

ACTIVATED SLUDGE RESPIRATION INHIBITION TEST

TEST SUBSTANCE

Identity: A mixture containing perfluorooctanesulfonate, which may also be referred to as PFOS, FC-95, or as a component of FC-603F. (1-Octanesulfonic acid) (CAS # 2795-39-3).

Remarks: The 3M production lot number was 501. The test sample is FC-603F. Current information indicates it is a mixture of 0.5% PFOS, 7% diethylene glycol butyl ether, 82.95% water, 2% acrylic foamer, 4% Alkylether sulfate, 1.45% Xanthan gum, 1.4% calcium acetate, 0.07% N-(3-Chloroallyl)hexaminium chloride, 0.5% triethanolamine, 0.05% sodium hydroxide, 0.03% cis-1-(3-Chloroallyl)-3,5,7-triaza-1-azoniaadamantane chloride, and 0.05% tolyltriazole.

The following summary applies to a mixture with incompletely characterized concentrations of impurities. Data may not accurately reflect the toxicity of the fluorochemical component of the test sample.

METHOD

Method: OECD Guideline 209

GLP: No

Year Completed: 1994

Analytical monitoring: Dissolved oxygen concentrations, temperature, and pH.

Statistical methods: Results were determined by calculation of the % inhibition in respiration rate.

Test organism source: Activated sludge mixed liquor collected from the Metro Wastewater Treatment Plant, St. Paul, MN.

Test condition:

Dilution water: Millipore Milli-Q™ water

Synthetic sewage: per OECD method

Reference and test solution preparation: A stock solution of the reference substance, 3,5-dichlorophenol, was prepared by dissolving 500 mg in 10mL 1N NaOH, diluted to 30mL with Millipore Milli-Q™ water, then brought to the point of incipient precipitation with 1N H₂SO₄ and diluted to 1L with Milli-Q™ water. The pH of the reference solution was measured to be 7.5. Test substance was added directly to the test vessels.

Test vessels: Not given.

Number of concentrations: one and a blank control, in duplicate, plus 1 reference substance

Temperature: 17.7 – 20.2 °C

Total suspended solids and pH on day of testing: 4.1 g/L (TSS in final: 1.6 g/L), initial pH 7.7 and final pH 7.1.

Element Basis: Respiration inhibition as determined by oxygen consumption.

Remarks: Values corrected to 20°C for calculations.

RESULTS

Nominal concentrations: Blank control, and 1000 mg/L, in duplicate.
Reference substance at 10 mg/L.

Element values: 30-minute EC₅₀ = >1000 mg/L
3-hour EC₅₀ = >1000 mg/L

Element values based on nominal concentrations.

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

CONCLUSIONS

The FC-603F 3-hour EC₅₀ for activated sludge respiration inhibition was determined to be >1000 mg/L, the highest concentration tested. Mean value of 10.1% inhibition at this concentration.

Submitter: 3M Company, Environmental Laboratory, P.O. Box 33331, St. Paul, Minnesota, 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 Company, Environmental Laboratory, St. Paul, MN, Lab Request number M1655, 1994.

OTHER

Last changed: 6/26/00

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ENVIRONMENTAL LABORATORY

ACTIVATED SLUDGE MIXED LIQUOR

LR MV655
Date 5-10-94
Analyst JBD
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Type Test SCD Activated Sludge Respiration Inhibition Test #209

ACTIVATED SLUDGE MIXED LIQUOR

Date Obtained 5-10-94

Date Used 5-10-94

Mixed Liquor Suspended Solids (MLSS) 4.1 g/L Adjusted to _____ g/L

MLSS in final test solution 1.6 g/L

Initial pH 7.7 (1st test soln. prepared)

Final pH 7.1 (last soln. prepared)

Comments _____

TEST SUBSTANCE

Sample Description FC-603F (Lot #501)

Initial pH of highest conc. tested (1000 mg/L) = 7.2

Adjusted to pH _____

Comments _____

REFERENCE SUBSTANCE

Sample Description 3,5-Dichlorophenol

Initial pH of stock solution (500 mg/L) = 7.5

Adjusted to pH _____

Comments _____

EXPOSURE CONDITIONS

Test Duration 3 hours

Test Temperature Range: 17.7 - 20.2 °C

Comments _____

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ENVIRONMENTAL LABORATORY

LR M1655

OECD ACTIVATED SLUDGE RESPIRATION INHIBITION TEST #209
TEST SOLUTIONS PREPARATION

Test Substance: FC 603F (Lot # 501)

Sample Description: Brown gel, miscible

Analyst: JTD

Date: 5-10-94

Test Conc. mg/L	mg added per 500 mL	mL MQ(1) water per 500 mL	mL syn. sewage feed per 500 mL	mL microbial inoculum per 500 mL	Total Volume mL
Controls	—	284	16	200	500
1000	0.5	284	16	200	500

Initial pH - highest conc. tested = 7.2

(1) Millipore Milli-QR high purity water

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M1655
LR M1665 (25)

ENVIRONMENTAL LABORATORY

OECD ACTIVATED SLUDGE RESPIRATION INHIBITION TEST #209

RAW DATA

Test Substance: FL 603F (Lot # 501)

Source of Activated Sludge Mixed Liquor: Water Waste Treatment Plant, St. Paul, MN

Date Received: 5-10-94

Date Used: 5-10-94

Analyst: JBS / RSW

30-MINUTE EXPOSURE

DISSOLVED OXYGEN CONCENTRATIONS, mg/L

Test Substance Concentration (mg/L)	TIME-MINUTES											Temp °C
	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5	
BK CONTROL I	6.4	6.1	5.7	5.4	5.1	4.7	4.3	4.0	3.6	3.2	2.9	20.1
1000	4.1	3.5	3.1	2.7	2.3	2.0	1.6	1.3	0.9	0.5	-	20.2
Dup	5.9	5.4	5.0	4.7	4.3	3.9	3.5	3.1	2.8	2.5	2.1	19.8
BK CONTROL II	3.0 (2.6)	7.7 4.0	7.4 5.3	7.1	6.8	6.5	6.1	5.9	5.6 (5.7)	5.3	5.0	17.7

3-HOUR EXPOSURE

DISSOLVED OXYGEN CONCENTRATIONS, mg/L

Test Substance Concentration (mg/L)	TIME - MINUTES											Temp °C
	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5	
BK CONTROL I	7.4	7.1	6.8	6.6	6.3	5.8	5.6	5.3	4.9	4.7	4.3	18.8
1000	6.4	6.0	5.7	5.3	5.0	4.7	4.4	4.0	3.7	3.4	3.0	19.5
Dup	7.6	7.3	7.1	6.8	6.5	6.3	6.0	5.7	5.5	5.2	4.8	19.0
BK CONTROL II	7.4	7.5	7.3	6.9	6.6	6.3	5.9	5.6	5.3	5.0	4.6	18.3

pH of activated sludge mixed liquor:

Initial: 7.7

Final: 7.4

pH of highest concentration tested:

Initial: 7.4

Final: 7.3

Test temperature: 17.7-18.2

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ENVIRONMENTAL LABORATORY
OECD ACTIVATED SLUDGE RESPIRATION INHIBITION TEST #209
CALCULATION OF RESULTS

Page 4 of 4
Lr: M1655
Date: 5/10/94

Test Substance: FC 603F (Lot #501)

Analyst: JBD

30-Minute Exposure												Dissolved Oxygen Concentrations, mg/L				Temp, °C	R (1)	Ra (2)	% Inh. (3)
mg/L	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5								
BK1	6.4	6.1	5.7	5.4	5.1	4.7	4.3	4.0	3.6	3.2	2.9	20.1	0.7152	0.7102					
												20.2	0.7367	0.7265	0				
1000.0	4.1	3.5	3.1	2.7	2.3	2.0	1.6	1.3	0.9	0.5		19.8	0.7358	0.7461	0				
Dup.	5.9	5.4	5.0	4.7	4.3	3.9	3.5	3.1	2.8	2.5	2.1								
BK2	8.0	7.7	7.4	7.1	6.8	6.5	6.1	5.9	5.6	5.3	5.0	17.7	0.6012	0.7055					

RaCm (5): 0.7078

% diff in controls (4): 0.7

EC50 (95% CI): > 1000 mg/L

3-Hour Exposure												Dissolved Oxygen Concentrations, mg/L				Temp, °C	R (1)	Ra (2)	% Inh. (3)
mg/L	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5								
BK1	7.4	7.1	6.8	6.6	6.3	5.8	5.6	5.3	4.9	4.7	4.3	18.8	0.6255	0.6799					
												19.5	0.6594	0.6827	3.2				
1000.0	6.4	6.0	5.7	5.3	5.0	4.7	4.4	4.0	3.7	3.4	3.0	19.0	0.5455	0.5847	17				
Dup.	7.6	7.3	7.1	6.8	6.5	6.3	6.0	5.7	5.5	5.2	4.8								
BK2	7.9	7.5	7.3	6.9	6.6	6.3	5.9	5.6	5.3	5.0	4.6	18.3	0.6497	0.7312					

RaCm (5): 0.7055

% diff in controls (4): 7.3

EC 50 (95% CI): > 1000 mg/L

Method: N/A

- (1) R= Oxygen uptake rate (mg O₂/L-min)
 (2) Ra= R adjusted to 20°C= [R/Ex]
 (3) % Inhibition= [(RaCm)-RaS/(RaCm)]100
 (4) % difference in controls: [(RaBk1-RaBk2)/RaCm]100 (Two controls must be within 15% of each other.)
 (5) Mean adjusted R, controls= (RaBk1+RaBk2)/2

α= 1.072

RaCm= mean adjusted R, controls

Bk= blank control, S= sample

Tobs= sample temp

x= Tobs-20°C

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m1655,

LR m1810-142

ENVIRONMENTAL LABORATORY

OECD ACTIVATED SLUDGE RESPIRATION INHIBITION TEST # 209
TEST SOLUTIONS PREPARATION

Test Substance: 3,5 - Dichlorophenol

Sample Description: crystalline solid, Aldrich Red Label
(Lot # 08210DLO)

Analyst: JBD

Date: 5-10-94

Stock Solution

Preparation: 1) Added 0.5g crystalline 3,5-DCP to a 1L volumetric flask
2) Added 10mL of 1N NaOH & stirred to dissolve DCP.
3) Added approx. 20mL Milli-Q
4) While stirring, added 1N H₂SO₄ to the point of incipient
precipitation
5) Added Milli-Q to dilute to a final volume of 1L.
Init. pH stock soln = 7.5

Test Conc. mg/l	mL added per 500 mL	mL MQ(1) water per 500 mL	mL syn. sewage feed per 500 mL	mL microbial inoculum per 500 mL	Total Volume mL
Controls	—	284	16	200	500
10	10	274	16	200	500

(1) Millipore Milli-Q high purity water

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M1655,
-R M1810-142

ENVIRONMENTAL LABORATORY

OECD ACTIVATED SLUDGE RESPIRATION INHIBITION TEST #209

RAW DATA

Test Substance: 3,5-Dichlorophenol

Source of Activated Sludge Mixed Liquor: Metrol Waste Treatment Plant, St. Paul, MN

Date Received: 5-10-94

Date Used: 5-10-94

Analyst: JBD/RJCO

30-MINUTE EXPOSURE

DISSOLVED OXYGEN CONCENTRATIONS, mg/L

Test Substance Concentration (mg/L)	TIME-MINUTES											Temp °C
	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5	
BK CONTROL I	6.4	6.1	5.7	5.4	5.1	4.7	4.3	4.0	3.6	3.2	2.9	20.1
10	7.5	7.3	7.0	6.8	6.5	6.3	6.1	5.8	5.6	5.4	5.2	19.3
BK CONTROL II	8.0 (JBD)	7.7 7.2	7.4	7.1	6.8	6.5	6.1	5.9	5.6 5.7	5.3	5.0	17.7 (JBD)

3-HOUR EXPOSURE

DISSOLVED OXYGEN CONCENTRATIONS, mg/L

Test Substance Concentration (mg/L)	TIME - MINUTES											Temp °C
	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5	
BK CONTROL I	7.4	7.1	6.8	6.6	6.3	5.8	5.6	5.3	4.9	4.7	4.3	18.8
10	8.2	8.0	7.8	7.6	7.4	7.1	7.0	6.8	6.7	6.5	6.3	19.7
BK CONTROL II	7.9	7.5	7.3	6.9	6.6	6.3	5.9	5.6	5.3	5.0	4.6	17.3

pH of activated sludge mixed liquor: Initial 7.1 Final 7.1
pH of highest concentration tested: Initial 6.9 Final 7.6
Test temperature: 17.7-20.1

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ENVIRONMENTAL LABORATORY
OECD ACTIVATED SLUDGE RESPIRATION INHIBITION TEST #209
CALCULATION OF RESULTS

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LR: M1695
M1810-142
Date: 5/10/94

Test Substance: 3,5 - Dichlorophenol Reference Toxicant

Analyst: JBD/RJW

30-Minute Exposure			Dissolved Oxygen Concentrations, mg/L										Temp. °C	R (1)	Ra (2)	% Inh. (3)
mg/L	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5					
BK1	6.4	6.1	5.7	5.4	5.1	4.7	4.3	4.0	3.6	3.2	2.9	20.1	0.7152	0.7102		
10.0	7.5	7.3	7.0	6.8	6.5	6.3	6.1	5.8	5.6	5.4	5.2	19.3	0.4733	0.4969	30	
BK2	8.0	7.7	7.4	7.1	6.8	6.5	6.1	5.9	5.6	5.3	5.0	17.7	0.6012	0.7055		
% diff in controls (4): 0.7															RaCm (5): 0.7078	

3-Hour Exposure			Dissolved Oxygen Concentrations, mg/L										Temp. °C	R (1)	Ra (2)	% Inh. (3)		
mg/L	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5							
BK1	7.4	7.1	6.8	6.6	6.3	5.8	5.6	5.3	4.9	4.7	4.3	18.8	0.6255	0.6799				
10.0	8.2	8.0	7.8	7.6	7.4	7.1	7.0	6.8	6.7	6.5	6.3	19.7	0.3733	0.3812	46			
BK2	7.9	7.5	7.3	6.9	6.6	6.3	5.9	5.6	5.3	5.0	4.6	18.3	0.6497	0.7312				
% diff in controls (4):															7.3	RaCm (5):		0.7055

- (1) R= Oxygen uptake rate (mg O₂/L-min)
- (2) Ra= R adjusted to 20°C= [R/vs Ex]
- (3) % Inhibition= [(RaCm)-RaS/(RaCm)]100
- (4) % difference in controls: [(RaBk1-RaBk2/RaCm)]100 (Two controls must be within 15% of each other.)
- (5) Mean adjusted R, controls= [RaBk1+RaBk2]/2

α= 1.072
RaCm= mean adjusted R, controls
Dk= blank control, S= sample
Tobs= sample temp
x= Tobs-20°C

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Chain of Custody Record

3M Environmental Laboratory

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3M Requester GAB	Page 4 of 4
3M Project Lead	
3M Project Title	
3M Project Number	
3M Department Number	
Lab Request/Template Number	

Form 35176 - PWO

3M Facility MWCC Pigs Eye	3M Project Title 3m mixed liquor P.U. May
Address	3M Facility Contact
City, State, Zip Code	3M Telephone Number

Sampled By (Print) Brad Jacobson		Contractor PACE Inc		Number Containers of each		Total Containers of Each Item	☐ Samples Split See Attachment Comments
Sample Signature <i>[Signature]</i>		Date 5/10/94		Preservatives			
Item #	Sample Description	Date	Time	Matrix			
1	Mixed Liquor	5/10/94	0735			1	sub. in - seal
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							

Item #	Relinquished By/Affiliation	Time	Date	Shipped Via	Received By/Affiliation	Time	Date
1	<i>[Signature]</i> / PACE	0756	5/10/94	SEIK	<i>[Signature]</i> / 3M	10:00	5/10/94

See Reverse Side For Instructions

Original - Accompanying Samples

Last Page - Originator

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