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Analysis of Surface Tris (2,3-Dibromopropyl) Phosphate on Chlorobutyl Rubber SCAPE Suits

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INTRODUCTION

The Self-Contained Atmospheric Protective Ensemble (SCAPE suit) is a coverall garment which provides personnel protection from toxic and vesicant substances, both liquid and vapor, such as hypergolic propellants. The suit is made from a chlorobutyl rubber coated "Nomex" aramid fabric and is worn over thermal underwear with a liquid air backpack inside the suit to provide air for breathing, suit pressurization, and ventilation.

Shortly before the U.S. Consumer Product Safety Commission banned (1)[1] the sale of children's clothing containing the flame-retardant chemical tris (2,3-dibromopropyl) phosphate, now commonly referred to as Tris, Kennedy Space Center was notified by the SCAPE suit manufacturer that the suits contained Tris, a suspected human carcinogen. The Tris was compounded directly into the chlorobutyl rubber and constituted approximately seven percent by weight of the coating.

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[1] Numbers in parentheses indicate Bibliography references.

Since the Consumer Product Safety Commission found that Tris could be absorbed through the skin, an investigation of the Tris present on the surface of SCAPE suits was conducted with the goal of providing a method of removing or isolating the Tris to reduce the hazards to the users so that the hundreds of suits available at KSC could still be used. However, the amount of Tris actually present on the surface of the suit, where it could be removed by body contact, was not known. No existing procedures for sampling and analyzing surface Tris under these conditions were found. It was therefore necessary to develop these methods.

The investigation was broken into five specific areas:

1. Develop a quantitative analytical method which is specific for Tris at low concentrations.

2. Determine if Tris is present on SCAPE suits now in use.

3. Provide an operational procedure for removing Tris from the surface.

4. Provide a barrier procedure by coating or lining the suits to provide protection between the Tris-containing-surface and the body.

5. Monitor the prescribed procedure under normal operational conditions for a period of time sufficient to see if the migration of Tris to the surface would create a problem in the future.

SAMPLING AND ANALYTICAL PROCEDURES

Sampling

Because of the intense investigation of Tris in children's sleepwear, the current literature details methods of sampling for this compound (2). However, the methods all involve solvent extraction of swatches of the fabric. These methods were not applicable to the determination of Tris in the nonporous SCAPE suits because they would have destroyed the suit. Consequently, an alternate method had to be developed.

The transfer of Tris from the SCAPE suit to the wearer, if it occurs, is probably by body contact exclusively; ingestion, as occurred when children chewed or sucked on their nightwear, is unlikely. The sampling method, therefore, was designed to simulate body contact with the suit. A 2.54 cm x 2.54 cm piece of absorbent cotton was wet with 0.35% sodium chloride solution (or normal saline) and then squeezed to remove excess liquid, approximating the sweaty undergarments wearers often have after periods in the SCAPE suits. Suits or fabric swatches were wiped with the damp cotton using moderate to heavy pressure and parallel strokes to cover the selected area (usually 15.24 cm x 15.24 cm) once in the horizontal direction and once in the vertical.

Further treatment of the cotton wipe was dictated by the analytical procedure to be used. For the X-ray analyses, the wipe was simply oven dried at 120°C. For the work with gas and liquid chromatography, Tris was extracted by covering the cotton with chloroform in a 50 ml beaker and placing the beaker in a hot-water-filled sonic cleaner for 10 minutes. The

cotton was then removed from the solution and rinsed with chloroform three times, draining all the rinses back into the 50 ml beaker. The beakers were placed in a hood and allowed to air dry. When dry, the residue was transferred to a 5 ml beaker by pipeting two portions of 2 ml each and one portion of 1 ml of chloroform into the 50 ml beaker and pouring into the 5 ml beaker after each wash. The 5 ml beaker was then air dried and held for analysis.

Analytical Procedures

Three analytical procedures were tested in the course of this work. The initial technique was an adaptation of X-ray fluorescence methods for the determination of Tris in sleepwear fabrics. The Tris content of the dried cotton wipe was determined by measuring the intensity of the Br Kα X-ray emission and reading the corresponding Tris concentration from a calibration curve. The limit of detection by this method was approximately 1 μg Tris. This procedure assumed that all the bromine on the cotton wipe was present as Tris, since Tris was the only bromine compound in the rubber formulation.

The second procedure, gas chromatography, was undertaken to confirm the results of X-ray fluorescence by measuring the molecular Tris rather than the element bromine. The Gas Chromatographic Analysis was performed on a Hewlett-Packard 5750 chromatograph using an electron capture detector (EC). The chromatographic column was a .635 cm O.D. glass tube 30.48 cm long packed with 25.4 cm of 5% OV 1 on Chromosorb GHP, 80 - 100 mesh, with the end voids filled with glass wool. The injection port was operated at

265°C, the column isothermally at 260°C, and detector at 295°C. Helium carrier gas was used at a flow rate of 200 ml/min. with 90 ml/min. split to the EC detector and 110 ml/min. to the flame ionization detector (FID) for dumping purposes only. The FID was operated during early runs and a peak corresponding to the EC peak used to measure Tris was observed for moderate to high concentrations, but it lacked the sensitivity required to measure the low levels which were encountered. Ten percent Methane in Argon was used as an EC purge gas at approximately 200 ml/min. The peak used to quantitate the Tris present eluted at 5 min. A series of standards were prepared ranging from 39 µg/µl to 0.039 µg/µl in chloroform. Sensitivity and reproducibility were good in this low range and averaged .0034 µg/sq. cm. Chromatograms at three concentrations are shown in Figure 1. The samples were diluted for injection by dissolving the contents of the 5 ml beaker in 100 µl of chloroform just prior to injection; a 4 µl sample quickly withdrawn from the dilution was injected into the gas chromatograph (GC). The areas of the 5 min. peaks were measured on all samples and standards with an Ott planimeter. Sample results were calculated as the total number of µg's of Tris removed from the surface of the sampling area.

The third analytical procedure tested, liquid chromatography, was performed on a Waters Associates ALC-202 Liquid Chromatograph equipped with a Differential UV Detector set at 254 nm. The chromatographic column contained 30 cm of µ Bondapak; the mobile phase was 80% methanol/20% water at a pressure of 850 psi and a flow rate of 1.3 ml/min. The instrument was operated at 25°C. The peak used to quantitate the Tris eluted at 13.2 min. The sensitivity was 14.3 µg/ sq. cm. The samples were diluted by dissolving

the contents of the 5 ml beaker (from the chloroform extraction) in 100 µl of methanol just prior to withdrawing a 50 µl portion for injection into the liquid chromatograph.

Comparison of Analytical Techniques

The comparison of X-ray and GC was made by submitting the wipes which had previously been analyzed by X-ray to the chloroform extraction procedure discussed in the preceding section. This comparison showed that the X-ray results for Tris were much greater than those from GC although standards run by both methods gave similar results. In order to resolve these differences and also to obtain some information about differences between cured and uncured rubber, formulations similar to the SCAPE suit coating were prepared, curing one batch and leaving another batch uncured, and analyzed by both X-ray and GC. Both analytical methods showed that the saline wiping procedure removed more Tris from the uncured material than from the cured. However, the differences between X-ray and GC which were noted earlier persisted. Repeated Soxhlet extractions of the cotton wipe with chloroform failed to reduce the amount of Tris indicated by X-ray fluorescence. Since Tris is known to be soluble in chloroform, this behavior indicated the bromine detected by X-ray was not part of the Tris molecule. Subsequent extraction of the cotton wipe with water reduced the amount of Tris indicated by X-ray to zero. Table 1 contains the data obtained in comparing cured and uncured materials measured by X-ray and GC. These data indicate that the X-ray method is not an appropriate analytical technique for this investigation.

The water extract which removed the bromine-containing material from the cotton wipe was evaporated to dryness, yielding a nearly colorless, glassy residue. Analysis of this material by infrared spectroscopy and induced electron emission spectrometry indicated that it was a brominated starch compound, presumably formed during the mixing and curing of the rubber. Because of this finding, X-ray fluorescence was abandoned as a method of analysis for this investigation.

Liquid chromatography confirmed the gas chromatographic analysis for Tris where quantities being observed were high, but was not sensitive enough to measure Tris at the low levels required in this investigation. Therefore, all further analyses in this study were done using the gas chromatographic procedure.

PRESENCE OF TRIS IN CURRENT SCAPE SUIT OPERATIONS

Kennedy Space Center has an inventory of several hundred SCAPE suits, all of which were manufactured in either 1966 or 1971. In normal operations, the suits are showered inside and outside with trichlorotrifluoroethane (Freon 113) after each use; the Freon used is held in a sump for reuse. Since Tris was found on the surface of unused SCAPE suit fabric in preliminary tests, several SCAPE suits and the Freon in the sump were analyzed for Tris.

Analysis of the SCAPE suits, shown in Table 2, indicated the 1966 suits had < 4.3 μg Tris/1000 sq. cm present while the 1971 suits had amounts

between 4 and 40 µg/ 1000 sq. cm. The Freon in the SCAPE suit sump was then analyzed for Tris and found to contain 5 mg/ℓ, which would indicate that although the Freon could still have a cleaning effect, it might also redeposite Tris on the suits at the 4 to 40 µg/1000 sq. cm range.

Since the solubility of Tris in Freon 113 was not available, it was determined by finding the nonvolatile residue of a saturated solution and by analyzing the saturated solution by gas chromatography. Both methods indicated that approximately 5 grams of Tris would dissolve in one liter of Freon 113.

CLEANING PROCEDURES

Since textile manufacturers had been able to reduce the Tris concentration in children's sleepwear by machine washing with detergent (2), the surface of the SCAPE suit fabric was scrubbed by hand and swatches were washed in a washing machine to determine how much Tris remained after cleaning. In addition, several samples were dipped into beakers containing Freon 113 and Methanol to see if rinsing with solvent alone would reduce the surface Tris.

Hand Washing Test

A series of nineteen 15.25 x 15.25 cm samples removed from a new bolt of material was prepared to test the efficiency of several hand washing methods for removing Tris from the surface. These samples were scrubbed with the solutions shown in Table 3 using a 2.54 cm x 1.27 cm soft

bristle brush except where noted. The results indicated that scrubbing the surface of the suits with a soft bristle brush and hot water-detergent solution followed by a hot water rinse removed the detectable surface Tris. Rinsing along with Methanol or Freon 113 reduced the quantity of Tris on the surface significantly. No migration of Tris back to the surface was detectable within 30 days.

Machine Washing Test

Four 91.5 cm x 91.5 cm pieces of new suit material were washed in a commercial washing machine using a normal hot water washing cycle repeatedly. A 30.5 cm x 30.5 cm piece was cut from each sample after each washing, with the first 30.5 x 30.5 cm piece cut before any washing to provide a baseline. The results obtained (Table 4) indicate that machine washing the material removed the surface Tris in one washing and that Tris did not migrate back to the surface within seventeen days.

Although no detectable Tris migrated to the surface within 17 days after washing, slow migration is considered probable with the rate increasing if the fabric is flexed. To approximate the effect that the lifetime usage of the suits would have on the migration of Tris to the surface, one of the washed and analyzed samples (1 - 4) was flexed in an automatic flexing machine and re-sampled for Tris. The results, also given in Table 4, indicated that flexing does increase the Tris level slightly, but not to a significant amount over its lifetime.

BARRIER PROCEDURES

Coatings

The following four candidate coatings were evaluated as barrier materials to prevent transfer of Tris from the surface of the SCAPE suit to the skin of a user.

(1) Bostik 1177, Neoprene Adhesive, manufactured by United Shoe and Machinery Corp.

(2) Witco 349Y, Urethane Coating, manufactured by Witco Chemical Corp.

(3) Uralane 5753, Urethane Coating, manufactured by Furane Plastic Co.

(4) Uralane 8267, Urethane Coating, manufactured by Furane Plastic Co.

Each material was applied to 0.84 square meters of new suit material and cured according to the manufacturer's instructions. Specimens were cut from each of the four coated samples and subjected to the following tests:

(1) Adhesion Test, ASTM-D-1876

(2) Flexure Test, 180° Bend Over 1/8" Radius, 5000 Cycles

(3) Flammability Test, Federal Test Method Standard 191, Method 5904

(4) Hypergolic Compatibility, Exposed Two Hours to N_2O_4, N_2H_4, Monomethyl Hydrazine, and Unsymetrical Dimethyl Hydrazine

Based on the results of these tests which are summarized in Table 5, the Bostik 1177 Neoprene Adhesive was the only material of the four tested to meet all of the required physical properties.

Additional tests were then conducted on the Bostik Adhesive to investigate methods of applying it to SCAPE suit material and to assess its potential for preventing Tris from reaching a SCAPE suit user.

Based on numerous tests it was determined that application of the coating was feasible by either brushing or spraying. However, with either method, it was found that drastic thinning was necessary to obtain uniform coating films. One part coating to one part thinner by weight was found to be about the optimum thinning ratio.

In the neoprene coating tests, which were designed to determine the coating's ability to protect the suit user from Tris, washed samples of new suit material from the machine washing tests previously described were coated with the neoprene material. The level of Tris detected on the washed samples before they were coated was < 1.1 μg/1000 sq. cm. However, analysis after coating showed greater concentrations, Tests 1 and 2 (Table 6) indicating that Tris was possibly migrating when the solvent used as the coating thinner came in contact with the material surface. Wiping during the sampling operation appeared to remove the Tris.

Test 3 (Table 6) results contradicted the conclusions of Tests 1 and 2, in that no Tris was found after coating. A greater number of specimens were then prepared and sampled (Test 4, Table 6). The results again indicated that Tris migration took place when the coating solvent came in contact with the material, but in six of seven cases removal of Tris by sampling was sufficient to prevent re-migration through an additional coating.

An actual SCAPE suit (No. 957) was coated both inside and out using the spray method. Although there were problems such as "cobwebbing" of the coating, and a tendency for the coated suit fabric to firmly stick to itself in creases and folds, spraying was determined to be a feasible method of coating the SCAPE suits.

Prior to spraying the suit, designated areas were cleaned with various solvents (defined in Table 7) to determine their effect on the adhesion of the coating and to assess the ability of these solvents for removing surface Tris. Wipe samples were taken for chemical analysis after the suit had been cleaned by the various solvents and again after it was coated. Results of the chemical analysis, presented in Table 7, show that the amount of Tris detected was consistently low both before and after coating. This indicated that this particular suit, which was later learned to be of the 1966 vintage, had been through the Freon cleaning cycles a sufficient number of times to remove the Tris to a depth not penetrated by the thinning solvent used in the coating.

Suit Liner

A suit liner was also suggested as a means of providing a barrier between the suit and user. A prototype liner, designed to attach to the inside of the SCAPE suit with Velcro tape at the neck, wrists, ankles and entrance zipper area, was constructed of a 3 oz. neoprene coated nylon. The liner would provide adequate protection for the wearer, but would not protect the support personnel who handled and cleaned the suits. If the suit liner were adopted for SCAPE suit operations, it would be necessary to provide protective clothing for the suit handlers.

CONCLUSIONS AND OPERATIONAL TEST

Conclusions

Based on the data obtained from the laboratory experiments, it is obvious that the most economical, expedient, and thorough method of providing protection to the fuel handlers is to wash the Tris from the surface of the SCAPE suits. Suits already fabricated can be scrubbed down with a brush and a hot water detergent mixture and then rinsed. Material on hand to make new suits and repair those already fabricated can either be scrubbed in the same fashion or machine washed.

Further, it was decided, since the 1966 vintage suits had no Tris on the surface and the 1971 vintage had less Tris on the surface than the new material, that the operational Freon showering had been beneficial. Laboratory tests have also shown that rinsing with Freon 113 removes Tris from the surface without affecting the physical properties of the material. It was therefore recommended that the following steps be carried out before further use of the SCAPE suits:

1. All suits would be scrubbed with a hot water-detergent mixture and a brush, followed by thorough rinsing.

2. The Freon 113 suit shower would be dismantled, cleaned and flushed with fresh Freon. Samples would be taken of the filled sump to be sure that no Tris remained.

Operational Test Design

As the suits were used and showered as in prior operations, each 8th suit would be sampled inside and out by laboratory personnel and a Tris analysis performed on the wipe samples. Each time a suit was sampled, the Freon sump would also be sampled to determine the Tris concentration in the Freon in order to determine when to replace the used solvent.

Operational Test Results

This test began on August 28, 1977 and to date 101 suits of the 1966 vintage and 85 suits of the 1971 have been showered without the Tris level on the inside or outside of the suits reaching the detectable limit. The level of Tris in the Freon sump (Figure 2) is rising slowly with each additional set of washings. Initial indications are that when the 1971 vintage suits are involved in the set, the Tris level increases at a faster rate.

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TABLE 1

Tris Concentration On The Surface Of KSC
Formulated Material In μg/1000 sq. cm

	X-Ray Analysis Wipe Before Extract	G.C. Analysis $CHCl_3$ Extract	X-Ray Analysis Wipe After $CHCl_3$ Extraction	X-Ray Analysis Wipe After Water Extract
Cured	129	4.1	125	0
Uncured	159	108	30.1	0

TABLE 2

Analysis Of 1966 And 1971 Vintage Used SCAPE Suits

Manufacture Date	Suit Identification	Tris, µg/1000 sq. cm Inside Suit	Tris, µg/1000 sq. cm Outside Suit
3/66	#257	< 4.3	< 4.3
3/66	#258	< 4.3	< 4.3
3/66	#966	< 4.3	< 4.3
3/66	#957	< 4.3	< 4.3
3/71	#604	9.0	11.6
3/71	#615	37.9	34.9
3/71	#616	20.2	23.2

Average Tris Found On Unused Material: 59.1 µg/1000 sq. cm

TABLE 3

Hand WAshing Test

Tris (μg/1000 sq. cm) remaining on surface of 15.25 cm x 15.25 cm samples of new material after brushing or rinsing with the indicated solutions.

Untreated Surface	Hot Detergent Wash		3 Hot Detergent Washes
	Immediately	After 30 Days	
60.3	< 4.3	< 4.3	< 4.3
64.6	< 4.3	< 4.3	< 4.3
1.5	< 4.3		< 4.3

Methanol Wash & Hot Detergent	2 Methanol Washes & Hot Detergent
< 4.3	< 4.3
< 4.3	< 4.3
< 4.3	< 4.3

Dip In Methanol No Brushing	Dip In Freon 113 No Brushing
< 4.3	5.6
17.2	8.6

TABLE 4

Machine Washing

Tris Detected In μg/1000 sq. cm

Unwashed		1 Washing		2 Washings		3 Washings	
Sample #	Result	Sample #	Result	Sample #	Result	Sample #	Result
1 - 1	49.5	1 - 2	< 1.1	1 - 3	1.1	1 - 4	< 1.1
2 - 1	24.8	2 - 2	< 1.1	2 - 3	1.1	2 - 4	< 1.1
3 - 1	92.6	3 - 2	< 1.1	3 - 3	1.1	3 - 4	< 1.1
4 - 1	100.1	4 - 2	< 1.1			4 - 4	< 1.1

Sample Tested After 17 Days

Sample #	Result
1 - 2	< 1.1
2 - 2	< 1.1

Extreme Flexing Test
(180° Bend 5,000 Times)

Sample #	Area	Result
1 - 4	Inside Flex	2.0
1 - 4	Outside Flex	1.5

TABLE 5

Comparative Evaluation Of Coatings For Use As Barrier Material

	Uncoated Fabric	WITCO 349Y	Uralane 5753	Uralane 8267	Bostik 1177
Adhesion Test, T Peel, ASTM D 1876 (Kilograms/sq. cm)	-	.105	.527	.178	.352
Flexure Test (Cycles to Failure)	-	> 5000	> 5000	< 100	> 5000
Flammability (Federal Test Method Standard 191, Method 5904)	Passed	Failed	Failed	Passed	Passed
Hypergolic Compatibility					
N_2O_4	Slight discoloration, some coating removed.	Slight discoloration, blistering.	Severe blistering, cracking, discoloration.	Moderately severe discoloration, blistering.	Moderately severe cracking, some butyl removed.
N_2H_4	Slight discoloration.	Some blistering, cracking, slight discoloration.	No visible discoloration.	Slight cracking and discoloration.	No visible reaction.
MMH	Slight discoloration.	Some blistering, moderate discoloration	Slight blistering and discoloration.	Slight blistering and discoloration.	No visible reaction.
UDMH	Slight discoloration.	Severe blistering, moderate discoloration.	Slight discoloration.	Moderate discoloration.	Moderate discoloration and cracking.

TABLE 6

Neoprene Coating Tests

Tris Detected In µg/1000 sq. cm

Test 1	After Coating	Rewipe	Scrub Then Rewipe
Sample #1 - 3	5.4	< 1.1	< 1.1
Sample #4 - 7	9.7	< 1.1	< 1.1

Test 2	Before Coating	3 Coats Oven Cure	3 Coats Air Cure
Sample #4 - 4	< 1.1	4.8	
Sample #3 - 4	< 1.1		2.0

TABLE 6 (Cont'd)

Tris Detected In µg/1000 sq. cm

Test 3	Before Coating	After First Coat 64 Hrs. Air Cure	After Second Coat 4 Hrs. Air Cure	After Second Coat 4 Hrs. Vacuum Cure
Sample #1 - 5	< 1.1			
2 - 5	< 1.1	< 1.1		< 1.1
3 - 5	< 1.1	< 1.1	< 1.1	
4 - 5	< 1.1	< 1.1	< 1.1	

Test 4	First Coat Air Cured For 72 Hours Then Recoated		
	After Second Coat 66 Hrs. Air Cure	WIPED	After Third Coat 30 Hrs. Air Cure
Sample #1 - 6	1.9	FOR	3.0
2 - 6	2.6	SAMPLING	< 1.1
3 - 6	4.1	BETWEEN	< 1.1
4 - 6	3.8	SECOND	< 1.1
1 - 7	4.0	AND	< 1.1
2 - 7	1.1	THIRD	< 1.1
3 - 7	< 1.1	COATS	< 1.1

TABLE 7

Washing And Neoprene Coating Of Suit #957 - 1966 Vintage

Tris Detected In μg/1000 sq. cm

Suit Interior	Before Wash	After Wash	After Coating
Left Arm Freon 113 Rinse	< 4.3	< 4.3	< 4.3
Right Arm Methanol Rinse	< 4.3	< 4.3	< 4.3
Left Leg Detergent Wash	< 4.3	< 4.3	< 4.3
Right Leg Acetone Wipe	< 4.3	< 4.3	< 4.3
Chest Untreated	< 4.3	< 4.3	< 4.3
Suit Exterior Untreated			< 4.3

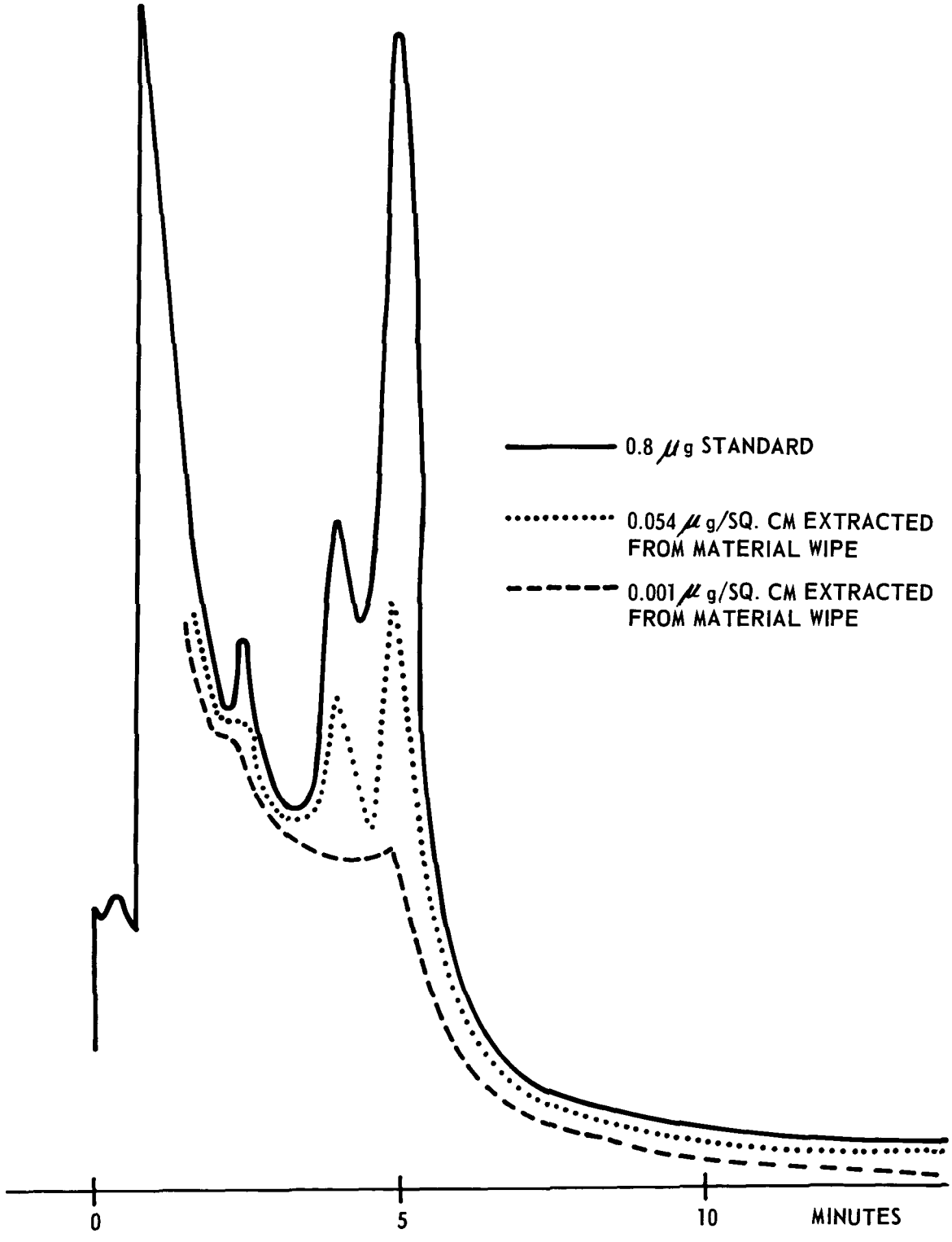

FIGURE 1. GAS CHROMATOGRAMS OF TRIS

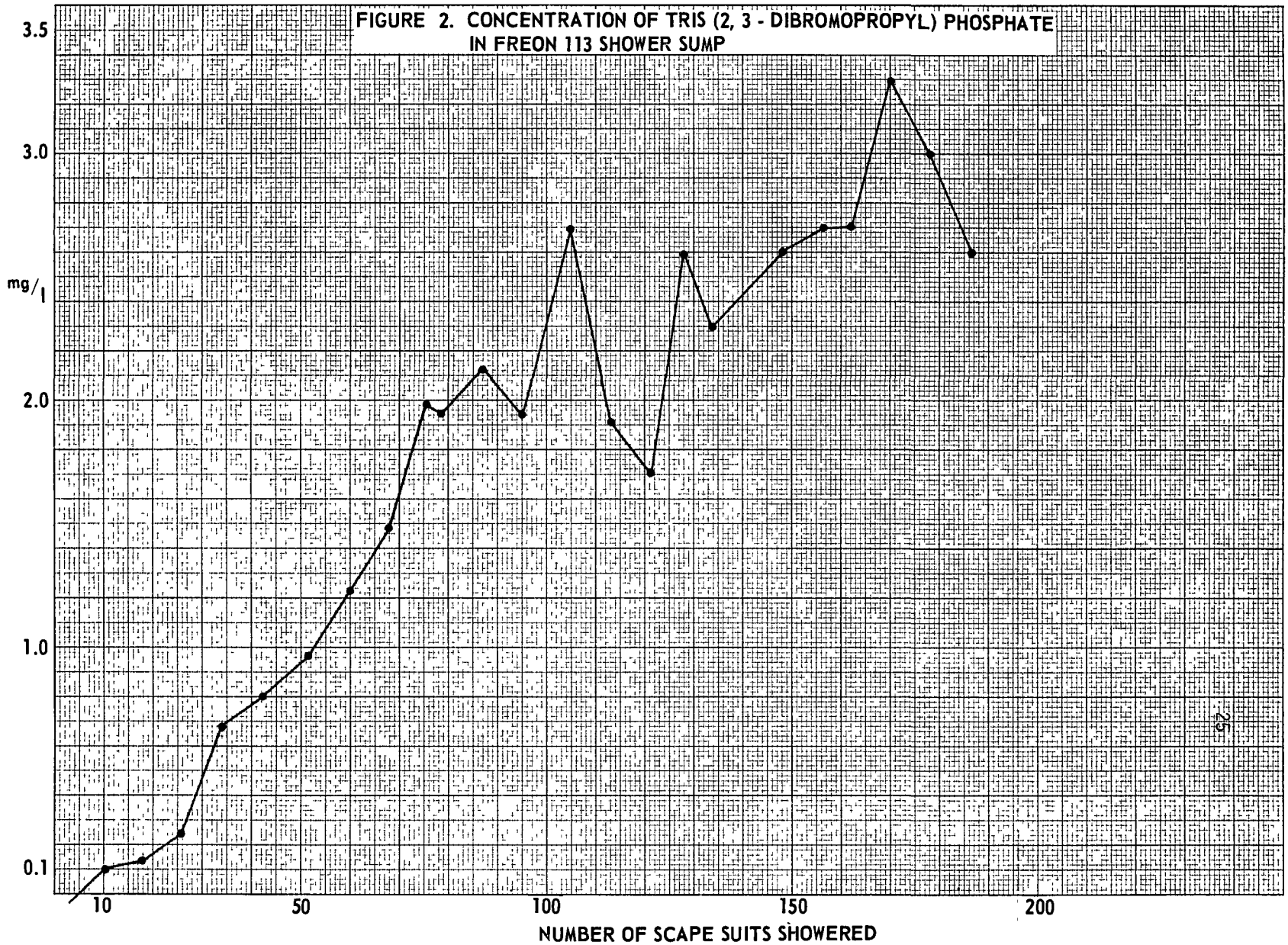
FIGURE 2. CONCENTRATION OF TRIS (2, 3 - DIBROMOPROPYL) PHOSPHATE IN FREON 113 SHOWER SUMP
3.5
3.0
mg/l
2.0
1.0
0.1
10
50
100
150
200
NUMBER OF SCAPE SUITS SHOWERED

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15 Supplementary Notes

16 Abstract

Tris (2,3-dibromopropyl) phosphate has been used to confer flame retardant properties on butyl rubber formulations destined for use in protective clothing. Self-Contained Atmospheric Protective Ensembles (SCAPE suits) constructed from a fabric coated with one such formulation have been in use at Kennedy Space Center in support of Apollo, Skylab, and Apollo-Soyuz missions since 1966.

The announcement that tris (2,3-dibromopropyl) phosphate was found to be mutagenic initiated an investigation of surface concentrations of the compound in SCAPE suits and potential methods of removing or isolating it. This report describes analytical procedures for determining surface concentrations of the tris compound on non-porous materials and finds that soap-and-water washing is the most efficient method of removing the compound from fabricated SCAPE suits and unused material.

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