

BIODEGRADATION (METHYLENE BLUE ACTIVE SUBSTANCE)

TEST SUBSTANCE

Identity: Perfluorooctanesulfonylamido(ethyl)acetate; may also be referred to as PFOSAA, FC-128 or as the major component of FC-127, FC-129, FC-109, FC-109-X or R1904. (Glycine, N-ethyl-N-[(heptadecafluorooctyl)sulfonyl]-, potassium salt, CAS # 2991-51-7)

Remarks: The test sample is FC-129 referred to by the test laboratory as sample G1208-1 and replicate analysis as sample G1208-4. Current information indicates it is a mixture of 42% test substance, 32% water, 14% 2-butoxyethanol, 4% ethanol, and 8% C₄-C₇ fluoroalkyl carboxylate compounds. Test material was from 3M production lot 564.

The following summary is abbreviated because the test sample is a mixture of the test substance in a water/organic solvent solution. Data does not accurately relate biodegradation of the test sample with that of the test substance.

METHOD:

Method: EPA Method 425.1
Type: Methylene Blue Active Substance
GLP: No
Year completed: 1989

RESULTS

G1208-1: 2200 mg/kg
G1208-4: 2800 mg/kg (duplicate of G1208-1)
Duplicate EPA LAS Reference samples: 60,000 mg/kg
60,000 mg/kg

CONCLUSIONS

This testing indicated that the test sample exhibited minimal activity in this MBAS study.

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

DATA QUALITY

Reliability: Klimisch ranking 2. This study lacks information on the methodology and testing parameters. Test substance purity within the test sample was not sufficiently characterized.

REFERENCES

This study was conducted at Pace Analytical of Minneapolis, Minnesota at the request of the 3M Company.

OTHER

Last changed: 5/17/00

LAB REQUEST NO. G1208

REQUESTOR NAME: EA REINER

DEPARTMENT: 0535

PROJECT NO: 9970010100

DATE RECEIVED: 02/03/1989

DESC: MBAS OF FC-129, TRITON H66

CONTRACT LAB: PACE

CONTRACT LAB COST: 162

3M E-L HOURS: 0

EXP COMP DATE: 02/17/1989

DATE COMPLETED: 02/15/1989

SAMPLES SHOULD BE DELIVERED IN MY NAME TO THE LAB. RUN A MEASUREMENT ON THE STANDARD LAS SOLUTION FOR COMPARISON. DO ALL EXTRACTIONS AND BACK WASH CALLED FOR IN PROCEDURE. FC-129 IS 50% SOLIDS. ITS MW IS 623. THE MW OF THE LAS STANDARD IS PROBABLE ABOUT 325. TEST FC-129 AT A MOLAR CONCENTRATION EQUAL TO THAT OF LAS.

G1208-3 IS AN EPA LAS REFERENCE MATERIAL: LOT #1138, AVE. M.W. 318, ACTIVE LAS 6.00 %, PREPARED BY EMSL CINCINNATI US EPA IN COOPERATION WITH SOAP AND DETERGENT ASSOCIATION, DATE 985.

Revised report shows correction in project description above.

SAMPLE	DATE	CODE	DESCRIPTION	RESULT	MIN DET LIMIT
1		FC-129	LOT 564		
		*SURFACTANTS - MBAS		2200 MG/KG	5
		*INORGANIC TESTS REPORT		SEE ATTACHED REPORT	
2		TRITON H66	RH-RM 6-1536		
		*SURFACTANTS - MBAS		1000 MG/KG	5
3		LAS STD.	EPA LOT 1138		
		*SURFACTANTS - MBAS		60000 MG/KG	5
4			DUPLICATE #1		
		*SURFACTANTS - MBAS		2800 MG/KG	5
5		DUPLICATE #2	~~~~~		
		*SURFACTANTS - MBAS		1200 MG/KG	5
6			DUPLICATE #3		
		*SURFACTANTS - MBAS		60000 MG/KG	5
		* = CONTRACT LAB			

SUBMITTED BY: *H. H. Korman* DATED: 2-23-89

February 10, 1989

G1208

Mr. Wade Scheil
3M Environmental Laboratory
935 Bush Avenue
Bldg. 2-3E
St. Paul, MN 55133

Rec 2/14/88
WAS

Dear Mr. Scheil:

Enclosed is the report of laboratory analyses for samples received
02/03/89.

If you have any questions concerning this report, please feel free
to contact Tom Halverson or me.

Sincerely,

Peggy F. Gaskill for

Roger C. Splinter, Ph.D.
Director, Laboratory Services

Enclosures



REPORT OF LABORATORY ANALYSIS

Offices:
Minneapolis, Minnesota
Tampa, Florida
Coralville, Iowa
Novato, California

3M Environmental Laboratory
935 Bush Avenue
Bldg. 2-3E
St. Paul, MN 55133

February 10, 1989
PACE Project Number: 890203509

Attn: Mr. Wade Scheil

G1208

Date Sample(s) Collected: By Client
Date Sample(s) Received: 02/03/89

PACE Sample Number:

Parameter

Units

MDL

032110

G1208-1

032120

G1208-2

032130

G1208-3

INORGANIC ANALYSIS

INDIVIDUAL PARAMETERS

Surfactants (1)

mg/kg

5

2200

1000

60000

MDL Method Detection Limit

REPORT OF LABORATORY ANALYSIS

Offices:
Minneapolis, Minnesota
Tampa, Florida
Coralville, Iowa
Novato, California

Mr. Wade Scheil
Page 2

February 10, 1989
PACE Project Number: 890203509

PACE Sample Number:

Parameter

Units

MDL

032140

G1208-4

032150

G1208-5

032160

G1208-6

INORGANIC ANALYSIS

INDIVIDUAL PARAMETERS

Surfactants (1)

mg/kg

5

2800

1200

60000

MDL Method Detection Limit
(1) Analysis performed by EPA Method 425.1.

The data contained in this report were obtained using EPA or other approved methodologies. All analyses were performed by me or under my direct supervision.


Thomas L. Halverson
Inorganic Chemistry Manager

ANALYSIS:

MBAS

DATE ANALYZED:

ANALYZED BY:

TIME:

DATA REVIEWED BY:

ENTERED BY:

FOR CLP USE ONLY?

CLIENT NAME:

PROJECT NUMBER:

PROJECT NAME:

FILE #:

DATE RECEIVED:

DATE COLLECTED:

[illegible]

BIODEGRADATION

TEST SUBSTANCE

Identity: Perfluorooctanesulfonylamido(ethyl)acetate; may also be referred to as PFOSAA, FC-128 or as the major component of FC-127, FC-129, FC-109, FC-109-X or R1904. (Glycine, N-ethyl-N-[(heptadecafluorooctyl)sulfonyl]-, potassium salt, CAS # 2991-51-7)

Remarks: The test sample is FC-129. The study-defined composition is 50% Potassium fluoro alkyl carboxylate, 18% butyl carbitol, 28% water, and 4% ethanol. This is a minor contradiction to the formulation available from proprietary 3M databases indicating it is a mixture of 42% test substance, 32% water, 14% 2-butoxyethanol, 4% ethanol, and 8% C₄-C₇ fluoroalkyl carboxylate compounds. Test material was from 3M Deutschland production batch 2304.

The following summary is abbreviated because the test sample is a mixture of the test substance in a water/organic solvent solution. Data does not accurately relate biodegradation of the test sample with that of the test substance.

METHOD:

Method: OECD 301E

Type: Aerobic

GLP: No

Year completed: 1984

Contact time: 28 days

Inoculum: Secondary Effluent of a domestic sewage plant

RESULTS

Mean DOC removal after 28 days: 90%

Remarks: Organic carbon content of the test material was determined to be 13.5 mg/100 mg sample by a carbon analyzer.

CONCLUSIONS

No reliable conclusions can be derived from this study.

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

DATA QUALITY

Reliability: Klimisch ranking 3. The digestive procedure of the instrument used in this study to determine TOC is not aggressive enough to break the carbon-fluorine bonds. The TOC is therefore inaccurate. The calculated TOC is 22% compared to the 13.5% obtained by analysis. These results are expected to be representative of the organic solvents present and not of the Perfluorooctanesulfonylamido(ethyl)acetate. Lack characterization of test substance and confirmation of test concentrations.

REFERENCES

This study was conducted at Research and Consulting Company AG, CH-4452 Itingen / Switzerland at the request of the 3M Company.

OTHER

Last changed: 5/17/00

ENVIRONMENTAL RISK ASSESSMENT OF FC-129

SUBJECT PRODUCT

Identity: FLUORAD Brand Fluorochemical Surfactant FC-129. Major component is Perfluorooctanesulfonylamido(ethyl)acetate, CAS # 2991-51-7

Remarks: The subject product is FC-129. Current information indicates it is a mixture of 42% test substance, 32% water, 14% 2-butoxyethanol, 4% ethanol, and 8% C₄-C₇ fluoroalkyl carboxylate compounds and is referred to in the assessment as "... a solution version of FC-128."

CONCLUSIONS

This assessment is extremely subjective. There is no data to support conclusions that it will "enter the environment in very low concentrations". Additionally, conclusions are based on limited hazard data.

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

REFERENCES

The subject assessment was conducted by E. A. Reiner of the 3M Environmental Laboratory, Env. Assessment Inquiry Number 80681, Sept. 25, 1981.

OTHER

Last changed: 5/17/00

Internal Correspondence

cc: R. L. Bohon - 21-2W-05

To: D. R. RICKER - COMMERCIAL CHEMICALS DIV. - 236-1B-10
From: E. A. REINER - ENVIRONMENTAL LABORATORY/EE & PC - 21-2W-5
Subject: OECD BIODEGRADATION TESTS ON ~~FC-129~~ AND FC-170C
Date: OCTOBER 24, 1984

3M

We have received copies of the OECD Screening Tests on FC-129 and FC-170C from D. Reese of 3M Germany.

The results of the FC-129 study incorrectly indicate that this product is readily degradable. This result does not accurately represent the actual susceptibility of this product to biodegradation because the instrument used to measure total organic carbon (TOC) uses a UV-persulfate digestion method. The OECD Screening test does not require a specific TOC method, but the digestive procedure of the instrument used in this study is not strong enough to break fluorine-carbon bonds. Therefore, these results, really only show that the butyl Carbitol® and ethanol in this product are biodegradable.

The results of the FC-170C study are more useful but still inaccurate. These results confirm our previous BOD tests that suggested partial degradation of the polyethylene glycol portion of this molecule. Again, however, the results obscure the fact that the perfluoro portion of the FC-170C molecule is almost certainly not biodegraded.

The data in these reports confirm the inability of the UV-persulfate digestion method used to degrade the perfluoro-carbon portion of these products. The measured TOC for FC-170C was 24 mg C per 100 mg product while the calculated TOC based on formulation information is 38 mg C per 100 mg product. The measured TOC for FC-129 was 13.5%; its calculated TOC is about 22%.

Because of the weak and inappropriate TOC method used, we are not going to add these results to our Product Environmental Data Sheets. Customers and regulatory agencies would likely misinterpret these results and think that these products are more biodegradable than they actually are.

E. A. Reiner

EAR/mb

FC-129 101

R C C

9/14/84

RESEARCH & CONSULTING COMPANY AG

*These results
are not appropriate
for use because the
persulfate oxidation method*

PROJECT 034244

READY BIODEGRADABILITY:
"MODIFIED OECD SCREENING TEST"
FOR FLUORAD FC-129

REPORT

P.O. Box CH-4452 Itingen Switzerland
Phone 061 / 98 52 52 Telex 966 136 RCC CH

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PREFACE

Project 034244

Sponsor 3M Deutschland GmbH
Postfach 643
D 4040 Neuss 1

Study monitor D. Reese

Testing facility R C C
Research and Consulting Company AG
CH-4452 Itingen/Switzerland

Study director Dr. H. Ellgehausen


Date: September 19, 1984 / hoh

Technical assistance V. Wuethrich

Start of experiments August 17, 1984

Date of completion September 14, 1984

Archiving Raw data, protocol, report,
duplicate of report:

R C C
Research and Consulting Company AG
CH-4452 Itingen/Switzerland

1 INTRODUCTION

The objectives of the present test were to follow the biodegradability of FLUORAD FC-129 in a 28 days biodegradation test by following the DOC of the chemical in the incubation flasks by colorimetric methods.

2 MATERIALS AND METHODS

2.1 Chemicals

The following chemicals were tested:

Standard compound:

Aniline
Purity: 99.5 %

Test compound:

Fluorad FC-129

Composition:

Potassium fluoro alkyl	
carboxylate	50 %
Butyl glycol	18 % Carbitol
Water	28 %
Ethanol	4 %

Batch: 2304

Water solubility:

>2 g/l at 25 degrees centigrade.

The test chemical was supplied by 3M Deutschland GmbH, on July 10, 1984.

2.2 Experimental design

The study was run according to the OECD method (1).

3 RESULTS

3.1 Degradation of test compound -----

The results are presented in Sample Form Sheet No 1 and Figure 1.

3.2 Degradation of standard compound (aniline) -----

The results are presented in Sample Form Sheet No 2 and Figure 2.

4 CONCLUSION

The test compound, Fluorad FC-129, was degraded under the test conditions within 28 days to 90 %.

The standard, aniline, was degraded within seven days to 97 %.

The compound is considered to be readily degradable.

5 REFERENCE

OECD Guideline for testing of Chemicals:

301 E: "Ready Biodegradability: Modified OECD Screening Test".

SAMPLE FORM SHEET FOR THE MODIFIED OECD SCREENING TEST: NO 1

Date of start of test: August 17, 1984
Test/Standard material: Fluorad FC-129
Theoretical test conc.: 19.8 mg C/l
Inoculum: sec. effluent of a domestic sewage plant
(ARA-Birsig, Therwil, CH)
Carbon analyser: Technicon Autoanalyser II, —
Method-No. 451-76 W

Per sulfate UV at 300°C
temp lamp bring it h

A: Organic carbon content of test material: 100 mg test material
correspond to: 13.5 mg organic carbon *

Stock solution of the test material (734.5 mg/100 ml, dilution
20 ml/1000 ml of nutrient solution)

based on
formulation
I calculate
product is
22% carbon
if it contains
14% butyl
Carbitol
or
25% carb
if 25% carb
butyl
Carbitol
10/17/84

B: Carbon determinations:

Culture medium	Flask No.	Analyses	Theor. conc. mg/l	DOC ** after x days					
				0 (CO)	7	14	21	27	28
Mineral nutrient solution with test material and with inoculum	1	a1	19.8	17.5	***	3.3	3.6	3.8	4.0
		a2	19.8	17.6	***	4.1	4.1	4.0	3.7
		a1+a2							
	2	m1=----- 2	19.8	17.6	***	3.7	3.9	3.9	3.9
		b1	19.8	17.7	***	3.8	3.6	4.1	3.6
		b2	19.8	17.7	***	3.8	3.5	4.2	3.6
Mineral nutrient solution without Blank test material but with inoculum		b1+b2							
		m2=----- 2	19.8	17.7	***	3.8	3.6	4.2	3.6
		c1	-	1.3	1.8	1.2	1.4	1.6	1.9
		c2	-	0.7	2.7	1.8	1.1	2.4	2.1
		c1+c2							
		m3=----- 2	-	1.0	2.3	1.5	1.3	2.0	2.0

* Value determined with carbon analyser.

** All DOC values determined after centrifugation.

*** Samples lost during analysis.

Sample form sheet No. 1 cont.

C: Evaluation of raw data:

	Flask No.	% DOC removal after x days						
		7	14	21	27	28		
DOC-concentrations minus blanks	1	D1 = 1 -	$\frac{m1t-m3t}{m1-m3}$	100	*** 87	84	89	89
	2	D2 = 1 -	$\frac{m2t-m3t}{m2-m3}$	100	*** 86	86	87	90
	mean	D =	$\frac{D1 + D2}{2}$		*** 87	85	88	90
				for day x				

*** Samples lost during analysis.

SAMPLE FORM SHEET FOR THE MODIFIED OECD SCREENING TEST: NO 2

Date of start of test: August 17, 1984
Test/Standard material: Aniline
Theoretical test conc.: 19.4 mg C/l
Inoculum: sec. effluent of a domestic sewage plant
(ARA-Birsig, Therwil, CH)
Carbon analyser: Technicon Autoanalyser II,
Method-No. 451-76 W

A: Organic carbon content of test material: 100 mg test material
correspond to: 77.4 mg organic carbon *

Stock solution of the test material (25 mg/l)

B: Carbon determinations:

Culture medium	Flask No.	Analyses	Theor. conc. mg/l	DOC ** after x days					
				0 (CO)	7	14	21	27	28

Mineral nutrient solution with test material and with inoculum	1	a1	19.4	12.4	0	2.1	1.2	1.8	1.9
		a2	19.4	11.9	2.4	1.8	1.1	1.6	2.3
		a1+a2							
		m1=----- 2	19.4	12.2	1.2	2.0	1.2	1.7	2.1

	2	b1	19.4	12.0	3.1	1.8	1.9	1.9	2.1
		b2	19.4	10.9	2.7	1.6	1.4	1.8	2.1
		b1+b2							
		m2=----- 2	19.4	11.5	2.9	1.7	1.7	1.7	2.1

Mineral nutrient solution without Blank test material but with inoculum		c1	-	1.3	1.8	1.2	1.4	1.6	1.9
		c2	-	0.7	2.7	1.8	1.1	2.4	2.1

		c1+c2							
		m3=----- 2	-	1.0	2.3	1.5	1.3	2.0	2.0

* Value calculated.

** All DOC values determined after centrifugation.

Sample form sheet No. 2 cont.

C: Evaluation of raw data:

	Flask No.			% DOC removal after x days				
				7	14	21	27	28
DOC-concentrations minus blanks	1	D1 = 1 -	m1t-m3t					
			m1 -m3	100	100	96	100	100
	2	D2 = 1 -	m2t-m3t					
			m2 -m3	100	94	98	96	100
	mean	D =	D1 + D2					
			$\frac{2}{\text{for day } x}$	97	97	98	100	99

Figure 1: DOC-removal
Test material: Fluorad FC-129

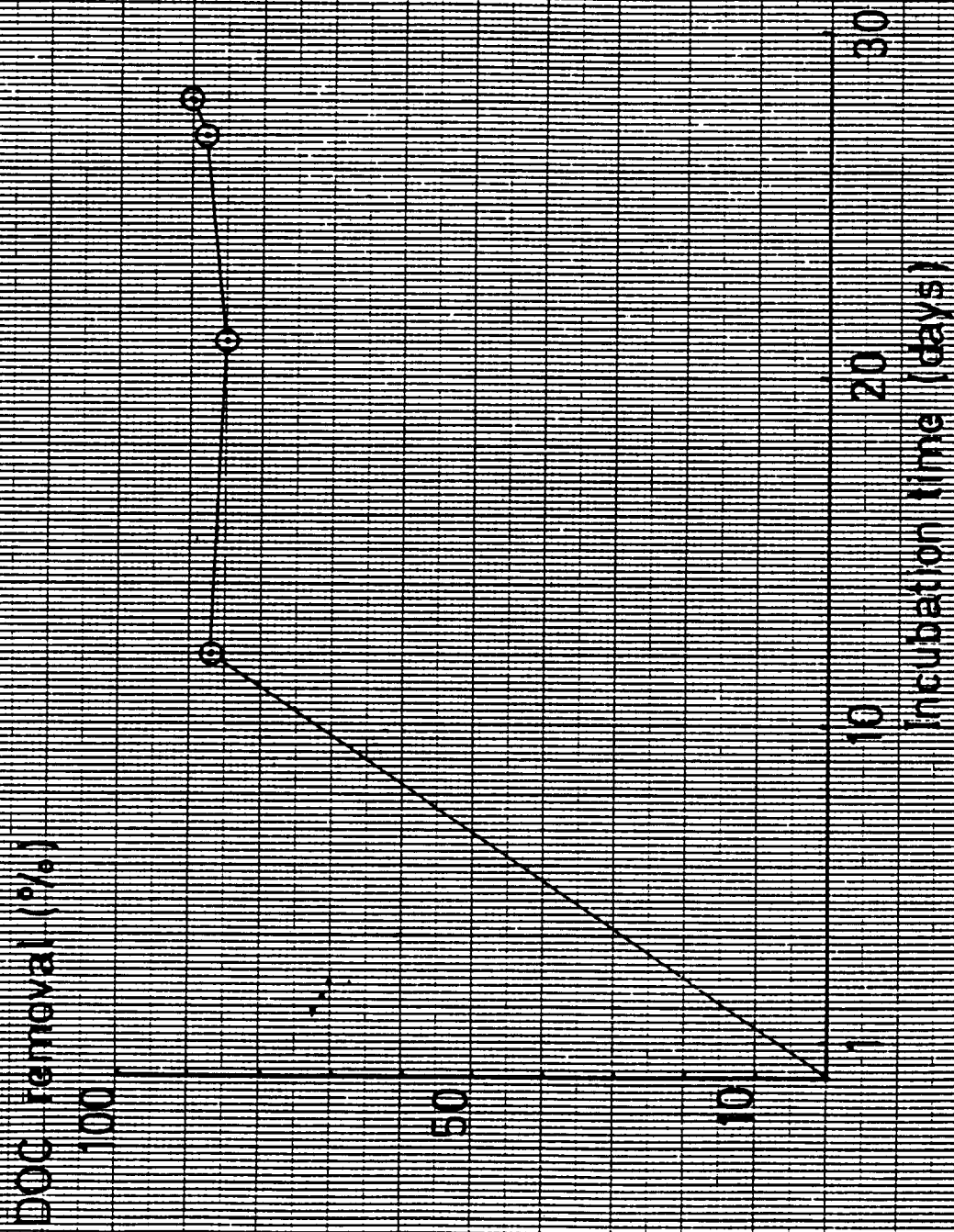
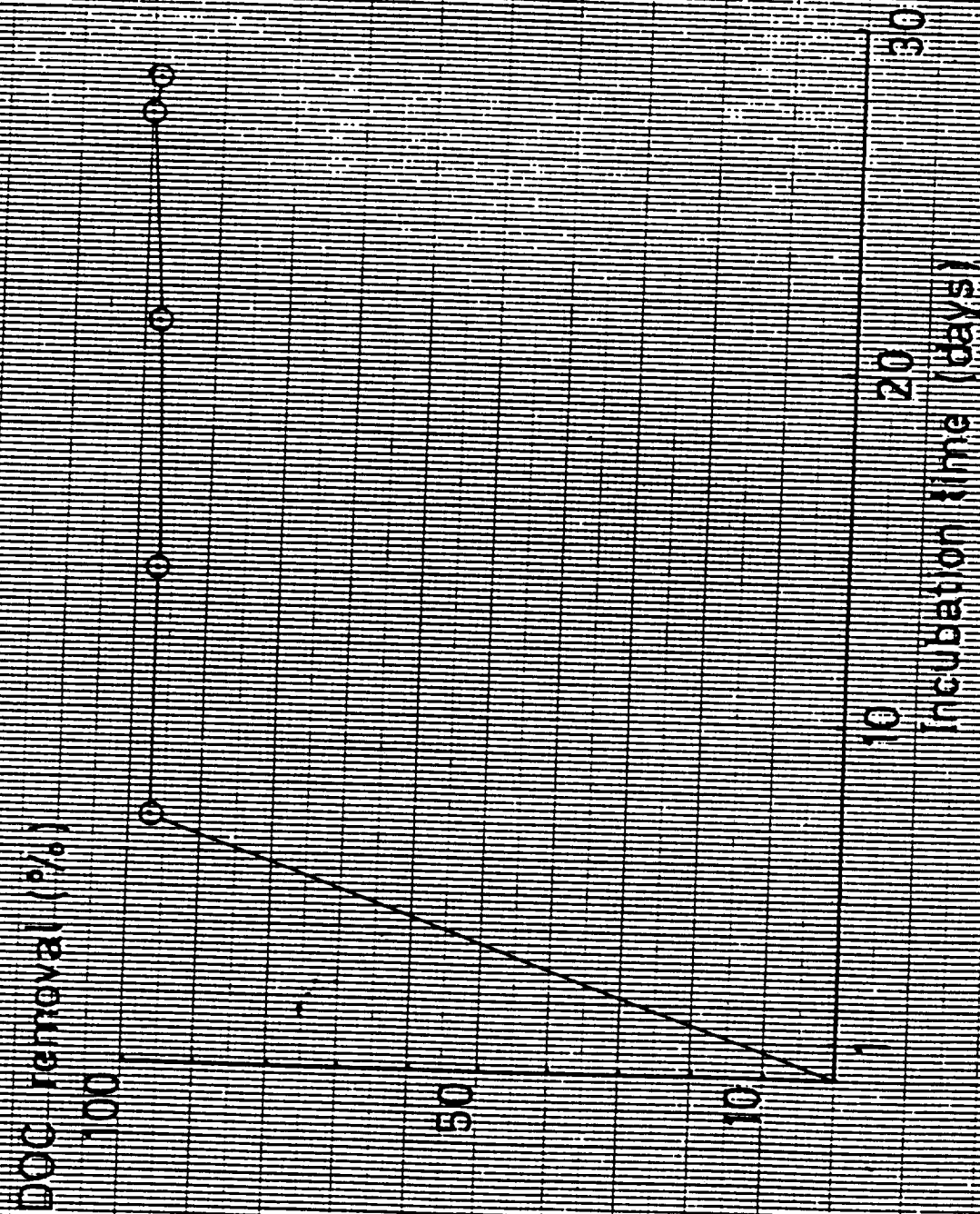


Figure 2: DOC-removal
Test material: Aniline



Sheet1

ThOD of FC-129						
1) 50% FC SOLIDS: C ₈ F ₁₇ SO ₂ N(C ₂ H ₅)CH ₂ COO-K ⁺						
11/91 NOTE: CARBON RATIOS ARE C ₄ ,C ₅ ,C ₆ ,C ₇ ,C ₈ =2.5%,0.5%,3%,2%,42%						
2) 14% BUTYL CELLOSOLVE						
3) 32% WATER						
4) 4% ETHANOL						
FRACTION	COMPONENTS			MW	MW CARBON	% Carbon
42.00%	C ₈ F ₁₇ SO ₂ N(C ₂ H ₅)CH ₂ COO-K ⁺			623	144	9.71%
2.00%	C ₇ F ₁₅ SO ₂ N(C ₂ H ₅)CH ₂ COO-K ⁺			573	132	0.46%
3.00%	C ₆ F ₁₃ SO ₂ N(C ₂ H ₅)CH ₂ COO-K ⁺			523	120	0.69%
0.50%	C ₅ F ₁₁ SO ₂ N(C ₂ H ₅)CH ₂ COO-K ⁺			473	108	0.11%
2.50%	C ₄ F ₉ SO ₂ N(C ₂ H ₅)CH ₂ COO-K ⁺			423	96	0.57%
14%	C ₄ H ₉ OCH ₂ CH ₂ OCH ₂ CH ₂ OH			162	96	8.30%
4%	CH ₃ CH ₂ OH			46	24	2.09%
32%	WATER			18	0	0.00%
100.00%	TOTAL					21.92%