

ACUTE AQUATIC TOXICITY

TEST SUBSTANCE

Identity: A mixture containing perfluorooctanesulfonate, which may also be referred to as PFOS, FC-95, or as a component of FC-203. (1-Octanesulfonic acid) (CAS # 2795-39-3).

Remarks: The 3M production lot numbers were 4 and 6. The test sample is FC-203. Current information indicates it is a mixture of 1.34% PFOS, 35% diethylene glycol butyl ether, 37.85% water, 20% ethylene glycol, 2.66 % Sultone foamer, 3% sodium octyl sulfate, 0.1% sodium lauryl sulfate, and 0.05% tolyltriazole.

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

METHOD:

Method: Not given.

Type: Various static and Continuous flow screening

GLP: No

Year completed: 1973 – 1974 (5 studies conducted)

Species: Not given.

Supplier: Not given.

Analytical monitoring: Temperature, and DO

Exposure period: 96-hours, 10 days, and 18 days

Statistical methods: TL₅₀/LC₅₀ values calculated using graphic interpolation.

Test organism age: Not given.

Pretreatment: Not given.

Test conditions: Various.

Lighting: Not given

Concentrations dosing rate: Various

Stability of the test chemical solutions: Not noted

Exposure vessels: Not given

Number of replicates: 1

Number of organisms per replicate: Not given.

Number of concentrations: five plus a blank control

Water chemistry during the study: Various

RESULTS

Nominal concentrations: Bk control and various concentrations

Element values:

Study 1 (w/3% concentrate Lot 4):

96-hour LC₅₀ = 0.75 mL
Study 2 (w/3% concentrate Lot 6):
96-hour LC₅₀ = 0.075% conc.
Study 3 (Lot 4):
96-hour LC₅₀ = >1000 mg/L
Study 4 (Lot 4):
96-hour LC₅₀ = 0.061%
Study 5 (Lot 4):
96-hour LC₅₀ = 0.1875%
Study 6 (Lot 4):
4-7 day LC₅₀ = 1875 mg/L
8 day LC₅₀ = 1525 mg/L
9-10 day LC₅₀ = 1250 mg/L
Study 7 (Lot 4):
96-hour TL₅₀ = 0.0438 (no units, assume % concentrate)

Element values based on nominal concentrations

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

CONCLUSIONS

The test sample LC₅₀ varied among the studies depending on the test type and duration.

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

DATA QUALITY

Reliability: Klimisch ranking = 3. Testing lacks record of methodology used. 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 2002 and 2009, 1973-1974.

OTHER

Last changed: 6/27/00

Date started; 2-27-73 Bioassay - FC-203 Lot-4 (3% Concentrate)

CONC.	24 hrs % Survival	pH	D.O. ppm	48 hrs. % Survival	pH	D.O. ppm	72 hrs. % Survival	pH	D.O. ppm	96 hrs. % Survival	pH
Control	100%		6.5	100%		5.0	100%		4.8	100%	
5.0ml 16	100%		5.4	100%		4.2	100%		3.5	100%	
10.0ml 17	100%		5.1	0%		3.9	0%	—	—	0%	—
15.0ml 18	90%		5.4	90%		4.2	0%	—	—	0%	—
20.0ml 19	70%		5.5	0%		2.7	0%	—	—	0%	—
25.0ml 20	50%		5.2	0%		2.0	0%	—	—	0%	—

Screening Test:

1.0ml one Dead
4.0ml All Dead
8.0ml All Dead

Two Dead
All Dead
All Dead

cc: LAB REQUEST BOOK - 42-5W

File: _____

Water and Wastewater

REQUEST FOR LABORATORY WORK

Lab Request No. 2002
1182

TO: ENVIRONMENTAL ENGINEERING LABORATORY - BUILDING 53-6N

Requestor	Lab Personnel
Name: <u>Dale Bacon</u> Request Date _____ Project No. _____ Prob. No. _____ Code # _____ Description: <u>* *</u> Sample Date: _____ Date Needed: _____ Hold Sample For _____ Days Est. Time: _____ Hrs.	Date Rec'd. Sample: _____ Analyst: <u>ME</u> Y <u>73</u> B <u>15</u> P <u>3</u> Date Completed: <u>12-30-73</u> Hrs. Spent: <u>20</u>

Sample Description	# 1	# 2	# 3	# 4	# 5
Total Hardness, mg/l as CaCO ₃					
Calcium Hardness, mg/l as CaCO ₃					
"P" Alkalinity, mg/l as CaCO ₃					
"M" Alkalinity, mg/l as CaCO ₃					
pH					
Sulfate, mg/l as SO ₄					
Nitrate, mg/l as NO ₃					
Chloride, mg/l as Cl					
Silica, mg/l as SiO ₂					
Fluoride, mg/l as F					
Ortho Phosphate, mg/l as P					
Total Phosphate, mg/l as P					
Total Chromium, mg/l as Cr					
Hex. Chromium, mg/l as Cr					
Total Cyanide, mg/l as CN					
Ferrocyanide, mg/l as CN					
Iron, mg/l as Fe					
Manganese, mg/l as Mn					
Surfactant, mg/l as LAS					
Phenol, mg/l					
BOD, mg/l					
COD, mg/l (Short, Long, Dilute)					
Total Suspended Solids, mg/l					
Volatile Suspended Solids, mg/l					
Turbidity, mg/l Silica					
Total Solids, mg/l					
Total Volatile Solids, mg/l					
Conductivity, mhos					
Total Bacteria, No./100 ml					
Total Coliform, No./100 ml					
Fecal Coliform, No./100 ml					
Fecal Streptococcus, No./100 ml					
Ammonia Nitrogen, mg/l as N					
Kjeldahl Nitrogen, mg/l as N					
Oil and Grease, mg/l					
Bioassay 96 hour LC 50 %	0.075	0.16	3.5	0.225	0.16

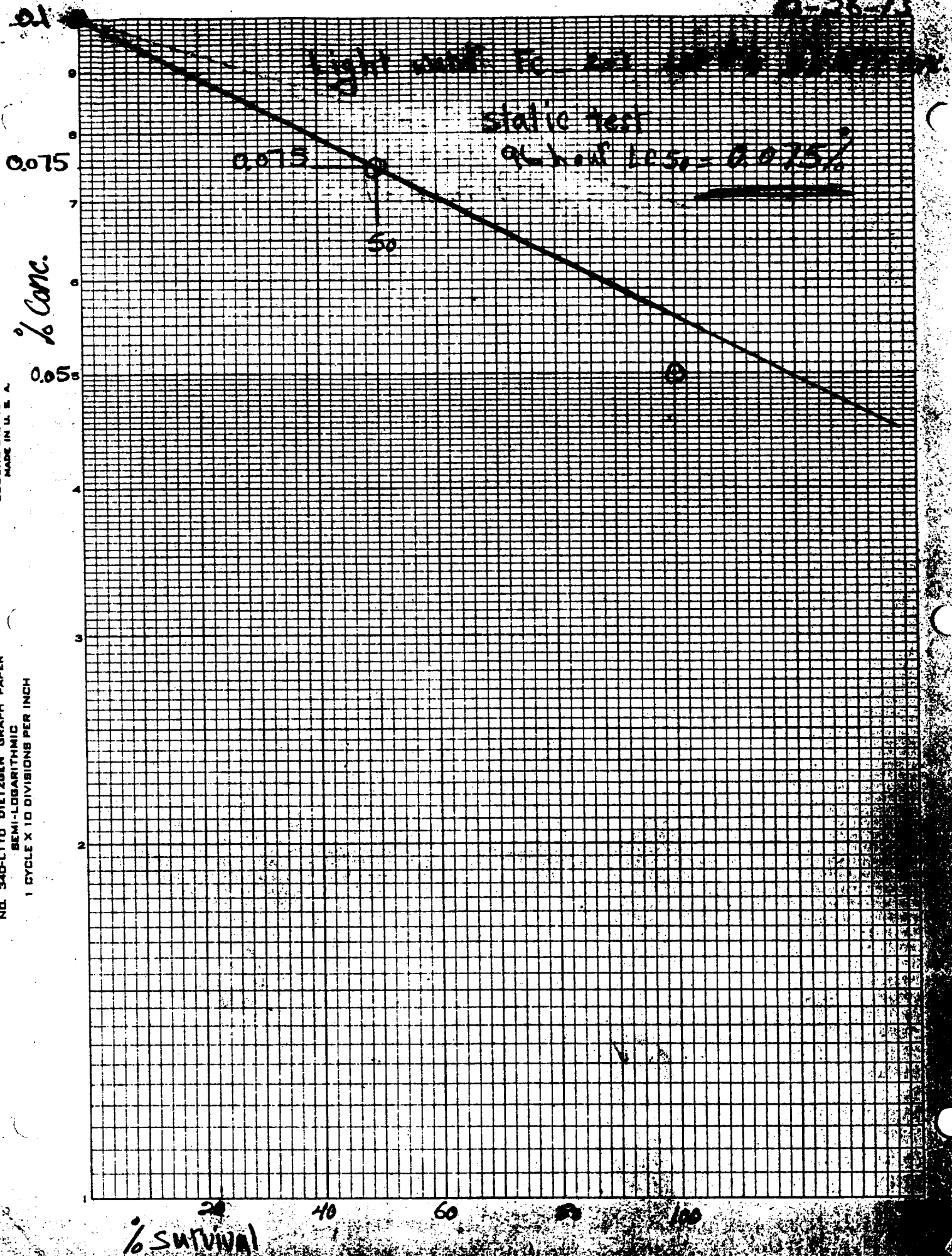
Comments: ***

1 FC-203 Lot 6 3%
#3 Ethylene glycol

#2 Butyl carbitol
#4 L-3243 (36670:48) 6%
#5 L-3243 (36670:50) 6%

EUGENE DIETZGEN CO.
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NO. 340-L110 DIETZGEN GRAPH PAPER
SEMI-LOGARITHMIC
1 CYCLE X 10 DIVISIONS PER INCH



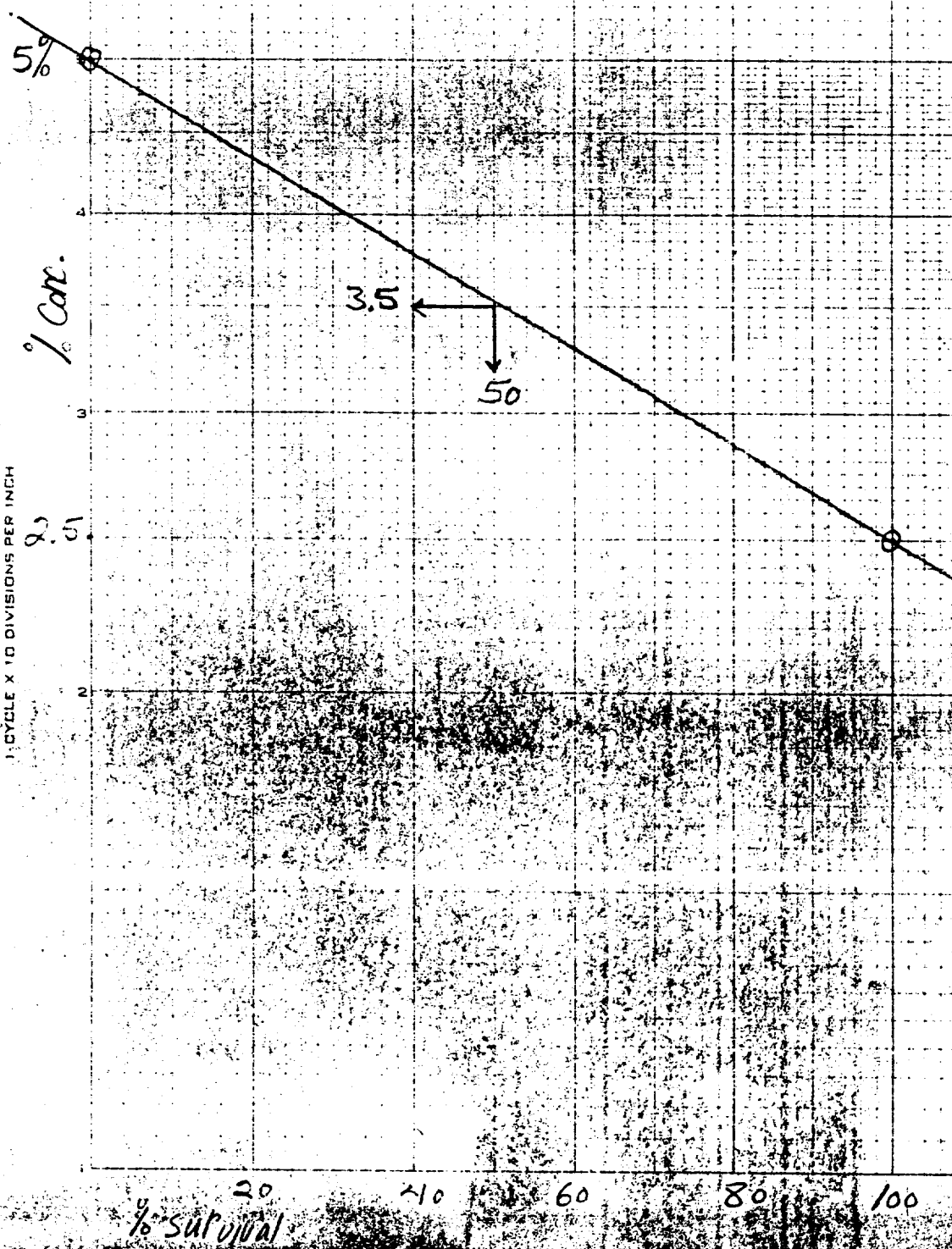
12-26-73

Ethylene Glycol
static test

96 hour $LC_{50} = \underline{\underline{3.5\%}}$

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Date started: 12-26-73

BIOASSAY - Ethylene Glycol

[illegible]

Screening Test:

12-10-73

1.0% 20ml / 2l

2.5" 50ml

5.0 100 m

No Dead

No Dead

No Dead

No Dead

No Dead

two Dead/3

```
* static-test
```

Date started: 12-26-73 Dioassay - Butyl carbitol

% CONC.	24 hrs % Survival	Temp	D.O. ppm	48 hrs. % Survival	Temp	D.O. ppm	72 hrs. % Survival	Temp	D.O. ppm	96 hrs. % Survival	Temp	D.O. ppm
Control	100	70	5.7	100	70	5.2	100	70	4.7	100	70	5.0
0.1	100	"	5.4	100	"	5.0	100	"	4.4	100	"	5.0
0.25	100	"	5.2	70	"	4.8	30	"	4.2	0	"	4.8
0.5	0	"	5.0	0	—	—	0	—	—	0	—	—
0.75	0	"	5.0	0	—	—	0	—	—	0	—	—
1.0	No Enough Sample											
160ml												

Screening Test:

12-10-73

0.01 %	0.2 ml	12	No Dead	No Dead
0.1 %	2.0 ml		No Dead	No Dead
1.0 %	2.0 ml		ALL Dead	ALL Dead

Static Test:

12-26-73

Butyl Carbitol
Static test

96 hour $LC_{50} = \underline{\underline{0.16\%}}$

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1 CYCLE X 10 DIVISIONS PER INCH

0.250

% Conc.

0.16

50

0.1

% Survival

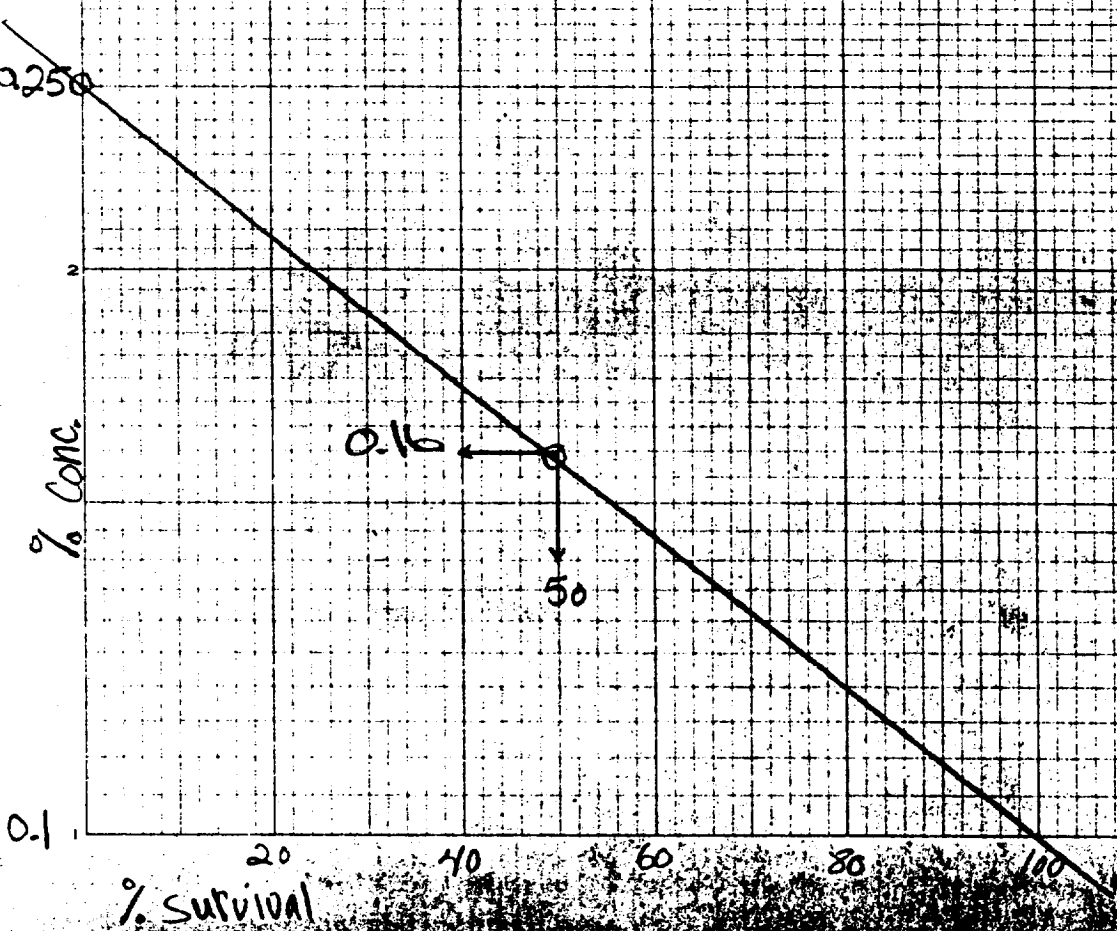
20

40

60

80

100



cc: LAB REQUEST BOOK - 42-5W

File: _____

Water and Wastewater

REQUEST FOR LABORATORY WORK

Lab Request No. ~~1984~~ 2004

TO: ENVIRONMENTAL ENGINEERING LABORATORY - BUILDING 53-6N

Requestor	Lab Personnel
Name: <u>Dale Bacon</u> Request Date _____	Date Rec'd. Sample: _____
Project No. _____ Prob. No. _____ Code # _____	Analyst: <u>ME</u>
Description: <u>FC-203 Lot #4</u>	Y <u>73</u> B <u>15</u> P <u>5</u>
Sample Date: _____ Date Needed: _____	Date Completed: <u>12-30-73</u>
Hold Sample For _____ Days Est. Time: _____ Hrs.	Hrs. Spent: <u>30</u>

Sample Description	X								
Total Hardness, mg/l as CaCO ₃									
Calcium Hardness, mg/l as CaCO ₃									
"P" Alkalinity, mg/l as CaCO ₃									
"M" Alkalinity, mg/l as CaCO ₃									
pH									
Sulfate, mg/l as SO ₄									
Nitrate, mg/l as NO ₃									
Chloride, mg/l as Cl									
Silica, mg/l as SiO ₂									
Fluoride, mg/l as F									
Ortho Phosphate, mg/l as P									
Total Phosphate, mg/l as P									
Total Chromium, mg/l as Cr									
Hex. Chromium, mg/l as Cr									
Total Cyanide, mg/l as CN									
Ferrocyanide, mg/l as CN									
Iron, mg/l as Fe									
Manganese, mg/l as Mn									
Surfactant, mg/l as LAS									
Phenol, mg/l									
BOD, mg/l									
COD, mg/l (Short, Long, Dilute)									
Total Suspended Solids, mg/l									
Volatile Suspended Solids, mg/l									
Turbidity, mg/l Silica									
Total Solids, mg/l									
Total Volatile Solids, mg/l									
Conductivity, mhos									
Total Bacteria, No./100 ml									
Total Coliform, No./100 ml									
Fecal Coliform, No./100 ml									
Fecal Streptococcus, No./100 ml									
Ammonia Nitrogen, mg/l as N									
Kjeldahl Nitrogen, mg/l as N									
Oil and Grease, mg/l									
Bioassay 96 hour LC50% * 0.1875									
** 0.061									
Comments: * C. Flow test									
** static test									

Light water FC-203 5 gal. pail Lot #4

Screening test

11-15-73

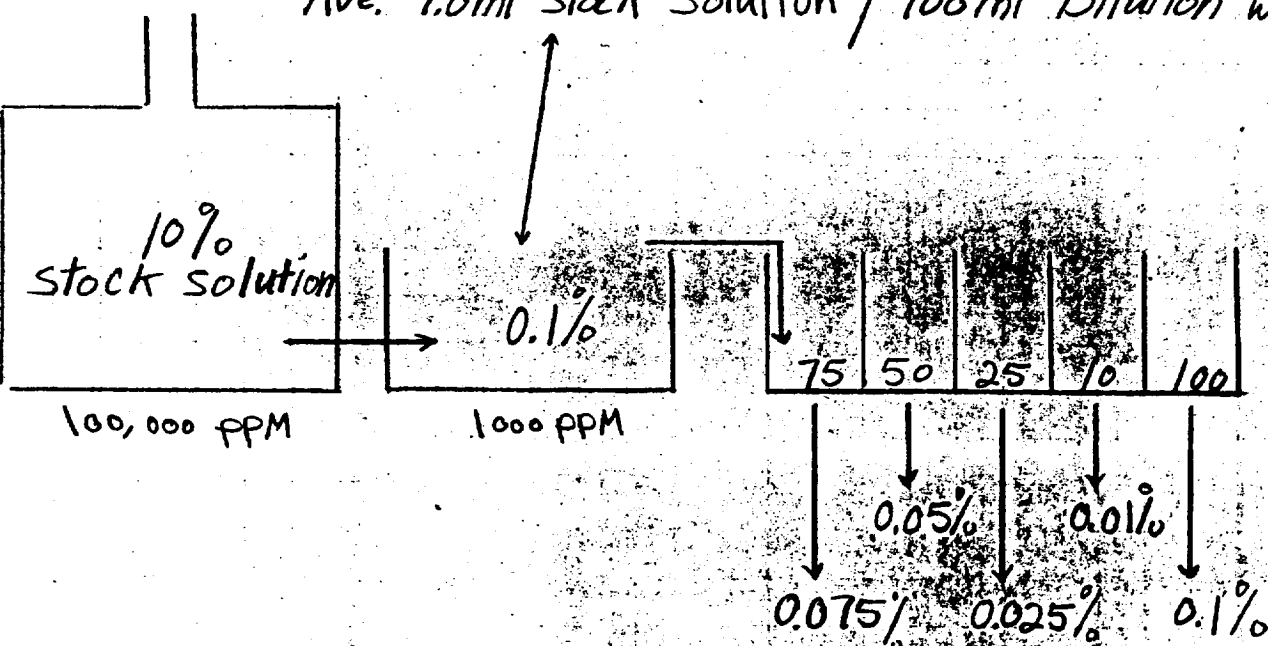
		<u>24 hour</u>	<u>48 hour</u>
Control		No Dead	No Dead
0.01%	0.2 ml	No Dead	No Dead
0.1%	2.0 ml	No Dead	2 Dead/3 *
1.0%	20 ml	ALL Dead	ALL Dead
10%	200 ml	ALL Dead	ALL Dead

* C.Flow test :-

PPM	100	250	500	750	1000
%	0.01	0.025	0.05	0.075	0.1
ML/16L	1.6	4	8	12	16

Every cycle :-

Ave. 7.0ml stock solution / 700ml Dilution water → 1% sol



PPM 100 250 500 750 1000

12-1-73

Light water FC-203 Lot #4

Screening test

			% survival	
			24 hour	48 hour
			100%	100%
#1	0.01 %	1.6 ml/16l		
#2	0.1	16 ml	10%	0%
#3	1.0	160 ml	0%	0%

Date started: 12-3-73 * Bioassay - Fc-203

Lot #4

CONC.	① day			②			③			④		
	24 hrs % Survival	Temp	D.O. ppm	48 hrs. % Survival	Temp	D.O. ppm	72 hrs. % Survival	Temp	D.O. ppm	96 hrs. % Survival	Temp	D.O. ppm
Control	100	60	6.2	100	60	6.0	100	62		100	66	6
0.01	100	60	6.0	100	"	5.9	100	"		100	66	5
0.025	100	60	5.8	100	"	5.8	100	"		100	66	5
0.05	100	60	5.6	100	"	6.0	100	"		100	66	6
0.075	100	60	5.5	100	"	5.7	100	"		100	66	5.7
0.1	100	60	5.5	100	"	6.0	100	"		100	66	5.7

Screening Test:

* c.flow test - 10% stock solution to start with

100 250 500 750 1000 ppm

Date started:

* Bioassay -

CONC.	^{(5) day} 24 hrs			⁽⁶⁾ 48 hrs.			⁽⁷⁾ 72 hrs.			⁽⁸⁾ 96 hrs.		
	% Survival	Temp	D.O. ppm	% Survival	Temp	D.O. ppm	% Survival	Temp	D.O. ppm	% Survival	Temp	D.O. ppm
Control	100	65	5.8	100			100	70	5.5	100	70	5.5
0.01	100	"	5.7	100			100	70	4.7	100	70	4.7
0.025	100	"	5.5	100			100	70	4.5	100	70	4.5
0.05	100	"	5.7	100			100	70	4.2	100	70	4.2
0.075	100	"	5.0	100			100	70	4.2	100	70	4.2
0.1	100	"	5.0	100			100	70	4.0	100	70	4.0

Screening Test:

* C. Flow test

Date started:

BIOASSAY -

CONC.	9 day			10 day			11 day			12 day		
	24 hrs % Survival	Temp	D.O. ppm	48 hrs. % Survival	Temp	D.O. ppm	72 hrs. % Survival	Temp	D.O. ppm	96 hrs. % Survival	Temp	
Control	100	67	5.9	100			100	70	5.2	100		
0.01	100	67	5.7	100			100	"	5.0	100		
0.025	100	67	5.0	100			100	"	4.7	100		
0.05	100	67	5.0	100			100	"	5.0	100		
0.075	100	67	5.2	100			100	"	5.0	100		
0.1	100	67	5.0	100			100	"	4.5	100		

Screening Test:

* C.Flow test

Date started:

BioAssy -

13 day				14			15			16	
CONC.	24 hrs % Survival	Temp	D.O. ppm	48 hrs. % Survival	Temp	D.O. ppm	72 hrs. % Survival	Temp	D.O. ppm	96 hrs. % Survival	Temp
Control	100			100	70	5.2	100			100	
	100			100	"	5.0	100			100	
	100			100	"	5.0	100			100	
	100			100	"	4.7	100			100	
	100			100	"	4.5	100			100	
	100			100	"	4.2	100			100	

Screening Test:

Date started: 12-19-73 **DIOASSY -**

CONC.	17 day			18								
	24 hrs % Survival	Temp	D.O. ppm	48 hrs. % Survival	Temp	D.O. ppm	72 hrs. % Survival	Temp	D.O. ppm	96 hrs. % Survival	Temp	D.O. ppm
Control	100	70	5.2	100	70	5.4						
0.05	100	70	5.0	100	"	4.7						
0.125	100	70	5.0	0	"	4.5						
0.25	100	70	5.0	0	"	4.6						
0.375%	100	70	4.7	0	"	4.5						
0.5%	10	70	4.9	0	"	4.2						

Screening Test: * C.Flow - 50% (500,000 ppm) stock solution to start with.

500 1,250 2,500 3,750 5,000 PPM
 0.05 0.125 0.25 0.375 0.5 % by vol.

Date started: 12-4-73

BIOASSY - FC-203: Lot #4

% CONC.	24 hrs % Survival	Temp	D.O. ppm	48 hrs. % Survival	Temp	D.O. ppm	72 hrs. % Survival	Temp	D.O. ppm	96 hrs. % Survival	Temp
Control	100	60	5.6	100	62	5.2	100	65	5.0	100	65
0.01	100	"	5.5	100	"	5.0	100	65	4.5	100	65
0.025	100	"	5.5	100	"	5.1	100	65	4.2	100	65
0.05	100	"	5.3	100	"	5.0	100	65	3.9	100	65
0.075	100	"	5.2	100	"	5.0	0	65	2.9	0	—
0.1	100	"	4.9	100	"	4.5	0	65	2.5	0	—

Screening Test:

PPM 100 250 500 750 1000

* static test

% 0.01 0.025 0.05 0.075 0.1

ML/16L 1.6 4 8 12 1

12-4-73

Light water: Fc-203 Lot #4
static test

96 hour $LC_{50} = \underline{\underline{0.061\%}}$

0.061
50%

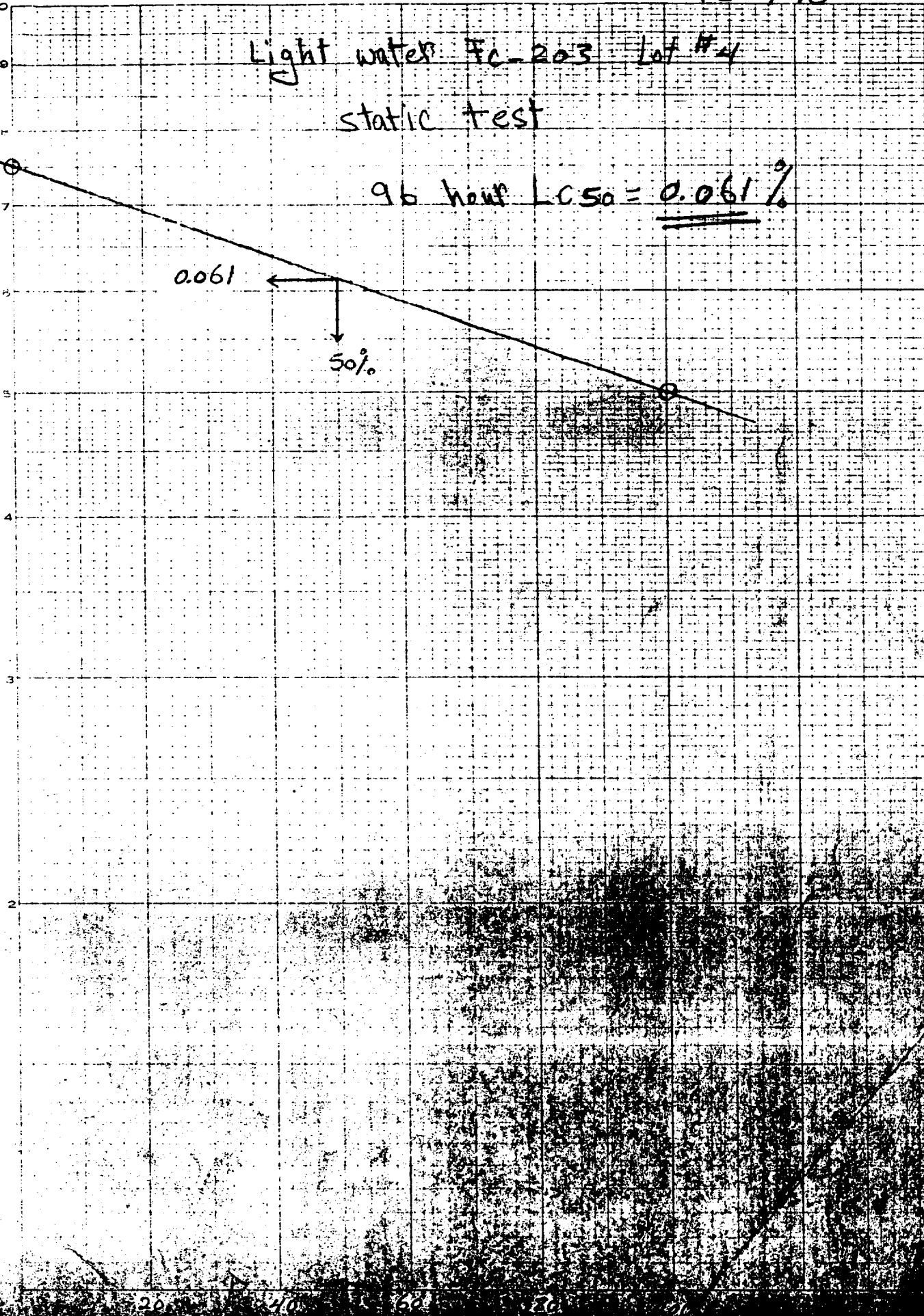
0.075

0.05

% CONC.

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NO. 340 L110 DIETZGEN GRAPH PAPER
SEMI-LOGARITHMIC
1 CYCLE X 10 DIVISIONS PER INCH



Date started: 12-26-73 **BIOASSAY** - Light water Ec 2.3 Lot #4

% CONC.	24 hrs % Survival	Temp	D.O ppm	48 hrs. % Survival	Temp	D.O ppm	72 hrs. % Survival	Temp	D.O ppm	96 hrs. % Survival	Temp
Control	100	70	5.9	100	70	5.5	100	70	5.3	100	70
0.0250	100	70	5.5	100	70	5.2	100	"	5.0	100	"
0.0625	100	70	5.7	100	70	5.0	100	"	5.1	100	"
0.1250	90	70	5.5	90	70	5.2	90	"	5.0	80	"
0.1875	90	70	5.0	50	70	5.0	50	"	4.5	50	"
0.2500	0	70	5.2	0	—	—	0	—	—	0	—

Screening Test: * C. Flow test 25% (250,000 ppm) solution to start with

ppm	250	625	1250	1875	2,500
%	0.025	0.0625	0.125	0.1875	0.25
ML/16L	4	10	20	30	40

22.58
10.58
1000

12-26-73

Light water Fc-203 Lot # 4

C-Flow test

96 hour LC50 = 0.1875%

DUPLICATE DIETZGEN CO.
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NO. 340-1110 DIETZGEN GRAPH PAPER
SEMI-LOGARITHMIC
1 CYCLE X 10 DIVISIONS PER INCH

% Conc.

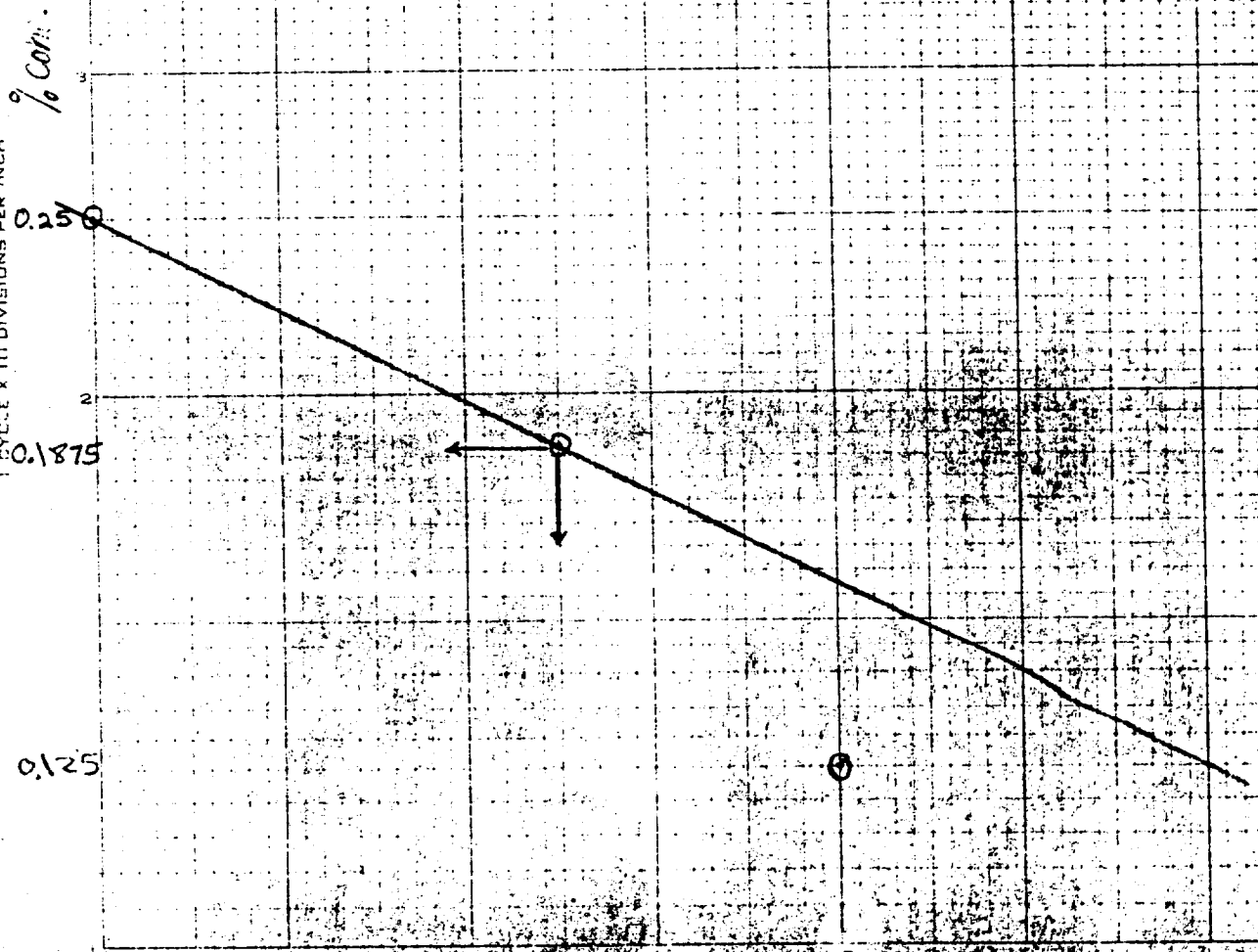
0.250

0.1875

0.125

20 40 60 80 100

% Survival



cc: LAB REQUEST BOOK - 42-5W

File: _____

Water and Wastewater

REQUEST FOR LABORATORY WORK

Lab Request No. _____

2009
~~1989~~

TO: ENVIRONMENTAL ENGINEERING LABORATORY - BUILDING 53-6N

Requestor	Lab Personnel
Name: <u>D. Bacon</u> Request Date: _____	Date Rec'd. Sample: _____
Project No. _____ Prob. No. _____ Code # _____	Analyst: <u>ME</u>
Description: <u>FC-203 Lot# 4</u>	Y <u>74</u> B <u>15</u> P <u>10</u>
Sample Date: _____ Date Needed: _____	Date Completed: <u>1-5-74</u>
Hold Sample For _____ Days Est. Time: _____ Hrs.	Hrs. Spent: <u>15</u>

Sample Description	4-7 day	8 day	9-10 day		
Total Hardness, mg/l as CaCO ₃					
Calcium Hardness, mg/l as CaCO ₃					
"P" Alkalinity, mg/l as CaCO ₃					
"M" Alkalinity, mg/l as CaCO ₃					
pH					
Sulfate, mg/l as SO ₄					
Nitrate, mg/l as NO ₃					
Chloride, mg/l as Cl					
Silica, mg/l as SiO ₂					
Fluoride, mg/l as F					
Ortho Phosphate, mg/l as P					
Total Phosphate, mg/l as P					
Total Chromium, mg/l as Cr					
Hex. Chromium, mg/l as Cr					
Total Cyanide, mg/l as CN					
Ferrocyanide, mg/l as CN					
Iron, mg/l as Fe					
Manganese, mg/l as Mn					
Surfactant, mg/l as LAS					
Phenol, mg/l					
BOD, mg/l					
COD, mg/l (Short, Long, Dilute)					
Total Suspended Solids, mg/l					
Volatile Suspended Solids, mg/l					
Turbidity, mg/l Silica					
Total Solids, mg/l					
Total Volatile Solids, mg/l					
Conductivity, mhos					
Total Bacteria, No./100 ml					
Total Coliform, No./100 ml					
Fecal Coliform, No./100 ml					
Fecal Streptococcus, No./100 ml					
Ammonia Nitrogen, mg/l as N					
Kjeldahl Nitrogen, mg/l as N					
Oil and Grease, mg/l					
Bioassay, LC50 PPM	1,875	1,525	1,250		

Comments: _____

FC-203 Lot #4

Bioassay study - C.F/w test

4-7 day $LC_{50} = \underline{\underline{1,875 \text{ PPM}}}$

8 day $LC_{50} = \underline{\underline{1,525 \text{ PPM}}}$

9-10 day $LC_{50} = \underline{\underline{1,250 \text{ PPM}}}$

EUGENE DIETZGEN CO.
MADE IN U.S.A.

NO. 340-1110, DIETZGEN GRAPH PAPER

SEMI-LOGARITHMIC

CYCLE X 10 DIVISIONS PER INCH

2,500
PPM

1,875
PPM

Conc.

1,250
PPM

% survival

40

60

80

100

4-7 days

8 days

9-10 days

Date 5 Wed; 12-26-73 * Bioassay - T - 203 Lot 4

5 day

#6

#7

#8

CONC.	24 hrs	Temp	D.O. ppm	48 hrs	Temp	D.O. ppm	72 hrs	Temp	D.O. ppm	96 hrs	Temp	D.O. ppm
	% Survival			% Survival			% Survival			% Survival		
Control	100	70	5.2	100	65	5.3	100	65	5.4	100	72	5.2
250 PPM	100	"	5.0	100	"	5.1	100	"	5.0	100	"	5.0
625	100	"	5.0	100	"	4.9	100	"	5.0	100	"	4.7
1,250	80	"	4.7	80	"	4.9	80	"	4.9	70	"	4.2
1,875	50	"	4.7	50	"	5.0	50	"	4.7	30	"	4.0
2,500	0	—		0	—		0	—		0		

~~Screening Test~~: * C-Flow test

25% solution to start with

2783

2258

%

0.0250

0.0625

0.1250

0.1875

0.2500

Date 5.11.61

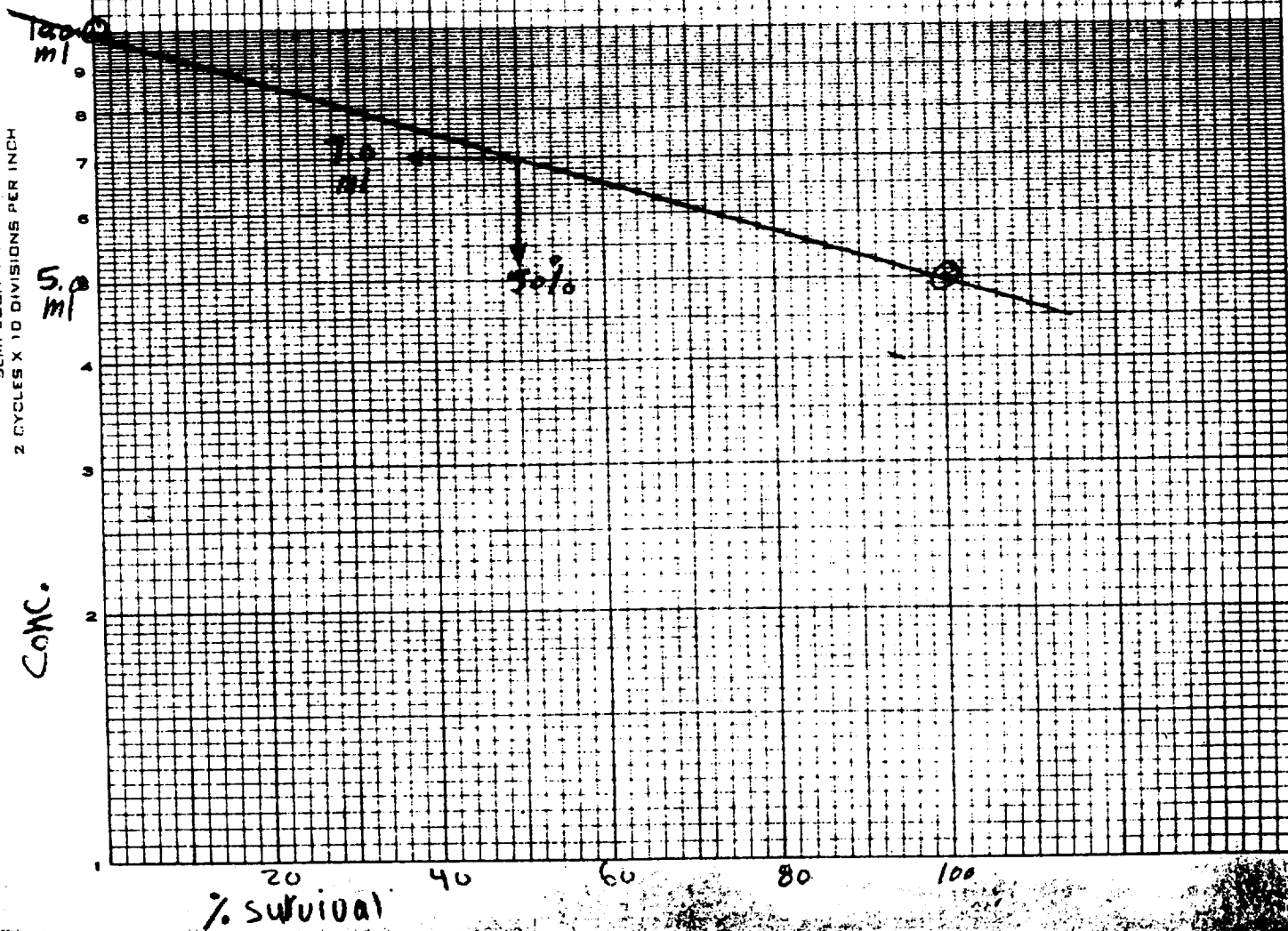
BIOASSAY - 3-203 Lot #4

9 day

CONC.	24 hrs	pH	D.O. ppm	48 hrs	pH	D.O. ppm	72 hrs	pH	D.O. ppm	96 hrs	pH	D.O. ppm
	% Survival			% Survival			% Survival			% Survival		
Control	100		5.0	100		5.0						
250 ppm	100		4.7	100		4.5						
625	100		4.5	100		4.2						
1,250	50		4.5	50		4.0						
1,875	0		4.2	0	—							
2,500	0	—		0	—							

* c.Flow test

CONC.



Stress: static test
FC-203

$$\frac{1}{50} \text{ hour} \quad \frac{1}{50} = \frac{70}{16,000} \times 100 =$$

0.0438