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FC-143 IN THE [redacted] PROCESS

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Period: March, 1981 - September, 1981 (Part Time)

Reference: SA-195

Previous Reports: SpAR 79-3

Textile Fibers Department
Spruance
Process Control Development
E. I. DuPont de Nemours & Co., Inc.
Richmond, Virginia

March, 1982

INTRODUCTION

In 1978, the 3M Company notified DuPont that the FC-143 surfactant (ammonium perfluorooctanoate) sold to DuPont for use in [redacted] polymerization may be teratogenic. Therefore, it was necessary to monitor its level in the [redacted] Process and work environment. Work done in a previous report (Ref. 1), showed that it was not present either in the work environment or in sintered yarn at an equipment limited lower detectable limit of 50 ppb. However, because of a proposed TLV in air of 0.5 ppb, it has been necessary to monitor the FC-143 level through the complete [redacted] process with respect to this lower level.

OBJECTIVES

- To measure the extent, if any, of personnel exposure to FC-143.
- To develop accurate methods for FC-143 in air, water, yarn and blood at TLV levels.
- To determine the reactions and final disposition of FC-143 in the [redacted] process.
- To determine the condition for and by-products of thermal treatment to remove FC-143 from dispersion and yarn.

SUMMARY AND CONCLUSIONS

The handling of coated yarns or direct contact with the [redacted] dispersion are the only potential areas that personnel can be exposed to significant levels of FC-143. The perfluorooctanoic acid of FC-143 was not detected in the spinning air environment. The level of perfluro acid in the spinning wash water was 1-4 ppm, but no limit has been defined and aqueous solutions have not been considered a potential means of personnel exposure. The perfluoro acid was detected in yarns coated with dispersion as in the case of yarns used for packing end uses. Levels ranged from 200-700 ppm. With uncoated yarns that come directly off the spinning machine, the levels of perfluoro acid were nondetectable. Thus, the potential for exposure of personnel is very limited, and this was confirmed by the low levels of perfluoro acid in blood. Levels did not exceed 0.09 ppm, which were well below the 0.4 ppm limit.

FC-143 is not found in the spinning area environment because of two reasons. Approximately 10% is washed off the yarn on the wash reel or module. The remainder decarboxylates, as the yarn goes over the heated rolls, giving off CO_2 and the 7 carbon perfluoro monohydride. This has been confirmed by IR analysis of FC-143 off gas as it is heated up to 200°C. The monohydride forms rather than the 7 carbon perfluoro olefin because the other surfactant present, Triton X100, provides the necessary hydrogen.

Because of the decarboxylation at 175-200°C, thermal treatment is an effective way to remove the FC-143. However, because of deleterious effects on yarn properties, thermal treatment is not considered to be a route to its removal.

A modified GC method was developed for higher sensitivity by using an electron capture detector and a new column packing. With these changes, FC-143 in air can be measured to 0.3 ppb and in blood to 0.02 ppm with a precision of ± 10%.

PATENT ACTION

No patent action based on this work is planned at present.

PUBLICATION

There are currently no plans to publish any of this work.

CODE LISTING

FC-143 - Ammonium perfluoro octanoate.

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I. INTRODUCTION

[redacted] dispersion is shipped to Spruance from the DuPont, Parkersburg, West Virginia PPD Plant as a 60% solids/water dispersion that contains 5% Triton X-100 and 1600 ppm FC-143 as surfactants. 3M, the FC-143 vendor, notified DuPont in May, 1978 that it was a possible teratogen. Therefore, it became necessary to measure the FC-143 levels in the process and work environment to determine the level of personnel exposure, if any. Initial work (Ref. 1) was equipment limited in sensitivity to a lower detection limit of 50 ppb. With a proposed TLV of 0.5 ppb, it became necessary to redetermine its levels with increased sensitivity and to determine its final disposition - whether it is washed out, decomposes or remains in the yarn. Also, since thermal treatment was a possible route to FC-143 removal from the dispersion, the condition and byproducts of thermal treatment must be determined.

II. ENVIRONMENTAL ANALYSIS

A. In-Air

Air samples were taken at eight locations around Teflon® spinning machines 2 and 3 (Figure I), and at four locations around the Finishing Area induction dryer. (Table I, Attachment I) Only one sample from inside the induction dryer stack inlet had a detectable level (3 ppb). Since this was inside the duct, there was no personnel hazard involved. The lower detection limit for in-air samples based on standards is 0.3 ppb. The lack of detection precludes any personnel exposure hazard since the exposure problem reported by 3M is due to air borne contamination.

B. Water

The FC-143 in [redacted] dispersion is changed to the sodium salt after the final pH adjustment step at Parkersburg. This is converted to the acid form in the regeneration bath, but it still has some water solubility. The wash water from the reel machine (SM 3) and wash module (SM 2) has been analyzed. Levels found are 1.3 ppm for SM-2 and 4.0 ppm for SM-3. The differences reflect differences in wash efficiency due to different equipment design. FC-143 in regenerated yarn from SM-3 (60 ppm) and SM-2 (490 ppm) support the differences in level. Combined sewer samples were 2 ppm for SM-3 and 1 ppm for SM-2. FC-143 concentration in water at these levels does not present an exposure hazard. (Table 1)

C. Yarn

Sintered yarn had non-detectable levels of FC-143. [redacted] and [redacted] products, where the yarn is dipped in the dispersion and dried, had levels ranging from 200-700 ppm. This variability is due to the uneven application and to the probable adsorption of the FC-143 [redacted] itself. Soxhlet extraction with methanol did not significantly improve recovery (Ref. 2).

D. Blood

The TLV for FC-143 in blood is 0.4 ppm. No blood samples from exposed personnel exceeded 0.02 ppm. Most had non-detectable levels. Method lower detection limits was ~ 0.02 ppm.

E. Disposition in the Process

Analysis has shown that ~10% of the FC-143 is washed off the yarn in spinning (Ref. 1). However, none is found on sintered yarn. Previous work (Ref. 3) has shown that yarn heated at 175°C has no extractable FC-143 remaining after 30 minutes. At 200°C, there is none after 10 minutes. This is because FC-143 can be removed thermally by decarboxylation, i.e., decomposition to CO_2 and the perfluoroseptamonohydride (Ref.. 4, 5). To confirm this, ~6 feet of [redacted] yarn was pulled into [redacted] tubing which was used as a GC column. The GC oven was temperature programmed from 125-250°C. The tube was purged with nitrogen and the off-gas analyzed by IR in a 10 cm. cell. The Nicolet MX-1 FTIR was set to run repetitive 1-minute scans. The [redacted] tubing itself was stable at these temperatures.

Two absorbance peaks appeared ~5 minutes after the 150°C oven temperature was passed. The hold-up time in the tubing from the GC to the IR cell and its volume was ~ 5 minutes at 20 ml/minute N_2 flow. A twin peak at 2340 cm^{-1} (CO_2) and a smaller C-F peak at ~ 1250 cm^{-1} were seen. These peaks increase in size until after ~ 5 minutes at 250°C, they begin to decrease. No carbonyl peaks at 1700-1750 cm^{-1} appear which would have indicated evaporation of the FC-143 (Figure 2).

Although these scans show that decarboxylation - not evaporation - occurs, they did not show whether the perfluoro by-product was the olefin (perfluoroheptene) or the hydride (perfluoro monohydride). Some concern was voiced over the potential hazard from the olefin. To determine the identity of the byproduct, an excess amount of the FC-143 sodium salt was mixed with Triton X-100 and poured into the [redacted] GC tube. The temperature was programmed from 150-225°C. The scans show a peak at 3010 cm^{-1}, which is indicative of the hydride. There is no evidence of the olefin which would have absorbed at 1790 cm^{-1} (Figure 3).

In order to determine the completeness of the decarboxylation, Draeger tubes were used. The gas evolved from the heating of the FC-143 sodium salt/Triton X-100 mixture was passed through the tube. This tube gave a specific colorimetric charge for CO_2. It showed 90+% of the theoretical CO_2 evolution.

III. METHOD DEVELOPMENT

The method used in this report for determining FC-143 levels is similar to that used in Reference I. The samples are extracted with methanol or freeze dried, esterified to get the volatile ester with BF_3, the reaction quenched with water, the ester extracted into hexane and the hexane injected in a GC. A new GC column packing (OV-202) was used to get better sensitivity. FC-143 is not a pure compound and consists of at least 5 identified isomers (Ref. 6). The OV-202 column consolidates these different compounds into one peak. A new Hewlett/Packard 5880 GC with a Ni_{63} electron capture detector was used to increase the method sensitivity to the low ppb range. A typical chromatogram is shown in Figure 4.

The following GC conditions were adopted for use:

Carrier Gas	Argon/Methane 5% @ 20 ml/Minute
Injection Temperature	180°C
Column Temperature	100°C
Detector Temperature	300°C
Sample Size	2 µl

External standards were used in each run and their peak area in the calculation of the sample concentration. Precision was ± 10% for any sample (Table 2).

A. Air Analysis

Air samples were taken for one hour at a rate of 3 liters per hour in 10 ml of water in an impinger. One ml of water was freeze dried and 1 ml BF_3/methanol added for esterification. After heating in sealed bottles for 15 minutes, the solution is quenched with water and 1 ml hexane added. The ester is extracted into the hexane and injected in the GC. Two micro liter samples were used. Method was sensitive to 0.3 ppb level, which is below the 0.5 ppb proposed TLV.

B. Yarn

Sintered yarn and [redacted] yarns were analyzed identically. Five ml of methanol was added to one gram of yarn. After 10-15 minutes, one ml was removed, and one ml BF_3/methanol esterfying reagent added. This was heated for 15 minutes, water quenched, extracted with hexane and the hexane injected into the GC. The [redacted] yarns had considerable variance between samples. This was due to either uneven application or to adsorption of the FC on the [redacted] itself. Soxhlet extraction with methanol did not reduce this variability. (Ref. 2).

C. Blood

Blood was analyzed identically to the water from the impinger air samples. S. Stafford, of PPD, prepared a series of spiked blood samples as an interlab check. These were analyzed with a precision of ± 10%. (Table 2) A set of spiked blood samples prepared at PCD were run with good precision.

IV. THERMAL TREATMENT

Thermal treatment can effectively remove the FC-143, but it is not the prime candidate because of deleterious effects on yarn properties. Previous work (Ref. 3) has shown that [redacted] yarns heated at 175°C for 30 minutes or at 200°C for 10 minutes no longer have any extractable FC-143. IR scans of off gas from yarn heated from 150° to 250°C confirm the presence of a fluorocarbon gas by the absorption peak at 1250 cm^{-1}. The presence of the CO_2 peak at 2350 cm^{-1} confirms CO_2 evolution, i.e., the FC is decarboxylating. There is no carboxyl peak at 1700-1750 cm^{-1} which would show evaporation. Further study of the gases evolved from 150-200°C from Triton X-100/FC-143 sodium salt mixture in a 5:1 ratio, show the presence of a C-H absorbance peak at 3010 cm^{-1} (Figure 3) This shows that the fluorocarbon yarn evolved in decarboxylation is the perfluoromonohydride. There is no evidence of an olefin (C = C, 1790 cm^{-1}) which is a possible decomposition product. The Triton supplies the necessary hydrogen to form the hydride (Ref. 4, 5). Analysis of the completeness of the decarboxylation was made by passing known amounts of off-gas through a Draeger tube. The tube, specific for CO_2, uses a color change to volumetrically measure the CO_2 evolved. The amount was 90 ± 10% of the theoretical, showing that no other major reaction occurs during thermal treatment.

REFERENCES

1. Forte, M. A. - SpAR 79-3.

2. Forte, M. A., Mokhtar, A. M. - PCD Monthly, April, 1981.

3. Forte, M. A., Mokhtar, A. M. - PCD Monthly, May, 1981.

4. LaZerte, J. D; JACS: 75, 4525 (1953).

5. Meschke, R. G. - PCRD-58-131.

6. Stafford, S. S. - PPD Analytical Method, "Determination of C_8 Fluorosurfactant in Water".

APPENDIX

TABLE I

I. [redacted] SPINNING IN-AIR SAMPLES

	LOCATION	LEVEL
1.	SM-2 - Roll 6	Non-Detectable (<0.3 ppb)
2.	SM-2 - Roll 23	Non-Detectable (<0.3 ppb)
3.	SM-2 - Roll 6	Non-Detectable (<0.3 ppb)
4.	SM-2 - Roll 23	Non-Detectable (<0.3 ppb)
5.	Operator's Desk	Non-Detectable (<0.3 ppb)
6.	Between SM-2 and 3, Roll 12	Non-Detectable (<0.3 ppb)
7.	SM-2 - Between Bath and Wash Module	Non-Detectable (<0.3 ppb)
8.	SM-3 - Between Bath and Wash Module	Non-Detectable (<0.3 ppb)
9.	SM-3 - Roll 6	Non-Detectable (<0.3 ppb)
10.	SM-3 - roll 23	Non-Detectable (<0.3 ppb)

Samples taken at estimated position of operator's head during work.

II. [redacted] SPINNING FC-143 WATER/YARN LEVEL

SM-2	Wash Water	1.2 ppm
	Sewer	1 ppm
	Regenerated Yarn	49 ppm
	Sintered Yarn	ND
SM-3	Wash Water	4.0 ppm
	Sewer	2.0 ppm
	Regenerated Yarn	60 ppm
	Sintered Yarn	ND

TABLE II

GC PRECISION

SAMPLE	GC COUNTS/THOUSANDS
5 ppm FC-143	232.5, 241.0, 234.1
1.0 ppm FC-143	63.2, 62.3, 65.2, 61.6, 59.0
0.5 ppm	33.4, 34.3, 38.8, 39.5
0.25 ppm	17.5, 15.5, 19.1, 18.4

BLOOD SAMPLES

ILC with PPD/S. Stafford

SAMPLE	SPIKED LEVEL (ppm F)	MEASURED LEVEL (ppm F)
1	.50	.56
2	1.5	1.9
3	Blank	.01
4	.027	0.026
5	0.17	0.145
6	1.0	1.2
7	Blank	.009
8	.054	.047
9	.34	.314
10	.054	.059

FIGURE 1

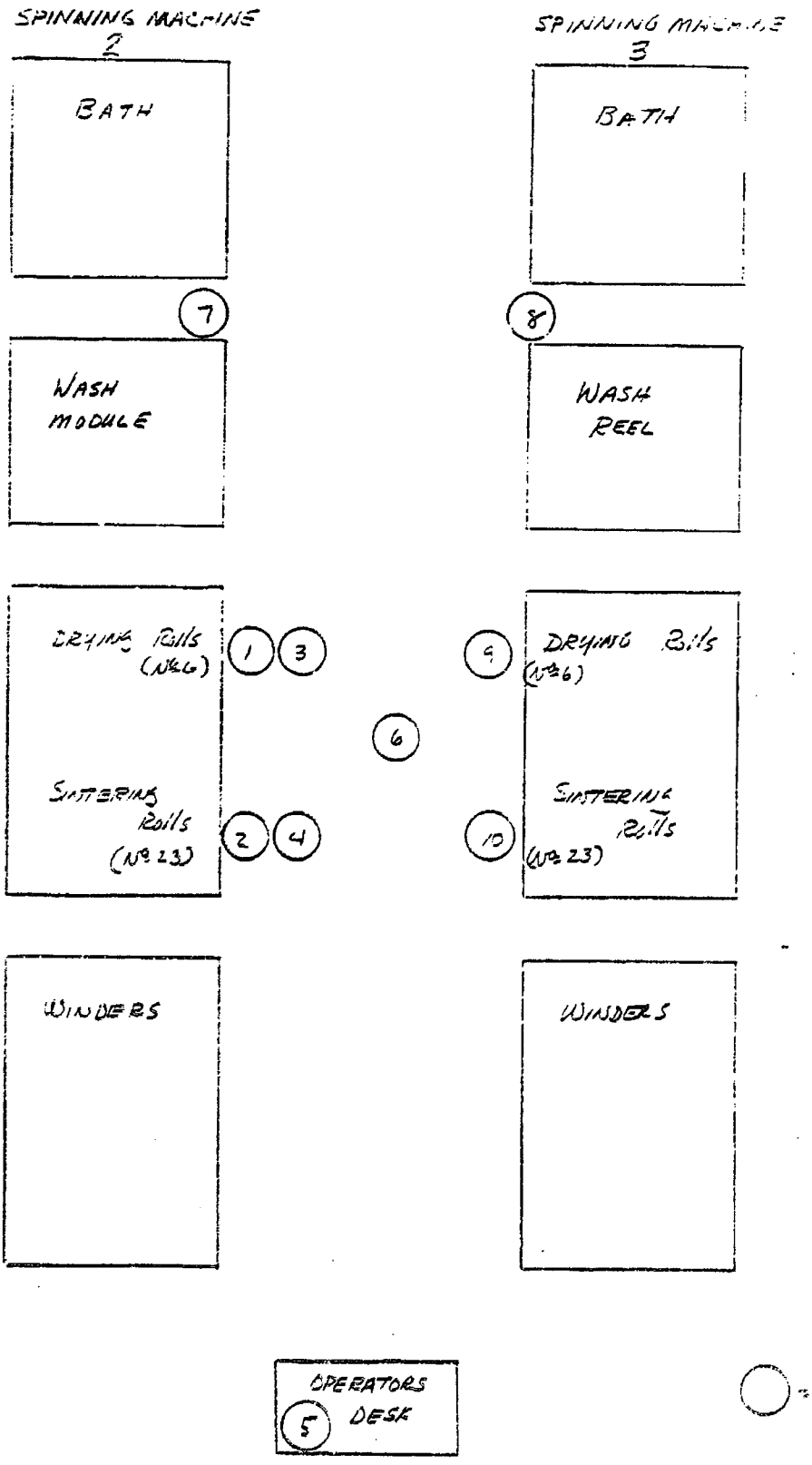
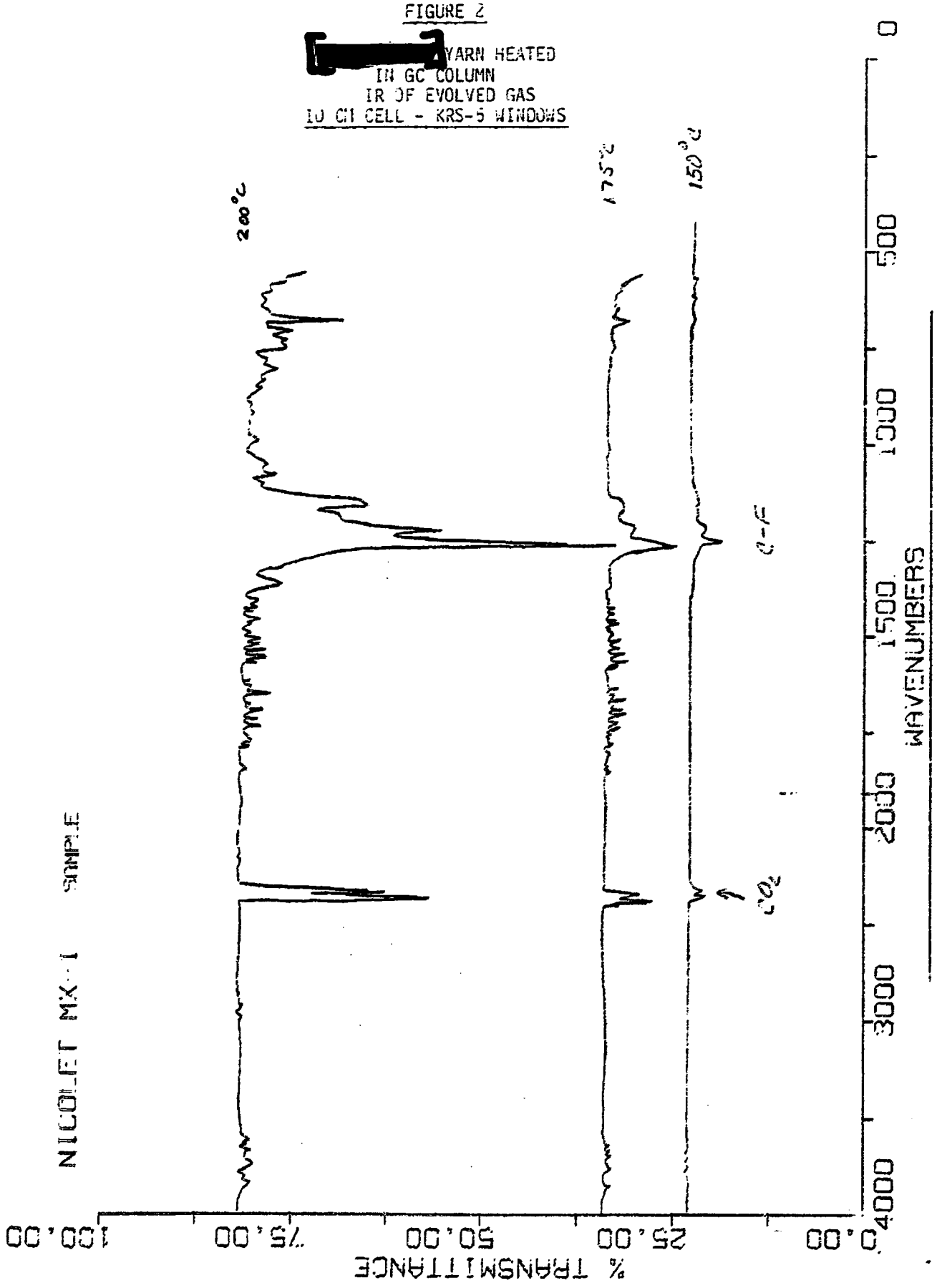


FIGURE 2

[redacted] YARN HEATED
IN GC COLUMN
IR OF EVOLVED GAS
IN CI CELL - KRS-5 WINDOWS



Company Sanitized. Does not contain TSCA CBI

CONCORD, CALIFORNIA
CHART NO. ZF-10T

FIGURE 3

FC-143 SODIUM SALT & TRITON X100
1:5 RATIO
EVOLVED GAS IR ANALYSIS
PERFLUOROMONOHYDRIDE ABSORBANCE PEAK

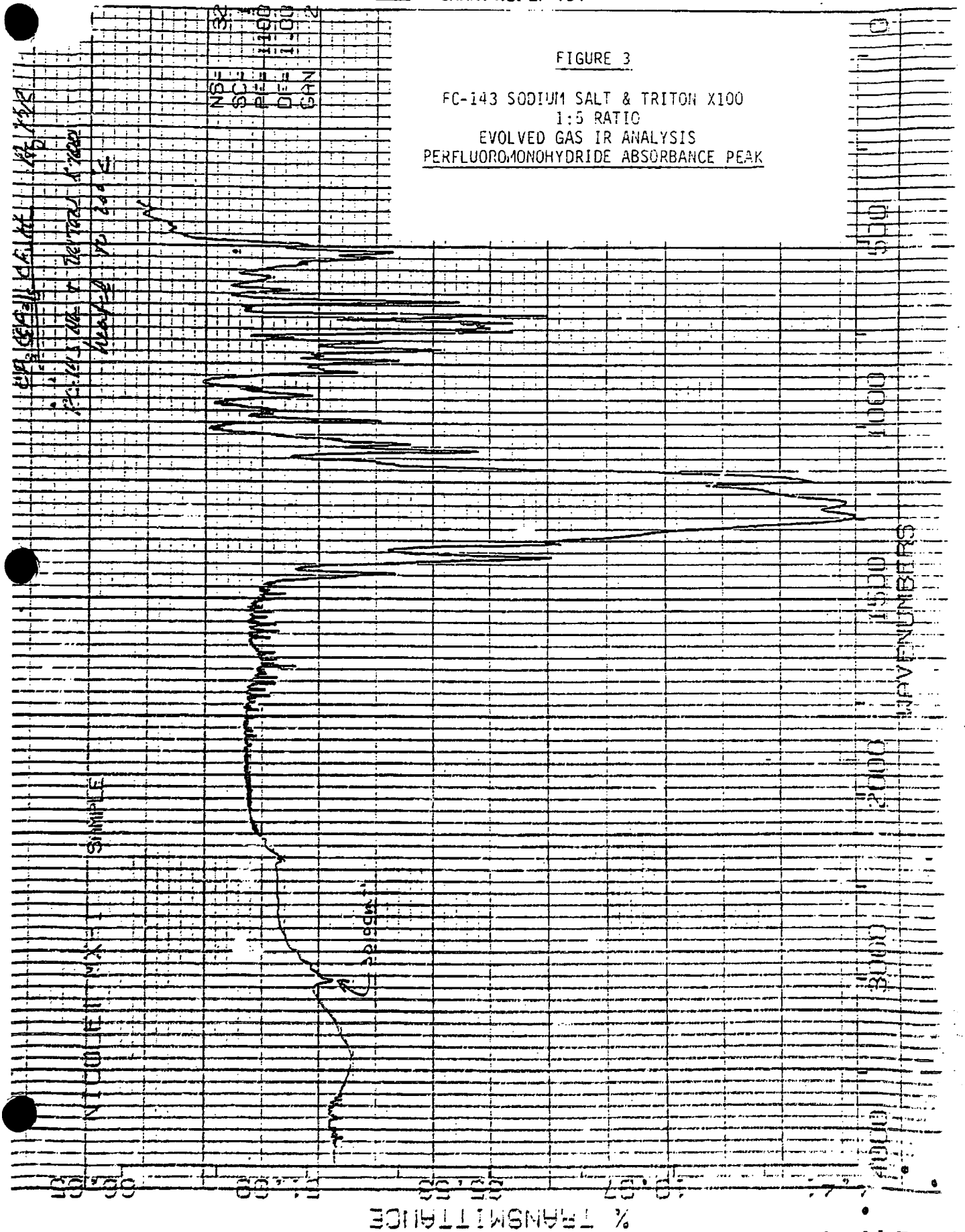
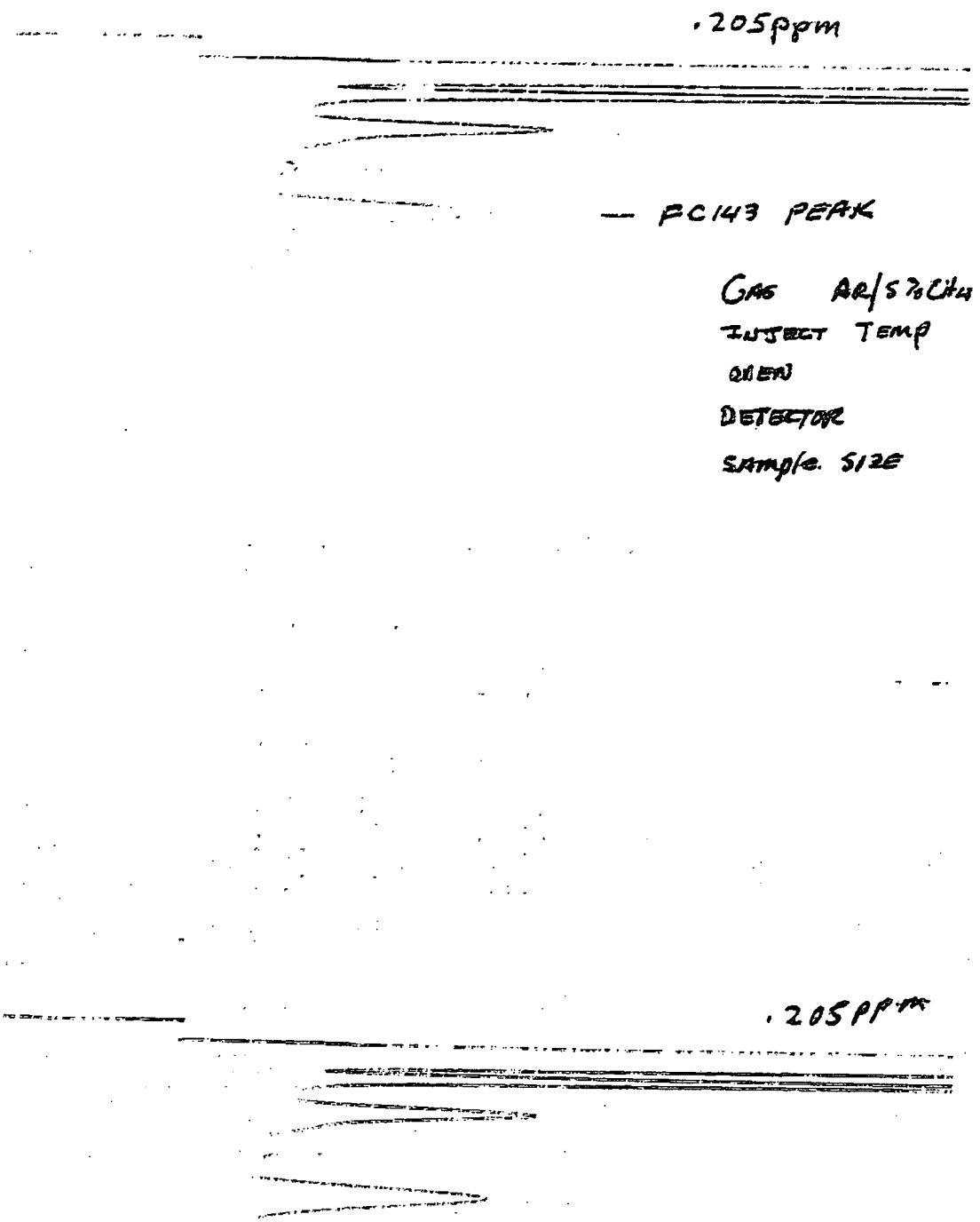


FIGURE 4

GC RUN OF FC-143
STANDARD 0.205 PPM



45 REV 12-79

ATTACHMENT I

cc: A. J. Dahl - 353
L. J. Papa - 269
W. C. Percival - 333
T. K. Wu - 333
Pral File
I.C.

E. I. DU PONT DE NEMOURS & COMPANY
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WILMINGTON, DELAWARE 19898

POLYMER PRODUCTS DEPARTMENT
EXPERIMENTAL STATION

ANALYTICAL REPORT

December 17, 1981

M. A. FORTE
SPRUANCE, TEXTILE FIBERS
RICHMOND, VA.

DETERMINATION OF PERFLUOROOCTANOATE IN AQUEOUS AIR IMPINGER SAMPLES
(Job No. 811-601; PRAL Nos. 81-2832-2835; Notebook No. E26162)

As reported by phone 7/2/81, the four aqueous air impinger samples submitted 6/81 were analyzed for perfluorooctanoate (C_8) using the gas chromatographic method ER-749. Results and sample identification are as follows:

Sample PRAL No		Identification	GC Analysis Date Analyzed	ug FC-143/mL (a)
81-2832		#1, Stack at Outlet	6/29-7/2/81	n.d. (b)
81-2833	(INSIDE)	#2, Stack at Inlet	"	0.032
81-2834		#3, Outlet of Dryer	"	n.d.
81-2835		#4, Inlet to Teflon Induction Dryer	"	n.d.

(a) FC-143 (3M ammonium perfluorooctanoate) was used as calibration standard, and C_8 concentration calculated as the salt. The lower limit for quantitation was 0.007 ug/mL.

(b) Chromatograms for sample #1 differed slightly in appearance from #3, #4, and the blanks; a longer air sampling time might have given a detectable level of C_8.

S. S. Stafford

jah

Key Words:
Perfluorooctanoate
FC-143
Air Analysis
GC

ATTACHMENT II

METHOD TM-0405-82T
DATE 3/16/82
PAGE 1 OF 10

DU PONT FIBERS PRODUCTS

[redacted]

FC-143 ANALYSIS

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Company Sanitized. Does not contain TSCA CBI

1. SCOPE

1.1 This method is used to describe sample preparation and GC analysis for FC-143 in air, water, blood and on yarn.

2. PRINCIPLE OF METHOD

2.1 Samples from in air, water or blood are first concentrated by freeze drying. Yarn samples are soaked in methanol to extract the FC-143. Then the FC-143 is esterified by heating with BF_3/methanol to form the volatile methyl ester.

$$CF_3(CF_2)COO^-Na^+ \cdot CH_3OH \xrightarrow{BF_3} CF_3(CF_2)_6COOCH_3$$

The reaction is quenched with water and the ester extracted into hexane. The hexane solution is shot into a GC with an electron capture detector. The FC-143 concentration is determined based on peak area comparison to standards.

3. SIGNIFICANCE AND USE

3.1 This method is used to determine FC-143 [redacted] yarn, in air, in water and in blood. The applicable ranges are:

Yarn - 50 - 1000 ppm
Air - 0.3 ppb - 1 ppm
Water - 0.02 ppm - 25 ppm
Blood - 0.02 ppm - 25 ppm

4. INTERFERENCES

4.1 Water must be removed from the samples to prevent reaction with the esterfying agent.

5. APPARATUS

5.1 Gas Chromatograph - Hewlett Packard 5880 with Ni_{63} electron capture detector and 5880 terminal Hewlett Packard Company.

5.2 Argon/Methane Carrier Gas 95/57 - Burdett.

5.3 Syringe - Hamilton - 701 N - 10 µl - Supelco Co.

5.4 Telematic Air Sampler - Taylor Parker Co., Norfolk, Virginia, Cat. No. 158.

5.5 Batteries, nickel-cadmium, 8.4 volts - Taylor Parker Co., Cat. No. 84Ta.

5.6 Vicils - 5 ml, glass - Fisher Cat. #03-338B.

5.7 Erlenmeyer Flask - 250 ml Glass Stoppered - Fisher Cat. No. 10-047C.

5.8 GC Column 10 glass, packed with OV-202 on 100/120 Supelco port - Supelco Co.

5.9 Heater - Fischer #11-495-50 or equivalent.

5.10 Sand Bath with copper tubes - Spruance Process Control Development.

5.11 Pipets 1 ml - Fisher Cat. #13-650B; 5 ml - Fisher Cat. #13-650F.

5.12 Manometer - Fisher Cat. #11-292 or equivalent.

5.13 Lyopilizer - Fisher Cat. #10-269-41 or equivalent.

5.14 Freeze Dry Flasks (4), 350 ml - Fisher Cat. #10-269-53.

5.15 Vacuum Pump - Two Stage Model D-25 - Fisher Cat. No. 01-183-31 or equivalent.

5.16 Pipet Aspirator Bulb - Fisher Cat. #14-070B.

5.17 Impinger Tube - LG-6890-100 - Scientific Glass Co., Vineland, N.J.

5.18 [redacted] cap liners - Supelco.

5.19 Dewar Flask, 1 quart size - Fisher Cat. #10-195B.

5.20 Vacuum system trap - LC-11025-102 - Scientific Glass co., Vineland, N.J.

6. REAGENTS AND MATERIALS

6.1 BF_3/Methanol - Pesticide grade in glass ampoules - Supelco Cat. No. 3-3041.

6.2 Methanol - Fisher Cat. #LA-936.

6.3 Hexane - Fisher Cat. #LH-301.

6.4 Dry Ice - Richmond Dry Ice Company.

6.5 Acetone - Fisher Cat. #LA-17-5.

7. PRECAUTIONS

7.1 BF_3/Methanol - Wear leather gloves when initially breaking the glass ampoule. Wear rubber gloves when pouring or pipeting.

7.2 Wear rubber gloves when handling methanol or hexane. Keep away from flame.

7.3 Wear leather gloves when handling vials or heater and sand bath.

7.4 Wear leather gloves when handling dry ice.

7.5 Blood. Rubber gloves must be worn when handling blood. When finished with blood, heat with concentrated bleach in a hot oven at ~ 90°C for one hour, then flush down drain with water. Use bleach in ~10:1 ratio over blood. Rinse all pipets, beaker and other equipment with bleach.

8. SAMPLING

8.1 In-Air

8.1.1 Place 10 ml DI water in an impinger tube. Set tube in Telematic and set turner for one hour. Flow should be set at 3 liters/minute. Set Telematic in area to be sampled.

8.2 Yarn

8.2.1 No special sampling required.

8.3 Blood

8.3.1 Obtain the necessary blood samples from Medical. Store in refrigerator until ready for use. See 7.5 for disposal.

9. CALIBRATION AND STANDARDIZATION

9.1 FC-143 Standards

9.1.1 Weigh 0.1000 g of FC-143 into a 100 ml volumetric flask. Make to volume with methanol. This is the 1000 ppm standard.

9.1.2 Take 1 ml of 9.1.1 and dilute to 50 ml with methanol. This is the 50 ppm standard.

9.1.3 Take 5 ml of 9.1.2 and dilute to 10 with methanol. This is the 25 ppm standard.

9.1.4 Take 1 ml of 9.1.1 and dilute to 1000 ml with methanol. This is the 10 ppm standard.

9.1.5 Take 5 ml of 9.1.4 and dilute to 10 ml with methanol. This is the 5 ppm standard.

9.1.6 Take 1 ml of 9.1.4 and dilute to 10 ml with meethenol. This is the 1 ppm standard.

9.1.7 Take 1 ml of 9.1.5 and dilute to 10 ml with methanol. This is the 0.5 ppm standard.

9.1.8 Take 1 ml of 9.1.6 and dilute to 10 ml with methanol. This is the 0.1 ppm standard.

10. PROCEDURE (References Attachment I, GC Conditions and Attachment II, Lyophilizer Operation)

10.1 Yarn - Sintered and [redacted]

10.1.1 For sintered yarn, weigh 0.5 g to 0.0001 and put into a glass vial. Add 1 ml methanol. For [redacted] yarn, weigh 0.05 g yarn to 0.0001 g, add to a glass vial and add 1 ml methanol.

10.1.2 Add 1 ml BF_3/Methanol. Heat in sand bath for 15-20 minutes at sand temperature of 65-70°C.

10.1.3 Remove from bath and allow to cool. Then add 1 ml DI H_2 and 1 ml hexane. Then shake for 30 seconds.

10.1.4 Allow layers to separate and shoot sample on GC (See Attachment I). FC-143 peak RT is ~2.9 minutes.

10.1.5 Shoot standards (9.1) that bracket the expected sample level.
Sintered - 0.5, 1.0, 5.0 ppm
[redacted] 10, 25, 50 ppm

10.1.6 See 11.1 for calculation.

10.2 In-Air Samples

10.2.1 Pipet one ml of the water from the impinger tube into a clean, glass vial. Carefully place it in a Freeze Dry Flask, put on the rubber top and hook it to the Lyophilizer (Attachment II). Leave on the Lyophilizer until all the water is gone (~2-3 hrs.).

10.2.2 Remove the vial from the flask and add one ml of BF_3/Methanol by pipet. Put a Teflon® gasket in the vial top, screw it tight and place in the sand bath for 15-20 minutes at 65-70°C.

10.2.3 Remove from the bath and allow to cool. Then add 1 ml DI H_2O and 1 ml Hexane. Then shake for 30 seconds.

10.2.4 Allow the layers to separate and shoot the hexane layer on the GC. (See Attachment I).

10.2.5 Shoot the standards that bracket the sample (0.1, 0.5, 1.0 ppm).

10.2.6 See 11.2 for calculation.

10.3 Water

10.3.1 Pipet 1 ml of water into a clean, glass vial. Then follow 10.2.1 - 10.2.5.

10.4 Blood (See 7.5 for handling)

10.4.1 Pipet 1 ml of blood into a clean, glass vial, freeze dry and run as in 10.2.1 - 10.2.5.

11. CALCULATION

11.1 Yarn

$$\text{FC-143 ppm} = \frac{A}{B} \times \frac{C}{W}$$

A = Sample GC peak area

B = Standard GC peak area

C = Standard concentration, ppm

D = Weight of sample

11.2 Air, Water, Blood

$$\text{FC-143 ppm} = \frac{A}{B} \times C$$

A = Sample GC peak area

B = Standard GC peak area

C = Standard concentration, ppm

12. PRECISION AND ACCURACY

12.1 Precision is $\pm$ 10%.

13. REFERENCE

13.1 PPD Analytical Method - Determination of C_8 Fluorosurfactant in Water GC, S. S. Stafford.

14. KEY WORD INDEX

14.1 FC-143 - in yarn, water, air and blood.

14.2 GC analysis.

ATTACHMENT I

HP 5880 GC CONDITIONS

Carrier Gas	Argon/Methane	95/5	20 ml/minute
Injection Temperature	180°C		
Oven Temperature	100°C		
Detector Temperature	300°C		
Sample Size	2 µl		
Column	6' Glass 1/8" OD		
Packing	OV-202 on 100/120 Supelcoport		

GC Terminal Conditions

- Run time - 0.1 minutes Tan Skim.
- Attenuation 2^6 (adjust as necessary for peak size)

FREEZE DRYING SAMPLES

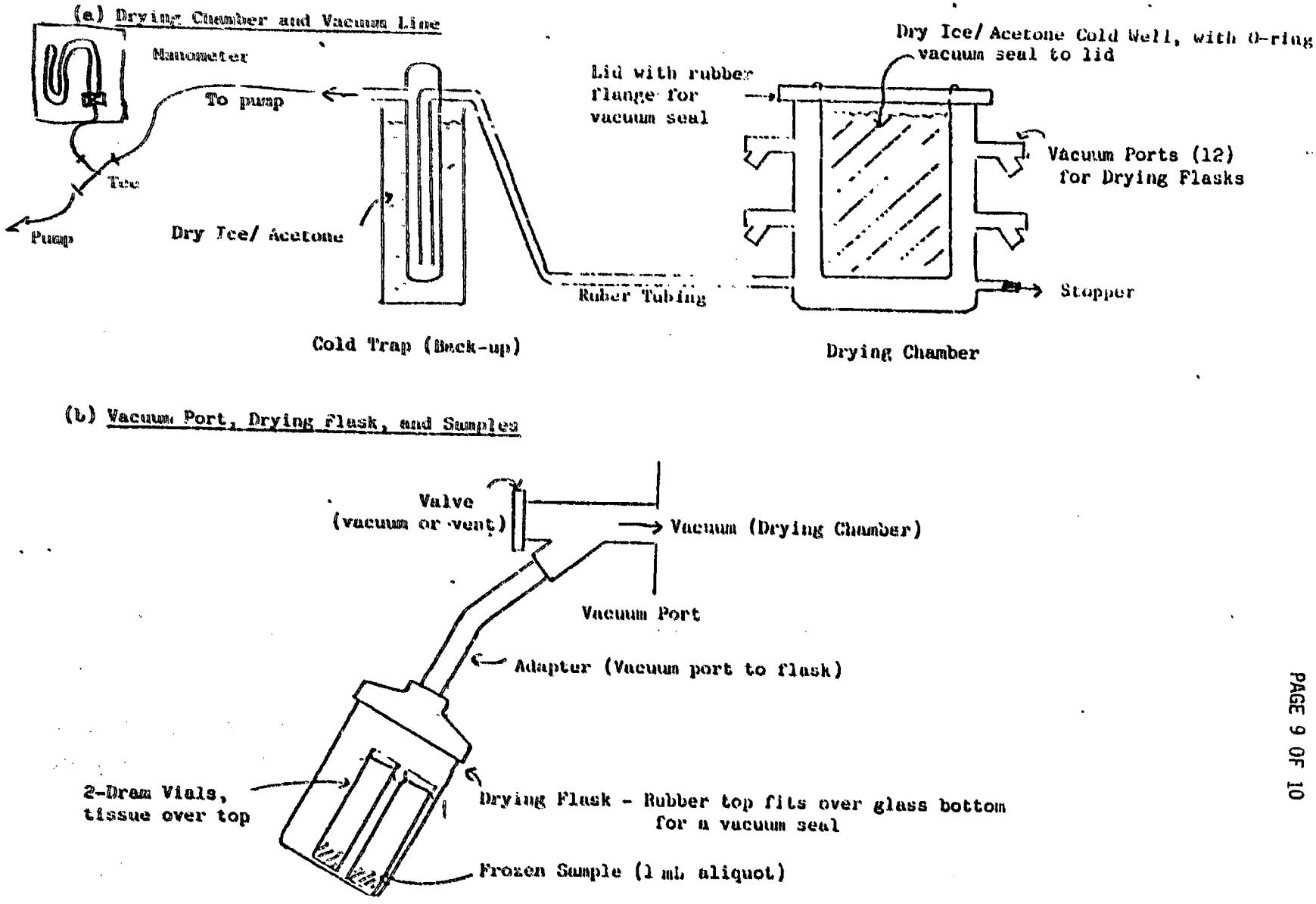
Equipment Setup

1. Put ~ 500 ml of acetone in the lyophilizer tank.
2. Carefully add enough dry ice to keep it as a solid. Replace lid.
3. Add ~ 50 ml acetone to vacuum trap Dewar. Set the vacuum trap in the Dewar and add dry ice to pack around it.
4. Hook up heavy wall rubber tubing from the lyophilizer to the trap to the vacuum pump. Tee in the manometer between the trap and pump.

Sample Setup

1. Take the samples in the glass vials and dip them in the lyophilizer dry ice/acetone mixture. Keep them immersed long enough for complete freezing.
2. Place the frozen vials in one of the freeze dry flasks. Put a fitler in the top of the rubber top, place on the flask and connect to the lyophilizer with the metal neck.
3. Open the connection to vacuum. Only those ports being used can be open to vacuum. All others must be on vent.
4. Cut on the pump, open the manometer conenctions. Vacuum must be ~ 0.1 mm.
5. After freezing, shut off manometer, carefully open sample lyopilizer port and cut off port.

FIGURE 1 - APPARATUS FOR FREEZE-DRYING SAMPLES



TEST METHOD APPROVAL

[redacted] - FC-143 Analysis"
TM-0405-82T

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Spruance
Site

Site	Authorized By	For (Organization)	Date
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Spruance	Chemist	Process Control Development	3/24/82
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ATM001.7

ABSTRACT

The FC-143 (ammonium perfluoro octanoate) surfactant used in the [redacted] polymerization process was a suspected teratogen. Methods were developed, and its level measured in air, water, blood and yarn. In air, water and blood levels were well below TLV's. None was present on sintered yarn, but I&T yarn, which is coated with dispersion, has 200-700 ppm levels. It was shown that FC-143 dicarboxylates when heated to 200°C giving off CO_2 and perfluoroseptamonohydride.

INDEXING SUBJECTS

1. Measurement of perfluorooctanoic acid (FC-143) in air, water, yarn.
2. Ammonium perfluorooctanoate dicarboxylation of.
3. Sodium perfluorooctanoate dicarboxylation of.

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