

MICROBICS MICROTOX® TOXICITY TEST

TEST SUBSTANCE

Identity: Mixture containing Perfluorooctanesulfonate; may also be referred to as PFOS, FC-95, or as a component of FC-3072. (1-Octanesulfonic acid) (CAS # 2795-39-3).

Remarks: The 3M production lot number was not noted. The test sample is FC-3072. Current information indicates it is a mixture of 1.67% test substance, 33.33% diethylene glycol butyl ether, 29.23% water, 20.83% urea, 5% sodium octyl sulfate, 3.33% sulfonated alkyl ether sulfate salts, 4.60% acrylic foamer, 1.67% triethanolamine, and 0.33% tolyl triazole.

The following summary applies to the test sample as a mixture of the test substance in water solution with incompletely characterized concentrations of impurities. Data may not accurately relate toxicity of the test sample with that of the fluorochemical component.

METHOD:

Method: Microbics Microtox® "BASIC" Procedure

Test type: Acute static

GLP: No

Year Completed: 1995

Species: *Photobacterium phosphoreum*

Analytical monitoring: pH and osmotic condition.

Exposure period: 30-minutes

Statistical methods: EC50 values calculated by statistical linear regression program provided for Microtox®

Test organism source: Microbics' Reagent.

Test conditions:

Diluent: Millipore Milli-Q™ water

Test temperature: Not noted.

Stock and test solution preparation: A 2000 mg/L primary stock solution was prepared by dissolving 200 mg test substance in 100 mL Millipore Milli-Q™ water. A secondary stock solution of 520 mg/L was prepared by diluting 26 mL of initial stock solution with water. The pH of the stock solution was 6.8. An osmotic adjustment was made using 200 mg NaCl dissolved in 10 mL stock solution. All test solutions were made by proportional dilutions

Exposure vessels: 4 mL cuvettes

Number of replicates: two

Number of concentrations: Four plus a blank control

Element basis: Percent light loss

RESULTS

Nominal concentrations: Blank control, 32.5, 65, 130, and 260 mg/L

Element value: 5-minute EC₅₀ = 305 (282 - 330) mg/L
 15-minute EC₅₀ = 184 (177 - 192) mg/L
 30-minute EC₅₀ = 139 (134 - 144) mg/L

Element values based on nominal concentrations.

Remarks: Testing was conducted on the mixture of the test substance as described in the test substance remarks field. The values reported apply to that mixture and not the fluorochemical proportion alone.

CONCLUSIONS

The test substance 30-minute EC₅₀ for *Photobacterium phosphoreum* was determined to be 139 mg/L with a 95% confidence interval of 134 to 144 mg/L.

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

DATA QUALITY

Reliability: Klimisch ranking = 2. This study meets the criteria for quality testing. However, sample purity was not properly characterized and the study lacks analytical confirmation of the amount of fluorochemical proportion in solution.

REFERENCES

This study was conducted by the 3M Environmental Laboratory, St. Paul, Minnesota, Lab Request number M3597, 1995.

OTHER

Last changed: 6/25/00.

LAB REQUEST NO. M3597
REQUESTOR NAME: SBS/KEN GOEBEL
DEPARTMENT: 3058
PROJECT NO: BILTWENVIR
DATE RECEIVED: 11/29/1994
DESC: FC-3072 AFFF BASE

CONTRACT LAB(S): ASCI
EXP COMP DATE: 02/06/1995
DATE COMPLETED: 03/24/1995
PROJECT LEAD: TL HATFIELD
PHONE NO: 651-778-7863
3M FAX NO: 651-778-6176

TESTING IS TO BE DONE IN-HOUSE.

Sample sent to contract lab for toxicity to fish evaluation due to problems with lab fish cultures. SAB 2/20/95
Results from Microtox testing:

Test Conc. mg/L	Percent Light Loss as Compared to the Control		
	5-min exp.	15-min exp.	30-min exp.
32.5	10	12	15
65	16	21	27
130	27	37	47
260	46	62	74
EC50	305	184	139
sab 3/16/95			

Results from Fathead minnow toxicity test:
After 96-hours of exposure, all test fish were still alive in 1000 mg/L test substance.

SAB 3/22/95

Activated Sludge respiration inhibition test results:
After 3-hours of exposure, there was 15% inhibition in respiration at 1000mg/L
SAB 3/22/95

Results from Daphnia magna toxicity test:
No mortality or immobilization was observed at 1000 mg/L after 48-hours.
SAB 3/23/95

SAMPLE	DATE	CODE	DESCRIPTION	RESULT	MDL or (95% C.I.)
1		FC-3072	LIQUID		
ASCI	48-HR-EC50	DAPHNIA ACUTE TEST		>1000 MG/L	(-)
	96-HR-LC50	*FISH ACUTE TEST - 96 HOUR		>1000 MG/L	(-)
	MICROTOX	MICROTOX - EC50		139 MG/L	(134 - 144)
	SLUDG-RESP	SLUDGE RESPIRATION - EC50		>1000 MG/L	(-)

* = CONTRACT LAB DATA

Second RUN

ENVIRONMENTAL LABORATORY: MICROTOX PREP SHEET

LR: 43597
 Date: 2/5/95
 Analyst: GWJ

TEST SUBSTANCE:

FC - 3072

1. Preparation of Stock Solution

- * Mass/Vol. Test Substance: 20.11 g (100.0%)
- * Final Dilution Volume: 20.11
- * Diluent: M.E. - 4-10
- * Final Concentration of Stock Solution: 20.11

2. pH Adjustment of Stock Solution

- * Initial pH of Stock Solution: 6.8
- * pH Adjusted to: —
- * pH Adjusted with: —

3. Osmotic Adjustment of Stock Solution

- * Osmotic Adjuster: NaCl
- * Comments: 200mg NaCl / 100ml Stock Soln

4. Appearance of pH and Osmotically Adjusted Stock Solution

- * CLEAR or OPAQUE (circle one)
- * Color: —

- * Comments: Sample foamed up slightly when mixed during diluti.

5. Comments

From the 20/L (2000mg/L) Stock Soln., 20.11 ml were added
to a 100.0 ml volumetric flask and brought to the mark.
This resulted in a 520mg/L Stock soln.

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LR# 3597

MICROTOX DATA REPORT

FILE NAME: GW03597.KS

TEST DATE: 3/15/95
TEST TIME: 2:00 PM

Investigator: GWJ

Sample Description: EC-3072

Procedure: E611

Initial Concentration: 200 mg/L

Test Time: 5 minutes

Operator: Christopher Hall
Dilution Factor: 1
Concentration Unit: mg/L

Initial	10/11	Conc.	Conc.
1	77.77	63.14	32.5000
2	77.46	62.63	32.5000
3	77.2	62.05	32.5000
4	76.8	60.87	32.5000
5	76.3	59.05	32.5000
6	74.3	49.14	32.5000
7	73.3	37.37	32.5000
8	71.3	27.64	32.5000
9	71.3	27.32	32.5000
10	71.3	27.32	32.5000
11	71.3	27.32	32.5000
12	71.3	27.32	32.5000
13	71.3	27.32	32.5000
14	71.3	27.32	32.5000
15	71.3	27.32	32.5000
16	71.3	27.32	32.5000
17	71.3	27.32	32.5000
18	71.3	27.32	32.5000
19	71.3	27.32	32.5000
20	71.3	27.32	32.5000
21	71.3	27.32	32.5000
22	71.3	27.32	32.5000
23	71.3	27.32	32.5000
24	71.3	27.32	32.5000
25	71.3	27.32	32.5000
26	71.3	27.32	32.5000
27	71.3	27.32	32.5000
28	71.3	27.32	32.5000
29	71.3	27.32	32.5000
30	71.3	27.32	32.5000
31	71.3	27.32	32.5000
32	71.3	27.32	32.5000
33	71.3	27.32	32.5000
34	71.3	27.32	32.5000
35	71.3	27.32	32.5000
36	71.3	27.32	32.5000
37	71.3	27.32	32.5000
38	71.3	27.32	32.5000
39	71.3	27.32	32.5000
40	71.3	27.32	32.5000
41	71.3	27.32	32.5000
42	71.3	27.32	32.5000
43	71.3	27.32	32.5000
44	71.3	27.32	32.5000
45	71.3	27.32	32.5000
46	71.3	27.32	32.5000
47	71.3	27.32	32.5000
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61	71.3	27.32	32.5000
62	71.3	27.32	32.5000
63	71.3	27.32	32.5000
64	71.3	27.32	32.5000
65	71.3	27.32	32.5000
66	71.3	27.32	32.5000
67	71.3	27.32	32.5000
68	71.3	27.32	32.5000
69	71.3	27.32	32.5000
70	71.3	27.32	32.5000
71	71.3	27.32	32.5000
72	71.3	27.32	32.5000
73	71.3	27.32	32.5000
74	71.3	27.32	32.5000
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76	71.3	27.32	32.5000
77	71.3	27.32	32.5000
78	71.3	27.32	32.5000
79	71.3	27.32	32.5000
80	71.3	27.32	32.5000
81	71.3	27.32	32.5000
82	71.3	27.32	32.5000
83	71.3	27.32	32.5000
84	71.3	27.32	32.5000
85	71.3	27.32	32.5000
86	71.3	27.32	32.5000
87	71.3	27.32	32.5000
88	71.3	27.32	32.5000
89	71.3	27.32	32.5000
90	71.3	27.32	32.5000
91	71.3	27.32	32.5000
92	71.3	27.32	32.5000
93	71.3	27.32	32.5000
94	71.3	27.32	32.5000
95	71.3	27.32	32.5000
96	71.3	27.32	32.5000
97	71.3	27.32	32.5000
98	71.3	27.32	32.5000
99	71.3	27.32	32.5000
100	71.3	27.32	32.5000

10. SLOPE OF LINE: 0.0071

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LR#m3597

STATISTICAL REPORT FOR DATA #1 - 10/10/77

ESTIMATED CORRELATION COEFFICIENT = 0.9170 - 0.9587

NUMBER OF DATA POINTS: 10

RESIDUAL STANDARD ERROR: 0.0029

EST. STANDARD ERROR OF ESTIMATE: 1.08115

CORRELATION COEFFICIENT: 0.9587

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CORRELATION COEFFICIENT: 0.9587

(± 7.7%)

INITIAL DATA

GWJ35-7.1.0

DATE	DESCRIPTION	AMOUNT	CHECK NO.	BANK	INTEREST
10/1/88	DEPOSIT	100.00			
10/5/88	PAYROLL	50.00	101		
10/10/88	RENT	200.00	102		
10/15/88	SALES	150.00	103		
10/20/88	UTILITIES	75.00	104		
10/25/88	DEPOSIT	120.00			
10/30/88	PAYROLL	50.00	105		
11/5/88	RENT	200.00	106		
11/10/88	SALES	150.00	107		
11/15/88	UTILITIES	75.00	108		
11/20/88	DEPOSIT	120.00			
11/25/88	PAYROLL	50.00	109		
11/30/88	RENT	200.00	110		
12/5/88	SALES	150.00	111		
12/10/88	UTILITIES	75.00	112		
12/15/88	DEPOSIT	120.00			
12/20/88	PAYROLL	50.00	113		
12/25/88	RENT	200.00	114		
12/30/88	SALES	150.00	115		
1/5/89	UTILITIES	75.00	116		
1/10/89	DEPOSIT	120.00			
1/15/89	PAYROLL	50.00	117		
1/20/89	RENT	200.00	118		
1/25/89	SALES	150.00	119		
1/30/89	UTILITIES	75.00	120		
2/5/89	DEPOSIT	120.00			
2/10/89	PAYROLL	50.00	121		
2/15/89	RENT	200.00	122		
2/20/89	SALES	150.00	123		
2/25/89	UTILITIES	75.00	124		
2/28/89	DEPOSIT	120.00			
3/5/89	PAYROLL	50.00	125		
3/10/89	RENT	200.00	126		
3/15/89	SALES	150.00	127		
3/20/89	UTILITIES	75.00	128		
3/25/89	DEPOSIT	120.00			
3/30/89	PAYROLL	50.00	129		
4/5/89	RENT	200.00	130		
4/10/89	SALES	150.00	131		
4/15/89	UTILITIES	75.00	132		
4/20/89	DEPOSIT	120.00			
4/25/89	PAYROLL	50.00	133		
4/30/89	RENT	200.00	134		
5/5/89	SALES	150.00	135		
5/10/89	UTILITIES	75.00	136		
5/15/89	DEPOSIT	120.00			
5/20/89	PAYROLL	50.00	137		
5/25/89	RENT	200.00	138		
5/30/89	SALES	150.00	139		
6/5/89	UTILITIES	75.00	140		
6/10/89	DEPOSIT	120.00			
6/15/89	PAYROLL	50.00	141		
6/20/89	RENT	200.00	142		
6/25/89	SALES	150.00	143		
6/30/89	UTILITIES	75.00	144		
7/5/89	DEPOSIT	120.00			
7/10/89	PAYROLL	50.00	145		
7/15/89	RENT	200.00	146		
7/20/89	SALES	150.00	147		
7/25/89	UTILITIES	75.00	148		
7/30/89	DEPOSIT	120.00			
8/5/89	PAYROLL	50.00	149		
8/10/89	RENT	200.00	150		
8/15/89	SALES	150.00	151		
8/20/89	UTILITIES	75.00	152		
8/25/89	DEPOSIT	120.00			
8/30/89	PAYROLL	50.00	153		
9/5/89	RENT	200.00	154		
9/10/89	SALES	150.00	155		
9/15/89	UTILITIES	75.00			

NUMBER	CONCENTRATION	LOG ₁₀	EFFECT	EFFECT
1	12.5000	0.111	10.00	
2	12.5000	0.111	10.12	10.08
3	65.0000	0.186	12.64	
4	65.0000	0.186	18.01	18.92
5	130.0000	0.187	27.40	
6	130.0000	0.187	25.98	27.40
7	260.0000	0.673	42.61	
8	260.0000	0.673	41.97	42.61

[illegible]

FINAL RESULTS

OW 3597.15

NUMBER 1 CONC 2 GAMMA 3 GAMMA EST 4 EFFECT

NUMBER	COND.	GAMMA	GAMMA EST.	% EFFECT
1	1	0.00	0.00	0.00
2	2	0.00	0.00	0.00
3	3	0.00	0.00	0.00
4	4	0.00	0.00	0.00
5	5	0.00	0.00	0.00
6	6	0.00	0.00	0.00
7	7	0.00	0.00	0.00
8	8	0.00	0.00	0.00
9	9	0.00	0.00	0.00
10	10	0.00	0.00	0.00
11	11	0.00	0.00	0.00
12	12	0.00	0.00	0.00
13	13	0.00	0.00	0.00
14	14	0.00	0.00	0.00
15	15	0.00	0.00	0.00
16	16	0.00	0.00	0.00
17	17	0.00	0.00	0.00
18	18	0.00	0.00	0.00
19	19	0.00	0.00	0.00
20	20	0.00	0.00	0.00
21	21	0.00	0.00	0.00
22	22	0.00	0.00	0.00
23	23	0.00	0.00	0.00
24	24	0.00	0.00	0.00
25	25	0.00	0.00	0.00
26	26	0.00	0.00	0.00
27	27	0.00	0.00	0.00
28	28	0.00	0.00	0.00
29	29	0.00	0.00	0.00
30	30	0.00	0.00	0.00
31	31	0.00	0.00	0.00
32	32	0.00	0.00	0.00
33	33	0.00	0.00	0.00
34	34	0.00	0.00	0.00
35	35	0.00	0.00	0.00
36	36	0.00	0.00	0.00
37	37	0.00	0.00	0.00
38	38	0.00	0.00	0.00
39	39	0.00	0.00	0.00
40	40	0.00	0.00	0.00
41	41	0.00	0.00	0.00
42	42	0.00	0.00	0.00
43	43	0.00	0.00	0.00
44	44	0.00	0.00	0.00
45	45	0.00	0.00	0.00
46	46	0.00	0.00	0.00
47	47	0.00	0.00	0.00
48	48	0.00	0.00	0.00
49	49	0.00	0.00	0.00
50	50	0.00	0.00	0.00
51	51	0.00	0.00	0.00
52	52	0.00	0.00	0.00
53	53	0.00	0.00	0.00
54	54	0.00	0.00	0.00
55	55	0.00	0.00	0.00
56	56	0.00	0.00	0.00
57	57	0.00	0.00	0.00
58	58	0.00	0.00	0.00
59	59	0.00	0.00	0.00
60	60	0.00	0.00	0.00
61	61	0.00	0.00	0.00
62	62	0.00	0.00	0.00
63	63	0.00	0.00	0.00
64	64	0.00	0.00	0.00
65	65	0.00	0.00	0.00
66	66	0.00	0.00	0.00
67	67	0.00	0.00	0.00
68	68	0.00	0.00	0.00
69	69	0.00	0.00	0.00
70	70	0.00	0.00	0.00
71	71	0.00	0.00	0.00
72	72	0.00	0.00	0.00
73	73	0.00	0.00	0.00
74	74	0.00	0.00	0.00
75	75	0.00	0.00	0.00
76	76	0.00	0.00	0.00
77	77	0.00		

245.0000	0.1882	0.1855	13.836
55.0000	0.1905	0.1855	18.031
130.0000	0.3869	0.3941	27.897
130.0000	0.3695	0.3941	26.981
250.0000	0.8729	0.8373	46.608
250.0000	0.8371	0.8373	45.586

ESTD 302513Z MAR 45/ CONFIDENCE RANGEL 8008571 TO 8007098

LR#M3597

FILE NAME: 000597.M00

TEST DATE: 2/15/95
TEST TIME: 2:00pm

100-111910-1 GWJ

Sample Descriptions: 1-5072

REFERENCE: BASIC

Optical Concentration : 260 mW/cm²

1000 - 11000: 15, 600, 000

OSMATIC Acoustics : 9607

De la 1^{re} à la 3^e année :

Cooperation: 14.000.000

Order	Customer	Product	Quantity	Unit Price	Total Price
1	John Doe	Widget A	10	\$1.50	\$15.00
2	Jane Smith	Widget B	5	\$2.00	\$10.00
3	Bob Johnson	Widget A	20	\$1.50	\$30.00
4	Alice Brown	Widget C	15	\$1.00	\$15.00
5	Charlie Davis	Widget B	10	\$2.00	\$20.00
6	Eve Wilson	Widget A	30	\$1.50	\$45.00
7	Frank Miller	Widget C	25	\$1.00	\$25.00
8	Grace Lee	Widget B	12	\$2.00	\$24.00

$$\text{RATIOS} = \frac{0.5627}{0.5825} = 0.9677$$

CORRECTION FACTOR = 0.7627

[illegible]

SSLOPE = 4.750

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11-11-68

CONCENTRATION 100

INDEPENDENCE BOND 6-7Z 078 0000

Pg 6 of 10
LR#m3597

STATISTICAL ANALYSIS OF 1941-1942 FILE ANALYSIS

STATISTICAL EQUATION: $\log C = 0.09 \times \log B + 1.1643$

NUMBER OF DATA PAIRS: 11

RELATIONSHIP: $\log C = 0.09 \times \log B + 1.1643$

RELATIONSHIP FACTOR: 1.1643

LOG OF LOG COEFFICIENT: 1.1643

LOG OF LOG OF LOG COEFFICIENT: 1.1643

LOG OF LOG OF LOG OF LOG COEFFICIENT: 1.1643

LOG C = 103.9911	LOG B = 100.0000	LOG C = 101.8481	($\pm 4.1\%$)
LOG C = 71.9681	LOG B = 50.0000	LOG C = 61.1004	***
LOG C = 41.5784	LOG B = 20.0000	LOG C = 49.8771	***
LOG C = 8.2025	LOG B = 0.0000	LOG C = 11.9211	***

*** EXTRAPOLATED RESULTS

pg 7 of 10
LR#M3597

INITIAL DATA

GW3359.K15

NUMBER	CONC.	GAMMA	% EFFECT
1	0.0000	0.100	11.94
2	20.0000	0.139	10.20
3	40.0000	0.267	10.63
4	60.0000	0.360	10.60
5	100.0000	0.615	10.10
6	130.0000	0.599	10.10
7	260.0000	1.678	62.87
8	260.0000	1.652	62.77

FINAL RESULTS

GW3359.K15

NUMBER	CONC.	GAMMA	GAMMA STD	% EFFECT
5	130.0000	0.6156	0.5991	10.10
6	130.0000	0.5830	0.5991	10.10
7	260.0000	1.6789	1.6654	62.87
8	260.0000	1.6520	1.6654	62.77

1971 07/05 195% CONFIDENCE RANGE 175.5528 TO 181.7645

LR# M3597

FILE NAME: 64159

RECEIVED DATE: 3/15/95

TEST TIME: 2:00 PM

[illegible]

Investigator: GWS

Sample No. 1011001 Box 1072

Procedures: 1A-200

Initial Concentration = 2.03 mg/ml.

Test Time: 30 minutes

05-00111. Instruments: NACI

... 1000 ...

Concentration Units: mg/L

NUMBER	IN	OUT	DIFF
1	21.707	21.27	0.437
2	21.861	21.54	0.321
3	21.807	21.20	0.607
4	21.857	21.04	0.817
5	21.117	21.85	-0.733
6	21.117	21.50	-0.383
7	21.117	21.4	-0.283
8	21.217	21.74	-0.523
CONTROL	21.117	21.79	-0.673
AVERAGE	21.117	21.54	-0.423
CORRECT IGM F-104	21.117	21.56	-0.443

[illegible]

pg 9 of 10
LR#m3597

STATISTICAL REPORT FOR DATA FILE GWJ3597.K00

ESTIMATING EQUATION: $\text{LOG C} = 0.6071 \times \text{LOG D} + 2.1424$

NUMBER OF DATA POINTS USED: 4

RESIDUAL VARIANCE: 0.00000

95% CONFIDENCE FACTOR: 2.05408 FOR EC50

CORRELATION COEFFICIENT: $r = 0.99984$

COEFFICIENT OF DETERMINATION: $R^2 = 0.99968$

EFFECTIVE CONCENTRATIONS (95% CONFIDENCE RANGE)

EC50 =	107.1109 mg/L	(	134.1715	)	(	144.1704	)
EC20 =	57.1101 mg/L	(	54.4891	)	(	65.9871	***
EC10 =	26.6465 mg/L	(	32.0316	)	(	41.9311	***
EC01 =	8.0463 mg/L	(	6.6420	)	(	10.9966	***

***EXTRAPOLATED RESULTS

pg 10 of 10
LR#M3597

INITIAL DATA

SNJ3597.K3

NUMBER	CONCENTRATION	GAMMA	% EFFECT
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1	32.5000	0.173	14.73
2	32.5000	0.182	15.07
3	65.0000	0.349	29.85
4	65.0000	0.574	27.23
5	130.0000	0.912	47.71
6	130.0000	0.379	46.73
7	260.0000	2.824	73.85
8	260.0000	2.775	73.51

PUSH SPACE BAR to continue..

FINAL RESULTS

SNJ3597.K3

NUMBER	CONC.	GAMMA	GAMMA EST	% EFFECT
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5	130.0000	0.9123	0.9123	47.707
6	130.0000	0.8773	0.8773	46.730
7	260.0000	2.8243	2.8243	73.851
8	260.0000	2.7745	2.7745	73.509

CONFIDENCE INTERVALS: 95% CONFIDENCE INTERVALS: 1715.70 100.27