

## BIODEGRADATION (301E)

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 number was not noted. 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 degradation potential of the fluorochemical component of the test sample.***

METHOD

**Method:** Modified OECD Screening Test, OECD 301E, with DOC Analysis, 1981 version.

**Test type:** Ready Biodegradability

**GLP:** No

**Year Completed:** 1982

**Analytical monitoring:** Dissolved organic carbon (DOC)

**Statistical methods:** Results were determined by calculation of the % DOC removal and graphic interpretation.

**Test organism source:** A 50:50 mix of soil extract and secondary effluent. The secondary effluent was the supernatant from an activated sludge aeration basin at the Metro Wastewater Treatment Plant, St. Paul, MN, while the soil was from the City of White Bear Lake, Ramsey County, MN.

**Test condition:**

**Dilution water:** Deionized water

**Mineral Nutrient Medium:** Nutrient medium per OECD 301E method (1981). Initial pH 7.0.

**Reference and test solution preparation:** The test material was prepared by dissolving 153 mg in two liters of mineral nutrient medium. This solution gives a final test concentration of 20 mg DOC/L. The reference substance, sodium benzoate, was prepared by dissolving 30.3 mg in one liter of mineral nutrient medium. This solution gives a final test concentration of 20 mg DOC/L.

**Test vessels:** Not given.

**Incubation conditions:** Constant dark conditions

**Temperature:** 23.5 – 24°C

**Agitation:** Continuously

**Number of concentrations:** 1 plus sodium benzoate (reference substance) and blank, all in duplicate.

**Inoculum condition on test initiation:** Not given .

**Element Basis:** Decrease in dissolved organic carbon compared to the blanks.

**Test substance flask conditions:** Not given.

## RESULTS

**Nominal concentrations:** Blank control, sodium benzoate at ~20 mg DOC/L and at ~20 mg DOC/L plus  $\text{HgCl}_2$  (inhibited), test material at ~20 mg DOC/L and at ~20 mg DOC/L plus  $\text{HgCl}_2$ , all in duplicate.

**Element values:** 27-day Degradation = 76.7% duplicate 1 and 81.3% duplicate 2. Mean value = 79%

**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 FC-203 % degradation based on the mean DOC removal value was 79 after 27 days.

**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 the solution.

## REFERENCES

The studies were conducted by the 3M Company, Environmental Laboratory, St. Paul, MN, Lab Request number 8483, 1982.

## OTHER

**Last changed:** 6/27/00

Internal Correspondence

File FC-203

cc: ✓ D. L. Bacon/R. L. Bohon - 21-2W-05  
✓ R. Gritzky - 3M Neuss  
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✓ J. A. Pignato - 236-2A-01

✓ To: K. F. SCHROEDER - 3M NEUSS, GERMANY

From: E. A. REINER - ENVIRONMENTAL LABORATORY/EE & PC - 21-2W-5

Subject: BIODEGRADATION TESTS

Date: APRIL 21, 1983

**3M**

Envir. Assess. Inq. #82326  
3M Neuss, Germany  
FC-203, FC-600

Attached is a copy of our report on the modified OECD screening tests on FC-600 and FC-203. While the results of this testing does not precisely repeat those which we obtained on FC-203 in 1980, they do confirm that FC-203 is readily biodegradable according to the test method criteria. The present results also show that FC-600 is readily biodegradable.

The reason that these results do not precisely match those of our previous testing is not known, but biodegradation test results are characteristically somewhat variable. This variability is often explained by one or more of the following three factors:

1. Differences in the bacterial species or strains present in the inocula of tests started at different times.
2. Variations in the formulation of a product from one lot to another.
3. The presence of inhibitory materials such as possibly toxic byproducts or inadvertent contaminants.

*Note This data  
not valid because error  
of low N in media*

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K. F. Schroeder  
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The percent biodegradation results given in the attached report are equally applicable to both the concentrated AFFF products, as sold, and the diluted products, as used. Dilution with pure water would have no effect. The reason for this is that testing is actually done on a very dilute portion of the concentrate (20 ppm as TOC). Both the concentrate or the 3 or 6% usage level would be diluted to this same concentration for testing. The dilution factor is not considered when calculating the results.

I have reviewed the table of biodegradation data that you sent us. Some of the variability in results which you show is not unexpected. This is particularly the case with the 5-day BOD test results. Biodegradation of AFFF products is usually only partially completed within the first five days of a BOD test and the percent of biodegradation that has been completed in this time period is highly dependent upon both the viability of the microbial inoculum and the fraction of the organisms in the inoculum which are capable of degrading the organic compounds.

Results of 20-day BOD tests are less variable because biodegradation is usually more nearly complete and proportion of organisms capable of degrading AFFF products has increased through growth. But, 20-day BOD tests are susceptible to another type of problem, "nitrification." In these longer tests, populations of bacteria can develop which can oxidize the ammonia in the BOD dilution water making it appear that the test material is more biodegradable than it actually is. In the Environmental Laboratory we are now taking precautions to avoid this problem by using "nitrification inhibitors," chemicals which inhibit the oxidation of ammonia.

A point of confusion in your table of BOD data is the reporting of different BOD<sub>5</sub> to COD ratios for both the undiluted and diluted AFFF products. This doesn't make sense because BOD and COD would decrease proportionally with dilution with the ratio remaining the same. These differences which you indicated were reported in Hellmann's work must therefore be due to variability between one BOD test run and another and not to dilution.

K. F. Schroeder  
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We hope the attached OECD test results and this explanation of the biodegradation data in the table which you provided are helpful to you in dealing with potential customers concerned about biodegradation. If you or your customers have any further questions about biodegradation test results on 3M AFFF products, feel free to contact us. We'll do our best in trying to get a satisfactory answer for you and your customers.

For our own benefit and at our expense, we are in the process of repeating the OECD screening tests on the FC-600 and FC-203 samples used in this testing. We are interested in seeing how repeatable our testing results are when the same samples are used in two separate tests. We will inform you of the results of this test.

EAR/mb

Attachment: Report on the Biodegradation of FC-600 and  
FC-203 by the OECD Screening Test

000448

# TECHNICAL REPORT SUMMARY

TO: TECHNICAL COMMUNICATIONS CENTER - 201-2C-12

(Important - If report is printed on both sides of paper, send two copies to TCC.) Guidelines on reverse side.

|                                                                                                               |  |                                                                                                                                                     |
|---------------------------------------------------------------------------------------------------------------|--|-----------------------------------------------------------------------------------------------------------------------------------------------------|
| Division<br>3M Environmental Laboratory (EE & PC) St. Paul Minnesota                                          |  | Dept. Number<br>0535                                                                                                                                |
| Project<br>3M Germany                                                                                         |  | Project Number<br>9972410100                                                                                                                        |
| Report Title<br>OECD Biodegradation Testing on FC-600 and FC-203                                              |  | Report Number<br>CO1                                                                                                                                |
| To<br>K. F. Schroeder, 3M Neuss, Germany                                                                      |  | Period Covered or Date<br>4/18/83                                                                                                                   |
| Author(s)<br>E. A. Reiner, Environmental Laboratory - 21-2W-05                                                |  | Employee Number(s)<br>47816                                                                                                                         |
| Notebook Reference<br>None - See 3M Environmental Lab Request 8483                                            |  | No. of Pages Including Coversheet<br>15                                                                                                             |
| SECURITY <input type="checkbox"/> Open Report & Summary <input type="checkbox"/> Closed Report - Open Summary |  | 3M CHEMICAL REGISTRY <input type="checkbox"/> Check box if new chemicals are reported. Use Chemical Registry Form 6092 to report all new substances |

KEYWORDS:  
Lab Code

Other Keywords

## CURRENT OBJECTIVE:

1. To measure the biodegradability of two "Light Water" products using the internationally recognized Modified OECD Screening Test.
2. To compare results with those previously obtained using the same test method.

REPORT ABSTRACT: This abstract information is distributed by the Technical Communications Center to alert 3M's Company R&amp;D.

This study used the Modified OECD Screening Test with dissolved organic carbon (DOC) analysis to measure the biodegradability of "Light Water" Brand AFFF products: FC-203 and FC-600. In 28 days, the DOC levels of FC-600 degraded by 80% and 92% for an average of 86% in two parallel tests. In 27 days the DOC of FC-203 degraded by 77% and 81% for an average of 79%. The FC-203 result differed from a similar test done in 1980 in which 93% DOC degradation occurred within 21 days. In the present study parallel sterile controls proved that the DOC loss was not due to chemical or physical processes such as adsorption, volatilization, or precipitation of the organic compounds.

Information Liaison  
Initials:

000449

### Introduction

The 3M Environmental Laboratory undertook this study of the biodegradability of "Light Water" products, FC-600 and FC-203, because of concern expressed by 3M Germany about inconsistencies in past biodegradation test results.

Modified OECD Screening Tests conducted in 1980 by the 3M Environmental Laboratory showed that three "Light Water" products were highly biodegradable. FC-203A, FC-206, and FC-3017 (FC-206 F) all were shown to have 90% of their dissolved organic carbon removed in the OECD Screening Test. These results, however, contradict those of H. Hellmann and D. Muller<sup>(1)</sup> using a similar yet not identical test method, the TOC Degradation Test<sup>(2)</sup>. In their study FC-206 and FC-3017 were found to have total organic carbon reductions of only 6% and 8%, respectively, in 21 days. These low biodegradation results of Hellmann, however, were also contradicted by his own work. His BOD<sub>5</sub>/COD ratios for FC-3017 and FC-206 were 0.5 and 0.4, respectively. This showed that 50% and 40% of the theoretical oxygen demand was expressed within only 5 days. These levels of biodegradation are much greater than the 8% and 6% TOC removals which he observed in the even longer (21 day) TOC degradation test. This is clearly an inexplicable discrepancy in his work.

Hellmann's TOC degradation results are also contradicted by BOD<sub>20</sub>/COD measurements made by the 3M Environmental Laboratory and other independent laboratories on 3M AFFF products. Such results typically show BOD<sub>20</sub> values to range between 60 and 90% of the COD. For example, a collection of such data on 3 U.S. military specification AFFF products made by 3M is shown in Table 1.

TABLE 1

BOD, CARBONACEOUS BOD, AND COD DATA ON MIL-SPEC APPE PRODUCT

| <u>Lab</u>             | <u>BOD<sub>20</sub></u><br><u>(mg/kg)</u> | <u>C-BOD</u><br><u>(mg/kg)</u> | <u>COD</u><br><u>(mg/kg)</u> | <u>BOD<sub>20</sub></u><br><u>COD</u> | <u>C-BOD<sub>20</sub></u><br><u>COD</u> |
|------------------------|-------------------------------------------|--------------------------------|------------------------------|---------------------------------------|-----------------------------------------|
| <u>FC-203C Lot 501</u> |                                           |                                |                              |                                       |                                         |
| 3M                     | 580,000                                   | 600,000                        | 730,000<br>740,000           | .79                                   | .81                                     |
| Pace                   | 800,000*                                  | 730,000*                       | 810,000<br>730,000           |                                       |                                         |
| Serco                  | 440,000                                   | 440,000                        | 737,000                      | .60                                   | .60                                     |
| Capsule                | 570,000                                   | 620,000                        | 950,000                      | .6                                    | .65                                     |
| Galbraith              | 616,000                                   | 671,000                        | 776,450                      | .79                                   | .86                                     |
| S & W Seed             |                                           |                                |                              |                                       |                                         |
| Galbraith              | 670,000                                   | 641,000                        | 776,450                      | .86                                   | .82                                     |
| Knoxville              |                                           |                                |                              |                                       |                                         |

|         |         |         |         |     |     |
|---------|---------|---------|---------|-----|-----|
| Average | 575,000 | 594,000 | 782,000 | .73 | .75 |
| S. D.   | 85,000  | 90,000  | 80,000  | .12 | .12 |

\* Calculated BOD's from different dilutions were very inconsistent. Pace agreed to repeat this work but inadvertently lost their repeated test results as well. We did not request a third testing nor did we include these values in the averages.

|                        |         |         |                    |      |      |
|------------------------|---------|---------|--------------------|------|------|
| <u>FC-206C Lot 502</u> |         |         |                    |      |      |
| 3M                     | 290,000 | 290,000 | 380,000<br>360,000 | 0.78 | 0.78 |
| Pace                   | 350,000 | 370,000 | 450,000            | 0.78 | 0.82 |
| Serco                  | 240,000 | 260,000 | 370,000            | 0.65 | 0.70 |
| Capsule                | 260,000 | 320,000 | 410,000            | 0.63 | 0.78 |
| Galbraith              | 416,500 | 411,000 | 407,250            | 1.02 | 1.01 |
| S & W Seed             |         |         |                    |      |      |
| Galbraith              | 395,500 | 374,000 | 407,250            | .97  | .92  |
| Knoxville              |         |         |                    |      |      |

|         |         |         |         |     |     |
|---------|---------|---------|---------|-----|-----|
| Average | 325,000 | 338,000 | 396,000 | .81 | .83 |
| S. D.   | 73,000  | 57,00   | 33,000  | .16 | .11 |

|                        |         |         |                    |     |     |
|------------------------|---------|---------|--------------------|-----|-----|
| <u>FC-780B Lot 501</u> |         |         |                    |     |     |
| 3M                     | 240,000 | 230,000 | 320,000<br>330,000 | .74 | .71 |
| Pace                   | 250,000 | 250,000 | 350,000            | .71 | .71 |
| Serco                  | 210,000 | 220,000 | 287,000            | .73 | .77 |
| Capsule                | 240,000 | 230,000 | 360,000            | .67 | .64 |
| Average                | 235,000 | 232,000 | 329,000            | .71 | .71 |
| S. D.                  | 17,000  | 13,000  | 28,000             | .03 | .05 |

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## Methods and Materials

### 1. Procedure

Testing was performed in accordance with the OECD Guideline for testing of chemicals adopted May 12, 1981.

### 2. Chemicals

The reference material used was laboratory grade sodium benzoate ( $C_7H_5NaO_2$ ) F.W. = 144.11. A 30.3 mg sample of this compound was diluted to 1.0 liters with the nutrient solution described by the method to yield a theoretical DOC of 20 mg/l. The "Light Water" samples used were supplied by G. Piessens, E.T.S.L. and consisted of FC-203 and FC-600. Lot numbers were not provided. Nutrient solutions of the "Light Water" products with DOC's of approximately 20 mg/l (based on calculations from the formulation) were prepared by bringing both 527 mg of FC-600 and 153 mg of FC-203 to 2.0 liters with nutrient solution.

### 3. Inoculum

The inoculum was a 50/50 mixture of the soil and secondary effluent inocula each prepared in accordance with the OECD procedure. The soil used was fresh loamy sand garden soil obtained from White Bear Lake, Minnesota, and the water for the soil extraction was chlorine-free well water. The secondary effluent sample was the supernatant from an activated sludge aeration basin at the St. Paul Metro Wastewater Treatment Plant. The combined inoculum was passed through Whatman® 54 filter paper prior to usage.

### 4. Sterile Controls

Replicate vessels containing 400 mg/l  $HgCl_2$  served as sterile controls for both test materials and the reference compound. The sterile controls were handled in the same way as the viable cultures.

### 5. Instrument

The organic carbon analyzer used was a Dohrmann DC-52A. Its sensitivity limit is approximately 2 mg of carbon per liter.

### 6. Sample Handling and Storage

The sample preservation method used was that prescribed by the TOC manufacturer. The method involved adding 1 drop of concentrated HCl per 10 ml of filtered sample. This brought the samples to less than pH 2. The samples were then stored under refrigeration in vials with aluminum foil-lined caps. Dohrmann literature indicates that the method can stabilize potassium hydrogen phthalate, a readily biodegradable material, for several months.

Due to problems with the sensitivity of the TOC analyzer, only half of the samples were analyzed within a month after sampling. Following 4-5 months of storage, all of the samples were reanalyzed. Comparison of the TOC analyses done within 1 month with those done after 4-5 months of storage showed that in only one stored sample had the TOC changed significantly. This sample was discarded.

### Results and Discussion

Table 2 summarizes the results of this study. Appendices 1 and 2 contain the actual data sheets and plots of the degradation as a function of time. Table 2 shows that the sodium benzoate reference compound was nearly completely degraded within 14 days. Degradation of both FC-600 and FC-203 continued throughout the course of the 28-day test period without plateauing. At 28 days the average TOC removal for FC-600 was 86% and at 27 days the average TOC removal for FC-203 was 79%. The test procedure recommends that chemicals giving a result greater than 70% loss of DOC (within 28 days) should be regarded as readily biodegradable. These 3M formulated AFFF products are thus even more completely degraded than is necessary for a chemical to be considered readily biodegradable.

TABLE 2  
Biodegradation (% DOC removal) of Sodium Benzoate,  
FC-203 and FC-206

| <u>Product</u>            | % Degradation at Day: |           |           |           |           |
|---------------------------|-----------------------|-----------|-----------|-----------|-----------|
|                           | <u>7</u>              | <u>14</u> | <u>21</u> | <u>27</u> | <u>28</u> |
| Sodium Benzoate           | 94.4                  | 94.7      | 93.2      | 93.5      | 94.5      |
| Sodium Benzoate Duplicate | 92.3                  | 96.3      | 99.6      | 97.7      | 100.0     |
| FC-203                    | 37.4                  | 59.2      | 68.4      | 76.7      | 78.0      |
| FC-203 Duplicate          | 43.0                  | 54.6      | 66.0      | 81.3      | *         |
| FC-600                    | 46                    | 63.5      | 73.5      | 82.1      | 79.9      |
| FC-600 Duplicate          | 47.2                  | 67.1      | 80.2      | 88.7      | 92.1      |

\*Sample discarded due to probable contamination.

The sterile control data in Table 3 provide further substantial evidence that soluble TOC loss from the test samples is not due to physical modes such as adsorption, volatilization, or precipitation. These samples were handled identically to the test vessels, except that they contained  $\text{HgCl}_2$  to prevent microbial growth. At the end of 28-day test period, these controls contained all of the initial DOC.

TABLE 3

Percent of Initial DOC in Sterile Controls at 28 days

| <u>Product</u> | <u>% DOC Remaining</u> |
|----------------|------------------------|
| FC-203         | 98.1                   |
| FC-600         | 101.3                  |

Conclusion

The present study demonstrates that Light Water products FC-203 and FC-600 are "readily biodegradable" according to the recommended 70% DOC removal criteria of the Modified OECD Screening Test. FC-203 and FC-206 had average losses of DOC of 79% and 86%, respectively.

References

1. Dr. H. Hellmann and D. Muller, "Environmental Impact of Fire Extinguishing Foam Compounds (Umweltgefahr durch Feuerlosch-Schaummittel). The study was carried out by the West German Institute of Hydrology (Bundesanstalt fur Gewasserkunde), April 18, 1979.
2. D. Muller and T. Tittizer, "The TOC Degradation Test - A Process to Test Organic Materials for Complete Biodegradation (Der TOC - Abbautest - ein Verfahren zur Prufung von Organischen Substanzen auf Vollstandigen Biologischen Abbau). Developed at the Bundesanstalt for Gewasserkunde, Koblenz, Z. F. Wasser-und Abwasser-Forschung (1979).
3. Organization for Economic Cooperation and Development, Guidelines For Testing of Chemicals, Section 301 E, Ready Biodegradability: Modified OECD Screening Test, May 12, 1981.
4. Dohrman DC-52A Operating Manual, 4th Ed., 1978.

Appendix 1

5. ANNEX

SAMPLE FORM SHEET FOR THE MODIFIED OECD SCREENING TEST

Exp. no.: LR # 8483  
 Date of start of test: 10-27-82  
 Standard material: SODIUM BENZOATE  
 Theoretical test conc.: 20 mg DOC/l  
 Inoculum: SECONDARY Soil  
 Carbon analyser: DOHRMANN DC-53A

|                                                                                                  |      |       |
|--------------------------------------------------------------------------------------------------|------|-------|
| A: Controls:                                                                                     | TOC* | DOC** |
| Stock solution of the test material<br>(1000 mg/l, dilution<br>.../1000 ml of nutrient solution) |      |       |

B: Carbon determinations:

SODIUM BENZOATE

| Culture medium                                                    | Flask no. | Analyses                                          | Theor. conc. mg/l | DOC - concentrations after x days |      |      |      |      |      |
|-------------------------------------------------------------------|-----------|---------------------------------------------------|-------------------|-----------------------------------|------|------|------|------|------|
|                                                                   |           |                                                   |                   | 0 (C <sub>0</sub> )               | 7    | 14   | 21   | 27   | 28   |
| Mineral nutrient solution with test material and with inoculum    | 1         | a <sub>1</sub>                                    | '                 | 20.3                              | 1.4  | 1.4  | 1.5  | 0.7  | 1.6  |
|                                                                   |           | a <sub>2</sub>                                    | '                 | 21.1                              | 2.2  | 1.4  | 2.0  | 1.1  | 1.5  |
|                                                                   |           | $a_1 = \frac{a_1 + a_2}{2}$<br>(C <sub>t</sub> )  |                   | 20.7                              | 1.8  | 1.4  | 1.75 | 1.8  | 1.55 |
|                                                                   | 2         | b <sub>1</sub>                                    | '                 | 19.7                              | 2.3  | 0.7  | 0.4  | 1.4  | 1.4  |
|                                                                   |           | b <sub>2</sub>                                    | '                 | 20.0                              | 2.0  | 1.4  | 0.6  | 0.5  | 1.5  |
|                                                                   |           | $b_2 = \frac{b_1 + b_2}{2}$<br>(C <sub>t</sub> )  |                   | 19.85                             | 2.15 | 1.05 | 0.5  | 0.95 | 0.45 |
| Mineral nutrient solution without test material but with inoculum | Blank #1  | c <sub>1</sub>                                    | '                 | 1.1                               | 0.3  | 0.8  | 0.7  | 0.1  | -0.1 |
|                                                                   |           | c <sub>2</sub>                                    | '                 | 0.9                               | 1.1  | 0.1  | 0.3  | 0.5  | 0.1  |
|                                                                   |           | $c_3 = \frac{c_1 + c_2}{2}$<br>(C <sub>b1</sub> ) | '                 | 1.0                               | 0.7  | 0.45 | 0.5  | 0.3  | 0.0  |
| Mineral nutrient solution without test material but with inoculum | Blank #2  | c <sub>1</sub>                                    | '                 | 1.3                               | -0.4 | 0.4  | 0.1  | 0.7  | 1.0  |
|                                                                   |           | c <sub>2</sub>                                    | '                 | 0.8                               | 1.8  | 0.1  | 0.6  | 0.8  | 0.8  |
|                                                                   |           | $c_3 = \frac{c_1 + c_2}{2}$<br>(C <sub>b2</sub> ) | '                 | 1.05                              | 0.7  | 0.25 | 0.35 | 0.75 | 0.9  |

\* Disagreement between DOC and TOC values points towards insufficient solubility of the test material.  
 \*\* All DOC values determined after membrane filtration.

|                             | DOC - AFTER X DAYS |     |      |      |      |      |
|-----------------------------|--------------------|-----|------|------|------|------|
|                             | 0                  | 7   | 14   | 21   | 27   | 28   |
| AVERAGE OF BLANKS #1 AND #2 | 1.02               | 0.7 | 0.35 | 0.42 | 0.52 | 0.45 |

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 W.G. Schul  
 10-05-83

"Ready Biodegradability:  
Modified OECD Screening Test"

LR # 8483

C: Evaluation of raw data:

SODIUM BENZOATE

| DOC - concentrations<br>minus<br>blanks | Flesh<br>no. |                                                                        | % DOC removal after x days |      |      |      |       |
|-----------------------------------------|--------------|------------------------------------------------------------------------|----------------------------|------|------|------|-------|
|                                         |              |                                                                        | 7                          | 14   | 21   | 27   | 28    |
| 1                                       |              | $D_1 = \left[ 1 - \frac{m_{1t} - m_{3t}}{m_1 - m_3} \right] \cdot 100$ | 94.4                       | 94.7 | 93.2 | 93.5 | 94.4  |
| 2                                       |              | $D_2 = \left[ 1 - \frac{m_{2t} - m_{3t}}{m_2 - m_3} \right] \cdot 100$ | 92.3                       | 96.3 | 99.6 | 97.7 | 100.0 |
| mean                                    |              | $D = \frac{D_1 + D_2}{2}$<br>for day x                                 | 93                         | 96   | 96   | 96   | 97    |

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W.A. Schick  
4-05-93

000456

5. ANNEX

SAMPLE FORM SHEET FOR THE MODIFIED OECD SCREENING TEST

Exp. no.: LR# 8483  
Date of start of test: 12-27-82  
Standard material: SODIUM BENZOATE  
Theoretical test conc.: mg DOC/l  
Inoculum: SECONDARY EFF. / SOIL  
Carbon analyser: DOHRMANN DC-R2A

A: Controls:

|                                                                                                  | TOC* | DOC** |
|--------------------------------------------------------------------------------------------------|------|-------|
| Stock solution of the test material<br>(1000 mg/l, dilution<br>.../1000 ml of nutrient solution) |      |       |

B: Carbon determinations:

TEST MATERIAL: FC-203

| Culture medium                                                    | Flask no. | Analyses                                          | Theor. conc. mg/l | DOC - concentrations after x days |       |      |      |      |      |
|-------------------------------------------------------------------|-----------|---------------------------------------------------|-------------------|-----------------------------------|-------|------|------|------|------|
|                                                                   |           |                                                   |                   | 0 (C <sub>0</sub> )               | 7     | 14   | 21   | 27   | 28   |
| Mineral nutrient solution with test material and with inoculum    | 1         | a <sub>1</sub>                                    | /                 | 22.5                              | 14.1  | 9.1  | 6.8  | 5.7  | 5.2  |
|                                                                   |           | a <sub>2</sub>                                    | /                 | 22.2                              | 14.0  | 9.0  | 7.5  | 5.3  | 5.1  |
|                                                                   |           | $a_1 = \frac{a_1 + a_2}{2}$<br>(C <sub>t</sub> )  |                   | 22.35                             | 14.05 | 9.05 | 7.15 | 5.5  | 5.15 |
|                                                                   | 2         | b <sub>1</sub>                                    | /                 | 23.0                              | 13.8  | 9.5  | 8.6  | 4.4  | 15.0 |
|                                                                   |           | b <sub>2</sub>                                    | /                 | 23.3                              | 12.8  | 11.3 | 7.3  | 4.9  | 15.4 |
|                                                                   |           | $b_2 = \frac{b_1 + b_2}{2}$<br>(C <sub>t</sub> )  |                   | 23.15                             | 13.3  | 10.4 | 7.95 | 4.65 | 15.2 |
| Mineral nutrient solution without test material but with inoculum | #1        | c <sub>1</sub>                                    | /                 |                                   |       |      |      |      |      |
|                                                                   |           | c <sub>2</sub>                                    | /                 |                                   |       |      |      |      |      |
|                                                                   |           | $c_3 = \frac{c_1 + c_2}{2}$<br>(C <sub>b1</sub> ) | /                 |                                   |       |      |      |      |      |
| Mineral nutrient solution without test material but with inoculum | #2        | c <sub>1</sub>                                    | /                 |                                   |       |      |      |      |      |
|                                                                   |           | c <sub>2</sub>                                    | /                 |                                   |       |      |      |      |      |
|                                                                   |           | $c_3 = \frac{c_1 + c_2}{2}$<br>(C <sub>b1</sub> ) | /                 |                                   |       |      |      |      |      |

- \* Disagreement between DOC and TOC values points towards insufficient solubility of the test material.  
\*\* All DOC values determined after membrane filtration.

SEE SODIUM BENZOATE PAGE FOR BLANK DATA,

BEST COPY AVAILABLE

000457 Wade A.S.  
4-05

# "Ready Biodegradability: Modified OECD Screening Test"

LR # 8483

C: Evaluation of raw data:

FC-203

| DOC - concentrations<br>minus<br>blanks | Fisk<br>no. |                                                                        | % DOC removal after x days |      |      |      |                   |
|-----------------------------------------|-------------|------------------------------------------------------------------------|----------------------------|------|------|------|-------------------|
|                                         |             |                                                                        | 7                          | 14   | 21   | 27   | 28                |
|                                         | 1           | $D_1 = \left[ 1 - \frac{m_{1t} - m_{3t}}{m_1 - m_3} \right] \cdot 100$ | 37.4                       | 59.2 | 68.4 | 76.7 | 78.0              |
|                                         | 2           | $D_2 = \left[ 1 - \frac{m_{2t} - m_{3t}}{m_2 - m_3} \right] \cdot 100$ | 43.0                       | 54.6 | 66.0 | 81.3 | *                 |
|                                         | mean        | $D = \frac{D_1 + D_2}{2}$<br>for day x                                 | 40                         | 57   | 67   | 79   | 78 <sup>(1)</sup> |

\* NO DATA - RESULTS APPEAR TO SHOW SAMPLE CONTAMINATION  
PRIOR TO DOC ANALYSES

(1) VALUE BASED ON ONE TEST SOLUTION RESULT.

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000458

W.A. Schmidt  
4-05-83

## S. ANNEX

## SAMPLE FORM SHEET FOR THE MODIFIED OECD SCREENING TEST

Exp. no.: LR # 8483  
 Date of start of test: 10-27-82  
 Standard material: SODIUM BENZOATE  
 Theoretical test conc.: mg DOC/l  
 Inoculum: SECONDARY EFF. / SOIL  
 Carbon analyser: DC-52A (DORMANN)

## A: Controls:

|                                                                                                   | TOC* | DOC** |
|---------------------------------------------------------------------------------------------------|------|-------|
| Stock solution of the test material<br>(1000 mg/l, dilution<br>.../1000 ml) of nutrient solution) |      |       |

## B: Carbon determinations:

TEST MATERIAL: FC-600

| Culture medium                                                    | Flesh no. | Analyses                                          | Theor. conc. mg/l | DOC - concentrations after x days mg/l | 0 (C <sub>0</sub> ) | 7    | 14   | 21   | 27   | 28 |
|-------------------------------------------------------------------|-----------|---------------------------------------------------|-------------------|----------------------------------------|---------------------|------|------|------|------|----|
| Mineral nutrient solution with test material and with inoculum    | 1         | a <sub>1</sub>                                    | /                 | 23.8                                   | 13.5                | 8.6  | 6.2  | 4.3  | 4.9  |    |
|                                                                   |           | a <sub>2</sub>                                    | /                 | 24.5                                   | 13.0                | 9.0  | 6.9  | 5.0  | 5.3  |    |
|                                                                   |           | $m_1 = \frac{a_1 + a_2}{2}$<br>(C <sub>t</sub> )  |                   | 24.15                                  | 13.25               | 8.8  | 6.55 | 4.65 | 5.1  |    |
|                                                                   | 2         | b <sub>1</sub>                                    | /                 | 24.0                                   | 12.4                | 7.9  | 4.6  | 3.3  | 2.4  |    |
|                                                                   |           | b <sub>2</sub>                                    | /                 | 23.7                                   | 13.1                | 7.8  | 5.3  | 2.9  | 2.1  |    |
|                                                                   |           | $m_2 = \frac{b_1 + b_2}{2}$<br>(C <sub>t</sub> )  |                   | 23.85                                  | 12.75               | 7.85 | 4.95 | 3.1  | 2.25 |    |
| Mineral nutrient solution without test material but with inoculum | Blank #1  | c <sub>1</sub>                                    | /                 |                                        |                     |      |      |      |      |    |
|                                                                   |           | c <sub>2</sub>                                    | /                 |                                        |                     |      |      |      |      |    |
|                                                                   |           | $m_3 = \frac{c_1 + c_2}{2}$<br>(C <sub>b1</sub> ) | /                 |                                        |                     |      |      |      |      |    |
| Mineral nutrient solution without test material but with inoculum | Blank #2  | c <sub>1</sub>                                    | /                 |                                        |                     |      |      |      |      |    |
|                                                                   |           | c <sub>2</sub>                                    | /                 |                                        |                     |      |      |      |      |    |
|                                                                   |           | $m_3 = \frac{c_1 + c_2}{2}$<br>(C <sub>b1</sub> ) | /                 |                                        |                     |      |      |      |      |    |

\* Disagreement between DOC and TOC values points towards insufficient solubility of the test material.

\*\* All DOC values determined after membrane filtration.

SEE SODIUM BENZOATE PAGE FOR BLANK DATA.

000459

W.A. Se  
4-0

"Ready Biodegradability:  
Modified OECD Screening Test"

LR# 8483

C: Evaluation of raw data:

FC-600

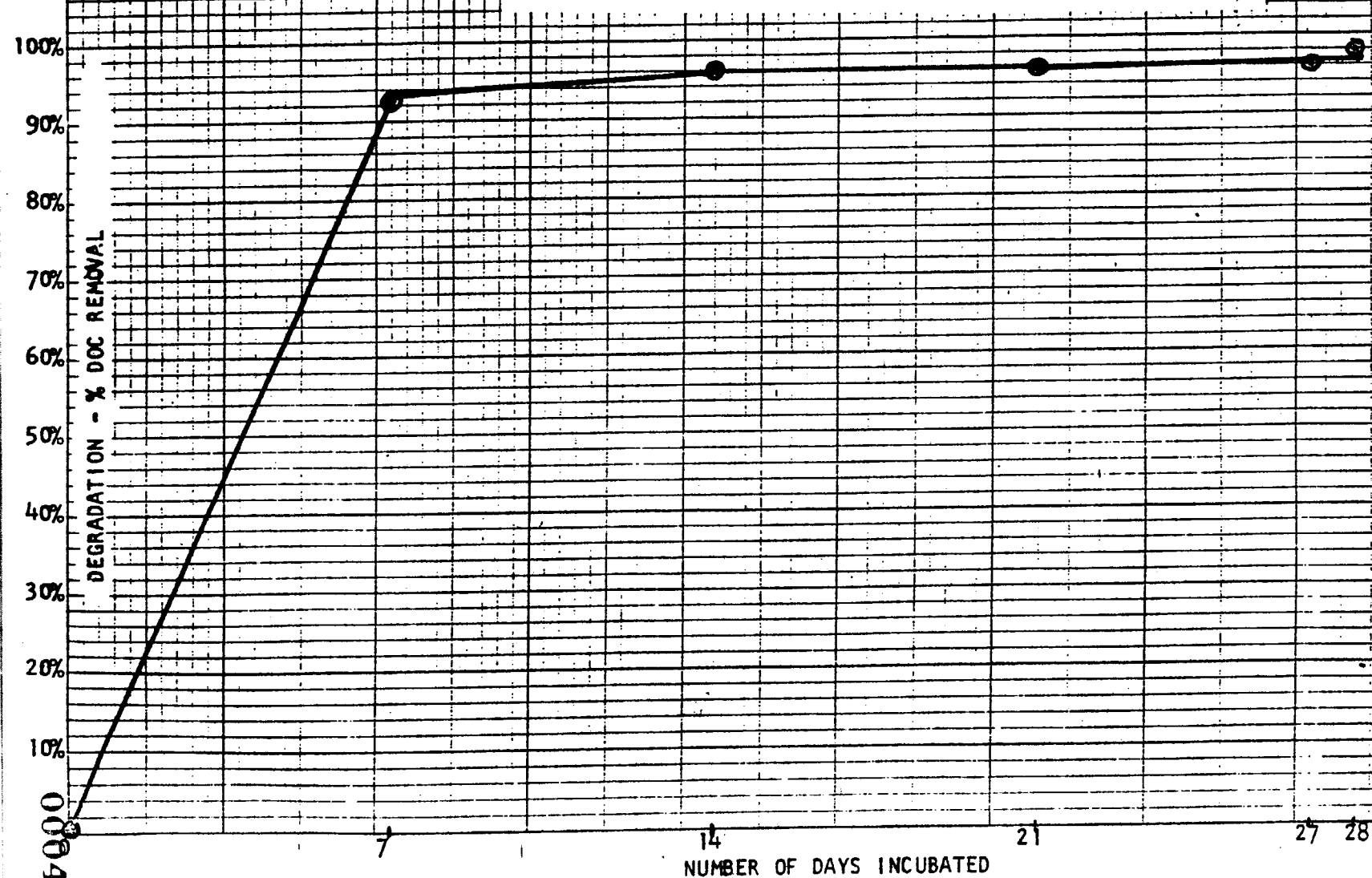
| DOC - concentrations<br>minus<br>blanks | Flesh<br>no. |                                                                        | DOC removal after x days |      |      |      |      |
|-----------------------------------------|--------------|------------------------------------------------------------------------|--------------------------|------|------|------|------|
|                                         |              |                                                                        | 7                        | 14   | 21   | 27   | 28   |
|                                         | 1            | $D_1 = \left[ 1 - \frac{m_{1t} - m_{3t}}{m_1 - m_3} \right] \cdot 100$ | 46.0                     | 63.5 | 73.5 | 82.1 | 79.9 |
|                                         | 2            | $D_2 = \left[ 1 - \frac{m_{2t} - m_{3t}}{m_2 - m_3} \right] \cdot 100$ | 47.2                     | 67.1 | 80.2 | 88.7 | 92.1 |
|                                         | mean         | $D = \frac{D_1 + D_2}{2}$<br>for day x                                 | 47-65                    | 77   | 85   | 86   |      |

000460

W.A.S.  
4-05-

3M Environmental Laboratory  
OECD Modified Screening Test with DOC Analysis  
Analyst: Wade A. Scheil  
Date of Start of Test: 10-27-82  
Lab Request No.: 8483

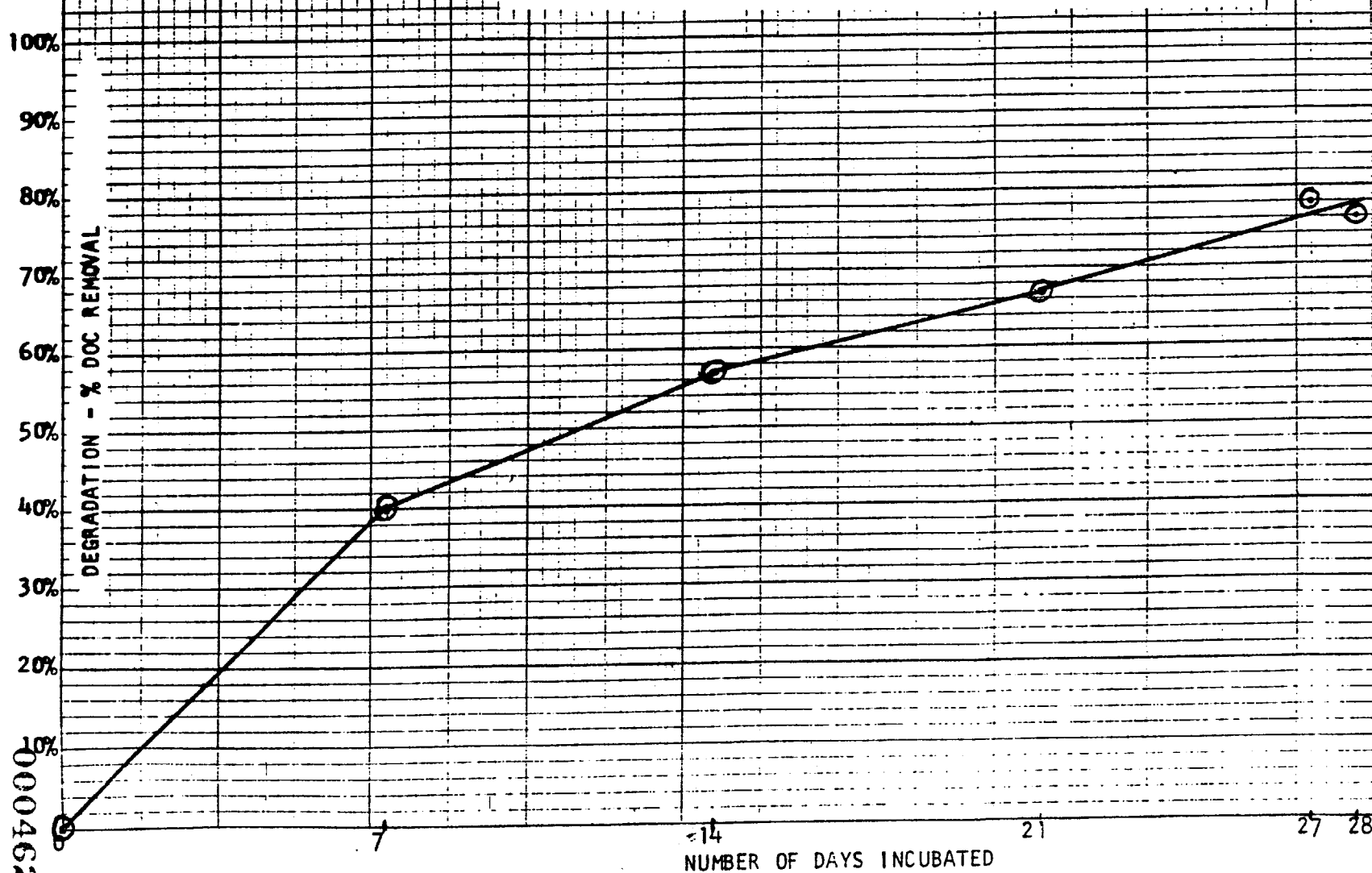
Test Material: SODIUM BENZOATE  
(REFERENCE COMPOUND)



W.A. Scheil  
4/05/83

3M Environmental Laboratory  
OECD Modified Screening Test with DOC Analysis  
Analyst: Wade A. Scheil  
Date of Start of Test: 10-27-82  
Lab Request No.: 8483

Test Material: FL-203 (ANTWERP)



W.A. Scheil  
10-05-83

3M Environmental Laboratory  
OECD Modified Screening Test with DOC Analysis  
Analyst: Wade A. Schell  
Date of Start of Test: 10-27-82  
Lab Request No.: 8483

Test Material: FC-600 (ANTWERP)

