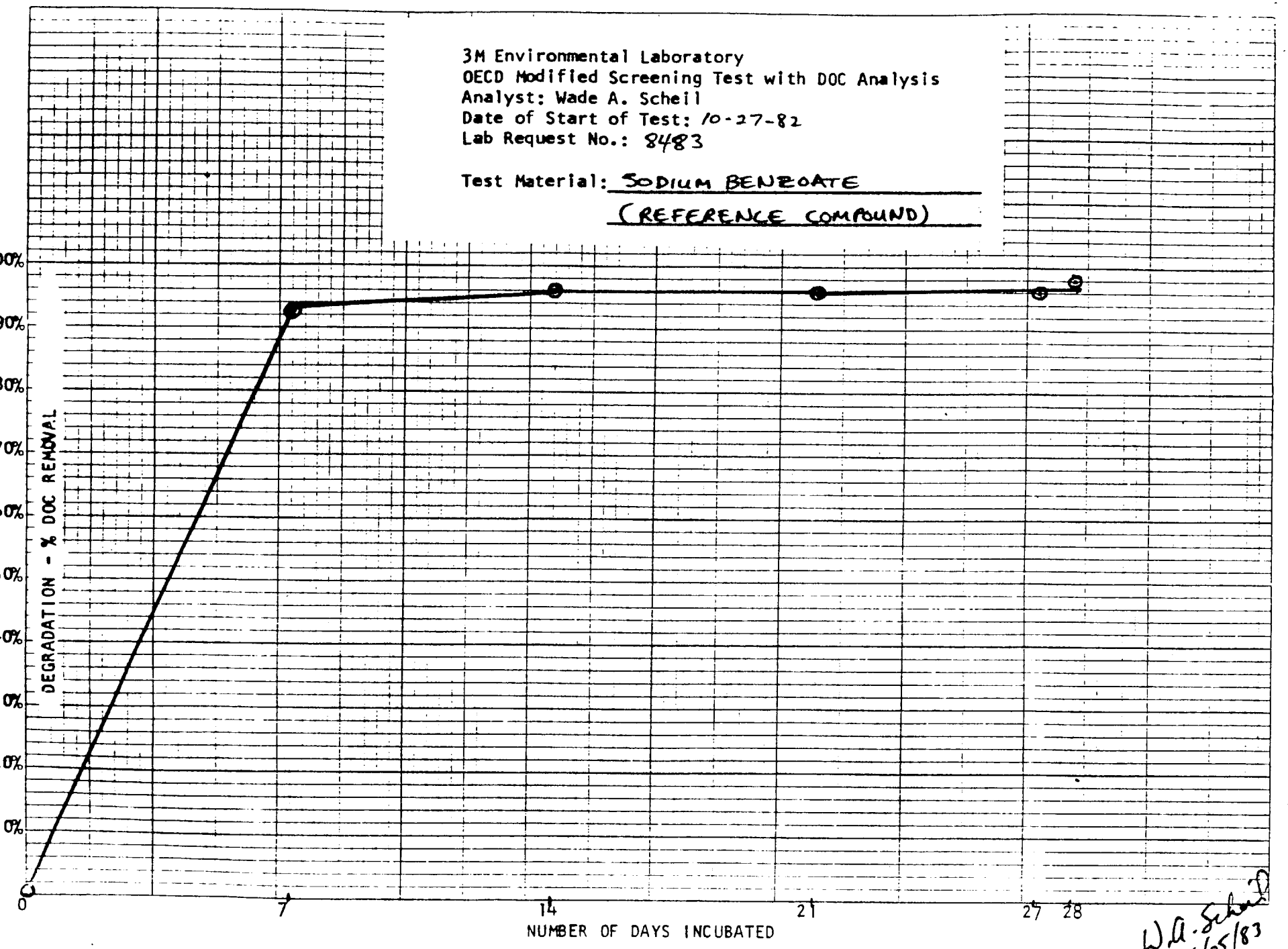
C: Evaluation of raw data:

FC-600

	Flask no.		% DOC removal after x days 7	14	21	27	28
DOC - concentrations minus blanks	1	$D_1 = \left[1 - \frac{m_{1_t} - m_{3_t}}{m_1 - m_3}\right] \cdot 100$	46.0	63.5	73.5	82.1	79.9
	2	$D_2 = \left[1 - \frac{m_{2_t} - m_{3_t}}{m_2 - m_3}\right] \cdot 100$	47.2	67.1	80.2	88.7	92.1
	mean	$D = \frac{D_1 + D_2}{2}$ for day x	47	65	77	85	86

W. A. Scheil
4-05-83

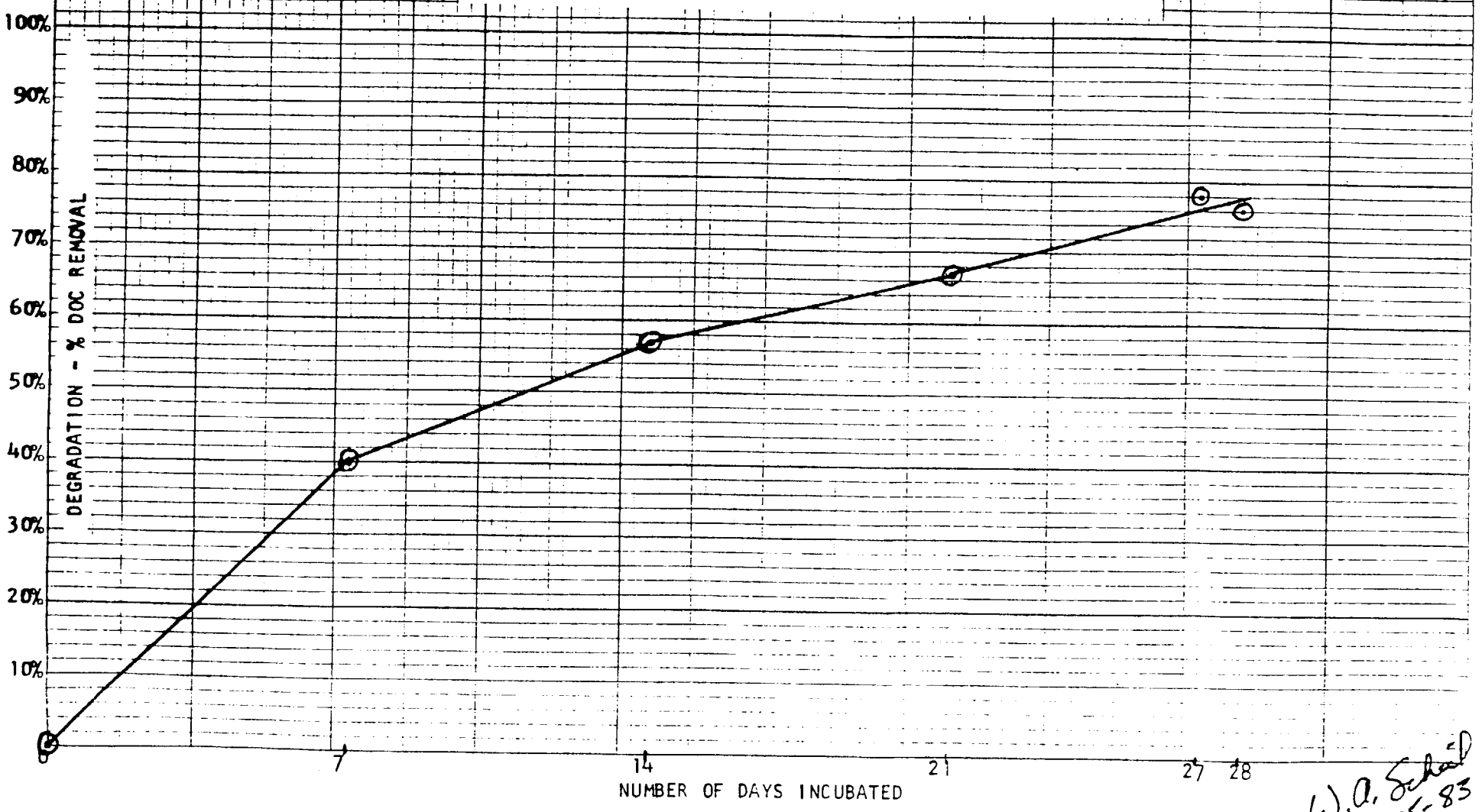
Appendix 2



W.A. Scheil
4/05/83

3M Environmental Laboratory
OECD Modified Screening Test with DOC Analysis
Analyst: Wade A. Scheil
Date of Start of Test: 10-27-82
Lab Request No.: 8483

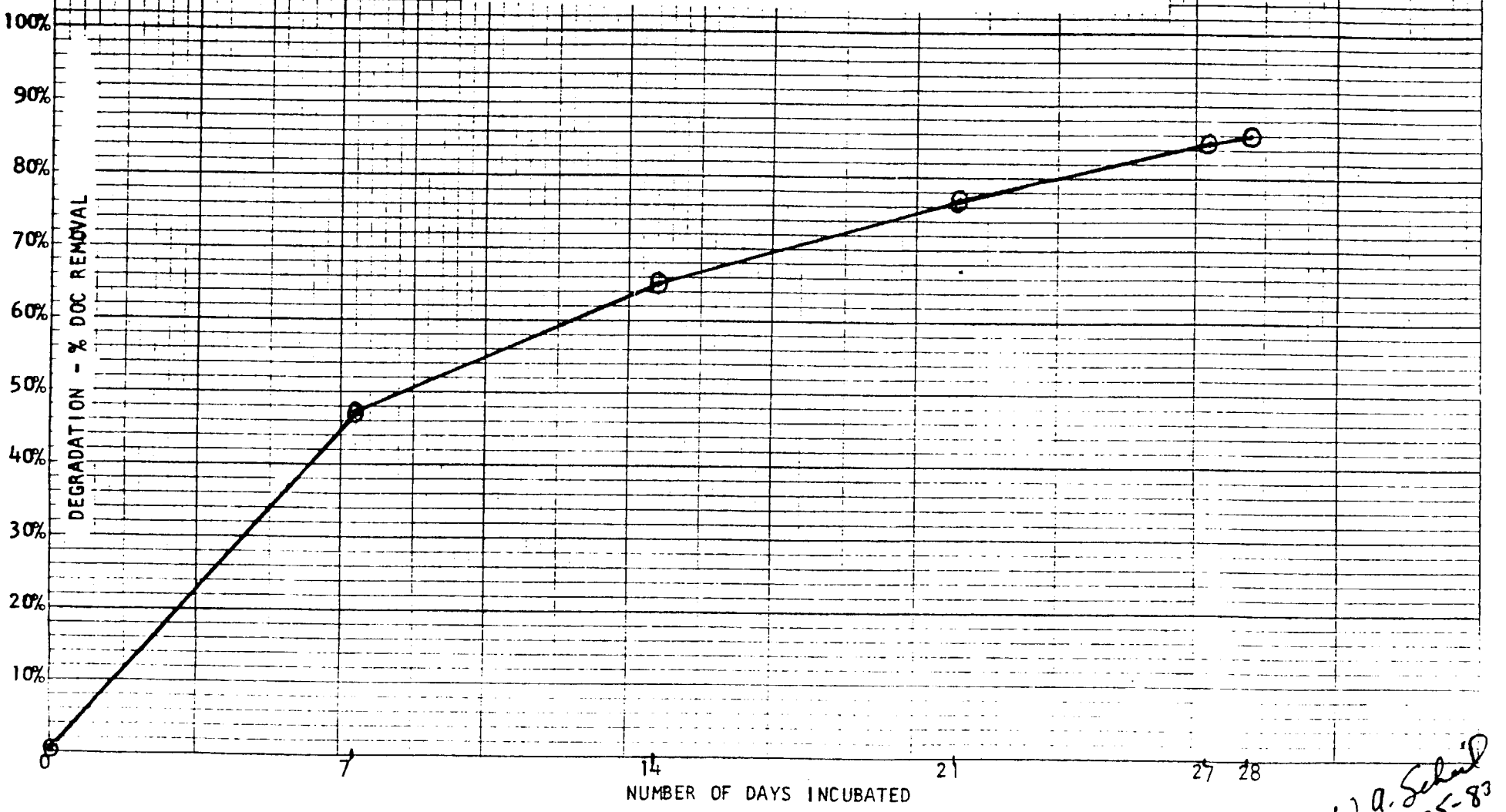
Test Material: FC-203 (ANTWERP)



W.A. Scheil
11-05-83

3M Environmental Laboratory
OECD Modified Screening Test with DOC Analysis
Analyst: Wade A. Scheil
Date of Start of Test: 10-27-82
Lab Request No.: 8483

Test Material: FC-600 (ANTWERP)



W. A. Scheil 11-05-82

Form 6747-11-D

TECHNICAL REPORT SUMMARY

TO: TECHNICAL COMMUNICATIONS CENTER – 201-2C-12

(Important – If report is printed on both sides of paper, send two copies to TCC.) Guidelines on reverse side.

Division: Environmental Laboratory (EE & PC) St. Paul, Minnesota	Dept. Number: 0535
Project: International	Project Number: 9970010100
Report Title: Modified OECD Screening Test on FC-203, FC-203A, and FC-600	Report Number: #003
To: D. Reese, Toxicology, 3M Germany	Period Covered or Date: 7/9/84
Author(s): E. A. Reiner - Environmental Laboratory - 21-2W-05	Employee Number(s): 47816
Notebook Reference: None - See Environmental Lab Request #9611	No. of Pages Including Coversheet: 12

SECURITY ▶ ☒ Open Report & Summary ☐ Closed Report–Open Summary

3M CHEMICAL REGISTRY ▶ ☐ Check box if new chemicals are reported. Use Chemical Registry Form 6092 to report all new substances.

KEYWORDS:
Lab Code

Other Keywords

CURRENT OBJECTIVE:

To test the repeatability of the modified OECD Screening Test in measuring the biodegradability of Light Water products.

REPORT ABSTRACT: This abstract information is distributed by the Technical Communications Center to alert 3M'ers to Company R&D.

This study again used the modified OECD Screening Test to evaluate the biodegradability of Antwerp Light Water products. The products studied in this set of tests were FC-203, FC-203 NFP (equivalent to FC-203A), and FC-600. Also tested was U.S. FC-203 Lot 3072. One of these products, FC-203 NFP, was also included in the previous set of tests done in 1980. The results of this testing were consistent with previous OECD Screening Test studies on Light Water products. The results indicate that all of the tested products are readily biodegradable.

cc: R.L.Bohon-21-2W-05
D.R.Ricker-236-1B-10
D.W.Kent-236-2A-04

Information Liaison Initials: EAR

MODIFIED OECD SCREENING TEST ON FC-203, FC-203A, AND FC-600

INTRODUCTION

This study was undertaken to demonstrate the biodegradability of two Light Water (L/W) products, FC-600 and FC-203, using the internationally recognzied Modified OECD Screening Test[1]. This testing was requested by 3M Germany because of inconsistency in the results of past biodegradation tests done on these products.

Modified OECD Screening Tests conducted in 1980 by the 3M Environmental Laboratory showed that three L/W products were highly biodegradable[2]. The products tested were FC-203A (previously identified as FC-203 NFP), FC-206, and FC-3017 (FC-206 freeze protected). Greater than 90% of the dissolved organic carbon (DOC) of these 3 products was removed during the 1980 testing. See Table 1 for a summary of these results.

Table 1

Results of 1980 Testing on Light Water Products

Product	% Degradation at Day: 7	14	21	27	28
FC-203 NFP[1]	13	85	93	91	93
FC-206	23	90	94	93	92
FC-3017[2]	37	94	96	96	95
Hydroquinone[3]	89	92	97	94	93

Footnotes:

[1] FC-203 NFP is nonfreeze protected FC-203 and is equivalent to FC-203A.
[2] FC-3017 is freeze protected FC-206.
[3] Hydroquinone was the reference product.

These 1980 results contradict those of H. Hellmann and D. Muller[3] who used a similar test method, the TOC Degradation Test[4]. Hellmann's TOC (total organic carbon) test results, however, are an exception from normally obtained results. His finding of 8% TOC removal for FC-3017 and 6% TOC removal for FC-206 in the 21-day long TOC Degradation Test contradict his own BOD_5/COD data. His 5-day biochemical oxygen demand (BOD) results were 50% and 40% of the chemical oxygen demand (COD) of FC-3017 and FC-206, respectively. This is a significant level of degradation for a 5-day BOD test. These opposite results within his own paper clearly are an inexplicable discrepancy.

Hellmann's TOC Degradation Test results are also inconsistent with BOD_{20}/COD measurements made by the 3M Environmental Laboratory and by other independent laboratories on 3M AFFF products. Such results typically show BOD_{20} values to range between 60% and 90% of the COD. For example, a collection of such data on 3 U.S. military specification AFFF products manufactured by 3M is shown in Table 2.

Table 2

BOD, Carbonaceous BOD, and COD Data on Mil-Spec AFFF Product

Lab	BOD_{20} (mg/kg)	C-BOD_{20} (mg/kg)	COD (mg/kg)	$\frac{BOD_{20}}{COD}$	$\frac{C\text{-}BOD_{20}}{COD}$
		FC-203 C Lot 501			
3M	580,000	600,000	730,000 740,000	.79	.81
Pace	800,000*	730,000*	810,000 730,000		
Serco	440,000	440,000	737,000	.60	.60
Capsule	570,000	620,000	950,000	.6	.65
Galbraith inoculum #1	616,000	671,000	776,450	.79	.86
Galbraith inoculum #2	670,000	641,000	776,450	.86	.82
Average	575,000	594,000	782,000	.73	.75
S. D.	85,000	90,000	80,000	.12	.12

* Calculated BOD's from different dilutions were very inconsistent.

		FC-206C Lot 502			
3M	290,000	290,000	380,000 360,000	0.78	0.78
Pace	350,000	370,000	450,000	0.78	0.82
Serco	240,000	260,000	370,000	0.65	0.70
Capsule	260,000	320,000	410,000	0.63	0.78
Galbraith inoculum #1	416,500	411,000	407,250	1.02	1.01
Galbraith inoculum #2	395,500	374,000	407,250	.97	.92
Average	325,000	338,000	396,000	.81	.83
S. D.	73,000	57,000	33,000	.16	.11

		FC-780B Lot 501			
3M	240,000	230,000	320,000 330,000	.74	.71
Pace	250,000	250,000	350,000	.71	.71
Serco	210,000	220,000	287,000	.73	.77
Capsule	240,000	230,000	360,000	.67	.64
Average	235,000	232,000	329,000	.71	.71
S. D.	17,000	13,000	28,000	.03	.05

METHODS AND MATERIALS

1. Procedure

Testing was performed in accordance with the OECD Guidelines for testing of chemicals adopted May 12, 1981.[1]

2. Chemicals

The reference materials used were laboratory grade sodium benzoate, ($C_7H_5NaO_2$) F.W. = 144.11 and linear alkylbenzene sulfonate (LAS). The LAS solution used was a 5.68% active solution supplied by the U.S.Environmental Protection Agency as a reference material for biodegradation testing. The Light Water (L/W) test mateials used were U.S. FC-203 Lot 3072 provided in 1983 by the Commercial Chemicals Division, FC-203 and FC-600 samples provided by E.T.S.L. Antwerp in 9/82 and FC-203 NFP provided by E.T.S.L. in 1980. Antwerp provided no lot numbers with their samples. FC-203 NFP (nonfreeze protected) is equivalent to FC-203A.

Prior to testing, the L/W and reference materials were dissolved in the OECD nutrient solution and brought to the following initial DOC concentrations: sodium benzoate 20 mg/l, LAS approximately 15 mg/l, and L/W products approximately 40 mg/l. The L/W TOC was higher than that used in previous testing (20 mg/l) because our experience has shown that L/W at 40 mg/l is not toxic. Also higher concentrations improve the accuracy of the TOC analysis.

For this study, nutrient solutions were prepared using both vitamin sources allowed by the OECD Screening Test, yeast extract and synthetic vitamin solution. This was done to determine if the vitamin source affected the extent of L/W degradation.

INOCULUM

The inoculum was a 50/50 mixture of soil extract and secondary effluent prepared in accordance with OECD procedures. The soil, which was extracted, was a mixture of fresh, sandy loam garden soils obtained from Ramsey and Washington Counties in Minnesota. The secondary effluent sample was the supernatant of an activated sludge mixed liquor sample taken from an aeration basin at the St. Paul Metro Wastewater Treatment Plant. The combined inoculum was passed through Whatman 54 filter paper prior to usage.

INSTRUMENT

The organic carbon analyzer used was a Dorhman DC-52A. Its sensitivity limit is approximately 2 mg of carbon per liter.

SAMPLE HANDLING AND STORAGE

The sample preservation method used was that prescribed by the manufacturer of the organic carbon analzyer.[5] The method involves adding 1 drop of concentrated HCl per 10 ml of filtered sample. This brings the samples to less than pH 2. The samples were stored under refrigeration in vials with aluminum foil lined caps. Our experience is that this storage method can preserve L/W containing samples for several months without affecting the DOC. All DOC analyses on samples from this study were done within one week of its completion.

RESULTS AND DISCUSSION

Table 3 summarizes the results of this study. This table shows that sodium benzoate, the reference compound, was nearly completely degraded (95%) within 7 days. Every replicate of all of the L/W products showed greater than 80% TOC removal within the 28-day test period. Seven of the ten test flasks containing the various L/W products showed greater than 90% TOC removal. The average DOC removal for all four L/W product was greater than 90%.

The results of this testing are shown graphically in Figures 1 and 2. These results are averaged, and because of this, do not show the actual pattern of degradation in the individual cultures. Figure 3 is an example graph of 2 cultures degrading the same product, FC-203 NFP. It shows that once degradation of this product began that it was very rapid, but that the lag prior to the onset of degradation was variable. The reason for this variability between the duplicate cultures was not known nor was it expected. It may have been due to the fact that the cultures received different levels of viable microorganisms capable of degrading the major components of the Light Water products. Thus, the population of bacteria with these capabilities had to build up in the cultures prior to the onset of rapid (exponential) degradation.

Review of the data in Table 3 indicates that results of replicate testing of the other L/W products also varied considerably at days 7, 14, and 21. Again, this variation appears to be due primarily to the length of the lag phase prior to the onset of biodegradation. However, due to the small number of samples taken, one frequently could not tell how long biodegradation of individual L/W containing cultures took following the initial lag.

Table 3

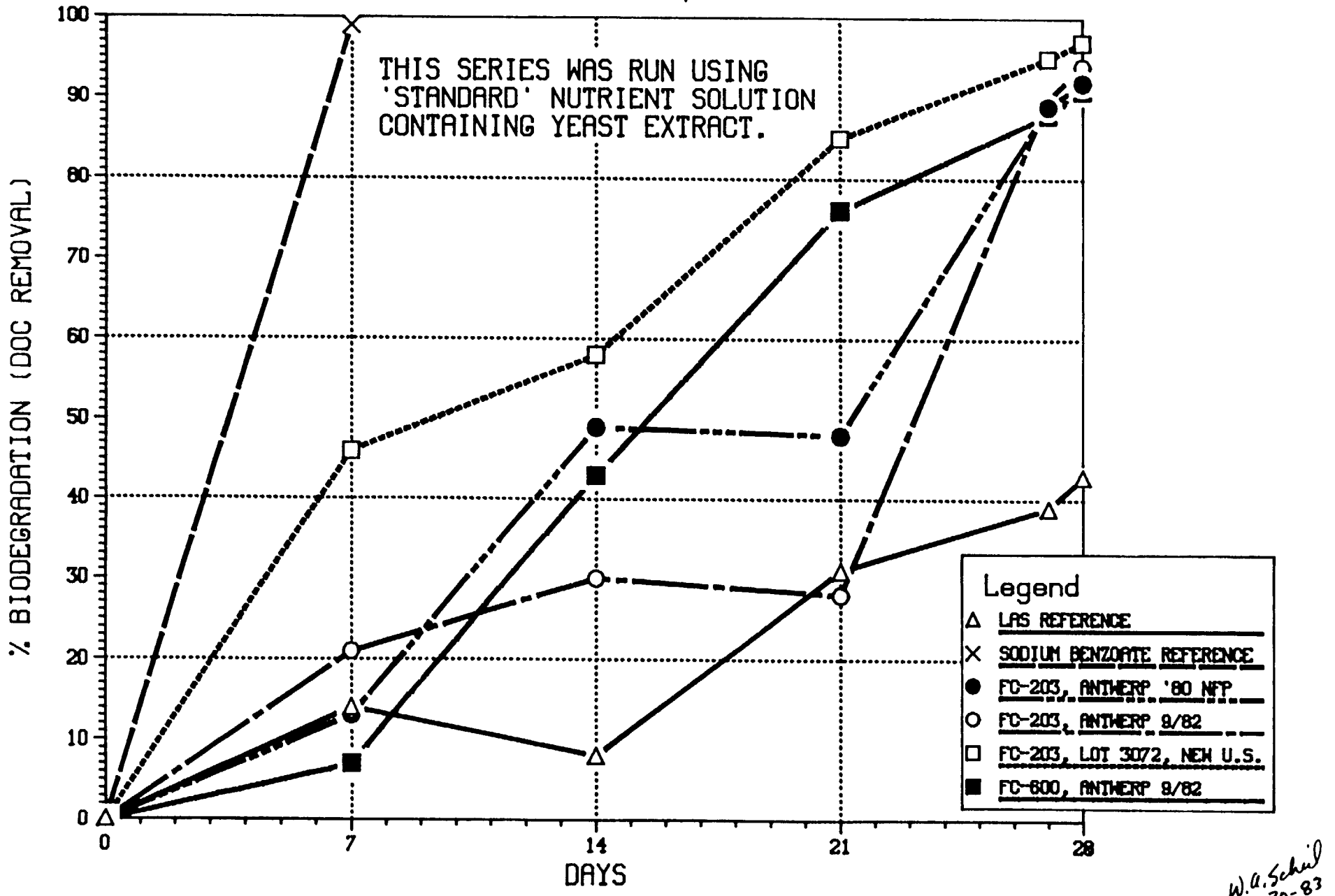
Biodegradation of LIGHT WATER Products and Refernce Materials

Product/Vitamin Source	% DOC Removal After X Days					
	7	14	17	21	27	28
Sodium Benzoate/Yeast-1[1]	98.8					
Sodium Benzoate/Yeast-2	99.6					
Sodium Benzoate/Vit.-1[2]	94.4					
Sodium Benzoate/Vit.-2	88.6					
Mean	95					
FC-203[3]/Yeast-1	11.7	30.5	--[4]	28.4	89.8	95.3
FC-203/Yeast-2	29.5	29.1	--	27.0	91.0	92.1
FC-203/Vit.-1	29.4	32.6	--	27.7	80.5	83.6
FC-203/Vit.-2	24.8	88.6	--	90.2	95.6	95.2
Mean						92
U.S. FC-203/Yeast-1	26.9	25.1	--	79.2	95.5	96.5
U.S. FC-203/Yeast-2	64.3	90.8	--	90.7	96.0	97.5
Mean						97
FC-203A[5]/Yeast-1	12.6	8.5	--	11.0	86.4	87.2
FC-203A/Yeast-2	13.2	89.1	--	85.1	92.5	96.0
Mean						92
FC-600[3]/Yeast-1	6.6	75.5	--	--[6]	89.3	94.2
FC-600/Yeast-2	7.7	10.5	--	76.3	87.0	88.8
Mean						91
LAS/Yeast-1	22.8	7.8	25.1	30.8	49.1	52.1
LAS/Yeast-2	5.9	8.8	20.4	--[6]	29.7	33.4
Mean						43

Footnotes:
(1) Used yeast extract as vitamin source.
(2) Used OECD synthetic vitamin solution as vitamin source.
(3) From Antwerp 1982.
(4) 17-day samples were only taken for LAS.
(5) From Antwerp 1980. Originally labelled FC-203 NFP.
(6) Sample contaminated, not used.

Figure 1

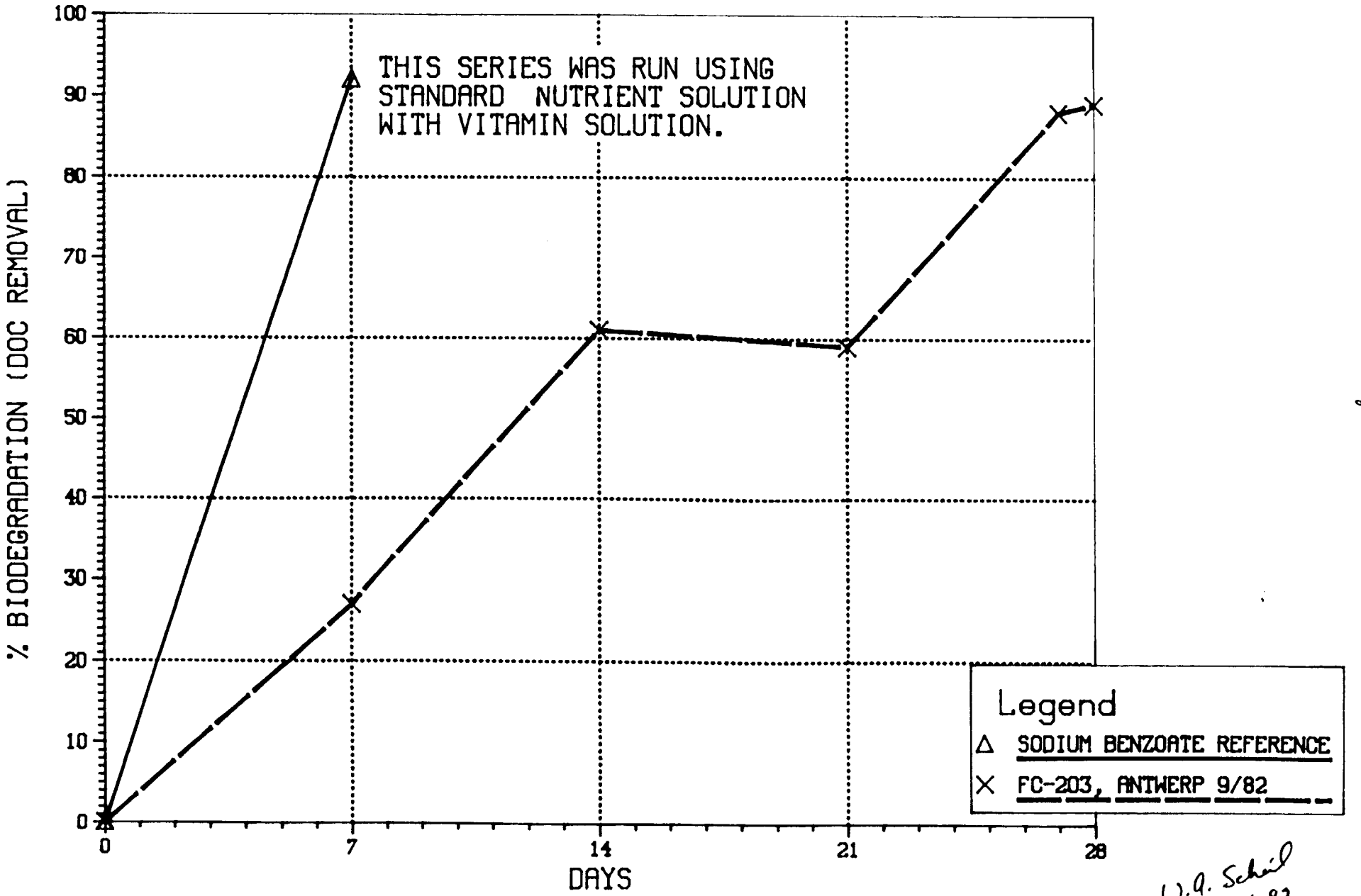
READY BIODEGRADABILITY: Modified Screening Test
OECD 301E -- Adopted 12 May 81
Environmental Lab Request No. 9611



W.A. Schul
9-30-83

Figure 2

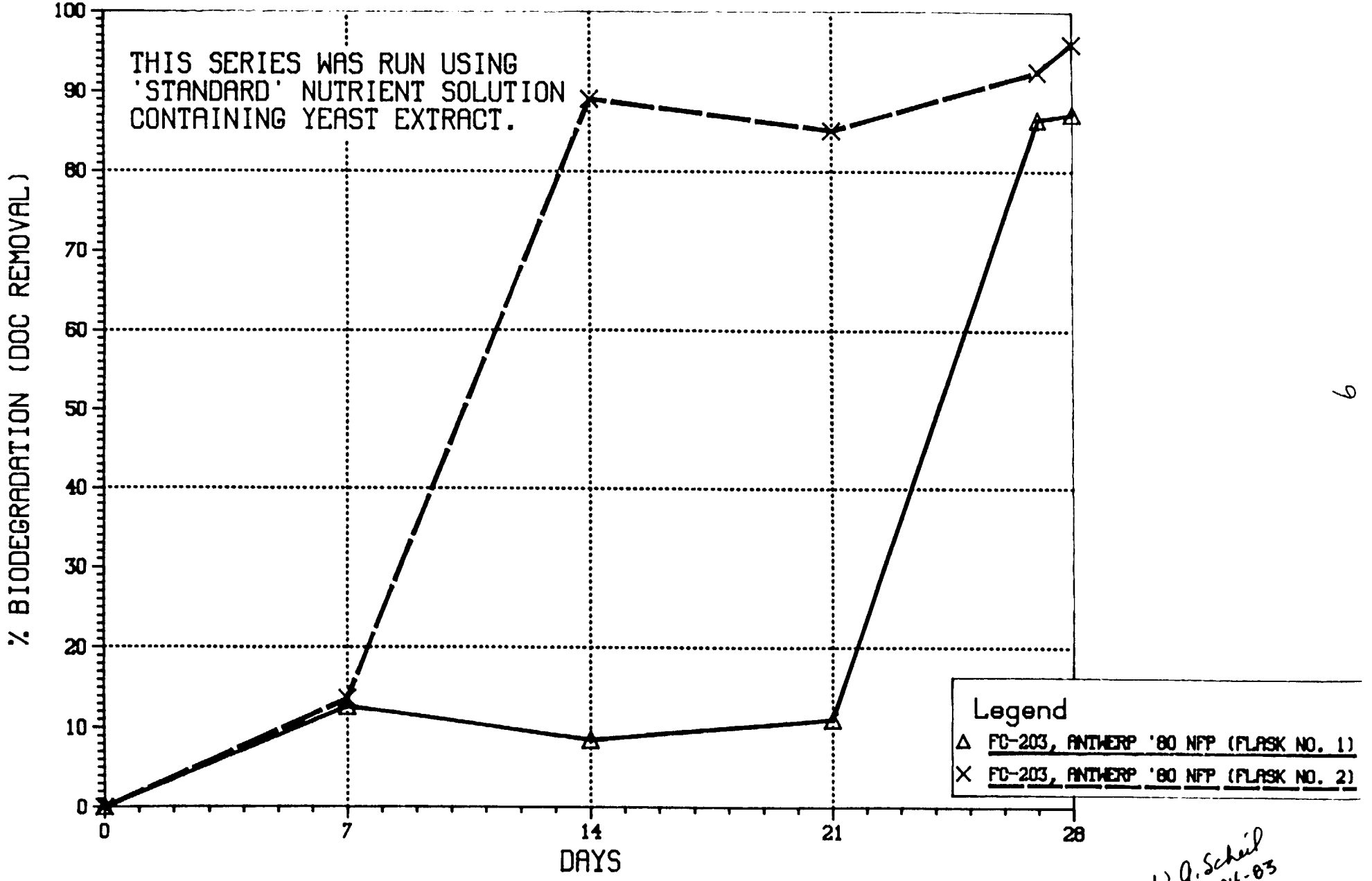
READY BIODEGRADABILITY: Modified Screening Test
OECD 301E -- Adopted 12 May 81
Environmental Lab Request No. 9611



W.A. Schiel
10-04-83

Figure 3

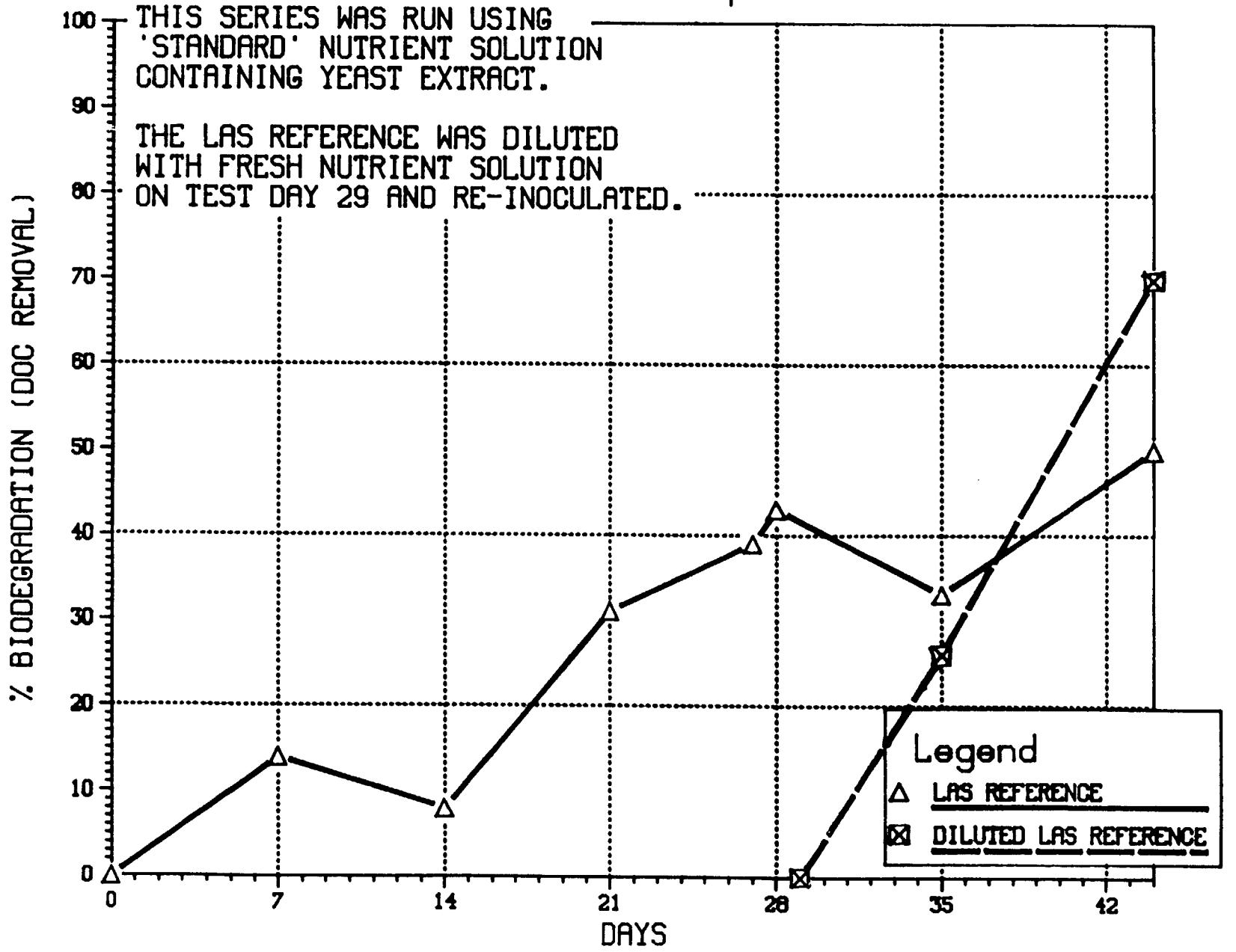
READY BIODEGRADABILITY: Modified Screening Test
OECD 301E -- Adopted 12 May 81
Environmental Lab Request No. 9611



W.A. Scheil
10-04-83

Figure 4

READY BIODEGRADABILITY: Modified Screening Test
OECD 301E -- Adopted 12 May 81
Environmental Lab Request No. 9611



The modified OECD Screening Test is intended for use on single chemical substances. L/W products, on the other hand, are aqueous solutions containing a mixture of chemicals. Pure chemicals, which have greater than 70% loss of DOC within 28 days, are regarded by the OECD guidelines as being readily biodegradable. Also, this result has to be reached within 10 days of the DOC removal first exceeding 10%. Only one of the replicates of each of the four L/W products tested met this second OECD requirement of 70% degradation within 10 days of first reaching 10% degradation. This requirement, however, is not realistically applicable to L/W or other formulated products (mixtures). The chemicals within mixtures will frequently have differing susceptibilities to biodegradation. Readily degradable chemical components of such products may start to degrade immediately, while the onset of degradation of other chemicals in these products may be delayed. Thus, disregarding this second criteria, which is not reasonable for mixtures of chemicals, the results show that the mixture of chemical components in each of these products is readily biodegradable.

Cultures tested with the synthetic vitamin solution showed, on average, a slightly lower level of DOC removed than cultures using yeast extract as the vitamin source. Future OECD screening tests should thus use yeast extract since the synthetic vitamin solution is more difficult to prepare and does not give improved degradation.

LAS, which is a readily biodegradable and commonly used surfactant in detergents, did not degrade fully under these test conditions. This occurred in spite of the fact that its concentration was only 15 mg/l of DOC compared to the 40 mg/l of DOC used for the Light Water products. The reason for the slow rate of LAS biodegradation appears to be that LAS inhibited microbial degradation at the test concentration[6]. To check this hypothesis, at the end of the 28-day test period, the remaining LAS containing test cultures were split and half were diluted to twice their volume with fresh inoculated nutrient media. At this point, the rate of degradation in the diluted cultures increased substantially. Seventy percent of the DOC remaining at the time of dilution was removed within 15 days. Results of this work on LAS are shown graphically in Figure 4. Another researcher, has similarly reported a low level of biodegradation of LAS in the modified OECD screening test. D. Liu found that Marlon A (dodecylbenzene sulfonate) had a half life of 39 days.[7]

CONCLUSIONS

The present studies repeated the findings of previous OECD Screening Tests on Light Water products done by the 3M Environmental Laboratory. Both sets of tests showed that L/W products are "readily biodegradable." In all cases, the average DOC removal for L/W products was greater than 90% after 28 days. L/W products were much more degradable than the very commonly used and "readily biodegradable" surfactant, LAS.

REFERENCES

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(3) Dr. H. Hellmann and D. Muller, "Environmental Impact of Fire Extinguishing Foam Compounds (Umweltgefahrdung durch Feuerlosch-Schaummittell)." The study was carried out by the West German Institute of Hydrology (Bundesanstalt fur gewasserkunde), April 18, 1979.

(4) D. Muller and T. Tittizer, "The TOC Degradation Test - A Process to Test Organic Materials for Complete Biodegradation (Der TOC - Abbautest - ein Verfahren zur Prufung von Organbischen Substanzen auf Vollstandigen Biologischen Abbau)." Developed at the Bundesanstalt for Gewasserkunde, Koblenz, Z. F. Waser-und Abwasser-Forschung (1979).

(5) Dohrman DC-52A Operating Manual, 4th Ed., 1978.

(6) R. D. Swisher, Surfactant Biodegradation, pages 16-17 and 147, Marcel Dekker, Inc., 1970.

(7) D. Liu, Study on Biodegradation of Aniline and Marlon A, Biodeterioration 5, Edited by T. A. Oxley and S. Barrry, 1983.

L/W TOC of FC-600

%in Prod	Components	Emperical Formula	MW	MWof C	TOC
2	Sultone foamer	C16 H23 F13 S2 06 N2	650	192	0.0059
1	Filmer (FC-95)	C8 F17 S 03 K	522	128	0.0025
1	Na octyl sulfate	C8 H17 04 S Na	232	96	0.0041
1	Na decyl sulfate	C10 H21 04 S Na	260	120	0.0046
0.1	Dowicide A	C12 H17 05 Na	264	144	0.0005
0.05	Benzotriazol	C6 H5 N3	119	72	0.0003
0.8	Kelco Keltrol	assume C6 H10 04	146	72	0.0039
0.8	Stanley H-277 Starch	assume C6 H10 04	146	72	0.0039
12	Butyl Carbitol	C8 H18 03	162	96	0.0711
81.25	DI Water	H2 0	18	0	0

Product TOC= 0.097 g/g

TOC
Calculated
10/4/88