

## 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-3017. (1-Octanesulfonic acid) (CAS # 2795-39-3).

**Remarks:** The 3M production lot number was not noted. The test sample is FC-3017. Current information indicates FC-3017 is a mixture of 0.67% PFOS, 20% diethylene glycol butyl ether, 55.41% water, 21% ethylene glycol, 1.33% Hydroxy foamer, 1% sodium octyl sulfate, 0.5% polyoxyethylene monoethylphenyl ether, 0.04% sodium lauryl sulfate, and 0.05% tolyltraizole.

***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:** OECD Guideline 209

**GLP:** No

**Year Completed:** 1995

**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 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 H<sub>2</sub>SO<sub>4</sub> and diluted to 1L with Milli-Q™ water. The pH of the reference solution was measured to be 7.7. Test substance was added directly to the test vessels.

**Test vessels:** Not given.

**Number of concentrations:** one plus blank control, in duplicate

**Temperature:** 18.9 – 20.6 °C

**Total suspended solids and pH on day of testing:** 3.8 g/L (TSS in final: 1.5 g/L), initial pH 6.4 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:** 2 Blank controls, 1000 mg/L plus 10 mg/L concentration for reference substance, all in duplicate.

**Element values:** 30-minute EC<sub>50</sub> = >1000 mg/L  
3-hour EC<sub>50</sub> = >1000 mg/L

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 substance 3-hour EC<sub>50</sub> for activated sludge respiration inhibition was determined to be >1000 mg/L, the highest concentration tested.

**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, the 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 N3188, 1995.

## OTHER

**Last changed:** 6/26/00

ENVIRONMENTAL LABORATORY  
ACTIVATED SLUDGE MIXED LIQUOR

LR N3188  
Date 9/29/95  
Analyst CNT  
Page 1 of 7

Type Test OECD Activated Sludge Respiration Inhibition Test #209

ACTIVATED SLUDGE MIXED LIQUOR

Date Obtained 9/29/95

Date Used 9/29/95

Mixed Liquor Suspended Solids (MLSS) 3.8 g/L Adjusted to      g/L

MLSS in final test solution 1.5 g/L

Initial pH 6.4 (1st test soln. prepared)

Final pH 7.1 (last soln. prepared)

Comments Sludge was allowed to settle out. Then ~1 L of the upper liquid layer was poured off. Sludge was then mix thoroughly and aerated.

TEST SUBSTANCE

Sample Description EC-3017 (Light Water)

Initial pH of highest conc. tested (1000 mg/L) = 7.3

Adjusted to pH     

Comments     

REFERENCE SUBSTANCE

Sample Description 3,5-Dichlorophenol

Initial pH of stock solution (10 mg/L) = 7.7

Adjusted to pH     

Comments     

EXPOSURE CONDITIONS

Test Duration 3 hrs.

Test Temperature Range: 18.9 to 20.6 °C

Comments

pg 2 of 7

LR N2188

ENVIRONMENTAL LABORATORY

OECD ACTIVATED SLUDGE RESPIRATION INHIBITION TEST #209  
TEST SOLUTIONS PREPARATION

Test Substance: FC-3017 (Light Water)

Sample Description: Clear, amber colored liquid.

Analyst: G.W.S

Date: 9/29/95

| Test Conc.<br>mg/L | mg added<br>per 500 ml | mL MQ(1) water<br>per 500 mL | mL syn. sewage<br>feed per 500 mL | mL microbial<br>inoculum per 500 mL | Total Volume<br>mL |
|--------------------|------------------------|------------------------------|-----------------------------------|-------------------------------------|--------------------|
| Control            | —                      | 254                          | 16                                | 200                                 | 500                |
| 1000               | 500                    | 254                          | 16                                | 200                                 | 500                |

In L. 1.5 H = 7.3

(1) Millipore Milli-QR high purity water

PG 3017

LR N3188

ENVIRONMENTAL LABORATORY

OECD ACTIVATED SLUDGE RESPIRATION INHIBITION TEST #209

RAW DATA

Test Substance: FL-3017 (Light Water)  
 Source of Activated Sludge Mixed Liquor: Metcalf Waste Water Treatment Plant St. Paul, MN  
 Date Received: 7/29/95 Date Used: 7/29/95  
 Analyst: GWJ

30-MINUTE EXPOSURE

DISSOLVED OXYGEN CONCENTRATIONS, mg/L

| Test Substance<br>Concentration (µg/L) | TIME-MINUTES |     |     |     |     |     |     |     |     |     |     | Temp<br>°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                           | 7.7          | 7.5 | 7.3 | 7.1 | 6.8 | 6.6 | 6.4 | 6.0 | 5.7 | 5.4 | 5.2 | 20.2       |
| 1000                                   | 6.6          | 6.3 | 5.9 | 5.4 | 5.1 | 4.7 | 4.3 | 3.9 | 3.5 | 3.0 | 2.6 | 20.6       |
| 100                                    | 7.1          | 6.6 | 6.2 | 5.9 | 5.4 | 5.2 | 4.7 | 4.2 | 3.8 | 3.4 | 3.0 | 20.3       |
|                                        |              |     |     |     |     |     |     |     |     |     |     |            |
|                                        |              |     |     |     |     |     |     |     |     |     |     |            |
|                                        |              |     |     |     |     |     |     |     |     |     |     |            |
| BK CONTROL II                          | 7.7          | 7.5 | 7.3 | 7.0 | 6.7 | 6.4 | 6.2 | 5.9 | 5.6 | 5.3 | 5.1 | 20.0       |

3-HOUR EXPOSURE

DISSOLVED OXYGEN CONCENTRATIONS, mg/L

| Test Substance<br>Concentration (µg/L) | TIME - MINUTES |     |     |     |     |     |     |     |     |     |     | Temp<br>°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                           | 8.0            | 7.8 | 7.6 | 7.5 | 7.2 | 7.0 | 6.7 | 6.5 | 6.3 | 6.0 | 5.8 | 19.3       |
| 1000                                   | 7.7            | 7.5 | 7.3 | 7.0 | 6.7 | 6.5 | 6.2 | 5.8 | 5.5 | 5.3 | 5.0 | 19.9       |
| 100                                    | 7.7            | 7.8 | 7.7 | 7.4 | 7.1 | 7.0 | 6.7 | 6.4 | 6.2 | 5.9 | 5.7 | 19.5       |
|                                        |                |     |     |     |     |     |     |     |     |     |     |            |
|                                        |                |     |     |     |     |     |     |     |     |     |     |            |
|                                        |                |     |     |     |     |     |     |     |     |     |     |            |
| BK CONTROL II                          | 7.9            | 7.8 | 7.6 | 7.4 | 7.1 | 6.9 | 6.7 | 6.4 | 6.2 | 6.0 | 5.7 | 18.7       |

pH of activated sludge mixed liquor: Initial 6.4 Final 7.1  
 pH of highest concentration tested: Initial 7.0 Final 6.7  
 Test temperature: 19.9

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

Lr: N3188  
Date: 9/29/95

Test Substance: EC-3017

Analyst: GWJ

30-Minute Exposure

Dissolved Oxygen Concentrations, mg/L

| mg/L   | 0.5 | 1.0 | 1.5 | 2.0 | 2.5 | 3.0 | 3.5 | 4.0 | 4.5 | 5.0 | 5.5 | Temp, °C | R (1)  | Ra (2) | % Inh. (3) |
|--------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|----------|--------|--------|------------|
| BK1    | 7.7 | 7.5 | 7.3 | 7.1 | 6.8 | 6.6 | 6.4 | 6.0 | 5.7 | 5.4 | 5.2 | 20.2     | 0.5285 | 0.5212 |            |
| 1000.0 | 6.6 | 6.3 | 5.9 | 5.4 | 5.1 | 4.7 | 4.3 | 3.9 | 3.5 | 3.0 | 2.6 | 20.6     | 0.8133 | 0.7801 | -46        |
| Dup.   | 7.1 | 6.6 | 6.2 | 5.9 | 5.4 | 5.2 | 4.7 | 4.2 | 3.8 | 3.4 | 3.0 | 20.3     | 0.8073 | 0.7906 | -48        |
|        |     |     |     |     |     |     |     |     |     |     |     |          |        |        |            |
|        |     |     |     |     |     |     |     |     |     |     |     |          |        |        |            |
|        |     |     |     |     |     |     |     |     |     |     |     |          |        |        |            |
| BK2    | 7.7 | 7.5 | 7.3 | 7.0 | 6.7 | 6.4 | 6.2 | 5.9 | 5.6 | 5.3 | 5.1 | 20.0     | 0.5479 | 0.5479 |            |

% diff in controls (4):

5.0

EC50 (95% CI):

RaCm (5): 0.5345

3-Hour Exposure

Dissolved Oxygen Concentrations, mg/L

| mg/L   | 0.5 | 1.0 | 1.5 | 2.0 | 2.5 | 3.0 | 3.5 | 4.0 | 4.5 | 5.0 | 5.5 | Temp, °C | R (1)  | Ra (2) | % Inh. (3) |
|--------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|----------|--------|--------|------------|
| BK1    | 8.0 | 7.8 | 7.6 | 7.5 | 7.2 | 7.0 | 6.7 | 6.5 | 6.3 | 6.0 | 5.8 | 19.3     | 0.4558 | 0.4785 |            |
| 1000.0 | 7.7 | 7.5 | 7.3 | 7.0 | 6.7 | 6.5 | 6.2 | 5.8 | 5.5 | 5.3 | 5.0 | 19.9     | 0.5697 | 0.5737 | -17        |
| Dup.   | 8.0 | 7.8 | 7.7 | 7.4 | 7.1 | 7.0 | 6.7 | 6.4 | 6.2 | 5.9 | 5.7 | 19.3     | 0.4836 | 0.5078 | -4         |
|        |     |     |     |     |     |     |     |     |     |     |     |          |        |        |            |
|        |     |     |     |     |     |     |     |     |     |     |     |          |        |        |            |
|        |     |     |     |     |     |     |     |     |     |     |     |          |        |        |            |
| BK2    | 7.9 | 7.8 | 7.6 | 7.4 | 7.1 | 6.9 | 6.7 | 6.4 | 6.2 | 6.0 | 5.7 | 18.9     | 0.4655 | 0.5024 |            |

% diff in controls (4):

4.9

EC50 (95% CI):

RaCm (5): 0.4905

(1) R= Oxygen uptake rate (mg O<sub>2</sub>/L-min)

(2) Ra= R adjusted to 20°C= [R/e.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

e= 1.072

RaCm= mean adjusted R, controls

Bk= blank control, S= sample

Tobs= sample temp

x= Tobs-20°C

LR N 3155

## ENVIRONMENTAL LABORATORY

OECD ACTIVATED SLUDGE RESPIRATION INHIBITION TEST #209  
TEST SOLUTIONS PREPARATIONTest Substance: 3,5-Dichlorophenol Reference SubstanceSample Description: Aldrich Chemical Co. Red Label Lot #CC41CKZAnalyst: G10J

Date: \_\_\_\_\_

## Stock Solution

Preparation: 0.5g/L; 1 mL = 0.5mg

weighed out 0.5g 3,5-DCP, then added to 10 mL of 1N NaOH and diluted up to ~30 mL with Milli-Q H<sub>2</sub>O, while stirring, until dissolved. Then added ~2 mL 1N H<sub>2</sub>SO<sub>4</sub> while stirring, until the point of precipitant precipitation. Then finally diluted to the mark of 1L with Milli-Q H<sub>2</sub>O.  
Initial pH = 7.7

| Test Conc.<br>mg/L | mL added<br>per 500 mL | mL MQ <sup>(1)</sup> water<br>per 500 mL | mL syn. sewage<br>feed per 500 mL | mL microbial<br>inoculum per 500 mL | Total Volume<br>mL |
|--------------------|------------------------|------------------------------------------|-----------------------------------|-------------------------------------|--------------------|
| Plank Controls     | —                      | 284                                      | 16                                | 200                                 | 500                |
| 10                 | 10                     | 274                                      | 16                                | 200                                 | 500                |

(1) Millipore Milli-QR high purity water

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LR NR155

# ENVIRONMENTAL LABORATORY

## OECD ACTIVATED SLUDGE RESPIRATION INHIBITION TEST #209

### RAW DATA

Test Substance: 2,5-Dichlorophenol Reference Substance  
 Source of Activated Sludge Mixed Liquor: Alto Lake Water Treatment Plant 50 mg/L  
 Date Received: 9/24/85 Date Used: 9/25/85  
 Analyst: GIN J

### 30-MINUTE EXPOSURE

### DISSOLVED OXYGEN CONCENTRATIONS, mg/L

| Test Substance<br>Concentration (mg/L) | TIME-MINUTES |     |     |     |     |     |     |     |     |     | Temp<br>°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                           | 77           | 75  | 73  | 71  | 69  | 67  | 65  | 63  | 61  | 59  | 57         | 20.0 |
| IC                                     | 80           | 78  | 76  | 74  | 72  | 71  | 70  | 69  | 66  | 64  | 63         | 20.1 |
|                                        |              |     |     |     |     |     |     |     |     |     |            |      |
|                                        |              |     |     |     |     |     |     |     |     |     |            |      |
|                                        |              |     |     |     |     |     |     |     |     |     |            |      |
|                                        |              |     |     |     |     |     |     |     |     |     |            |      |
|                                        |              |     |     |     |     |     |     |     |     |     |            |      |
| BK CONTROL II                          | 77           | 75  | 73  | 70  | 67  | 64  | 62  | 59  | 56  | 53  | 51         | 20.0 |

### 3-HOUR EXPOSURE

### DISSOLVED OXYGEN CONCENTRATIONS, mg/L

| Test Substance<br>Concentration (mg/L) | 0.5 | 1.0 | 1.5 | 2.0 | TIME - MINUTES |     |     | 4.0 | 4.5 | 5.0 | 5.5 | Temp<br>°C |
|----------------------------------------|-----|-----|-----|-----|----------------|-----|-----|-----|-----|-----|-----|------------|
|                                        |     |     |     |     | 2.5            | 3.0 | 3.5 |     |     |     |     |            |
| BK CONTROL I                           | 86  | 78  | 76  | 75  | 72             | 71  | 67  | 65  | 63  | 60  | 58  | 19.2       |
| IC                                     | 82  | 81  | 80  | 80  | 79             | 78  | 77  | 76  | 76  | 75  | 74  | 20.0       |
|                                        |     |     |     |     |                |     |     |     |     |     |     |            |
|                                        |     |     |     |     |                |     |     |     |     |     |     |            |
|                                        |     |     |     |     |                |     |     |     |     |     |     |            |
|                                        |     |     |     |     |                |     |     |     |     |     |     |            |
| BK CONTROL II                          | 79  | 78  | 76  | 74  | 71             | 69  | 67  | 64  | 62  | 60  | 57  | 18.9       |

pH of activated sludge mixed liquor: Initial 6.4 Final 7.1  
 pH of highest concentration tested: Initial 7.2 Final 7.6  
 Test temperature: 18.9 to 20.2

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

L.R N3188  
Date: 9/29/95

Test Substance: 3,5-DICHLOROPHENOL

Analyst: GWJ

**30-Minute Exposure**

**Dissolved Oxygen Concentrations, mg/L**

| mg/L | 0.5 | 1.0 | 1.5 | 2.0 | 2.5 | 3.0 | 3.5 | 4.0 | 4.5 | 5.0 | 5.5 | Temp. °C | R (1)  | Ra (2) | % Inh. (3) |
|------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|----------|--------|--------|------------|
| BK1  | 7.7 | 7.5 | 7.3 | 7.1 | 6.8 | 6.6 | 6.4 | 6.0 | 5.7 | 5.4 | 5.2 | 20.2     | 0.5285 | 0.5212 |            |
| 10   | 8.0 | 7.8 | 7.6 | 7.5 | 7.3 | 7.1 | 6.9 | 6.7 | 6.6 | 6.3 | 6.1 | 20.1     | 0.3745 | 0.3720 | 30         |
|      |     |     |     |     |     |     |     |     |     |     |     |          |        |        |            |
|      |     |     |     |     |     |     |     |     |     |     |     |          |        |        |            |
|      |     |     |     |     |     |     |     |     |     |     |     |          |        |        |            |
| BK2  | 7.7 | 7.5 | 7.3 | 7.0 | 6.7 | 6.4 | 6.2 | 5.9 | 5.6 | 5.3 | 5.1 | 20.0     | 0.5479 | 0.5479 |            |

% diff in controls (4): 5.0

RaCm (5): 0.5345

**3-Hour Exposure**

**Dissolved Oxygen Concentrations, mg/L**

| mg/L | 0.5 | 1.0 | 1.5 | 2.0 | 2.5 | 3.0 | 3.5 | 4.0 | 4.5 | 5.0 | 5.5 | Temp. °C | R (1)  | Ra (2) | % Inh. (3) |
|------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|----------|--------|--------|------------|
| BK1  | 8.0 | 7.8 | 7.6 | 7.5 | 7.2 | 7.0 | 6.7 | 6.5 | 6.3 | 6.0 | 5.8 | 19.3     | 0.4558 | 0.4785 |            |
| 10   | 8.2 | 8.1 | 8.0 | 8.0 | 7.9 | 7.8 | 7.7 | 7.6 | 7.6 | 7.5 | 7.4 | 20.0     | 0.1552 | 0.1552 | 68         |
|      |     |     |     |     |     |     |     |     |     |     |     |          |        |        |            |
|      |     |     |     |     |     |     |     |     |     |     |     |          |        |        |            |
|      |     |     |     |     |     |     |     |     |     |     |     |          |        |        |            |
| BK2  | 7.9 | 7.8 | 7.6 | 7.4 | 7.1 | 6.9 | 6.7 | 6.4 | 6.2 | 6.0 | 5.7 | 18.9     | 0.4655 | 0.5024 |            |

% diff in controls (4): 4.9

RaCm (5): 0.4905

- (1) R= Oxygen uptake rate (mg O<sub>2</sub>/L-min)
- (2) Ra= R adjusted to 20°C= [R/a.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

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

PS  
LR N3188  
7047

**Chain of Custody Record**  
3M Environmental Laboratory

02298

|                               |
|-------------------------------|
| 3M Requester<br><b>SAB</b>    |
| 3M Project Lead<br><b>SAB</b> |

Form 10176 PWD

|                                            |                                                      |                                         |
|--------------------------------------------|------------------------------------------------------|-----------------------------------------|
| 3M Project Title<br><b>MCLWS Piggy Eye</b> | 3M Project Title<br><b>Mixed Liquor P.U. 9/29/95</b> | 3M Project Number<br><b>20-0222</b>     |
| Address                                    | 3M Facility Contact                                  | 3M Department Number<br><b>0222</b>     |
| City/State/Zip Code                        | 3M Template Number                                   | Lab Request/Template Number<br><b>—</b> |

| Sampled By (Print)<br><b>Brad Jacobson</b> |                      | Contractor<br><b>PACE</b> |      | Number Containers of each |      | Total Containers of Each Item | <input type="checkbox"/> Samples Split See Attachment<br>Comments |      |     |  |  |   |
|--------------------------------------------|----------------------|---------------------------|------|---------------------------|------|-------------------------------|-------------------------------------------------------------------|------|-----|--|--|---|
| Sample Signature                           |                      | Date<br><b>9/29/95</b>    |      | Preservatives             |      |                               |                                                                   |      |     |  |  |   |
| Item #                                     | Sample Description   | Date                      | Time | Matrix                    | None | HNO <sub>3</sub>              | H <sub>2</sub> SO <sub>4</sub>                                    | NaOH | VOC |  |  |   |
| 1                                          | Mixed Liquor 9/29/95 | 9/29/95                   | 7:30 | Sludge                    | 1    |                               |                                                                   |      |     |  |  | 1 |
| 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      | <b>Brad Jacobson / PACE</b> | <b>0953</b> | <b>9/29/95</b> | <b>5-10</b> | <b>Brad Jacobson / 3M</b> | <b>0953</b> | <b>9/29/95</b> |
|        |                             |             |                |             |                           |             |                |
|        |                             |             |                |             |                           |             |                |
|        |                             |             |                |             |                           |             |                |

On... Accompanying Samples

Last Page - Original

See Reverse Side For Instructions