

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-206CE. (1-Octanesulfonic acid) (CAS # 2795-39-3).

Remarks: The 3M production lot number was 502. The test sample is FC-206CE. Current information indicates it is a mixture of 0.92% PFOS, 15% diethylene glycol butyl ether, 76.09% water, 4% urea, 1.28% Sultone foamer, 1.55% sodium octyl sulfate, 1.05% polyoxyethylene monooctylphenyl ether, 0.06% sodium lauryl sulfate, and 0.05% tolytriazole. Antifoamer SWS 214 was added at a nominal concentration of 200 mg/L.

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 209

Test type: Static acute

GLP: No

Year Completed: 1990

Analytical monitoring: Dissolved oxygen concentrations, pH and temperature.

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

Test organism source: Activated sludge mixed liquor collected from the Metro Waste 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 7.2. Test substance was added directly to the test vessels. Antifoamer SWS 214 was added to the test vessels at 200 mg/L from a stock solution prepared by diluting 2000 mg of SWS 214 in 500 mL Milli-Q™ water. The solutions appeared miscible in water.

Test vessels: Not given.

Number of concentrations: three plus 1 reference substance and 2 blank controls

Temperature: 18 – 19 °C

Total suspended solids and pH on day of testing: 7.853 g/L (TSS), initial pH 6.7 and final pH 6.9. Initial TSS in 1000 mg/L concentration was 3.16 g/L, initial pH 6.7, final pH 6.8, and MLSS 3.38.

Element Basis: Respiration inhibition as determined by oxygen consumption.

Remarks: Values corrected to 20 °C for calculations.

RESULTS

Nominal concentrations: 2 Blank controls, reference substance, 320, 560 and 1000 mg/L

Element values: 30-minute $EC_{50} = >1000$ mg/L
3-hour $EC_{50} = >1000$ mg/L

Element values based on nominal concentrations.

Remarks: There was minimal foaming in the 1000 mg/L exposure concentration when compared to FC-206CE without antifoamer. 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-206CE 3-hour EC_{50} for activated sludge respiration inhibition was determined to be >1000 mg/L.

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

DATA QUALITY

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

REFERENCES

This study was conducted by the 3M Company, Environmental Laboratory, St Paul, MN, Lab Request number H1480-4, 1990.

OTHER

Last changed: 6/26/00

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Lab Request No. H1480-4

Environmental Lab — Work Sheet

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Sample Description

Date: 3/23/90OECD ACTIVATED SLUDGE
RESPIRATION INHIBITION TEST #209Analyst: [Signature] 3

Test Solutions Preparation

FC206CE Lot 502 (2/16/89) with Antifoamer SWS 214 (200mg/L)FC206CE Lot 502 individual weights.SWS 214 at 200mg/L refer to page 1 of 3 of SWS 214Stock Solution preparation sheet.

Test Conc. mg/L	mg added per 500 ml	ml ^{SW214} water per 500 ml	ml syn. sewage feed per 500 ml	ml microbial inoculum per 500 ml	Total volume ml
320	1160	+ 2.59	+ 2.5	+ 16	+ 200 = 500
560	2.80	+ 2.59	+ 2.5	+ 16	+ 200 = 500
1000*	500	+ 2.59	+ 2.5	+ 16	+ 200 = 500

Bk Controls shared with LR H1480-2.* Minimal foaming.This test solution was duplicated. Used in measuring TSS.

ENVIRONMENTAL LABORATORY
OECD ACTIVATED SLUDGE RESPIRATION INHIBITION TEST #209
RAW DATA

Test Substance: EC 2016 CE Lot 502 (2/16/89) with Antifoamer SWS 214 (200mg/L)
Source of Activated Sludge Mixed Liquor: Metro Waste Treatment Plant, St. Paul, Mn
Date Received: 3/23/90 Date Used: 3/23/90
Analyst: Rochelle Rbideau

30 - MINUTE EXPOSURE

30 - MINUTE EXPOSURE		DISSOLVED OXYGEN CONCENTRATIONS mg/L											TEMP °C
Test Substance Concentration (mg/L)	TIME-MINUTES												
	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5		
CONTROL I	7.4	6.5	5.8	5.3	4.7	4.2	3.7	3.1	2.6	2.1	1.6	18	
320	3.0	2.0	1.4	0.8	2:18 0.5	-						18	
560	2.3	1.2	0.5									18	
1000	1.6	0.5										18	
CONTROL II	5.8	5.2	4.5	4.0	3.5	2.9	2.5	1.9	1.4	0.9	5:27 0.5	18	

3 - HOUR EXPOSURE

3 - HOUR EXPOSURE		DISSOLVED OXYGEN CONCENTRATIONS mg/L											TEMP °C
Test Substance Concentration (mg/L)	0.5	1.0	1.5	2.0	2.5	TIME-MINUTES		4.0	4.5	5.0	5.5		
CONTROL I	6.5	6.1	5.6	4.9	4.4	3.8	3.2	2.4	2.0	1.5	1.0	19	
320	4.5	3.9	3.2	2.6	2.0	1.4	0.9	3:32 0.5				19	
560	4.2	3.4	2.8	2.2	1.6	1.1	0.5					19	
1000	6.1	5.7	5.2	4.6	4.1	3.5	3.0	2.4	1.9	1.3	0.8	19	
CONTROL II	5.8	4.9	4.4	3.8	3.3	2.7	2.4	1.7	1.2	0.5		18	

pH of Activated Sludge Mixed Liquor: Initial 6.7 Final 6.9
pH of highest concentration tested: Initial 6.7 Final 6.8
Test Temperature: 18-19°C

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ENVIRONMENTAL LABORATORY

ACTIVATED SLUDGE RESPIRATION INHIBITION TEST #209
CALCULATION OF RESULTSLR H1480-4
Date 3/23/90
Analyst 383
page 30Test Substance EC 206 CE Lot 502 (2/16/89) with Antifoamer SWS 214 (200mg/L)

30 - MINUTE EXPOSURE

Test Substance Concentration (mg/L)	Oxygen Uptake Rate R	R Corrected ⁽¹⁾ to 20°C	% Inhibition in Respiration Rate ⁽²⁾
CONTROL I	1.07164	1.1803	
320	1.11633	1.2755	— -48.40 ()
560	1.4000	1.5351	— (27)
1000	*		—
CONTROL II	1.1297	1.2387	

Percent Difference in Controls⁽³⁾ 4.8% 1.2095EC₅₀ (95% Confidence Limits) mg/L _____

3 - HOUR EXPOSURE

Test Substance Concentration (mg/L)	Oxygen Uptake Rate R	R Corrected ⁽¹⁾ to 20°C	% Inhibition in Respiration Rate ⁽²⁾
CONTROL I	1.1939	1.2502	
320	1.1768	1.2323	— (1.4)
560	1.1543	1.2087	0.5 0.35 0.
1000	1.0970	1.1487	5.5
CONTROL II	1.07167	1.1806	

Percent Difference in Controls⁽³⁾ 5.7% 1.2154EC₅₀ (95% Confidence Limits) mg/L _____

$$(1) \text{ Temp. Correction} = \frac{R_{T, \text{obs}}}{\theta^x} \quad (\text{mg O}_2/\text{L-min.})$$

$$(3) \% \text{ Difference} = \frac{|RC_1 - RC_2|}{\left(\frac{RC_1 + RC_2}{2}\right)} \times 100$$

$$(2) \% \text{ Inhibition} = \frac{\left(\frac{RC_1 + RC_2}{2}\right) - R_S}{\left(\frac{RC_1 + RC_2}{2}\right)} \times 100$$

The two control respiration rates should be within 15% of each other

Where:

R = Oxygen uptake rate (mg O₂/L-min.)T_{obs} = Sample temp., °Cθ = 1.047, x = T_{obs} - 20°C

C = Control

* Rate not calculable; oxygen depleted too rapidly, in less than 1 minute.

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LR H1480-S

Date 3/23/90

Analyst J3

ENVIRONMENTAL LABORATORY
ACTIVATED SLUDGE MIXED LIQUOR

Type Test Activated Sludge Respiration Inhibition Test (OECD)#209

Source Metro Wastewater Treatment Plant, St. Paul, Mn

Date Obtained 3/23/90 Date Used 3/23/90

Mixed Liquor Suspended Solids (MLSS) 7.853 g/L Adjustment g/L

pH initial 6.7, final 6.9

Comments _____

Test Substance

Sample Description SWIS 214 Antifoamer (4/7/86) Silicones Corporation, Adrian, MI
highest concentration tested, 1000 mg/L

Sample pH initial 6.7, final 6.8 Adjustment _____

Comments Stock solution pH 6.7

Reference Substance

Sample Description 3,5-Dichlorophenol
highest concentration tested, 10 mg/L

Sample pH initial 6.8, final 7.0 Adjustment _____

Comments Stock solution pH 7.2

Exposure Conditions

Test Duration 3 Hours

Test Temperature 18-19 °C

Comments _____

001780

Lab Request No. H1480-5

Environmental Lab — Work Sheet

Page 1 of 3

Sample Description

OECD ACTIVATED SLUDGE
RESPIRATION INHIBITION TEST #209Date: 3/23/90Analyst: [Signature]

Stock and Test Solutions Preparation

SWS 214 Antifoamer (4/7/86) Silicones Corporation, Adrian, Mi.STOCK SOLUTION: 2000 mg/500 mL Each 1 mL = 4 mg= 2000 mg SWS 214 / 500 mL Milli-Q water.(SWS 214 very miscible). Stock soln. pH 6.7.

Test Conc. mg/L	ml stock soln per 500 ml	ml MQ water per 500 ml	ml syn. sewage feed per 500 ml	ml microbial inoculum per 500 ml	Total volume ml
<u>1000</u>	<u>125</u>	<u>+</u>	<u>159</u>	<u>+</u>	<u>16</u>
				<u>+</u>	<u>200</u>
					<u>= 500</u>

Shared BK Controls with LTR H1480-2 and

ENVIRONMENTAL LABORATORY

LR H1480-5
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OECD ACTIVATED SLUDGE RESPIRATION INHIBITION TEST #209

RAW DATA

Test Substance: SW5214 Antifoamer (4/7/86) Silicones Corporation, Adrian, Mi.Source of Activated Sludge Mixed Liquor: Metrom Waste Treatment Plant, St. Paul, MnDate Received: 3/23/90Date Used: 3/23/90Analyst: Rechele Rabideau

30 - MINUTE EXPOSURE

Test Substance Concentration (mg/L)	DISSOLVED OXYGEN CONCENTRATIONS mg/L											TEMP °C
	TIME-MINUTES											
	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5	
CONTROL I	7.4	6.5	5.8	5.3	4.7	4.2	3.7	3.1	2.6	2.1	1.6	18
1000	3.0	1.9	1.3	0.8	2:17 0.5	-						18
CONTROL II	5.8	5.2	4.5	4.0	3.5	2.9	2.5	1.9	1.4	0.9	5:27 0.5	18

3 - HOUR EXPOSURE

Test Substance Concentration (mg/L)	DISSOLVED OXYGEN CONCENTRATIONS mg/L											TEMP °C
	TIME-MINUTES											
	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5	
CONTROL I	6.5	6.1	5.6	4.9	4.4	3.8	3.2	2.4	2.0	1.5	1.0	19
1000	3.0	2.2	1.5	1.0	2.7 0.5							19
CONTROL II	5.8	4.9	4.4	3.8	3.3	2.7	2.4	1.7	1.2	0.5		18

pH of Activated Sludge Mixed Liquor: Initial 6.7
 pH of highest concentration tested: Initial 6.7
 Test Temperature: 18-19°C

Final 6.9
 Final 6.8

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ENVIRONMENTAL LABORATORY

ACTIVATED SLUDGE RESPIRATION INHIBITION TEST #209
CALCULATION OF RESULTSLR H1480-5
Date 3/23/90
Analyst 3
Page 3 of 3Test Substance SWS 214 Antifoamer (4/4/86) Silicones Corporation, Adrian, MI.

30 - MINUTE EXPOSURE

Test Substance Concentration (mg/L)	Oxygen Uptake Rate R	R Corrected ⁽¹⁾ to 20°C	% Inhibition in Respiration Rate ⁽²⁾
CONTROL I	1.0764	1.1803	
1000	1.0849	1.1896	1.6
CONTROL II	1.1297	1.2387	

Percent Difference in Controls⁽³⁾ 4.8%EC₅₀ (95% Confidence Limits) mg/L _____

3 - HOUR EXPOSURE

Test Substance Concentration (mg/L)	Oxygen Uptake Rate R	R Corrected ⁽¹⁾ to 20°C	% Inhibition in Respiration Rate ⁽²⁾
CONTROL I	1.1939	1.2502	
1000	1.1556	1.2101	
CONTROL II	1.0767	1.1806	

Percent Difference in Controls⁽³⁾ 5.7%EC₅₀ (95% Confidence Limits) mg/L _____

$$(1) \text{ Temp. Correction} = \frac{R_{T,obs}}{\theta^x} \quad (\text{mg O}_2/\text{L-min.})$$

$$(3) \% \text{ Difference} = \frac{|RC_1 - RC_2|}{\left(\frac{RC_1 + RC_2}{2}\right)} \times 100$$

$$(2) \% \text{ Inhibition} = \frac{\left(\frac{RC_1 + RC_2}{2}\right) - R_s}{\left(\frac{RC_1 + RC_2}{2}\right)} \times 100$$

The two control respiration rates should be within 15% of each other

Where:

R = Oxygen uptake rate (mg O₂/L-min.)T_{obs} = Sample temp., °Cθ = 1.047, x = T_{obs} - 20°C

C = Control

TSS Solids [TS] [TSS] [TDS]
3M Environmental Laboratory - Raw Data Sheet

(17th ed) pp. 2-75 to 2-77, Sec. 2540 D.

Method Reference: Std. Methods (14th ed.) pp. 89-98, sec. 200

Metro Waste Treatment Plant, St. Paul, Mn
 Mixed liquor (obtained & used 3/23/90)
 For LR H1480-2, 4, 5 & 3, 5-DGP

Calculation: $\text{mg/l Residue} = \frac{\text{mg residue} \times 1000}{\text{ml sample}}$

No. Tests	
No. Hours	
Analyst and Date Analyzed	<i>[Signature]</i> 3/23/90
QA Check	

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Sample Description	Reported Result mg/l	ml Sample	Gross Weight after 103 °C	pan + P. Filter Crucible Weight (g)	Residue Weight (g)	Residue mg/l	Comments
Initial Sludge Metro Waste Treatment Plant 1 St. Paul, Mn.		5	1.1549	1.1156	0.0393	7860	} $\bar{X} = 7853 \pm 12 \text{ mg/L}$
2		5	1.1589	1.1197	0.0392	7840	
3		5	1.1645	1.1252	0.0393	7860	
Initial in FC206CE 1000 mg/L test solution		5	1.1418	1.1263	0.0155	3100	} $\bar{X} = 3140 \pm 40 \text{ mg/L}$
4		5	1.1365	1.1208	0.0157	3140	
5		5	1.1365	1.1206	0.0159	3180	
Initial in FC206CE 500 mg/L with 205214 500mg/L test solution		5	1.1352	1.1194	0.0158	3160	} $\bar{X} = 3143 \pm 12 \text{ mg/L}$
6		5	1.1334	1.1175	0.0159	3180	
7		5	1.1341	1.1184	0.0157	3140	
500 in FC206CE 500 mg/L test solution		5	1.1338	1.1171	0.0167	3340	} $\bar{X} = 3340 \pm 40 \text{ mg/L}$
8		5	1.1384	1.1219	0.0165	3300	
9		5	1.1355	1.1186	0.0169	3380	
500 in FC206CE 500 mg/L with 205214 200mg/L test solution		5	1.1322	1.1154	0.0168	3360	} $\bar{X} = 3380 \pm 20 \text{ mg/L}$
10		5	1.1358	1.1189	0.0169	3380	
11		5	1.1380	1.1210	0.0170	3400	
12							
13							
14							
15							

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