

PHOTODEGRADATION (SOIL)

TEST SUBSTANCE

Identity: N-ethylperfluorooctane sulfonamidoethanol; may also be referred to as N-EtFOSE Alcohol or FM-3422. (1-Octanesulfonamide, N-ethyl-1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-N-(2-hydroxyethyl)-, CAS # 1691-99-2)

Remarks: Material is an off-white, waxy solid of uncharacterized purity.

The following summary is abbreviated due to the fact that a new study is pending.

METHOD

Method/guideline followed: Procedure developed by 3M to study the photolysis of N-EtFOSE alcohol sorbed to soil.

Type (test type): Sunlight exposure

GLP (Y/N): No

Year: 1979

Light Source: Minnesota sunlight as it passes through an east-facing window in the 3M Environment Laboratory from February 26 to October 18, 1979.

Light Spectrum (nm): Not recorded

Intensity: Not recorded

Spectrum of substance (max lambda, max epsilon and epsilon 295): Not determined.

Remarks field:

- **Test medium (air, water, soil, other - specify):** Soil
- **Duration:** 233 days
- **Positive Controls:** None
- **Negative Controls:** None
- **Blank Controls:** One

RESULTS

Concentration of Substance: One gram N-EtFOSE alcohol on 20 grams of soil

Temperature °C: Not recorded

Degradation %: Not quantified

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. Study is invalidated by a lack of proper methodology and record keeping. Test substance purity was not properly characterized.

REFERENCES

3M Technical Report "Photolysis of FM 3422 on Soil." James E Gagnon and Arthur Mendel, Fate of Fluorochemicals, Project 9770612600, Report Number 015, December 10, 1979

Review of Technical Report Summary. Photolysis of FM 3422 on Soil. Professor Stephen A. Boyd, Michigan State University, May 19, 1993.

OTHER

Last changed: 5/18/00

Attached are comments on the 3M Technical Report "Photolysis of FM-3422 on Soil. J. Gagnon and A. Mendel, Project 9970612600, Report No. 15, Dec. 10, 1979" made by Professor Stephen A. Boyd, Michigan State University, dated May 19, 1993.

Review of Technical Report Summary
Photolysis of FM 3422 on Soil

The report lacks any description of the experiments. The Materials and Methods section is missing. The fact that FM 3422 is virtually absent in the control soil is worrisome. Of course, an exact description of the control isn't given, but I assume it is the same coated soil not exposed to sunlight. At any rate the control looks invalid, which essentially invalidates the whole study. The study should be re-designed in consultation with a photochemist and repeated.



Form 6747-11-A

TECHNICAL REPORT SUMMARY

Date
Dec. 10, 1979

TO: TECHNICAL COMMUNICATIONS CENTER - 201-2CN

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

Division Environmental Laboratory (EE & PC)		Dept. Number 0535
Project Fate of Fluorochemicals		Project Number 9970612600
Report Title Photolysis of FM 3422 on Soil		Report Number 015
To Dale L. Bacon		
Author(s) James E. Gagnon, Arthur Mendel <i>12-11-79</i>		Employee Number(s) 213531, 043939
Notebook Reference 51567, 52692		No. of Pages Including Coversheet
SECURITY ► ☐ Open (Company Confidential) ☐ Closed (Special Authorization)	3M CHEMICAL REGISTRY ► New Chemicals Reported ☐ Yes ☒ No	

KEYWORDS:
(Select terms from 3M
Thesaurus. Suggest other
applicable terms.)Env. Lab
FM-3422
Photolysis

CURRENT OBJECTIVE:

Determine if FM 3422 (FM 3923) coated onto soil would
degrade in sunlight.

REPORT ABSTRACT: (200-250 words) This abstract information is distributed by the Technical Communications Center to alert 3M'ers to Company R&D. It is Company confidential material.

Soil coated with FM 3422 and control soil were exposed
to sunlight for 7 1/2 months. Methanol extracts of the
respective soils were then analyzed by gas chromatography.cc: D. Ricker
A. N. WelterInformation Liaison
Initials: CKL

Introduction

The complex of FM 3422 with sludge and the photolysis of FM 3422 in water were reported earlier^{1,2}. It was of interest to study the fate of FM 3422 deposited on soil and then exposed to sunlight since photolysis, if any, under these conditions may differ from aqueous photolysis.

Results and Discussion

A soil sample coated with FM 3422 and a control soil sample were exposed to Minnesota sunlight from 26 February to 18 October 1979 and then extracted with methanol. The respective extracts were analyzed by electron capture gas chromatography (Figures 1 - 4) for possible photolysis products. Three unknown peaks showed up in the FM 3422 soil sample (Table 1) which were not present in the soil control. Owing to the small amount of products present no attempt was made to isolate and identify these compounds. The peak at $T_R = 2.13$ minutes (Table 1) in the control sample was absent^R from the coated sample and is therefore unlikely to be a photolysis product or a sample preparation artifact. Again because of its low concentration no attempt at identification was undertaken.

FM 3422 was also detected in the control sample. The amount detected was much less than in the FM 3422 coated soil. This could have been caused by cross contamination: 1) during sample preparation 2) from sublimation of FM 3422 from soil 3) during extraction step.

TABLE 1

G.C. of Methanol Extracts of FM 3422 from soil

	T_R (Min)					
	2.13	2.44	4.38*	5.14*	8.43	9.87
Methanol Blank	ND	ND	ND	ND	ND	ND
Control	6.1**	ND	ND	93.9	ND	ND
FM 3422 Soil Extract	ND	2.3	3.5	93.5	0.4	0.3
FM 3422 Standard	ND	ND	6.8	93.2	ND	ND

* = Average retention time

ND = Not detected

** = Results are reported as relative area percent per sample.

This assumes the same response factor for each component.

Future Work

In the future, a scale-up of this experiment should confirm the presence of the unknowns and allow sufficient quantities of samples for subsequent identification.

HP 3380A
 DLY 2.
 MV/M 30

STOP 30
 ATTN 64

REJECT 100

sample

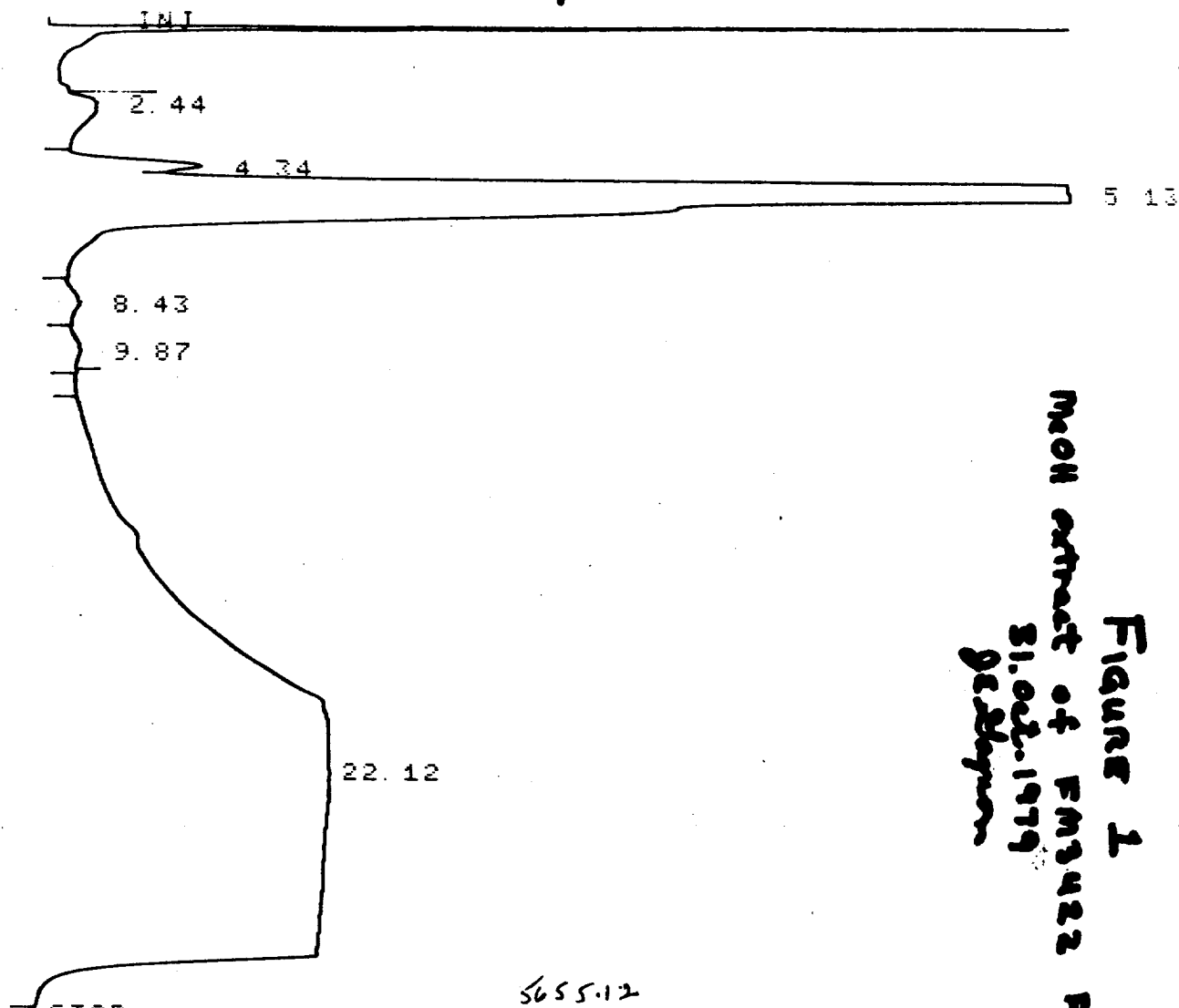


Figure 1
 Mon extract of FM342 from soil
 51.02.1979
 J. S. S. S. S.

STOP
 BTL # 35

RT	TYPE	AREA	AREA %	Corrected Area
2.44	TM	133485	2.29	.780 8
4.34	TM	204485	3.51	1.196
5.13	M	5450639	93.49	31.88
8.43	M	24694	0.42	.144 4
9.87	M	16700	0.29	.097 69
22.12		11265540	65.9	

5655.12

5030003

HP 3380A
 DLY 2.
 MV/M 30

STOP 30
 ATTN 64

REJECT 100

HP 3280A
DLY 2
MV/M 10

STOP 30
ATTN 64

REJECT 100

CUTL

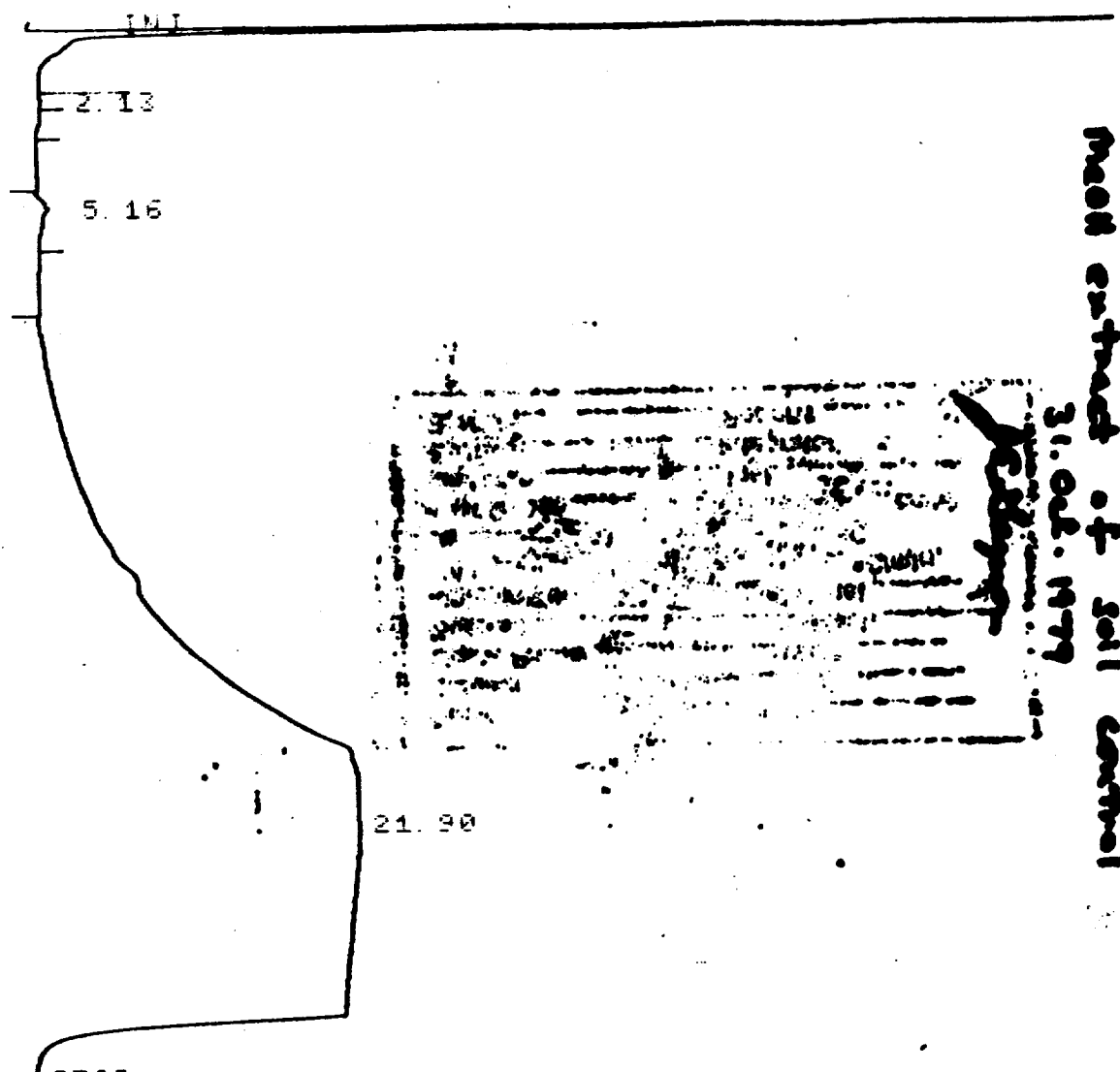


FIGURE 2

mean extract of soil control
31.02.1979

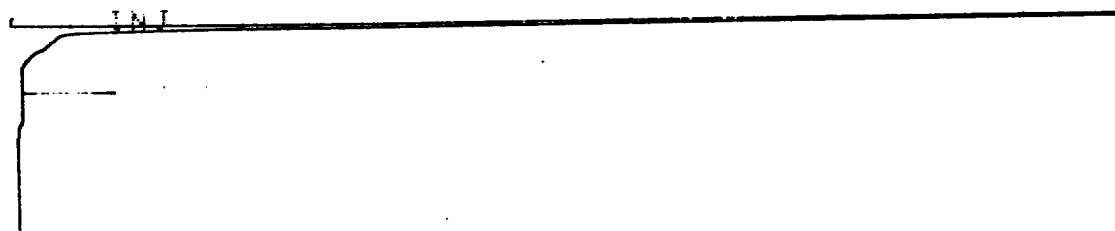
STOP		BTL # 29		AREA M	
RT	TYPE	AREA			
2.13	T	1561		012	15
5.16		23913		185	2
21.90		12817145		99.8	

HP 3280A
DLY 2
MV/M 10

STOP 30
ATTN 64

REJECT 100

MEAN BLK



HP 3380A
DLV 2
MV/M 30

STOP 30
ATTN 64

REJECT 100

10 ppm 178

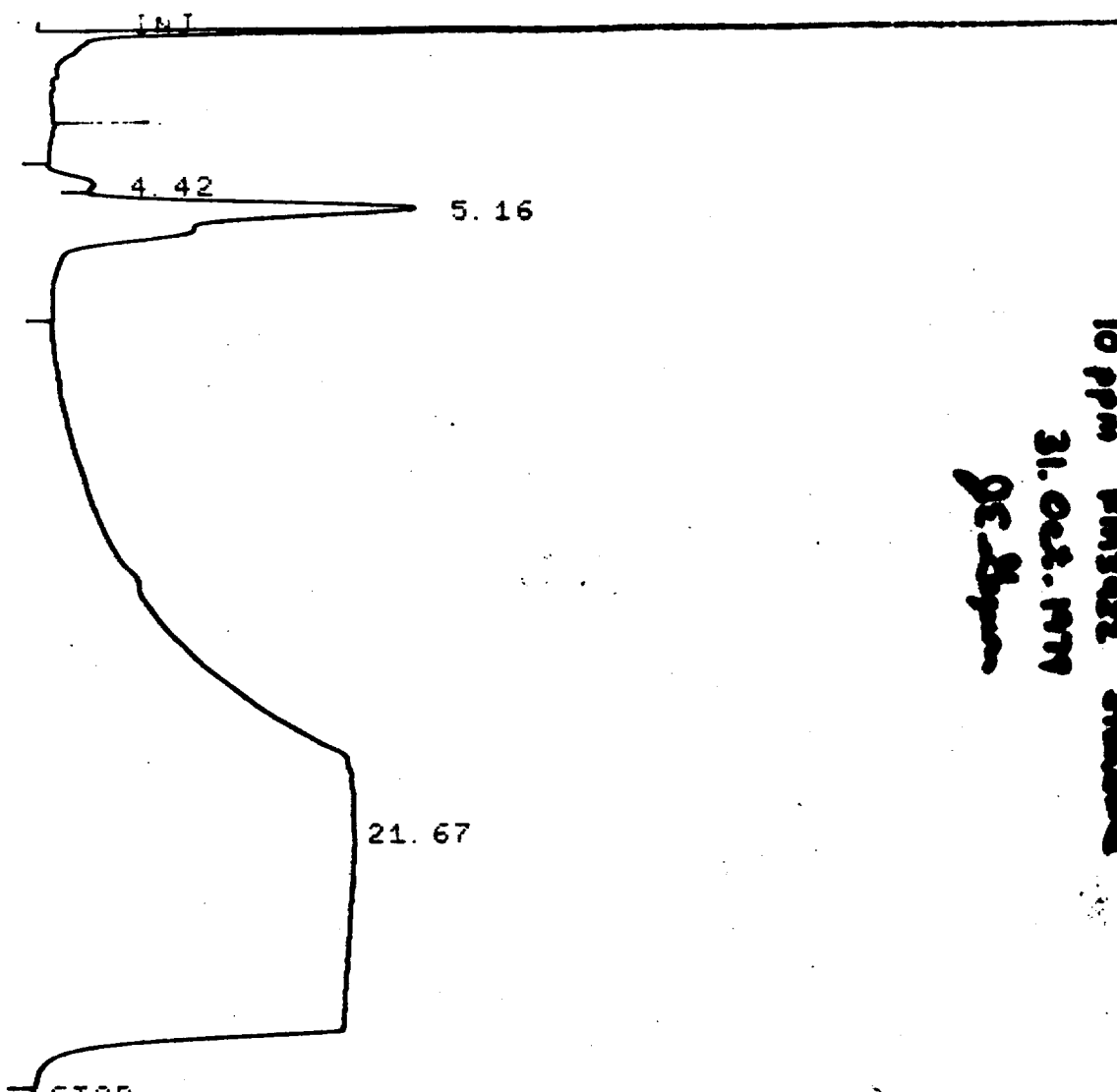


Figure 3
10 ppm PMSU2 standard
31. Oct. 1979
JC. Hynes

STOP		BTL # 32	1011.01	AREA %
RT	TYPE	AREA		
4.42	T	70342		519.7
5.16	TM	967670		7.149
21.67	TM	12498157		92.33

HP 3380A
DLV 2
MV/M 30

STOP 30
ATTN 64

REJECT 100

10 ppm 178



HP 3380A
DLY 2
MV/M 30

STOP 30
ATTN 64

REJECT 100

meth BLK

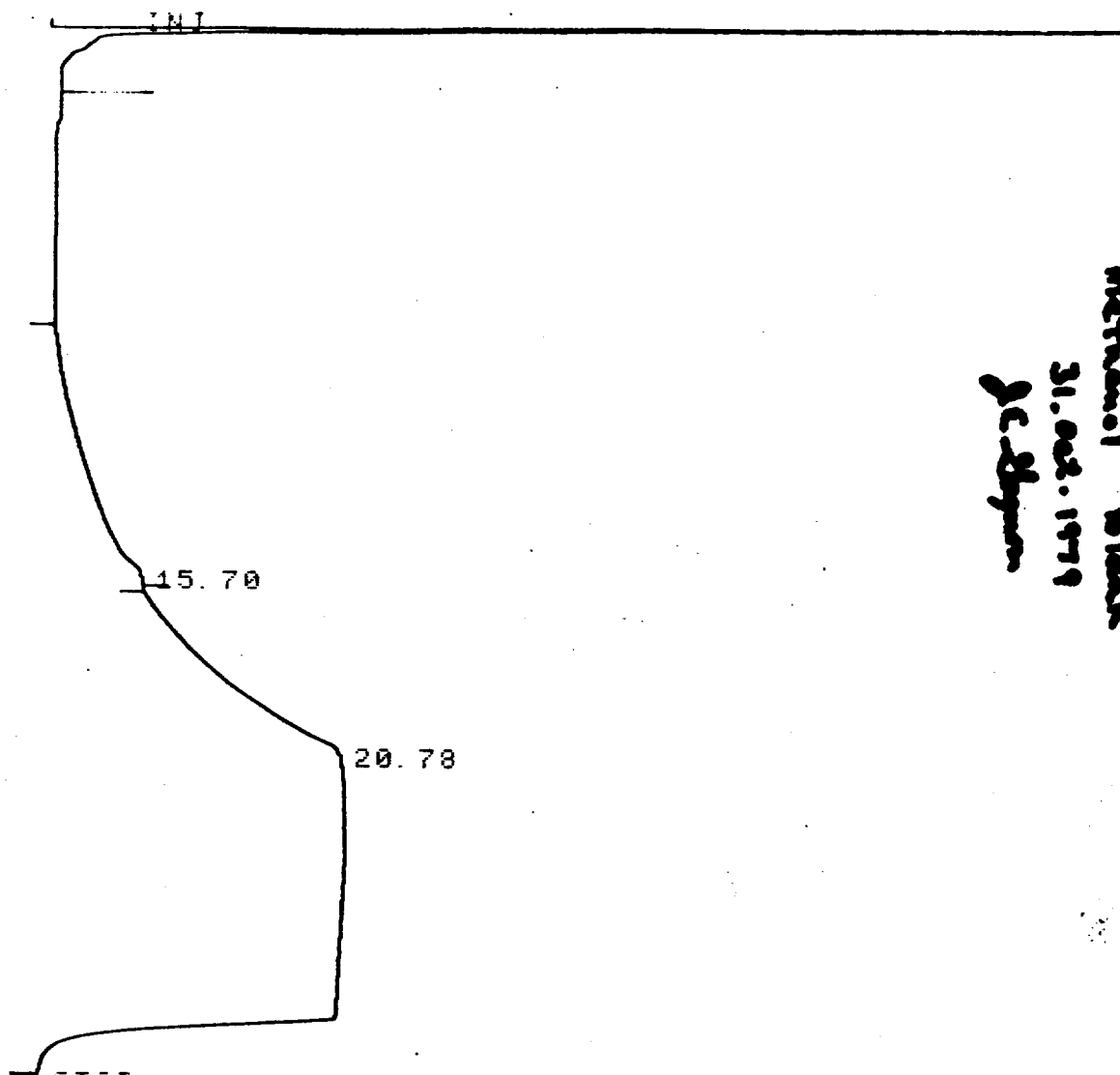


Figure 4
methanol Blank
31.03.1979
JC. Brown

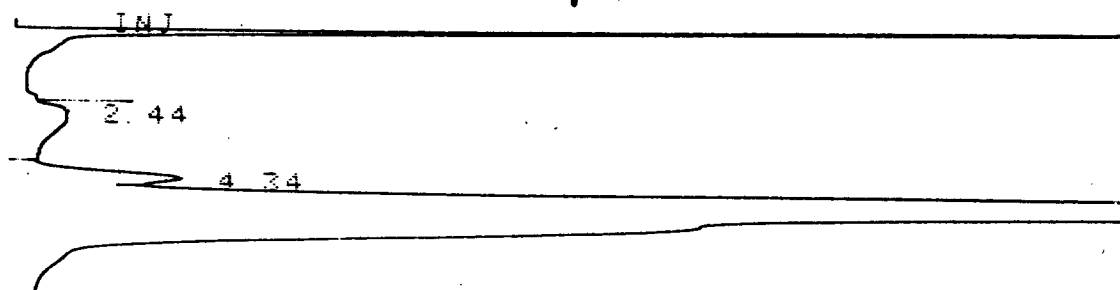
STOP	BTL # 34	AREA %
RT	TYPE	AREA
20.78		8749498 100.

HP 3380A
DLY 2
MV/M 30

STOP 30
ATTN 64

REJECT 100

sample



5.13

Every attempt should be made to eliminate possible cross contamination, such as preparing and analyzing the controls first. An attempt should also be made to physically separate the control and sample during photolysis to eliminate the chance of cross contamination by sublimation, volatilization/adsorption or other means.

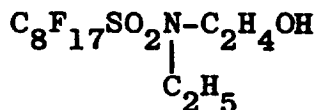
Experimental

1. Sample Materials

Soil - 250 μ sieved greenhouse soil

FM 3422

FM 3422 is N-ethylperfluorooctanesulfonamide-N-ethylalcohol:



Lot E-788 CG-745-2 received from D Ricker of 3M's Commercial Chemicals Division was sublimed at 60°C and 0.1 mm Hg before use.

Methanol - "Lichrosolv" grade available from MC/B Manufacturing Chemists, 2909 Highland Avenue, Norwood, OH 45212.

2. Analysis Instruments/Materials

Extraction Thimbles: Cellulose double thickness available from Curtin Scientific

Gas Chromatograph:

Chromatograph - Hewlett-Packard Model 5713 GC.
Integrator - Hewlett-Packard Model 3380A integrator-printer.

Both of the above available from Hewlett-Packard Co., 150 Page Mill Road, Palo Alto, CA 94304.

Column - Six-foot, 1/8 inch OD, stainless steel, packed with 10% CW20M on 60/80 Chromasorb W-AW.

Column Temperature - 170°C-220°C at 4°C/min. 8 minute post injection interval and upper limit hold

Injector - On-column at 200°C.

Detector - Electron Capture at 300°C.

Flow - ~40 cc/minute of Argon:Methane (95/5).

3. Procedure

Sample Preparation

The control plate was prepared by thoroughly shaking 20 grams of soil with 20 mL of methanol. This slurry was evenly spread to a thickness of approximately 0.5 mm with a glass rod onto a precleaned glass plate.

The FM 3422 slurry was prepared by dissolving one gram of FM 3422 in 20 mL of methanol and shaking with 20 grams of soil. This slurry was applied to a glass plate as described above and both were air dried.

Both plates were placed in an east facing window of the environmental laboratory (no manmade obstruction to sunlight) on 26 February, 1979. The plates were separated from each other by a distance of about 30 cm.

Sample Extraction

All extraction thimbles and silanized glass wool were Soxhlet extracted for 7 hours and stored in methanol rinsed aluminum foil until used.

The samples were removed from the window on 17 October, 1979 (233 days of exposure). Each sample was completely scrapped off the glass plate, placed in separate extraction thimbles, and Soxhlet extracted (methanol) for 7 hours (1 cycle per 15 minutes).

The respective extracts were then concentrated to 1.5 mL in a Kuderna-Danish (K-D) evaporater using steam heat. The control sample was a dark yellow (some of the soil had been transferred from the Soxhlet) while the FM 3422 sample was a light yellow.

Sample Analysis

Approximately 0.1 mL of each sample was diluted to 100 mL with methanol. A 10 ppm standard, the control and sample (1 μ L each) were injected onto the gas chromatograph. Two 1 μ L injections of a methanol blank were injected between each run.

References

- 1) A. Mendel, 3M Technical Report Summary, 30 August 1978 to R. L. Bohon, "Fate of Fluorochemicals Project - Progress Report."
- 2) Report in progress by J. W. Todd of Agricultural Chemicals.