BIODEGRADATION (OECD 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 toxicity of the fluorochemical component of the test sample.

METHOD

Method: Modified OECD Screening Test, OECD 301E, with DOC Analysis
Test type: Ready Biodegradability.
GLP: No
Year Completed: 1983
Analytical monitoring: Dissolved organic carbon (DOC)
Statistical methods: Results were determined by calculation of the % DOC removal.
Test organism source: A 50:50 mix of soil extract and secondary effluent. The secondary effluent was from the Metro Wastewater Treatment Plant, St. Paul, MN while the soil was from the town of White Bear Lake, Ramsey County, MN.

Test condition:

Dilution water: Not given.
Reference and test solution preparation: Solutions of the test and reference material, sodium benzoate, were prepared on a weight/volume basis with nutrient solution.
Test vessels: Not given.
Number of concentrations: 1 plus sodium benzoate standard and blank, all in duplicate.
Temperature: Not given.
Total suspended solids and pH on day of testing: Not given.
Element Basis: Decrease in dissolved organic carbon compared to the blanks.

RESULTS

Nominal concentrations: Blank control, sodium benzoate standard, and test material at ~ 40 mg DOC/L and test material inhibited.

Element values: 28-day degradation = 66% mean DOC removal

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

FC-203 degradation based on the mean DOC removal value was 66% after 28 days.

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

DATA QUALITY

Reliability: Klimisch ranking = 3. Study lacks description and records of the method followed. The inoculum is not properly characterized. 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, Lab Request number 9191, 1983.

OTHER

Last changed: 6/28/00

Form 6747-11-E

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: Environmental Laboratory (EE & PC), St. Paul, MN	Dept. Number: 0535
Project: New Method Development	Project Number: 9970030000
Report Title: Repeat OECD Biodegradation Test on FC-600 and FC-203	Report Number:
To: R. L. Bohon, Environmental Laboratory/EE & PC, 21-2W-05	Period Covered or Date: 6/16/83
Author(s): E. A. Reiner, Environmental Laboratory/EE & PC, 21-2W-05	Employee Number(s): 47816
Notebook Reference: None - See Environmental Lab Request #9191	No. of Pages Including Coversheet:

SECURITY ▶ ☒ Open Report & Summary ☐ Closed Report–Open Summary

3M CHEMICAL REGISTRY ▶ ☐ Check box if new chemicals are reported. Use Chemical Registry Form 6092 to report all new substances.

KEYWORDS:
Lab Code

Other Keywords

CURRENT OBJECTIVE:

To test repeatability of modified OECD Screening Tests in measuring the biodegradabiity of "LIGHT WATER" products.

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

This study repeated the modified OECD Screening Tests on Antwerp "LIGHT WATER" products FC-203 and FC-600. The same product lots were used as had been used in the previous test (see Lab Request #8483). The results of this testing showed poor repeatabiity both between the two sets of tests and between the duplicates in the current test. The report discusses possible causes for the variability and suggest testing to determine ways of reducing variability in the future.

~~Note FC-203 TFP~~

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Information Liaison Initials: _____

INTRODUCTION

The Environmental Lab has conducted two previous modified OECD Screening Tests to determine the biodegradability of "LIGHT WATER" products (1,2). This testing was conducted in accordance with the guidelines current at the time of testing (3,4). The differences between the 1979 and 1981 modified OECD Screening Test protocols were of a type that they would not have significantly affected the extent of biodegradation.

The first set of testing, that was conducted under LR #5441, showed nearly complete DOC removal ($\geq$ 92%) for all "LIGHT WATER" products tested. The results also showed little variability between the duplicates done on each "LIGHT WATER" product (<2%). LR #8483 was undertaken at the request of K. F. Schroeder of 3M Germany in order to confirm the previous biodegradation test results. He made this request because a large amount of variability existed among the biodegradation test results on "LIGHT WATER" products. This second test showed less complete dedgradation of "LIGHT WATER" products. Due to difficulties with the analytical equiment, from samples from the LR8483 degradation study were analyzed four to 5 months after they were taken). The samples from this study, when eventually analyzed, gave very consistent results and results which duplicated those done prior to the onset of difficulties with the DOC analyzer.

Nevertheless, it was decided to repeat the study because of concerns about the reliability of the results after long-term sample storage and because the results showed biodegradation levels less complete than that previously found.

This report describes the results of a third set of modified OECD Screening Tests on "LIGHT WATER" products. It is an attempt to repeat the findings of LR #8483 and is done using the same "LIGHT WATER" samples and the same reference compound.

METHODS AND MATERIALS

The methods and materials used in this screening study were identical to those used in the previous study on FC-600 and FC-203 (2), except that the initial DOC of the "LIGHT WATER" products was initially adjusted to 40 mg/l instead of 20 mg/l. This was done because the first OECD test on "LIGHT WATER" biodegradation (1) showed that 40 mg/l "LIGHT WATER" concentrations were not toxic, and because it was felt that a higher "LIGHT WATER" concentration would improve when proved the sensitivity of the TOC analysis.

Inocula for the study which was started on 3/30/83, which consisted of garden soil from White Bear Lake, MN, and secondary effluent from the St. Paul Metro Wastewater Treatment Plant were collected on 3/30/83. All TOC analyses on samples from the study were done within a week of completion of the study.

RESULTS AND DISCUSSIONS

Table 1 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. Degradation of the reference compound, sodium benzoate, was nearly complete within 7 days. FC-203 degradation continued gradually to Day 27 but remained thereafter plateaued at about 65% throughout a 14-day extension of the test. FC-600 reached an average degradation level of 73% during the 28-day test period, and degradation slowly proceeded to the 14-day extension of the test to 85%

The OECD test procedure stipulates that chemicals should reach 70% DOC removal during the 28-day test period to be considered readily biodegradable. In this set of tests, the average result for FC-600 indicated that it just bearly reached the stipulated level of degradation. The average FC-203 DOC removal did not reach the required 70% level in either the 28-day standard test period or the 42-day extended test period. However, one of the FC-203 duplicates did just exceed this required level of degradation.

The modified OECD Screening Test procedure also stipulates the chemicals considered to be readily biodegradable should complete 70% DOC removal within 10 days of reaching 10% DOC removal. This stipulation was not met by either of the products. However, this recommendation of the test procedure appears to be intended for pure chemicals and does not make sense when applied to a formulated product such as "LIGHT WATER" since some components may be readily degradable thus allowing a 10% DOC removal quite early in the test period, while other components may be more resistant to degradation, and no degradation may have occurred to

these components during the time that 10% of the DOC total product was removed

The _____ controlled data in Table 3 demonstrates the soluble loss from the test samples is not due to physical modes such adsorption, volatilization, or precipitation. These samples were handled identically to the test vessels, except that they contained HgCl to prevent microbial growth. The table shows that at the end of the 28-day test period, these controls contained all of the initial DOC.

Tables 3 and 4 summarize the results of the three OECD screening tests on "LIGHT WATER" products. The first test on "LIGHT WATER products on Lab Request #5543 showed that 3 "LIGHT WATER" products were very degradable. However, these tests were run on samples of European made "LIGHT WATER" products. However, more recent testing on the LR #8483, as well as the data presented in this report, come under LR #9191, showed that "LIGHT WATER" products provided in 1982 were not as degradable. Table 4 shows that the differences between the duplicates in the OECD screening tests also increased with the latter testing. The reasons for this decreased degradation and increased variability are not known. Contamination of the samples during testing is not the likely cause since great care was taken in washing the glass. Glasses were washed first with soap and then were acid washed with a strong acid dichromate solution and then were triply rinsed with tap and deionized water. Analytical errors are also not a likely cause of the lower levels of degradation observed or the increased variability between duplicates. This can be stated

because TOC measurements were very precise. Of 50 duplicate TOC measurements made during the test, the main difference was only 0.38 ppm or dissolved organic carbon with the standard deviation of 0.33 ppm. In a 20 ppm test such as that done with the reference compound, this would only account for a 1.9% error plus or minus 3.3% with a 95% confidence level. In a 40 ppm test such as that done with the two "LIGHT WATER" products, this variability could only have accounted for a 1% error with a 95% confidence interval of $\pm$1.7%. Also calculated and measured DOC values for the reference compound were identical

Some possible causes for the variability could be the differences between the lots of "LIGHT WATER" products tested. The new lot may contain some toxic components which inhibit degradation or it could contain chemicals components, which are more resistant to degradation than those tested in 1980. A second possibility is that the extract, which was from the same bottle of material used in the 1980 study, may have lost some of its necessary cofactors through age. A third possibility is that the inocula, although taken from the sources, may have had lower levels of viable microorganisms or may have not included organisms with capabilities necessary for rapidly degrading the "LIGHT WATER" products

Possible ways in which these reasons for reduced biodegradation could be tested would be to run parallel tests using not only these samples which we have found to give low levels of degradation but also samples of lots which are known to be within specifications for "LIGHT WATER" products, as well as the FC-203

LIGHT WATER" sample used in the 1980 test, which showed high levels of degradation. Another way to test the results would be to run the screening test, both with the vitamin solution and with the yeast extract. The extract could be used both at the levels called for by the procedure and at higher levels which could provide larger quantities of cofactors during the "LIGHT WATER" degradation process. In addition, this parallel study using an inoculum from an alternate waste treatment and soil source could be run to determine if this has any effect on degradability. Hopefully, such a series of tests would enable us to find the causes of low degradability in the higher levels of variability found in the current testing and testing done under LR #8483.

CONCLUSIONS

Present study did not repeat the findings from previous OECD screening tests on "LIGHT WATER" products which show these products to be "readily biodegradable." In the present study, FC-203 was found to be slightly below the guideline level of 70% DOC removal required for a material to be considered readily biodegradable, while FC-600 showed an average level of DOC removal just slightly above this required level. Possible reasons for this variability in test results and in ways of determining the cause of this variability are discussed.

REFERENCES

1. E. A. Reiner, Biodegradation of "LIGHT WATER" Products in OECD Test 6/80, 3M Technical Report No. 40, Dept. 0535 Project 9970012600, 6/30/80.

2. E. A. Reiner, OECD Biodegradation Testing on FC-600 and FC-203, 3M Technical Report No. 001, Dept. 0535, Project 9972410100, 4/18/83.

3. Modified OECD Screening Test with DOC Analysis, OECD-Chemicals Testing Programme Expert Group C, Degradational Accumulation, Tokyo, December 1, 1979.

4. Ready Biodegradability: Modified OECD Screening Test, 301E, OECD Guideline for Testing of Chemicals Adopted, May 12, 1981.

TABLE 1

BIODEGRADATION (% DOC REMOVAL) OF SODIUM BENZOATE FC-203, AND FC-206

	% Degradation at Day:					
Product	7	14	21	27	28	42
Sodium Benzoate #1	93.8	92.4	94.8	-	-	-
Sodium Benzoate #2	94.5	94.5	99.8	-	-	-
FC-203 #1	31.9	46.4	59.5	70.4	71.7	73.8
FC-203 #2	31.4	42.5	56.3	61.1	60.3	59.5
FC-600 #1	38.6	54.0	67.5	74.8	78.4	87.8
FC-600 #2	32.8	42.1	56.3	66.9	67.5	82.0

TABLE 2

PERCENT OF INITIAL DOC IN STERILE CONTROLS AT DAY 28

Product	% DOC Remaining
FC-203	100
FC-600	101

TABLE 3

COMPARISON OF PERCENT DOC REMOVAL[a]
IN OECD SCREENING TESTS ON "LIGHT WATER" PRODUCTS

Product	LR 5541	LR 8483	LR 9191
FC-203	93%	79%[b]	66%
FC-206	92%	-	-
FC-600	-	86%	73%
FC-3017	95%	-	-
Sodium Benzoate	-	97%	97%[c]
Hydroquinone	93%	-	-

(a) Percent DOC removal was measured at 28 days.

(b) Determined at 27 days.

(c) Determined at 21 days.

TABLE 4

DIFFERENCE[a] BETWEEN DUPLICATES IN THE THREE MODIFIED OECD SCREENING TESTS ON "LIGHT WATER" PRODUCTS

Product	LR[b]5541	LR 8483	LR 9191
FC-203	0	4.6[c]	11.4
FC-206	0.9	-	-
FC-600	-	12.2	10.9
FC-3017	1.6	-	-
Sodium Benzoate	-	5.6	5[d]
Hydroquinone	3.7	-	-

(a) Difference is expressed as % of original DOC at 28 days.

(b) LR 5541 is the Environmental Laboratory Request Number of the biodegradation test.

(c) Difference at 27 days.

(d) Difference at 21 days.

References

(8)

1) E. A. Reiner, Biodegradation of "Light Water" Products in OECD Test 6/80, 3M Technical Report No 40, Dept 0535, project 9970012600, 6/30/80

2) E. A. Reiner, OECD Biodegradation Testing on FC-600 and FC-203, 3M Technical Report No 001, Dept 0535, project 9972410100, 4/18/83

3) Modified OECD Screening Test w.r.t DOC analysis, OECD-Chemicals Testing Programme Expert Groups, Degradation/Accumulation, Tokyo, December 1, 1979

4) Ready Biodegradability: Modified OECD Screening Test, 301 E, OECD Guidline for Testing of Chemical Adopted May 11, 12, 1981.