

BIODEGRADATION (MANOMETRIC RESPIROMETRY – AEROBIC)

TEST SUBSTANCE

Identity: A mixture containing perfluorooctanesulfonate, which may also be referred to as PFOS, FC-95, or as a component of FC-3041. (1-Octanesulfonic acid) (CAS # 2795-39-3).

Remarks: The 3M production lot number was not noted. The test sample is FC-3041. Current information indicates it is a mixture of 6% PFOS, 36% diethylene glycol butyl ether, 36.05% water, 12% Sultone foamer, 9% sodium octyl sulfate, 0.55% sodium lauryl sulfate, and 0.4% tolyltriazole.

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

METHOD

Method: Manometric Respirometry Test

Test type: Ready biodegradability

GLP: No

Year Completed: 1993

Analytical monitoring: Oxygen consumption determined by measurement of the amount of oxygen generated electrolytically to maintain a constant gas volume in the respiration flask. Depletion of dissolved oxygen was registered automatically. Amount of oxygen taken up is given (after comparison with the blank inoculum) as a percentage of the chemical oxygen demand.

Statistical methods: Results were determined by calculation of the percentage biodegradation.

Inoculum source: Activated sludge collected from a community sewage plant (Schmallenberg, Germany) that was fed almost exclusively with residential wastewater.

Test condition:

Dilution water: Not noted.

Mineral nutrient medium: Mineral Basal Medium

Stock and test solution preparation: Solutions of the test and reference materials were prepared at 100 mg/L with mineral basal medium.

Test vessels: Closed flasks kept at a constant temperature. Each vessel is connected gastight to the pressure regulator corresponding to it.

Incubation Conditions: Per manometric method

Temperature: 22 ± 2 °C

Agitation: Continuous stirring

Number of concentrations: Test sample, test sample inhibited, test sample plus aniline, aniline (reference substance) and blank inoculum, all in duplicate.

Inoculum condition on test initiation: Initial solids 3 g/L, 30 mg solid matter/L.

Element Basis: Biological degradation by means of oxygen consumption, mg O₂/L.

Test substance flask conditions:

pH: Not noted.

D.O.: Automatically measured.

RESULTS

Nominal concentrations: Blank control, test material at 100 mg/L, and aniline (reference substance) at 100 mg/L.

Element values: 14-day Degradation with Test Material = 93.8% based on average of two replicates minus the blank.
14-day Degradation with Test Material plus Aniline = 88% based on average of two replicates minus the blank.
14-day Degradation with Test Material Inhibited = 8.5% based on average of two replicates minus the blank.

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

CONCLUSIONS

The test material % degradation based on the mean of two replicates minus the oxygen consumption of the blank control was 93.8% after 14 days.

The reference substance obtained 82% degradation by 14 days, thus the validation criterion of the test was fulfilled (>60% after 14 days). The test was terminated on day 14 since the validity criterion of the test was fulfilled and the oxygen uptake reached a plateau.

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

DATA QUALITY

Reliability: Klimisch ranking 3. Lacks specific methodology used and documentation. 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 Fraunhofer Institute for Environmental Chemistry and Ecotoxicology, D-57377 Schmallenberg, Germany at the request of the

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OTHER

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FINAL REPORT

**ECO-TOXICOLOGICAL EFFECT OF
FLUORO-SURFACTANTS**

Grant No.:

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Client:

**Bundesamt für Werktechnik und Beschaffung, Koblenz
(Federal Bureau for Material Technology and Acquisition)**

Investigating Institute:

**Fraunhofer Institute
for Environmental Chemistry and Eco-toxicology
D-57377 Schmallenberg**

Institute Manager:

Prof. Dr. W. Klein

Project leader:

Dr. R. Debus

Project authors:

Dr. A. Fliedner

Dr. K. Hund

Dipl. Biol. R. Niemann

Dr. A. Remde

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**Fraunhofer Institute
for Environmental Chemistry and Eco-toxicology
D-57377 Schmallenberg**

**Investigation for
the Eco-toxicological Evaluation of the Fluoro-surfactant Base FC-3041**

Aerobic Degradation

Part f

by

Dr. A. Remde

AEROBIC DEGRADATION

The determination of the easy biological degradability of Base FC-3041 under aerobic conditions was done in the manometric-respirometry test (8).

MATERIAL AND METHODS

Test substance:

Base FC-3041 (combination of fluoro-surfactants in aqueous solution;
Manufacturer: 3M Company Germany, Neuss).

Test criterion:

Measurement of the biological degradability by means of the oxygen consumption.

Test method:

A defined volume of an inoculated mineral medium with a known concentration of the test substance (100 mg/L) is stirred in a closed flask at a constant temperature ($22\text{ }^{\circ}\text{C} \pm 2$). The oxygen consumption is determined through the measurement of the amount of oxygen generated electrolytically that is necessary to maintain a constant gas volume in the respiration flask. The generated carbon dioxide is absorbed into a solution of potassium hydroxide. The amount of oxygen taken up by the test substance is given (after considering the corresponding value in the inoculum blank test performed in parallel) as percentage of the CSB¹ (chemical oxygen demand).

Test procedure:

10 test vessels were prepared for the performance of the test:

Vessel 1 and 2: mineral basal medium + inoculum

Vessel 3 and 4: mineral basal medium + reference substance (100 mg/L)
+ inoculum (30 mg solid matter/L)

¹in English: COD. I will preserve the German abbreviations since they are present in formulas and tables.

- Vessel 5 and 6: mineral basal medium + test substance (100 mg/L)
+ inoculum (30 mg solid matter/L)
- Vessel 7 and 8: mineral basal medium + reference substance (100 mg/L)
+ test substance (100 mg/L)+ inoculum (30 mg solid matter/L)
- Vessel 9 and 10: mineral basal medium + test substance (100 mg/L)
+ inoculum (30 mg solid matter/L) + mercury chloride
solution (0.001% final concentration)

The vessels were closed and placed into the respirometer. After temperature and pressure compensation (approx. 0.5 hours), each vessel was connected gastight to the pressure regulator belonging to it. During the test period the depletion of dissolved oxygen was registered automatically.

The bio-degradation was determined according to the following equation:

$$\text{Percentage bio-degradation} = \frac{(\text{BSB} - \text{B})}{\text{CSB or ThSB}} \times 100$$

BSB = biochemical oxygen consumption of the test substance (mg)

B = depletion of dissolved oxygen of the basal medium with inoculum (mg)

CSB = chemical oxygen consumption² (mg)

ThSB = theoretical oxygen demand (mg)

Inoculum:

Active sludge from a community sewage treatment plant (Schmallenberg) which was fed almost exclusively with residential waste water, was used as inoculum. The active sludge was aerated during the transport. The inoculum was adjusted with isotonic Ringer's solution to a solids content of 3 g/L and aerated. The sample collection occurred on 10/28/1993.

²in the text above, CSB was *chemischer Sauerstoffbedarf*, which translates to *chemical oxygen demand*. In this case the same abbreviation CSB is used for *chemischer Sauerstoffverbrauch* which translates to *chemical oxygen consumption*.

Analysis:

To determine the dissolved oxygen depletion, a sapromate of the Voith Company (Heidenheim) was used. The oxygen consumption was automatically registered as BSB/L.

Reference substance: aniline (Merck, reagent grade)

Test period: 10/29/1993 - 11/12/1993

RESULTS

The results are represented in Tab. f1 and Fig. f1.

During the incubation period of 14 days, 82% of the reference substance aniline was degraded. The validity criteria of the test (degradation of aniline >40% after 7 days and >60% after 14 days) were fulfilled.

93.8% of the test substance Base FC-3041 was degraded in the same period of time. Since already 88.6% of the substance had degraded within 10 days counted from the day the degradation for the first time surpassed 10%, the substance may be categorized as easily biologically degradable.

The test could be terminated on day 14 since all validity criteria of the test had been fulfilled and a plateau was reached for the oxygen uptake.

EVALUATION

Based on the present results, the test substance Base FC-3041 was categorized as easily biologically degradable.

Table f1: BSB and % of biological degradation of the test substance FC-3041 and of the reference substance aniline

Start of the test: 10/29/1993
 Test substance (TS): Base FC-3041
 Test concentration: 100 mg/L
 Chemical oxygen consumption (CSB): 0.88 mg/mg substance
 Purity: -
 Reference substance (RS): aniline
 Test concentration: 100 mg/L
 Theoretical oxygen consumption (ThSB; NH₃): 2.41 mg/mg substance
 Inoculum active sludge

Day ³	Inoculum	Reference substance		Test substance		RS + TS		TS + HgCl ₂
	Σ BSB 1) mg O ₂ /l	Σ BSB 2) mg O ₂ /l	% degradation	Σ BSB 3) mg O ₂ /l	% degradation	Σ BSB 3) mg O ₂ /l	% degradation	Σ BSB 4) mg O ₂ /l
1	9,5	0,0	0,0	10,5	11,9	10,0	3,0	2,0
2	14,0	0,0	0,0	17,0	19,3	14,5	4,4	3,0
3	18,0	1,0	0,4	22,5	25,6	33,0	10,0	3,0
4	20,5	14,8	6,0	34,0	38,6	172,0	52,3	4,0
5	22,5	57,0	23,7	53,0	60,2	238,0	72,3	5,0
6	24,5	114,5	47,5	66,5	75,6	256,5	78,0	5,0
7	26,0	165,5	68,7	71,0	80,7	266,8	81,0	5,0
8	28,0	175,5	72,8	74,0	84,1	272,0	82,7	6,0
9	29,5	184,0	76,3	76,5	86,9	276,5	84,0	6,0
10	30,5	188,5	78,2	78,0	88,6	281,0	85,4	7,0
11	32,0	191,5	79,5	79,5	90,3	284,0	86,3	7,0
12	32,5	194,5	80,7	81,0	92,0	287,0	87,2	7,0
13	34,5	196,0	81,3	82,5	93,8	289,5	88,0	8,0
14	34,5	197,5	82,0	82,5	93,8	289,5	88,0	8,5

1) Inoculum control

2) Average value from two experimental runs, minus the oxygen consumption by the inoculum control

3) Average value from two experimental runs, minus the oxygen consumption by the inoculum control

4) Test substance with inoculum and HgCl₂ solution, average value from two experimental runs

³In the original only g is visible. By analogy to table g1 on page g/6, the heading of this column must be Tag which translates as day.

Fig. f1: Biological degradation of the test substance BASE FC-3041 and of the reference substance aniline in the manometric-respirometry test

