WATER SOLUBILITY

TEST SUBSTANCE

Identity: N-methylperfluorooctane sulfonamidoethanol; may also be referred to as N-MeFOSE Alcohol, FC-790, or FM-3925. (1-Octanesulfonamide, N-methyl-1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-N-(2-hydroxyethyl)-, CAS # 24448-09-7)

Remarks: Material is an off-white, waxy solid. Sample was taken from 3M lot number 505. Purity information was not recorded.

METHOD:

Method followed: The Veith-Comstock technique, G. D. Veith and V. M. Comstock, J. Fish Res. Board Can., 32, 1849 (1975)

GLP: No

Year study was performed: 1979

RESULTS:

Value (mg/L) at 18 ± 1°C: 0.82

Remarks: Initial values for saturated solutions resulting from circulating the system for 24 hours indicated a solubility of approximately 2.3 mg/L. It was suspected that this number was inaccurate as it was considerably higher than the solubility of the closely related material, N-EtFOSE alcohol. Since it's known that water-soluble impurities and some homologues are preferentially solubilized by this technique, the saturated water was replaced with fresh water and was allowed to recirculate for 5 hours. Replicate solubility values of the recirculated solution then indicated a solubility of 0.82 ppm.

CONCLUSIONS:

The test substance is relatively insoluble in water.

DATA QUALITY:

Reliability: Klimisch ranking = 3. This study does not meet criteria for quality testing. This study lacks sufficient characterization of the test substance purity. The properties of the test water were not noted. Additionally, the second circulation of fresh water for only 5 hours without any sampling during that time period or for extended times is inadequate to definitively conclude that the solubility is 0.82 ppm. Although solubility

apparently plateaued between 4 and 6 hours initially in the first 24 hour cycle, it doesn't follow that the same effect will occur in the absence of impurities or homologues. Additionally, there was no identification of the homologues or impurities in the sample. Their presence is assumed, thus they are only possibly interfering with the solubilization of N-methyl FOSE alcohol. The extraction efficiency with ethyl acetate was not characterized. The analytical method was not validated. Electron capture detector is not analyte specific to the target analyte (FM-3925), therefore peak used for quantitation may not be target analyte.

REFERENCES:

This study was conducted by the 3M Company, Environmental Laboratory, 1979.

OTHER

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

Last changed: 5/17/00

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TECHNICAL REPORT SUMMARY

Date: 1/8/79

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: Premanufacturing Notice FC-790	Project Number: 9970012600
Report Title: Solubility of FM 3925	Report Number: 014
To: V. Pothapragada	
Author(s): A. N. Welter/E. A. Reiner	Employee Number(s): 09362/47816
Notebook Reference: NB 46269-53; NB 44191-43,44; NB 42669-41; NB 48277-8; See Environmental Lab Request 4197	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.)

EE & PC Div. (Environmental Lab)

Fluorochemical Solubility Studies (Veith Technique)

CURRENT OBJECTIVE:

To determine the water solubility of FM 3925 (Lot 505).

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.

The solubility of FM 3925 was determined by a method in which water was recirculated from a reservoir through a column containing glass beads coated with FM-3925. Analysis of water samples showed that the FM 3925 concentration reached a plateau of ∿2.3 mg/l at 4 to 6 hours. Since water soluble impurities and more soluble chemical homologs are preferentially solubilized by this technique, the saturated water was replaced with fresh water. The second sequential circulation of water gave an FM 3925 solubility of 0.82 mg/l. This solubility value is considered more realistic.

Information Liaison Initials: SKW

Introduction

FM 3925, based on its structural similarity to FM 3422, may reasonably be expected to possess similar physicochemical characteristics. The latter compound is minimally water soluble (0.05 ppm) and lipophilic.

Techniques have been developed for determining the solubilities of so-called hydrophobic materials while at the same time providing saturated solutions for exposure to aquatic organisms.

The data in this report reflects the results of studies; wherein, a modification of the Veith-Comstock technique has been used in determining the water solubility of FM 3925 (1).

Methods

The method and apparatus used in determining water solubility was modeled after that of Veith and Comstock (1). In brief, water in a reservoir is continuously saturated with the chemical by circulating the water through a bed of an inert substrate impregnated with the chemical under investigation.

Water solubility studies were carried out at 18+ 1°C. in a dark temperature-controlled chamber. 200 mg FM 3925 was dissolved in reagent-grade acetone. This solution was then added to 2 mm solid glass beads and allowed to evaporate off in room air. The coated glass beads were then packed into a glass column fitted on each end with rubber stoppers containing suitable glass-to-tubing connectors (2). (Attachment 1, Environmental Laboratory Protocol.)

One hundred (100) ml water samples were withdrawn at 1, 2, 4, 6, 8, and 24 hours after beginning the experiment. At 24 hours fresh water was added to the system and following 5 hours recirculation, replicate samples were withdrawn for analysis. All water flowed were retrograde: from bottom to top of column.

Samples were analyzed using the following methodology: Hewlett-Packard Model 5713 gas chromatograph with electron capture using ^{63}Ni as the source. Columns 6' x 1/8" O.D. SS, packed with 10% Carbowax 20M on Chromosorb W-AW 60/80 mesh. Temperatures - Detector 300° C., Injection temperature 200° C., Oven - 180° C. Isothermal; flow: 40 cc/min. 95% Argon/5% Methane. Absolute lower detection limit - 0.5 nanograms.

Results and Discussion

Water solubilities of FM 3925 as a function of exposure time com prise TABLE 1. Chromatographic traces, 10 ppm std., 6, 8, and 24 hours, of these results are appended as Attachment 2. The replicated values are in good agreement,

TABLE 1

WATER SOLUBILITY OF FM 3925 - VEITH TECHNIQUE

Sample Time, Hrs.	ppm FM 3925
1	1.72, 1.82
2	1.90, 1.87
4	2.10, 2.17
6	2.33, 2.30
8	2.34, 2.35
24	2.17, 2.14

When fresh water was added to the system and allowed to recirculate for five hours, replicate solubility values of 0.82, 0.82 ppm were obtained (Attachment 3).

The variability in solubilities obtained during the 24-hour run versus that obtained after the addition of fresh water may be explained by the presence of trace impurities in the 24-hour run samples. It is known that when testing materials of inherently low solubilities, it is possible that the trace impurities may be more soluble than the material under study. The recirculated water will then tend to solubilize the impurities, hence higher analytical values. Upon the addition of fresh water to the system and in the probable absence of trace impurities, a more realistic water solubility value of 0.82 ppm FM 3925 was recorded.

References:

(1) Veith, G. D. and V. M. Comstock. Apparatus for Continuously Saturating Water with Hydrophobic Organic Chemicals. J. Fish Res. Board Can. 32:1849-1851, 1975.

(2) Environmental Laboratory Water Solubility Protocol.

ANW/EAR/cen

Attachments: 1, 2, 3

3M ENVIRONMENTAL LABORATORY PROTOCOL FOR THE DETERMINATION OF WATER SOLUBILITY

Discussion:

A true solution may be defined as a "physically homogeneous mixture of two or more substances" (1). In a true solution (particle diameter of less than 1 nm), no heterogeneity can be detected and solutes cannot be separated from each other by mechanical means. In general, most organic compounds are not soluble in water and many tend to form colloids. In colloidal dispersions (particle diameters between 1 nm and 1μm), the particles may not be readily discernible, but they will scatter light (Tyndall effect) and can be separated by physical means.

A saturated solution in water is defined as one in which no more solute will dissolve at a given temperature, or the chemical potential of the undissolved solute equals the chemical potential of the dissolved solute.

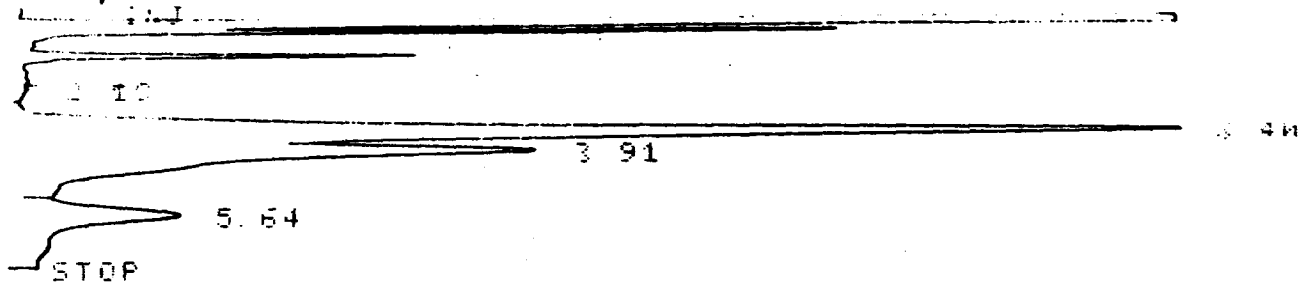
The principal method for determining the solubility of a chemical at saturation is to analyze the solution by some suitable physical or chemical method. No single method is available to cover the entire range of solubilities in water from relatively soluble organic chemicals to very insoluble chemicals. Therefore, several methods can be used to cover the range of solubilities. The method and apparatus chosen was modeled after that of Veith and Comstock (2). Briefly, water in a reservoir is continuously saturated with the chemical by circulating the water through a bed of an inert substrate impregnated with the chemical under investigation. Experiments are carried out in duplicate.

For very hydrophobic chemicals which form colloids, concentration time studies are conducted to ensure that true saturated equilibrium solubility has been reached as evidenced by a plateau in such studies.

The apparatus for these solubility determinations is housed in a temperature-controlled walk-in environmental room whose constant temperature (usually 18°C) is kept below that of room temperature to prevent hydrophobic compounds from precipitating, congealing, or otherwise coming out of solution once it is transferred from this environmental room to the laboratory for further analyses. The possibility of such an event happening would be more likely if the room temperature is below that of the environmental chamber.

HP 3380A
DLY 2. STOP 20 REJECT 1000
MV/M 10 ATTN 128

Again
FM 3925 SOL. ΔT = 6 HR. 48277-4

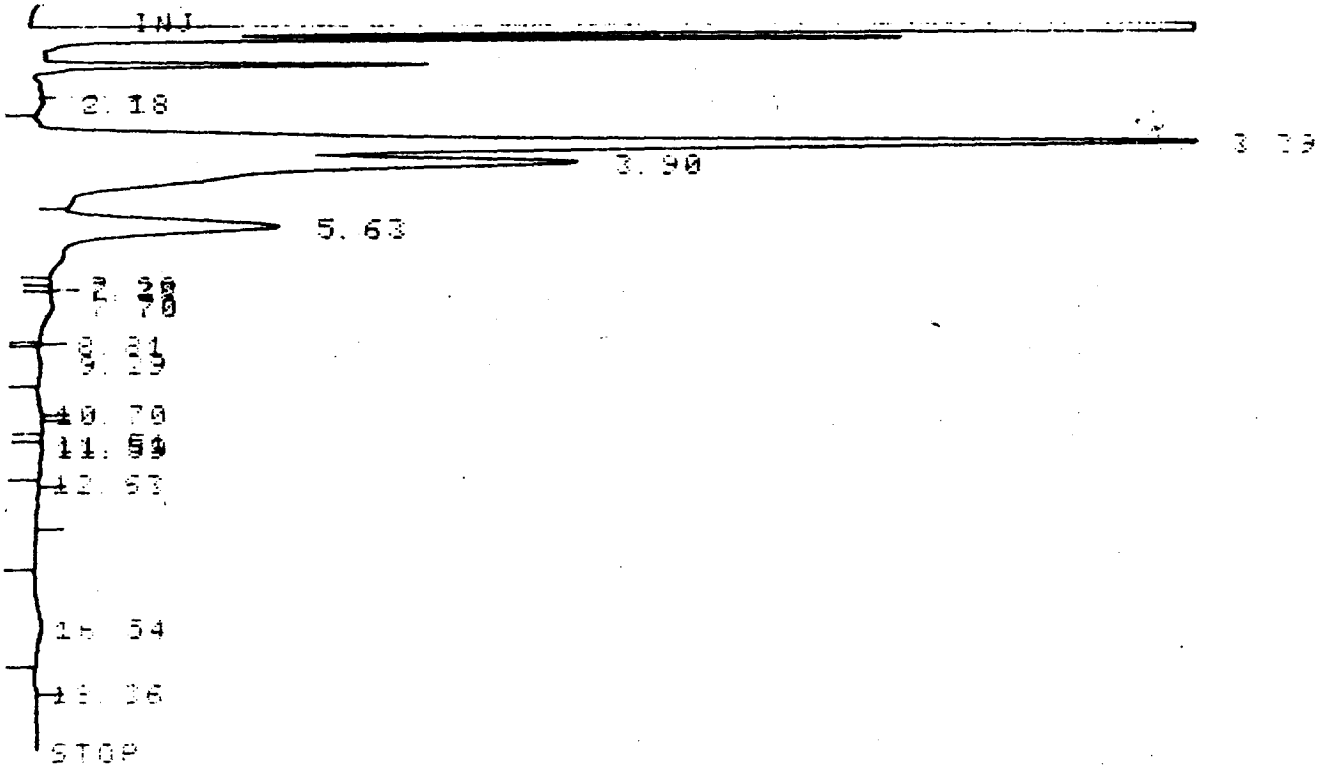


AREA %

RT	TYPE	AREA		
2.10	TM	6419		.123 1
3.40	TM	2764349	2.34 pp	53.
3.91	TM	1786127		34.24
5.64	TM	659296		12.64

HP 3380A
DLY 2. STOP 20 REJECT 1000
MV/M 10 ATTN 128

FM 3925 SOL. ΔT = 8 HRS. 48277-8

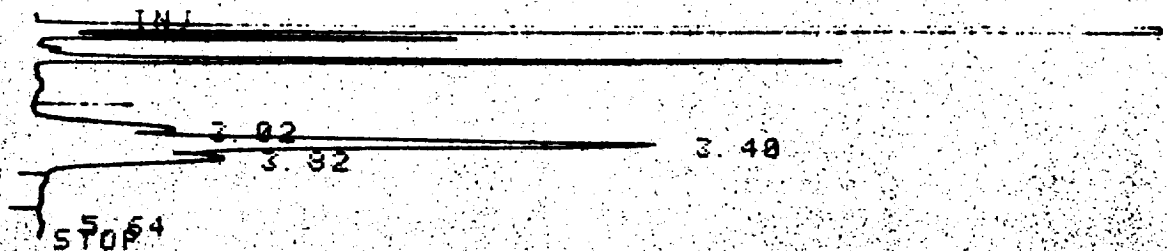


AREA %

RT	TYPE	AREA		
2.18	TM	10485		.191 3
3.40	TM	2791804	2.34 pp	50.93
3.90	TM	1837775		33.53
5.63	TM	779537		14.22

HP 3380A
DLY 2. STOP 20 REJECT 1000
MV/M 10 ATTN 128

10 ppm FM-3925

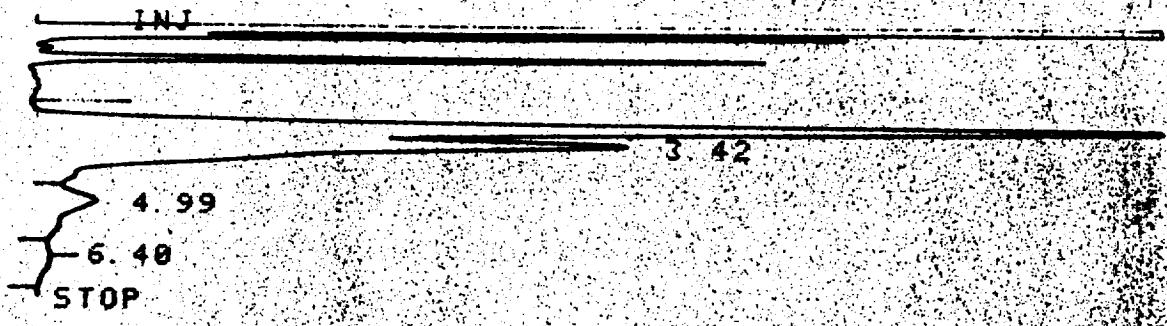


AREA %

RT	TYPE	AREA		
3.02	TM	227990		12.24
3.40	TM	1261420		67.75
3.82	TM	362249	1851659	19.46
5.64	IT	10320		554.2

HP 3380A
DLY 2. STOP 15 REJECT 1000
MV/M .10 ATTN 128

FM3925 SOL. ΔT=24 HRS. 48277-9

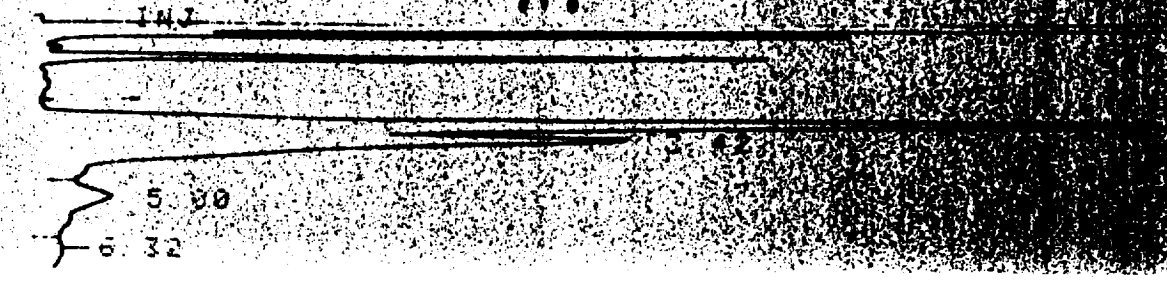


AREA %

RT	TYPE	AREA		
3.01	TM	2390907	2.17 ppm	52.96
3.42	TM	1876717		41.57
4.99	TM	244283		5.411
6.40	TM	2443		.054 12

HP 3380A
DLY 2. STOP 10 REJECT 1000
MV/M 10 ATTN 128

Again



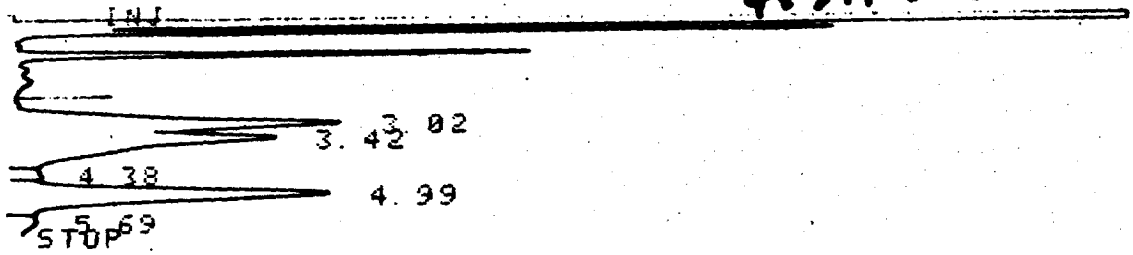
STOP

AREA %

	TYPE	AREA		
	TM	2757409		32 97
	TM	1156827	2.14ppm	41 21
	TM	153645		5 829
	TM	1528		014 02

HP 3380A
DL= 5 STOP 10 REJECT 1000
MW = 10 ATTN 128

FM3925 Sol. 2nd Batch ΔT 6HRS. HP 377-8 (EVAP.) 5ml

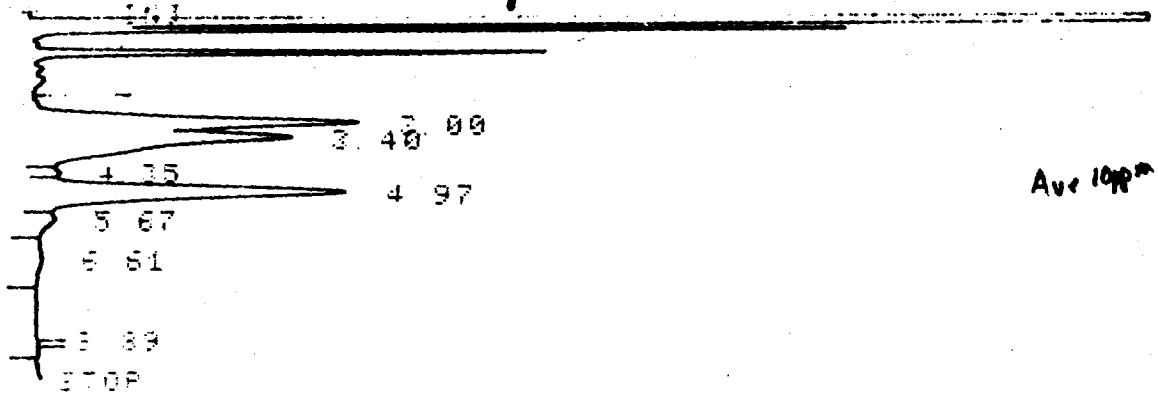


AREA %

RT	TYPE	AREA		
3.02	TM	773577		29.93
3.42	TM	843776	0.82ppm	32.64
4.38	TM	52324		2.024
4.99	TM	851356		32.94
5.69	ITM	63902		2.472

HP 3380A
DL= 2 STOP 10 REJECT 1000
MW = 10 ATTN 128

Aquin 5ml



Ave 10ppm: 963,710

AREA %

	TYPE	AREA		
	TM	779128	1606849	32 98
	TM	811771		32 17
	TM	49701		1 996
	TM	833990	0.82ppm	33 42
	TM	55522		2 186
	TM	35690	4 PK	1 33
	TM	1513		056 17

3M

Form 6747 11-A

TECHNICAL REPORT SUMMARY

Date: 1/17/79

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: 9970612643
Report Title: Analytical Methodology and Support	Report Number: 008
To: D. L. Bacon	
Author(s): Arthur Mendel	Employee Number(s): 043939
Notebook Reference: 41947, 44191, 46269, 47703, 48277, 49400	No. of Pages Including Coversheet: 22

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.)

EE & PC-Div.
Fluorochemicals

Soil
TLC

CURRENT OBJECTIVE:

Progress Report

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.

Information Liaison Initials: JKW

TLC OF Biodegradation Studies on Radioactive FC-95, FC-143, and FM 3422 (47703; 6-14, 21, 25-27, -35; 39-43, 46)

Details of the biodegradation studies on labeled FC-95, FC-143, FM 3422, and controls are described in E. A. Reiner's report (14). In the present work, the nineteen samples each from sludge, water, and ethyl acetate extracts generated from biodegradation studies were applied directly to plates (20x20 cm, E. Merck GF 254 silica gel). The plates were developed in ethyl acetate then examined first by ultraviolet light (long and short wavelength) and then by autoradiography. The plates were exposed to Kodak x-ray film for one week, and the film was then developed and examined. Comparison with control samples indicated no significant differences.

In a second part of this experiment, a portion of the ethyl acetate extract from the various samples was methylated with diazomethane to convert any nonvolatile components to GC volatile species. Both the unmethylated and methylated samples were then analyzed by gas chromatography. No differences were noted between these and control samples. GC conditions are detailed in Notebook 47703-43.

Determination of the Solubility of N-Methyl FOSE Alcohol FM 3925 (48277-8; 46269-53; 42669-4)

About 20 g of FM 3925 Lot 505 was melted (steam bath) to obtain a homogeneous sample. The cooled, solid material, 200 mg, was dissolved in about 200 ml of reagent grade acetone, and this solution was added to precleaned 2 mm diameter solid glass beads contained in a beaker. Beads were precleaned successively with heptane, chloroform, acetone, water, methanol, and then air dried. The FM 3925 coated glass beads were placed in a glass column (350x18 mm) and supported by glass wool (pretreated with Glastreat^{R}). The end rubber stoppers were connected to Bev-Line V^{R} tubing and a peristaltic pump was used to circulate deionized water through the column. The entire setup was conducted in the dark in a temperature-controlled walk-in environmental room (18±1^{o}). Prior to the start of this experiment, water was pumped for 5 hours through the setup, and this water was discarded. The purpose of this was to remove any impurities that would be as soluble as, or more soluble than, FM 3925. Fresh water was then placed into the system and at intervals (see Table 3), 100 ml (volumetric pipet) of water was withdrawn from the main 2-liter storage chamber. Each of the 100 ml samples was quantitatively transferred to a separatory funnel, and 10 ml of saturated aqueous sodium chloride was added followed by 15 ml of ethyl acetate. The mixture was extracted and the separated organic phase was quantitatively transferred to a 10-ml volumetric flask. The aqueous phase was reextracted with 4 ml of ethyl acetate, and the organic phase was added to the 10-ml volumetric flask whose volume was finally brought to the mark with ethyl acetate. An aliquot was used for gas chromatography using an electron capture detector and a six-foot (1.8 meters) column of Carbowax CW20M run at 180^{o}. The results are reported in Table 3.

Table 3

Solubility of FM 3925 in Water

Time of Water Circulation (hrs.)	FM 3925 (ppm)
1	1.72; 1.82
2	1.90; 1.87
4	2.10; 2.17
6	2.33; 2.30
8	2.34; 2.35
24	2.17; 2.14

Avg. (Hrs. 4-24) = 2.3 ppm

The solubility of FM 3925 is forty-six times that of FM 3422. The retention time of FM 3925 is close to that of FM 3422, and the gas chromatographic patterns (see Figure 2 in reference 1) are essentially the same.

REFERENCES

(1) "Analytical Methodology on FM 3422," A. Mendel's progress report to D. L. Bacon, dated 11/15/77, p. 8.

(2) D. B. Easty and B. A. Wabers, Analytical Letters, 10, 857 (1977).

(3) Interoffice correspondence of G. A. Vraspir to D. L. Bacon, March 26, 1976, entitled "Gas Chromatographic Analyses of FM 3422." The distribution coefficient is defined as the ratio of the concentration of compound soluble in n-octanol phase to the concentration of all species in the aqueous phase at a given pH (usually 7).

(4) A. Otsuki and K. Fuwa, Talanta, 24, 584 (1977).

(5) G. T. Wallace, Jr., I. S. Fletcher, and R. A. Duce, J. Environ. Sci. Health, A12 493 (1977).

(6) G. D. Veith and V. M. Comstock, J. Fish. Res. Board Can., 32, 1849 (1975).

(7) R. F. Heine and R. R. Burford, C. G. Klaus, J. D. LaZerte, J. W. Sargent. (Unpublished)

(8) A. Mendel report on Fate of Fluorochemicals to D. L. Bacon, Nov. 15, 1977.

(9) C. T. Chiou, V. H. Freed, D. W. Schmedding and R. L. Kohnert, Environmental Science and Technology, 11, 475 (1977).