MESERAN Test Results for Elimination of Flammable Solvents in Wipe Applications at Pantex

Federal Manufacturing & Technologies

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M. G. Benkovich, Project Leader

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Abstract

In recent years, efforts have been made within the nuclear weapons complex (National Nuclear Security Administration) of the Department of Energy (DOE) to replace Resource Conservation and Recovery Act (RCRA) regulated solvents (i.e., flammable, toxic, corrosive, and reactive) and ozone-depleting chemicals (ODC) with more benign alternatives. Within the National Nuclear Security Administration (NNSA) and the Department of Defense (DoD) sectors, these solvents are used for cleaning hardware during routine maintenance operations. A primary goal of this study is to replace flammable solvents for wiping applications. Two cleaners, including a hydrofluoroether (HFE) and an azeotrope of the HFE and isopropyl alcohol (IPA), have been studied as potential replacements for flammable solvents. Cleaning efficacy, short-term and long-term materials compatibility, corrosion, drying times, flammability, environment, safety and health (ES&H) issues and accelerated aging studies are among the tests that are being conducted and that are used to screen candidate solvents by the interagency team performing this work. The results are compared to the traditionally used isopropyl alcohol, which serves as the baseline cleaner. This report details the results of MESERAN (Measurement and Evaluation of Surfaces by Evaporative Rate ANalysis) testing performed at the Kansas City Plant (KCP) to quantify the cleaning efficacy on samples contaminated with the various contaminants and cleaned by wiping with the various solvents being evaluated.

Summary

Cleaning hardware during routine maintenance operations has historically been accomplished with RCRA-regulated or ODC solvents within the National Nuclear Security Administration (NNSA). In an effort to curtail the use of these toxic and flammable solvents for wipe applications, a study has been conducted to identify and qualify a more benign alternative or alternatives. The primary goal is to systematically reduce flammable and combustible solvents used in proximity to and in nuclear explosive operations through solvent elimination, minimization or substitution.

Two solvents, HFE-7100 hydrofluoroether and HFE-7100/IPA azeotrope, were evaluated and compared to IPA for their abilities to remove contamination from metal substrates using a wipe cleaning application. The four metal alloys evaluated in this portion of the study were 2024-aluminum, 304L stainless steel, passivated 304L stainless steel, and 303 stainless steel. The following three contaminants were selected because they were a representative cross section of those found on weapon surfaces: Krytox 240-AD fluorinated grease, Dow Corning 4 (DC-4) silicone insulating compound and dust sebum emulsion (a simulated fingerprint oil).

MESERAN (Measurement and Evaluation of Surfaces by Evaporative Rate ANalysis) Analysis was performed at the Kansas City Plant (KCP) to quantify the cleaning efficacy on samples contaminated with the various contaminants and cleaned by wiping with the various solvents

being evaluated. MESERAN testing was performed both before applying the contaminants and then again after wiping with IPA, HFE-7100, or HFE-7100/IPA azeotrope. The substrates included 2024 aluminum, 304L stainless steel, passivated 304L stainless steel, and 303 stainless steel. Based on calibration curves developed for these specific contaminants on these specific substrates, the MESERAN slopes could be converted to equivalent contamination levels.

For the two alternative solvents evaluated, the HFE-7100/IPA azeotrope is much more effective than HFE-7100 for removing the three contaminants from the four substrates evaluated.

MESERAN data indicates that the HFE-7100/IPA azeotrope was able to remove all three of the contaminants from all of the substrates to a level below $1 \mu\text{g}/\text{cm}^2$ (the cleanliness level called out by most aerospace companies for metal cleaning operations). In some cases, the HFE-7100/IPA azeotrope was even able to remove the contaminants below $100 \text{ ng}/\text{cm}^2$ (the cleanliness level LLNL calls out for some of their NIF cleaning operations). However, no defined cleanliness levels are called out for the product being wipe cleaned at BWXT Pantex Plant. Therefore, these evaluations centered on determining if either HFE-7100 or HFE-7100/IPA azeotrope could clean as well as or better than the baseline solvent (IPA).

Generally, for Krytox 240-AD and dust sebum emulsion, wiping six times with HFE-7100/IPA azeotrope approached the same cleanliness achieved with wiping three times with IPA. For some substrates, wiping three times with HFE-7100/IPA azeotrope was roughly equivalent to wiping three times with IPA. However, for other substrates, wiping six times was necessary.

For Krytox 240-AD, three wipes with HFE-7100/IPA azeotrope was equivalent to three wipes with IPA.

For dust sebum emulsion, wiping six times with HFE-7100/IPA azeotrope approaches the same cleanliness achieved with wiping three times with IPA.

IPA is the most effective solvent evaluated for removing DC-4 silicone. In initial evaluations, wiping six times with HFE-7100 approaches the same cleanliness achieved with wiping three times with IPA; however, the results could not be repeated when the HFE-7100 evaluations were performed again. The HFE-7100/IPA azeotrope was not as effective as IPA, even when six wipes were used.

DC-4 silicone was the hardest contaminant for the HFE-7100/IPA azeotrope to remove. Wiping six times with HFE-7100/IPA azeotrope did not remove the DC-4 silicone as well as IPA. Wiping six times with HFE-7100/IPA azeotrope was able to clean the DC-4 silicone from all of the substrates to a level below $1 \mu\text{g}/\text{cm}^2$. However, it was unable to clean it to submonolayer levels. It must be determined whether the cleanliness level achieved is good enough or if additional wipes are necessary.

HFE-7100 Premoistened Wipers were not effective in removing contamination. In addition, they appeared to leave large amounts of contamination on the samples. Either the HFE-7100 in the premoistened wipers is not very clean or the HFE-7100 is extracting something from the wipers or the bags they are stored in.

Wipe cleaning is an operator-dependent process and can lead to variability in cleaning results. Even when the same operator cleans all of the samples, significant variability will exist. Generally, wipe cleaning is good for removing gross amounts of contamination, but will not consistently remove low levels of contamination unless the contaminants being cleaned are soluble in the solvent used for wiping.

Discussion

Introduction

Kansas City Plant, Sandia National Laboratories, BWXT Pantex Plant, and other National Nuclear Security Administration (NNSA) sites have teamed to identify suitable replacements for Resource Conservation and Recovery Act (RCRA) solvents and ozone-depleting chemicals (ODC) used in nuclear maintenance operations. The primary goal is to systematically reduce the usage of flammable solvents (principally isopropyl alcohol) used in proximity to and in nuclear explosive operations through a risk-cost benefit assessment of solvent and combustible material elimination, minimization or substitution. The cost benefits to all programs at BWXT Pantex Plant by removing a flammable solvent, such as isopropyl alcohol (IPA), would include (1) the savings of significant process time if the requirements for task exhaust, bonding and dry times were lifted due to the use of nonflammable solvents; (2) other operational efficiencies, such as the use of facilities that are not currently available for weapons work due to the absence of task exhaust, could be optimized if the requirement was removed; (3) waste disposal costs could be reduced for both the NNSA/BWXT Pantex Plant and the Department of Defense (DoD) since the waste generated has the potential of not being categorized as hazardous or mixed; and, most importantly, (4) safety would be greatly enhanced, i.e., the elimination of flammable solvents would remove the fuel component necessary to support a fire.

As mandated by NNSA [ref. 1], the BWXT Pantex Plant is currently in the process of implementing a two-stage approach that will eliminate the use of RCRA (especially flammable) and ODC solvents in one pilot program. The first stage is to reduce the number of cleaning steps where these solvents are used, and the second stage is to substitute a nonflammable/noncombustible solvent, where it is not possible to eliminate solvent use, or implement an alternative cleaning process. To date, most of the Freon TF (CFC-113), methyl ethyl ketone and 1,1,1 trichloroethane in the pilot program have been eliminated, primarily by replacement with IPA. Although BWXT Pantex Plant is in the process of eliminating several cleaning steps where IPA is used, complete elimination is impossible. Therefore, a nonflammable alternate solvent(s) must be found.

Initially, one candidate solvent, HFE-7100 hydrofluoroether, [ref. 2], was extensively studied as a potential replacement. Additional testing occurred on HFE-7100/IPA azeotrope, [ref. 3].

HFE-7100 is an inseparable mixture of methoxynanofluorobutane and methoxynanofluoroisobutane. The HFE-7100/IPA azeotrope evaluated consisted of 95.5 wt.% HFE and 4.5 wt.% IPA. Isopropyl alcohol was the principal baseline solvent in this study.

The solvents were evaluated not only for their inability to support combustion, but on other criteria that include: ES&H issues, e.g., toxicity, ozone depletion potential, global warming potential etc.; cleaning efficacy; corrosion of metal alloys; materials compatibility of polymers; nonvolatile residue; high explosive compatibility; cure inhibition of polymers; and short-term/long-term compatibility concerns.

Three contaminants were evaluated on several metal substrates. The three contaminants included Krytox 240-AD [ref. 4] fluorinated grease, Dow Corning 4 (DC-4) silicone insulating compound [ref. 5] and dust sebum emulsion (a simulated fingerprint oil) [ref. 6]. The contaminants selected were a representative cross section of those found on weapon surfaces.

Scope and Purpose

The purpose of the work detailed in this report was to perform MESERAN (Measurement and Evaluation of Surfaces by Evaporative Rate ANALysis) Analysis to quantify the cleaning efficacy on samples contaminated with the three contaminants discussed above and cleaned by wiping with the various solvents being evaluated.

Activity

Solvents Evaluated

Isopropyl alcohol was the principal baseline solvent evaluated in this study. The other two solvents evaluated as potential replacements for IPA were HFE-7100 hydrofluoroether and HFE-7100/IPA azeotrope. A comparison of some of the physical properties of IPA, HFE-7100, and HFE-7100/IPA azeotrope are shown in Table 1.

Table 1. Solvent Properties

Solvent Properties	IPA	HFE-7100	HFE-IPA Azeotrope
Boiling Point @ 760 mmHg	82°C	61°C	54.8°C
Vapor Pressure	96 mm Hg @ 25°C	202 mm Hg at 25°C	207 mm Hg at 25°C
Flash Point	12 °C	None	None
Time Weighted Average (8hr.)	400 ppm	750 ppm	750/400 ppm
Global Warming Potential		320 (lb equivalent CO ₂)	310 (lb equivalent CO ₂)
Ozone Depletion Potential	0	0	0

The IPA used was Fisher Scientific 2-Propanol, Optima Grade, Lot number 023388, with a stated residue after evaporation of 1 ppm. The HFE-7100/IPA azeotrope was made up by mixing 95.5 wt.% of HFE-7100 and 4.5 wt.% IPA. The original bottle of HFE-7100 used was 3M Novec™ HFE-7100, Lot number 24205, Mfg. Date: 7/03. This bottle was used for all of the HFE-7100 wiping evaluations and the majority of the HFE-7100/IPA azeotrope evaluations. After the original bottle of HFE-7100 was depleted, another bottle was ordered. This material was labeled 3M Novec™ HFE-7100DL, Lot number 20154, Mfg. Date: 12/04. This is a higher purity version of Novec™ HFE-7100. Novec™ HFE-7100DL is tightly-controlled for ions, metals, and non-volatile residue. Additional azeotrope evaluations (all azeotrope evaluations for wiping six times to remove DC-4 silicone) were performed by mixing the HFE-7100DL with IPA.

Substrates

Substrates were 1.5” diameter metal disks that were ¼” thick and machined to a 16 rms finish. The four metal alloys evaluated using MESERAN Analysis were 2024-aluminum, 304L stainless steel, passivated 304L stainless steel, and 303 stainless steel. Initial evaluations were attempted on anodized 2024-aluminum and alodined-2024 aluminum, but MESERAN Analysis was not suitable for directly testing these substrates. Their increased surface condition due to the porosity of the anodized and alodined surfaces makes direct testing using MESERAN Analysis impractical.

Precleaning of Substrates

All of the disks were precleaned prior to contamination to create a uniform initial substrate surface. The disks were spray cleaned, ultrasonic cleaned, and vapor degreased under vacuum in trichloroethylene (TCE) using the F-1 Near Zero Emission Cleaner. This process involves warm (50°C) TCE spray cleaning for 5 minutes, warm (50°C) TCE ultrasonic cleaning for 5 minutes, cold (25°C) TCE spray cleaning for 5 minutes, cold (25°C) TCE ultrasonic cleaning for 5 minutes (this 4-stage cycle is repeated three times), vapor degreasing for 5 minutes, and vacuum drying.

The stainless steel disks were then additionally cleaned using the following process: (1) ultrasonic cleaned in Dirl Lum 603 (30g per liter concentration) for 5 minutes at 140°F, (2) rinsed in flowing DI water for 15 – 30 seconds, (3) DI water rinsed in ultrasonic cascade rinse station with 3 tanks (30 seconds in each tank) at 110 – 115°F, (4) blown dry with filtered nitrogen, (5) baked for 30 minutes minimum at 220°F in a HEPA filtered convection oven with nitrogen flowing into the oven. The disks were then packaged in nylon bags and heat sealed. Aqueous cleaning was not performed on the aluminum disks due to the possibility of micro-etching the aluminum with the aqueous alkaline ultrasonic cleaning process.

Contaminants

The contaminants selected were a representative cross section of those found on weapon surfaces. The three contaminants included Krytox 240-AD fluorinated grease, Dow Corning 4 (DC-4) silicone insulating compound, and dust sebum emulsion (a simulated fingerprint oil).

The contaminants were applied as follows. For the dust sebum, approximately 4 microliters (μL) [i.e., 4 milligrams (mg), assuming a density of 1 gram/milliliter (g/mL)] of the simulated fingerprint oil were deposited and spread over the center of each disk using a toothpick. The dust sebum emulsion consists primarily of organic acids, paraffin, and oils. Since a fingerprint weighs ca. 0.1 milligrams, each deposit corresponds to 40 superimposed fingerprints. The Krytox 240-AD fluorinated grease and the DC-4 silicone insulating compound were each deposited by squirting a 0.05 grams (g) [or 50 milligrams] on a disk and then lightly spreading the contaminant across the entire surface of the disk using a tongue depressor. After the

contaminant was applied to the disk, the samples were allowed to dry for 30 minutes before being wiped clean and evaluated with the MESERAN Analyzer. All of the samples were contaminated by Eddie Lopez, Sandia National Laboratories/New Mexico (SNL/NM).

Wipe Cleaning Process

The contaminants were removed by wiping with either IPA, HFE-7100, or HFE-7100/IPA azeotrope using a Kaydry impregnated with roughly 5 mL of solvent. One wipe consists of applying a light pressure with the middle and forefingers on the wiper and dragging it across the surface of the metal disk. A second wipe consists of using a fresh Kaydry impregnated also with ca. 5 mL of solvent, etc. In some cases, up to six wipes were necessary to remove the contaminant from the surface, regardless of the type of solvent used. Removal of the three contaminants from the four metal substrates was evaluated by the MESERAN Analyzer. Replicates of five samples per condition were performed. All of the samples were cleaned by Eddie Lopez, SNL/NM.

Some evaluations were performed on Millimoist® Anticon Gold premoistened polyester wipers saturated with HFE-7100. These wipers were packaged three to a bag (vapor barrier bag) with ca. 90 mL of HFE-7100 in each bag. The wiping process was performed as above except the wipers were already saturated with the HFE-7100. A fresh wipe was used each time.

MESERAN Analysis

General MESERAN Analysis Information [ref. 7-22]

The MESERAN (Measurement and Evaluation of Surfaces by Evaporative Rate ANalysis) Analyzer was used at KCP for determining cleanliness. The precision analytical technique known as MESERAN Analysis permits quantitative measurement of the level of preexisting organic residue on a substrate from <1 nanogram/centimeter² (ng/cm²) to > 100 micrograms/centimeter² (μg/cm²) in 2 minutes. The characterization is performed by depositing a radiochemical solution onto the test surface and then observing the rate at which the radiochemical disappears. The radiochemical solution covers approximately 1 cm² on the surface being tested. The rate at which the radiochemical evaporates is a function of the amount of residue on the surface. The quicker the radiochemical evaporates (higher slope), the cleaner the surface and the slower it evaporates (lower slope), the greater the contamination amount. Further details of the MESERAN Analysis method are shown in the Appendix.

Formulation of Calibration Solutions

Extensive solubility evaluations were conducted to determine the appropriate solvent to use in dissolving the contaminants so that calibration solutions of these contaminants could be made. Methylene chloride proved to be the best solvent for dissolving DC-4 silicone insulating compound. CFC-113 proved to be the best solvent for dissolving Krytox 240-AD fluorinated grease. Methylene chloride proved to be the best solvent for dissolving dust sebum (a true solution could not be made with the dust sebum, but we achieved a thoroughly mixed

suspension). Calibration solutions of these contaminants were made so that calibration curves for the contaminants could be developed on the various substrates. MESERAN results obtained on the samples tested can then be compared to the calibration curves to determine quantitative amounts of contamination detected.

Volumetric dilutions were used to make contamination solutions for depositing known amounts of the organic contaminant on the substrates. These calibrations were performed in the following manner. A master calibration solution was prepared in a 10-mL volumetric flask by dissolving 100 mg of the organic contaminant in 10 mL of solvent (e.g., methylene chloride or CFC-113 that has been double distilled in an all-glass still with no grease in the joints – NVOR of these solvents are approximately 10 ppb). The master calibration solution was thoroughly mixed and 1 mL of this solution was placed in another 10-mL volumetric flask. The second volumetric flask was then diluted with the double distilled solvent until the solution level was at 10 mL and this solution was thoroughly mixed. Subsequent dilutions were carried out in a similar fashion. Ten microliters (μL) of each calibration solution were deposited on the precleaned substrates and allowed to evaporate. This resulted in the following amounts of contamination on the substrates: 1 ng, 10 ng, 100 ng, 1 μg , 10 μg , and 100 μg . In some cases, intermediate levels were obtained by depositing 3 μL and 5 μL of the calibration solutions. The volumetric dilution process for making calibration solutions is shown in Figure 1.

Figure 1. Volumetric Dilution Process for Formulating Calibration Solutions

Radiochemicals Chosen for Evaluations

When testing samples that were contaminated with DC-4 silicone insulating compound, the radiochemical solution consisted of tridecane C-14 radiochemical dissolved in methylene chloride. When testing samples that were contaminated with Krytox 240-AD fluorinated grease, the radiochemical solution consisted of tridecane C-14 radiochemical dissolved in CFC-113. When testing samples that were contaminated with dust sebum emulsion, the radiochemical solution consisted of tridecane C-14 radiochemical dissolved in cyclopentane.

Cleanliness Levels

No defined cleanliness levels are called out for the product being cleaned. Therefore, these evaluations centered on determining if either HFE-7100 or HFE-7100/IPA azeotrope could clean as well as or better than the baseline solvent (IPA). It is important to remember that the cleanliness analysis method used at KCP (MESERAN Analysis) is extremely sensitive for detecting organic contamination. MESERAN Analysis can detect levels of contamination in the nanogram range, which is up to 100 – 1000 times lower than many other standard cleanliness detection methods. Most companies in the aerospace industry use gravimetric analysis for quantifying cleanliness. Gravimetric analysis is not very accurate below microgram levels of contamination.

IEST-STD-CC1246D (Product Cleanliness Levels and Contamination Control Program) [ref. 23] allows users of this document to define desired cleanliness levels. The cleanliness levels called out in this document are shown in Table 2.

Table 2. IEST-STD-CC1246D Cleanliness Levels

AA5 = 10 pg/cm ²	AA3 = 1 ng/cm ²	A = 1 µg/cm ²
AA4.7 = 20 pg/cm ²	AA2.7 = 2 ng/cm ²	B = 2 µg/cm ²
AA4.3 = 50 pg/cm ²	AA4.3 = 5 ng/cm ²	C = 3 µg/cm ²
AA4 = 100 pg/cm ²	A/100 = 10 ng/cm ²	D = 4 µg/cm ²
AA3.7 = 200 pg/cm ²	A/50 = 20 ng/cm ²	E = 5 µg/cm ²
AA4.3 = 500 pg/cm ²	A/20 = 50 ng/cm ²	F = 7 µg/cm ²
	A/10 = 100 ng/cm ²	G = 10 µg/cm ²
	A/5 = 200 ng/cm ²	H = 15 µg/cm ²
	A/2 = 500 ng/cm ²	J = 25 µg/cm ²

Note: pg = picograms (10⁻¹² grams),
ng = nanograms (10⁻⁹ grams), and
µg = micrograms (10⁻⁶ grams)

Many aerospace companies call out a cleanliness level of A (1 µg/cm²) for their metal cleaning applications. However, there are some applications where they call out lower levels. Lawrence Livermore National Laboratory (LLNL) calls out A/10 (100 ng/cm²) for some of their National Ignition Facility (NIF) cleaning processes. Depending upon the contaminant and the surface roughness of the substrate, one monolayer of organic contamination is approximately 100 - 300 ng/cm².

Results and Discussion

MESERAN testing was performed both before applying the contaminants and then again after wiping with IPA, HFE-7100, or HFE-7100/IPA azeotrope. The substrates included 2024 aluminum, 304L stainless steel, passivated 304L stainless steel, and 303 stainless steel. Based on calibration curves developed for these specific contaminants on these specific substrates, the MESERAN slopes could be converted to equivalent contamination levels.

Krytox 240-AD Fluorinated Grease Cleaning Evaluations

Krytox 240-AD Fluorinated Grease on 2024 Aluminum Disks

The calibration of Krytox 240-AD fluorinated grease on 2024 aluminum disks is shown in Figure 2.

Figure 2. Calibration of Krytox 240-AD Fluorinated Grease on 2024 Aluminum Disks

The results of the cleaning evaluations performed to remove the Krytox 240-AD fluorinated grease from 2024 aluminum disks are shown in Table 3.

Table 3. MESERAN Results and Equivalent Contamination Amounts for Krytox 240-AD Fluorinated Grease on 2024 Aluminum Disks

	Gross Contamination (Not Cleaned)		Cleaned by Wiping with IPA (5 mL/Kaydry) – 3 Times		Cleaned by Wiping With HFE-7100 (5 mL/Kaydry) – 3 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	233	Est. >100,000	2271	1030	1314	4155
2	297	Est. >100,000	2433	336	1293	4284
3	400	Est. >100,000	3032	<1	1148	5456
4	179	Est. >100,000	3545	<1	898	9541
5	164	Est. >100,000	2171	1191	972	8086
Avg.	255	Est. >100,000	2690	511	1125	6304
Std. Dev.	96.6		582.9	566.7	186.7	2401.6
% CV	37.9		21.7	110.8	16.6	38.1

Est. = Values estimated outside of calibration curve

	Cleaned by Wiping With HFE-7100 (5 mL/Kaydry) – 6 Times		Cleaned by Wiping With HFE-7100/IPA Azeotrope (5 mL/Kaydry) – 3 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	1652	2538	2704	33
2	1160	5311	2779	16
3	1074	6437	2620	75
4	1371	3824	2275	1024
5	1367	3846	2984	<1
Avg.	1325	4391	2672	230
Std. Dev.	224.2	1507.0	259.9	445.0
% CV	16.9	34.3	9.7	193.8

The samples that were contaminated and not cleaned had an average slope value of 255 and an average contamination level of >100,000 nanograms per square centimeter (ng/cm²). After wiping three times with IPA, the average slope was 2690 and the average contamination level

was 511 ng/cm². Two of the IPA cleaned samples were very clean; however, two of the samples evaluated exhibited fairly large amounts of contamination (greater than 1µg/ cm²). Cleaning with HFE-7100 was not as effective at removing the Krytox as cleaning with IPA. The average slope value after wiping three times with the HFE solvent was 1125 and the average contamination value was 6304 ng/cm². Wiping six times with HFE-7100 was slightly more effective than wiping three times. The average slope was 1325 and the average contamination value was 4391 ng/cm². Wiping three times with the HFE-7100/IPA azeotrope was the most effective for removing the Krytox grease. The average slope was 2672 and the average contamination value was 230 ng/cm². Four of the five samples cleaned with the HFE-7100/IPA azeotrope were very clean; however, one sample did exhibit a large amount of contamination (greater than 1µg/ cm²). The HFE-7100/IPA azeotrope cleaned samples also showed the most consistency.

Krytox 240-AD Fluorinated Grease on 304L Stainless Steel Disks

The calibration of Krytox 240-AD fluorinated grease on 304L stainless steel disks is shown in Figure 3.

Figure 3. Calibration of Krytox 240-AD Fluorinated Grease on 304L Stainless Steel Disks

The results of the cleaning evaluations performed to remove the Krytox 240-AD fluorinated grease from 304L stainless steel disks are shown in Table 4.

Table 4. MESERAN Results and Equivalent Contamination Amounts for Krytox 240-AD Fluorinated Grease on 304L Stainless Steel Disks

	Gross Contamination (Not Cleaned)		Cleaned by Wiping with IPA (5 mL/Kaydry) – 3 Times		Cleaned by Wiping With HFE-7100 (5 mL/Kaydry) – 3 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	347	Est. >100,000	3144	<1	742	>10000 (Est. 30000)
2	403	Est. >100,000	2904	<1	1280	3730
3	291	Est. >100,000	3400	<1	1668	1903
4	112	Est. >100,000	2549	372	1434	2856
5	453	Est. >100,000	2877	<1	1610	2104
Avg.	321	Est. >100,000	2975	74	1347	8119
Std. Dev.	131.7		318.3	166.4	370.9	12253.2
% CV	41.0		10.7	223.6	27.5	150.9

Est. = Values estimated outside of calibration curve

	Cleaned by Wiping With HFE-7100 (5 mL/Kaydry) – 6 Times		Cleaned by Wiping With HFE-7100/IPA Azeotrope (5 mL/Kaydry) – 3 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	1584	2201	3435	<1
2	1223	4117	3164	<1
3	1644	1984	2707	141
4	1710	1769	2916	<1
5	1379	3141	2430	505
Avg.	1508	2642	2930	129
Std. Dev.	201.8	976.5	390.4	218.8
% CV	13.4	37.0	13.3	169.3

The samples that were contaminated and not cleaned had an average slope value of 321 and an average contamination level of $>100,000 \text{ ng/cm}^2$. After wiping three times with IPA, the average slope was 2975 and the average contamination level was 74 ng/cm^2 . Four of the IPA cleaned samples were very clean. Cleaning with HFE-7100 was not as effective at removing the Krytox as cleaning with IPA. The average slope value after wiping three times with the HFE solvent was 1347 and the average contamination value was 8119 ng/cm^2 . Wiping six times with HFE-7100 was slightly more effective than wiping three times. The average slope was 1508 and the average contamination value was 2642 ng/cm^2 . Wiping three times with the HFE-7100/IPA azeotrope approached the equivalency of wiping three times with IPA. The average slope was 2930 and the average contamination value was 129 ng/cm^2 . Three of the five samples cleaned with the HFE-7100/IPA azeotrope were very clean.

Krytox 240-AD Fluorinated Grease on Passivated 304L Stainless Steel Disks

The calibration of Krytox 240-AD fluorinated grease on passivated 304L stainless steel disks is shown in Figure 4.

Figure 4. Calibration of Krytox 240-AD Fluorinated Grease on Passivated 304L Stainless Steel Disks

The results of the cleaning evaluations performed to remove the Krytox 240-AD fluorinated grease from passivated 304L stainless steel disks are shown in Table 5.

Table 5. MESERAN Results and Equivalent Contamination Amounts for Krytox 240-AD Fluorinated Grease on Passivated 304L Stainless Steel Disks

	Gross Contamination (Not Cleaned)		Cleaned by Wiping with IPA (5 mL/Kaydry) – 3 Times		Cleaned by Wiping With HFE-7100 (5 mL/Kaydry) – 3 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	358	Est. >100,000	2805	1	1047	5075
2	248	Est. >100,000	2351	500	1047	5075
3	326	Est. >100,000	2878	<1	1730	1343
4	420	Est. >100,000	2525	157	733	>10000 (Est. 14118)
5	476	Est. >100,000	2682	11	1585	1779
Avg.	366	Est. >100,000	2648	134	1228	5478
Std. Dev.	87.5		213.3	215.2	415.3	5141.9
% CV	23.9		8.1	160.8	33.8	93.9

Est. = Values estimated outside of calibration curve

	Cleaned by Wiping With HFE-7100 (5 mL/Kaydry) – 6 Times		Cleaned by Wiping With HFE-7100/IPA Azeotrope (5 mL/Kaydry) – 3 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	1616	1675	3166	<1
2	2060	769	3102	<1
3	1200	3753	2653	20
4	1353	2789	2878	<1
5	1322	2962	2329	517
Avg.	1510	2390	2826	107
Std. Dev.	342.7	1170.8	343.3	229.1
% CV	22.7	49.0	12.1	213.3

The samples that were contaminated and not cleaned had an average slope value of 366 and an average contamination level of $>100,000 \text{ ng/cm}^2$. After wiping three times with IPA, the average slope was 2648 and the average contamination level was 134 ng/cm^2 . Three of the IPA cleaned samples were very clean. Cleaning with HFE-7100 was not as effective at removing the Krytox as cleaning with IPA. The average slope value after wiping three times with the HFE solvent was 1228 and the average contamination value was 5478 ng/cm^2 . One of the samples (test 4) showed a slope that was outside of the highest contamination level of the calibration curve (i.e., slope < 820 , contamination $> 10000 \text{ ng/cm}^2$). The data was extrapolated to estimate the contamination value as 14118 ng/cm^2 . Wiping six times with HFE-7100 was slightly more effective than wiping three times. The average slope was 1510 and the average contamination value was 2390 ng/cm^2 . Wiping three times with the HFE-7100/IPA azeotrope was slightly better than wiping three times with IPA. The average slope was 2826 and the average contamination value was 107 ng/cm^2 . Four of the five samples cleaned with the HFE-7100/IPA azeotrope were very clean.

Krytox 240-AD Fluorinated Grease on 303 Stainless Steel Disks

The calibration of Krytox 240-AD fluorinated grease on 303 stainless steel disks is shown in Figure 5.

Figure 5. Calibration of Krytox 240-AD Fluorinated Grease on 303 Stainless Steel Disks

The results of the cleaning evaluations performed to remove the Krytox 240-AD fluorinated grease from 303 stainless steel disks are shown in Table 6.

Table 6. MESERAN Results and Equivalent Contamination Amounts for Krytox 240-AD Fluorinated Grease on 303 Stainless Steel Disks

	Cleaned by Wiping with IPA (5 mL/Kaydry) – 3 Times		Cleaned by Wiping With HFE-7100 (5 mL/Kaydry) – 6 Times		Cleaned by Wiping With HFE-7100/IPA Azeotrope (5 mL/Kaydry) – 3 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	3044	3	3030	4	2888	18
2	2848	32	3312	<1	3102	2
3	2622	532	1876	2346	3191	<1
4	2772	92	1780	2654	3146	1
5	2449	1125	2595	657	2932	10
Avg.	2747	357	2519	1132	3052	6
Std. Dev.	225.5	480.0	681.0	1281.6	134.1	7.5
% CV	8.2	134.5	27.0	113.2	4.4	117.2

After wiping three times with IPA, the average slope was 2747 and the average contamination level was 357 ng/cm². Three of the IPA cleaned samples were pretty clean (<100 ng/cm²); however, one was 532 ng/cm² and another sample exhibited a larger amount of contamination (greater than 1µg/cm²). Cleaning six times with HFE-7100 was not as effective at removing the Krytox as cleaning with IPA. The average slope value after wiping six times with the HFE solvent was 2519 and the average contamination value was 1132 ng/cm². Two of the samples were very clean and two of the samples exhibited large amounts of contamination (greater than 1µg/cm²). Wiping three times with the HFE-7100/IPA azeotrope was significantly better than wiping three times with IPA. The average slope was 3052 and the average contamination value was 6 ng/cm². All five samples cleaned with the HFE-7100/IPA azeotrope were very clean.

Krytox 240-AD Fluorinated Grease Conclusions

A graphical representation of the MESERAN slope data for all of the Krytox 240-AD fluorinated grease cleaning evaluations for each substrate is shown in Figure 6. A graphical representation of the equivalent contamination amounts determined by MESERAN Analysis for all of the Krytox 240-AD fluorinated grease cleaning evaluations for each substrate is shown in Figure 7.

Wiping three times with the HFE-7100/IPA azeotrope is essentially equivalent to wiping three times with IPA. HFE-7100 is not as effective as either IPA or the HFE-7100/IPA azeotrope (even when six wipes are used).

Figure 6. MESERAN Slope Data for Krytox 240-AD Fluorinated Grease Cleaning Evaluations

Figure 7. Equivalent Contamination Amounts Determined by MESERAN Analysis for Krytox 240-AD Fluorinated Grease Cleaning Evaluations

Dow Corning 4 (DC-4) Silicone Insulating Compound Cleaning Evaluations

DC-4 Silicone Insulating Compound on 2024 Aluminum Disks

The calibration of DC-4 silicone insulating compound on 2024 aluminum disks is shown in Figure 8.

Figure 8. Calibration of DC-4 Silicone Insulating Compound on 2024 Aluminum Disks

The results of the cleaning evaluations performed to remove the DC-4 silicone insulating compound from 2024 aluminum disks are shown in Table 7.

Table 7. MESERAN Results and Equivalent Contamination Amounts for DC-4 Silicone Insulating Compound on 2024 Aluminum Disks

	Cleaned by Wiping With HFE-7100 Premoistened Polyester Wipers 3 Times		Cleaned by Wiping with IPA (5 mL/Kaydry) – 3 Times		Cleaned by Wiping With HFE-7100 (5 mL/Kaydry) – 6 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	849	3807	2364	138	2401	124
2	853	3782	3095	10	2731	39
3	854	3776	3405	<1	2149	257
4	1159	2309	3050	12	2732	39
5	1147	2354	3172	4	2939	18
Avg.	972	3206	3017	33	2590	95
Std. Dev.	164.9	798.2	389.9	59.0	313.1	99.1
% CV	17.0	24.9	12.9	179.9	12.1	103.9

	Cleaned by Wiping With HFE- 7100/IPA Azeotrope (5 mL/Kaydry) – 3 Times		Cleaned by Wiping With HFE-7100/IPA Azeotrope (5 mL/Kaydry) – 6 Times		Cleaned by Wiping With HFE-7100/IPA Azeotrope (5 mL/Kaydry) – 6 Times Retests	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	1605	1124	2212	215	2436	113
2	2312	161	2225	207	2025	368
3	1958	446	1874	568	2117	282
4	1422	1510	2174	239	2081	313
5	2157	251	2262	186	1581	1169
6	1299	1842				
Avg.	1792	889	2149	283	2048	449
Std. Dev.	411.4	704.6	157.1	160.5	306.2	413.6
% CV	23.0	79.3	7.3	56.8	15.0	92.2

The samples that were cleaned by wiping three times with HFE-7100 saturated premoistened polyester wipers remained significantly contaminated. The average slope was 972 and the average contamination level was 3206 ng/cm². Additional wipes with the HFE-7100 saturated premoistened polyester wipers (data not shown) did not improve the cleanliness levels. Additional tests were run by wiping a “clean – not contaminated” aluminum disk three times with the HFE-7100 saturated premoistened polyester wipers. This resulted in a slope of 1534 and a contamination level of 1261 ng/cm². This result indicates that wiping clean substrates with the HFE-7100 saturated premoistened polyester wipers leaves a significant amount of contamination. The contamination is either because the HFE-7100 used to saturate the polyester wipers is not very clean in terms of nonvolatile residue or the HFE-7100 is extracting something from the polyester wipers or the bags they are stored in. After wiping three times with IPA, the average slope was 3017 and the average contamination level was 33 ng/cm². Four of the five IPA cleaned samples were very clean. Because the HFE-7100 saturated premoistened polyester wipers did not do an adequate job of removing the DC-4 silicone (and left a residue on even clean substrates), it was decided to evaluate HFE-7100 in the same manner as IPA (5 mL/Kaydry). The average slope value for wiping six times with HFE-7100 was 2590 and the average contamination level was 95 ng/cm². Three of the samples were very clean. Wiping three times with the HFE-7100/IPA azeotrope was not as good as wiping three times with IPA or six times with HFE-7100. The average slope was 1792 and the average contamination value was 889 ng/cm². Three of the six samples cleaned three times with the HFE-7100/IPA azeotrope showed significant contamination amounts. Wiping six times with the HFE-7100/IPA azeotrope was an improvement over wiping three times. The average slope increased to 2149 and the average contamination level decreased to 283 ng/cm². This still was not as effective as wiping three times with IPA or six times with HFE-7100. Evaluation of six wipes with the HFE-7100/IPA azeotrope was repeated. The average slope obtained for these tests was 2048 and the average contamination amount was 449 ng/cm². The first four tests fell in line with the original tests of the HFE-7100/IPA azeotrope; however, the fifth test exhibited a significant amount of contamination which increased the average contamination for this group of tests. The average contamination amount for the first four tests of the repeat evaluations was 269 ng/cm² which compares favorably with the 283 ng/cm² average of the initial tests. The HFE-7100/IPA azeotrope (6 wipes) was not as effective as the IPA (three wipes) or the HFE-7100 (6 wipes).

DC-4 Silicone Insulating Compound on 304L Stainless Steel Disks

The calibration of DC-4 silicone insulating compound on 304L stainless steel disks is shown in Figure 9.

Figure 9. Calibration of DC-4 Silicone Insulating Compound on 304L Stainless Steel Disks

The results of the cleaning evaluations performed to remove the DC-4 silicone insulating compound from 304L stainless steel disks are shown in Table 8.

Table 8. MESERAN Results and Equivalent Contamination Amounts for DC-4 Silicone Insulating Compound on 304L Stainless Steel Disks

	Cleaned by Wiping With HFE-7100 Premoistened Polyester Wipers 3 Times		Cleaned by Wiping with IPA (5 mL/Kaydry) – 3 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	1178	3237	3630	<1
2	1114	3766	3292	<1
3	1431	1779	2992	<1
4	960	5247	4079	<1
5	1133	3600	3109	<1
Avg.	1163	3526	3420	<1
Std. Dev.	170.7	1241.0	440.1	
% CV	14.7	35.2	12.9	

	Cleaned by Wiping With HFE- 7100/IPA Azeotrope (5 mL/Kaydry) – 3 Times		Cleaned by Wiping With HFE-7100/IPA Azeotrope (5 mL/Kaydry) – 6 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	2360	198	2787	3
2	2327	215	2587	112
3	2680	19	2076	402
4	2529	130	2419	171
5	1802	754	2467	152
Avg.	2340	263	2467	168
Std. Dev.	332.2	285.2	260.8	145.8
% CV	14.2	108.4	10.6	86.8

The samples that were cleaned by wiping three times with HFE-7100 saturated premoistened polyester wipers remained significantly contaminated. The average slope was 1163 and the average contamination level was 3526 ng/cm². After wiping three times with IPA, the average

slope was 3420 and the average contamination level was $<1 \text{ ng/cm}^2$. All five IPA cleaned samples were very clean. Wiping three times with the HFE-7100/IPA azeotrope was not as good as wiping three times with IPA. The average slope was 2340 and the average contamination value was 263 ng/cm^2 . One of the five samples cleaned three times with the HFE-7100/IPA azeotrope was very clean and one showed a significant contamination amount. Wiping six times with the HFE-7100/IPA azeotrope was a slight improvement over wiping three times. The average slope increased to 2467 and the average contamination level decreased to 168 ng/cm^2 . This still was not as effective as wiping three times with IPA.

DC-4 Silicone Insulating Compound on Passivated 304L Stainless Steel Disks

The calibration of DC-4 silicone insulating compound on passivated 304L stainless steel disks is shown in Figure 10.

Figure 10. Calibration of DC-4 Silicone Insulating Compound on Passivated 304L Stainless Steel Disks

As can be seen from Figure 10, the calibration was only conducted down to 10 ng. Any slopes obtained on samples tested and compared to this calibration that are higher than 2860 are reported as $<10 \text{ ng}$. Estimates can be made by extrapolating the curve and estimating a value for 1 ng. Any estimates shown will be indicated as such.

The results of the cleaning evaluations performed to remove the DC-4 silicone insulating compound from passivated 304L stainless steel disks are shown in Table 9.

Table 9. MESERAN Results and Equivalent Contamination Amounts for DC-4 Silicone Insulating Compound on Passivated 304L Stainless Steel Disks

	Cleaned by Wiping With HFE-7100 Premoistened Polyester Wipers 3 Times		Cleaned by Wiping with IPA (5 mL/Kaydry) – 3 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	1189	1787	2905	<10 (Est. 6)
2	1022	2277	2666	27
3	1043	2209	2307	142
4	1189	1787	2448	81
5	1074	2111	2457	78
Avg.	1103	2034	2557	67
Std. Dev.	80.3	233.2	233.1	53.1
% CV	7.3	11.5	9.1	79.4

Est. = Value estimated outside of calibration curve

	Cleaned by Wiping With HFE- 7100/IPA Azeotrope (5 mL/Kaydry) – 3 Times		Cleaned by Wiping With HFE-7100/IPA Azeotrope (5 mL/Kaydry) – 6 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	2421	93	2335	128
2	1975	396	2028	349
3	1963	408	2401	102
4	2008	366	1702	763
5	1842	545	1927	445
Avg.	2042	362	2079	357
Std. Dev.	221.1	165.2	290.3	269.0
% CV	10.8	45.7	14.0	75.3

The samples that were cleaned by wiping three times with HFE-7100 saturated premoistened polyester wipers remained significantly contaminated. The average slope was 1103 and the average contamination level was 2034 ng/cm². After wiping three times with IPA, the average slope was 2557 and the average contamination level was 67 ng/cm². Wiping three times with the HFE-7100/IPA azeotrope was not as good as wiping three times with IPA. The average slope was 2042 and the average contamination value was 362 ng/cm². Wiping six times with the HFE-7100/IPA azeotrope did not show any improvement over wiping three times. The average slope was 2079 and the average contamination level was 357 ng/cm². Even with increased wipes, the HFE-7100/IPA azeotrope was not as effective at removing the DC-4 silicone from the passivated 304L stainless steel as the IPA.

DC-4 Silicone Insulating Compound on 303 Stainless Steel Disks

The calibration of DC-4 silicone insulating compound on 303 stainless steel disks is shown in Figure 11.

Figure 11. Calibration of DC-4 Silicone Insulating Compound on 303 Stainless Steel Disks

The results of the cleaning evaluations performed to remove the DC-4 silicone insulating compound from 303 stainless steel disks are shown in Table 10.

Table 10. MESERAN Results and Equivalent Contamination Amounts for DC-4 Silicone Insulating Compound on 303 Stainless Steel Disks

	Cleaned by Wiping With HFE-7100 Premoistened Polyester Wipers 3 Times		Cleaned by Wiping with IPA (5 mL/Kaydry) – 3 Times (Initial Tests)		Cleaned by Wiping With HFE-7100 (5 mL/Kaydry) – 6 Times (Initial Tests)	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	944	3452	2761	40	3112	<1
2	1041	2860	2936	10	2539	330
3	1168	2236	3440	<1	2799	30
4	900	3760	3246	<1	2164	612
5	1280	1800	2842	21	3209	<1
Avg.	1067	2822	3045	14	2765	195
Std. Dev.	157.5	816.4	287.3	16.8	427.5	271.6
% CV	14.8	28.9	9.4	118.5	15.5	139.6

	Cleaned by Wiping With HFE- 7100/IPA Azeotrope (5 mL/Kaydry) – 3 Times		Cleaned by Wiping With HFE-7100/IPA Azeotrope (5 mL/Kaydry) – 6 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	2325	534	2106	643
2	1765	858	2430	462
3	1853	796	1569	1028
4	1577	1012	1679	922
5	1479	1223	2517	353
Avg.	1800	885	2060	681
Std. Dev.	328.8	256.0	428.4	289.5
% CV	18.3	28.9	20.8	42.5

Table 10 (continued). MESERAN Results and Equivalent Contamination Amounts for DC-4 Silicone Insulating Compound on 303 Stainless Steel Disks

	Cleaned by Wiping With HFE-7100DL (5 mL/Kaydry) – 6 Times (Retests)		Cleaned by Wiping with IPA (5 mL/Kaydry) – 3 Times (Retests)		Samples Not Contaminated Cleaned by Wiping With HFE-7100DL (5 mL/Kaydry) – 3 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	1847	800	2689	72	2922	11
2	1803	830	2547	322	2265	562
3	1680	921	2753	43	3114	<1
4	1490	1198	3154	<1	2745	46
5	1177	2197	2894	14	2683	76
Avg.	1599	1189	2807	90	2746	139
Std. Dev.	273.7	584.9	230.4	132.3	316.9	238.3
% CV	17.1	49.2	8.2	146.3	11.5	171.1

The samples that were cleaned by wiping three times with HFE-7100 saturated premoistened polyester wipers remained significantly contaminated. The average slope was 1067 and the average contamination level was 2822 ng/cm². After wiping three times with IPA in the initial tests performed, the average slope was 3045 and the average contamination level was 14 ng/cm². All of the samples cleaned with IPA in the initial group exhibited low contamination levels. The IPA cleaning tests were performed again at a later date and the average slope obtained for the retests was 2807 and the average contamination level was 90 ng/cm². One of the samples exhibited more contamination than the other four samples, which increased the average contamination level for the retest group. The initial tests performed by wiping six times with HFE-7100 (5 mL/Kaydry) yielded an average slope of 2765 and an average contamination level of 195 ng/cm². Three of the samples evaluated exhibited very low contamination levels and the other two samples exhibited higher contamination levels. All of these samples still looked somewhat contaminated. These tests were performed again at a later date using the new bottle of HFE-7100DL, and the average slope was 1599 and the average contamination level was 1189 ng/cm². All of the samples tested exhibited high levels of contamination. Samples that had not been intentionally contaminated with DC-4 silicone were cleaned by wiping three times with the HFE-7100DL to make sure that the solvent was not contaminated. The average slope was 2746 and the average contamination amount was 139 ng/cm². Four of the five samples tested pretty clean; however, one sample exhibited a larger amount of contamination. It is postulated that this sample must not have been properly precleaned prior to these tests and the HFE-7100DL was unable to completely remove whatever contamination had previously been on the sample. It does not appear that the HFE-7100DL has a large nonvolatile residue issue. There is no clear-cut

answer as to why the initial tests on HFE-7100 yielded much cleaner surfaces than the subsequent retests. The evaluations conducted on the HFE-7100/IPA azeotrope indicate that it does not remove the DC-4 silicone as well as IPA. Wiping three times with the HFE-7100/IPA azeotrope yielded an average slope of 1800 and an average contamination level of 885 ng/cm². Wiping six times with the HFE-7100/IPA azeotrope slightly improved the results. The average slope increased to 2060 and the average contamination level decreased to 681 ng/cm². All of the samples tested with three and six wipes with the HFE-7100/IPA azeotrope showed significant contamination amounts. Even with increased wipes, the HFE-7100/IPA azeotrope was not as effective at removing the DC-4 silicone from the 303 stainless steel as the IPA.

DC-4 Silicone Insulating Compound Conclusions

A graphical representation of the MESERAN slope data for all of the DC-4 silicone insulating compound cleaning evaluations for each substrate is shown in Figure 12. A graphical representation of the equivalent contamination amounts determined by MESERAN Analysis for all of the DC-4 silicone insulating compound cleaning evaluations for each substrate is shown in Figure 13.

Figure 12. MESERAN Slope Data for DC-4 Silicone Insulating Compound Cleaning Evaluations

DC-4 Silicone Insulating Compound Cleaning Evaluations
Equivalent Contamination Amounts Determined by MESERAN Analysis

Figure 13. Equivalent Contamination Amounts Determined by MESERAN Analysis for DC-4 Silicone Insulating Compound Cleaning Evaluations

IPA is the most effective solvent evaluated for removing DC-4 silicone. In initial evaluations, wiping six times with HFE-7100 approaches the same cleanliness achieved with wiping three times with IPA; however the results could not be repeated when the HFE-7100 evaluations were performed again. The HFE-7100/IPA azeotrope was not as effective as IPA, even when six wipes were used.

DC-4 silicone was the hardest contaminant for the HFE-7100/IPA azeotrope to remove. Wiping six times with HFE-7100/IPA azeotrope did not remove the DC-4 silicone as well as IPA. Wiping six times with HFE-7100/IPA azeotrope was able to clean the DC-4 silicone from all of the substrates to a level below $1 \mu\text{g}/\text{cm}^2$ (the cleanliness level called out by most aerospace companies for metal cleaning operations). However, it was unable to clean it to submonolayer levels. It must be determined whether the cleanliness level achieved is good enough or if additional wipes are necessary.

Dust Sebum Emulsion Cleaning Evaluations

Dust Sebum Emulsion on 2024 Aluminum Disks

The calibration of dust sebum emulsion on 2024 aluminum disks is shown in Figure 14.

Figure 14. Calibration of Dust Sebum Emulsion on 2024 Aluminum Disks

As can be seen from Figure 14, the calibration was only conducted down to 10 ng. Any slopes obtained on samples tested and compared to this calibration that are higher than 2873 are reported as <10 ng. Any slopes obtained that are lower than 344 are reported as >10,000 ng. Estimates can be made by extrapolating the curve and estimating a value for 1 ng and a value for something higher than 10,000 ng. Any estimates shown will be indicated as such.

The results of the cleaning evaluations performed to remove the dust sebum emulsion from 2024 aluminum disks are shown in Table 11.

Table 11. MESERAN Results and Equivalent Contamination Amounts for Dust Sebum Emulsion on 2024 Aluminum Disks

	Cleaned by Wiping With HFE-7100 Premoistened Polyester Wipers 3 Times		Cleaned by Wiping with IPA (5 mL/Kaydry) – 3 Times		Cleaned by Wiping With HFE-7100 (5 mL/Kaydry) – 6 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	297	>10000 (Est. 17737)	2953	<10 (Est. 4)	2357	230
2	338	>10000 (Est. 10759)	2539	130	1517	751
3	553	4213	1922	482	1459	799
4	528	4436	2862	11	1221	1062
5	656	3406	3037	<10 (Est. 1)	1427	827
Avg.	474	8110	2663	126	1596	733
Std. Dev.	151.7	6134.0	455.0	206.4	439.7	306.2
% CV	32.0	75.6	17.1	164.3	27.5	41.7

Est. = Values estimated outside of calibration curve

	Cleaned by Wiping With HFE- 7100/IPA Azeotrope (5 mL/Kaydry) – 3 Times		Cleaned by Wiping With HFE-7100/IPA Azeotrope (5 mL/Kaydry) – 6 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	2399	201	2935	<10 (Est. 5)
2	2810	18	3202	<10 (Est. <1)
3	2526	136	2372	219
4	2123	367	2273	298
5	2633	92	2831	15
Avg.	2498	163	2723	107
Std. Dev.	258.3	132.1	391.1	140.9
% CV	10.3	81.2	14.4	131.2

Est. = Values estimated outside of calibration curve

The samples that were cleaned by wiping three times with HFE-7100 saturated premoistened polyester wipers remained significantly contaminated. The average slope was 474 and the

average contamination level was 8110 ng/cm². All of the samples showed significant amounts of contamination. Two of the samples exhibited slopes lower than the bottom range of the calibration (i.e., lower than 344 which correlate to > 10,000 ng/cm²). Their contamination amounts were estimated by extrapolating the curve beyond 10,000 ng based upon likely values seen in other calibrations. The samples cleaned by wiping three times with IPA had an average slope of 2663 and an average contamination level of 126 ng/cm². Three of the five samples were very clean and one sample had significantly more contamination than the other samples. The samples cleaned by wiping six times with HFE-7100 (5 mL/Kaydry) yielded an average slope of 1596 and an average contamination level of 733 ng/cm². Wiping three times with the HFE-7100/IPA azeotrope approached the equivalency of wiping three times with IPA. The average slope was 2498 and the average contamination value was 163 ng/cm². Wiping six times with the HFE-7100/IPA azeotrope increased the average slope to 2723 and decreased the average contamination level to 107 ng/cm². Three of the five samples wiped six times with the HFE-7100/IPA azeotrope were very clean. Wiping six times with the HFE-7100/IPA azeotrope was slightly better than wiping three times with IPA.

Dust Sebum Emulsion on 304L Stainless Steel Disks

The calibration of dust sebum emulsion on 304L stainless steel disks is shown in Figure 15.

Figure 15. Calibration of Dust Sebum Emulsion on 304L Stainless Steel Disks

As can be seen from Figure 15, the calibration was only conducted down to 10 ng. Any slopes obtained on samples tested and compared to this calibration that are higher than 2513 are

reported as <10 ng. Estimates can be made by extrapolating the curve and estimating a value for 1 ng. Any estimates shown will be indicated as such.

The results of the cleaning evaluations performed to remove the dust sebum emulsion from 304L stainless steel disks are shown in Table 12.

Table 12. MESERAN Results and Equivalent Contamination Amounts for Dust Sebum Emulsion on 304L Stainless Steel Disks

	Cleaned by Wiping With HFE-7100 Premoistened Polyester Wipers 3 Times		Cleaned by Wiping with IPA (5 mL/Kaydry) – 3 Times		Cleaned by Wiping With HFE-7100 (5 mL/Kaydry) – 6 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	1154	483	2844	<10 (Est. <1)	2861	<10 (Est. <1)
2	1374	425	2930	<10 (Est. <1)	2354	32
3	767	895	3027	<10 (Est. <1)	3060	<10 (Est. <1)
4	1024	566	3205	<10 (Est. <1)	2713	<10 (Est. <1)
5	360	6881	3222	<10 (Est. <1)	2770	<10 (Est. <1)
Avg.	936	1850	3046	<10 (Est. <1)	2752	7
Std. Dev.	389.7	2818.3	166.5		258.4	14.3
% CV	41.6	152.3	5.5		9.4	207.5

Est. = Values estimated outside of calibration curve

	Cleaned by Wiping With HFE- 7100/IPA Azeotrope (5 mL/Kaydry) – 3 Times		Cleaned by Wiping With HFE-7100/IPA Azeotrope (5 mL/Kaydry) – 6 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	2720	<10 (Est. <1)	2508	10
2	3103	<10 (Est. <1)	3019	<10 (Est. <1)
3	2814	<10 (Est. <1)	2968	<10 (Est. <1)
4	2185	109	2839	<10 (Est. <1)
5	2576	<10 (Est. 5)	3059	<10 (Est. <1)
Avg.	2680	23	2879	2
Std. Dev.	337.0	48.0	223.1	4.6
% CV	12.6	209.2	7.8	223.6

Est. = Values estimated outside of calibration curve

The samples that were cleaned by wiping three times with HFE-7100 saturated premoistened polyester wipers remained significantly contaminated. The average slope was 936 and the average contamination level was 1850 ng/cm². All of the samples showed significant amounts of contamination, especially sample five. The samples cleaned by wiping three times with IPA had an average slope of 3046 and an average contamination level of <1 ng/cm². All of the samples were very clean. The samples cleaned by wiping six times with HFE-7100 (5 mL/Kaydry) yielded an average slope of 2752 and an average contamination level of 7 ng/cm². All of the samples were very clean. Wiping three times with the HFE-7100/IPA azeotrope approached the equivalency of wiping three times with IPA and three times with HFE-7100. The average slope was 2680 and the average contamination value was 23 ng/cm². Four of the five samples were extremely clean while one sample exhibited a little more contamination. Wiping six times with the HFE-7100/IPA azeotrope increased the average slope to 2879 and decreased the average contamination level to 2 ng/cm². All of the samples were very clean. Both the HFE-7100 and the HFE-7100/IPA azeotrope appear to be able to clean the 304L stainless steel disks to similar levels as the IPA.

Dust Sebum Emulsion on Passivated 304L Stainless Steel Disks

The calibration of dust sebum emulsion on passivated 304L stainless steel disks is shown in Figure 16.

Figure 16. Calibration of Dust Sebum Emulsion on Passivated 304L Stainless Steel Disks

The results of the cleaning evaluations performed to remove the dust sebum emulsion from passivated 304L stainless steel disks are shown in Table 13.

Table 13. MESERAN Results and Equivalent Contamination Amounts for Dust Sebum Emulsion on Passivated 304L Stainless Steel Disks

	Cleaned by Wiping With HFE-7100 Premoistened Polyester Wipers 3 Times		Cleaned by Wiping with IPA (5 mL/Kaydry) – 3 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	1060	851	1741	529
2	691	1568	3085	<1
3	604	2081	2448	240
4	914	942	2588	127
5	1775	517	2011	423
Avg.	1009	1192	2375	264
Std. Dev.	464.6	625.7	521.8	214.5
% CV	46.1	52.5	22.0	81.2

	Cleaned by Wiping With HFE-7100 (5 mL/Kaydry) – 6 Times		Cleaned by Wiping With HFE-7100/IPA Azeotrope (5 mL/Kaydry) – 3 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	2517	175	3707	<1
2	2798	14	2532	164
3	2778	18	2954	2
4	2851	7	3392	<1
5	2399	300	3283	<1
Avg.	2669	103	3174	33
Std. Dev.	198.5	130.7	448.3	73.1
% CV	7.4	127.2	14.1	220.3

The samples that were cleaned by wiping three times with HFE-7100 saturated premoistened polyester wipers remained significantly contaminated. The average slope was 1009 and the average contamination level was 1192 ng/cm². All of the samples showed significant amounts of contamination, especially samples two and three. The samples cleaned by wiping three times with IPA had an average slope of 2375 and an average contamination level of 264 ng/cm². Only one of the samples was extremely clean. The samples cleaned by wiping six times with HFE-7100 (5 mL/Kaydry) yielded an average slope of 2669 and an average contamination level of 103 ng/cm². Three of the samples were very clean and the other two samples had more contamination. Wiping three times with the HFE-7100/IPA azeotrope yielded an average slope of 3174 and the average contamination value of 33 ng/cm². Four of the five samples were extremely clean while one sample exhibited a little more contamination. Both the HFE-7100 (six wipes) and the HFE-7100/IPA azeotrope (three wipes) appear to be able to consistently clean the passivated 304L stainless steel disks to lower levels than the IPA.

Dust Sebum Emulsion on 303 Stainless Steel Disks

The calibration of dust sebum emulsion on 303 stainless steel disks is shown in Figure 17.

Figure 17. Calibration of Dust Sebum Emulsion on 303 Stainless Steel Disks

The results of the cleaning evaluations performed to remove the dust sebum emulsion from 303 stainless steel disks are shown in Table 14.

**Table 14. MESERAN Results and Equivalent Contamination Amounts
for Dust Sebum Emulsion on 303 Stainless Steel Disks**

	Cleaned by Wiping With HFE-7100 Premoistened Polyester Wipers 3 Times		Cleaned by Wiping with IPA (5 mL/Kaydry) – 3 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	483	3717	2856	19
2	1886	391	2655	75
3	642	2484	3033	3
4	1691	460	3121	<1
5	768	1805	2881	16
Avg.	1094	1771	2909	23
Std. Dev.	645.7	1407.3	179.2	30.3
% CV	59.0	79.5	6.2	131.9

	Cleaned by Wiping With HFE-7100 (5 mL/Kaydry) – 6 Times		Cleaned by Wiping With HFE-7100/IPA Azeotrope (5 mL/Kaydry) – 3 Times	
Test	Low Variance Slope	Equivalent Contamination (ng/cm ²)	Low Variance Slope	Equivalent Contamination (ng/cm ²)
1	2323	217	2605	102
2	2652	77	2924	12
3	2581	109	3071	2
4	2484	141	3262	<1
5	2604	102	2575	111
Avg.	2529	129	2887	45
Std. Dev.	130.3	54.2	296.9	56.0
% CV	5.2	41.9	10.3	123.5

The samples that were cleaned by wiping three times with HFE-7100 saturated premoistened polyester wipers remained significantly contaminated. The average slope was 1094 and the average contamination level was 1771 ng/cm². Three of the five samples showed significant amounts of contamination. The samples cleaned by wiping three times with IPA had an average slope of 2909 and an average contamination level of 23 ng/ cm². Four of the five samples were

very clean. The samples cleaned by wiping six times with HFE-7100 (5 mL/Kaydry) yielded an average slope of 2529 and an average contamination level of 129 ng/cm². These samples visually appeared to have islands of contamination present. It is speculated that the majority of the visual contamination was inorganic material (dust) from the dust sebum emulsion (i.e., MESERAN testing only detects organic contamination – this would explain why the slopes are relatively high even though visual contamination is present). Wiping three times with the HFE-7100/IPA azeotrope yielded an average slope of 2887 and the average contamination value of 45 ng/cm². Three of the five samples were extremely clean while the other two samples exhibited a little more contamination. All of the samples cleaned with the HFE-7100/IPA azeotrope appeared to be visually clean. The HFE-7100/IPA azeotrope approaches the cleanliness achieved with the IPA.

Dust Sebum Emulsion Conclusions

A graphical representation of the MESERAN slope data for all of the dust sebum emulsion evaluations for each substrate is shown in Figure 18. A graphical representation of the equivalent contamination amounts determined by MESERAN Analysis for all of the dust sebum emulsion cleaning evaluations for each substrate is shown in Figure 19.

Figure 18. MESERAN Slope Data for Dust Sebum Emulsion Cleaning Evaluations

Figure 19. Equivalent Contamination Amounts Determined by MESERAN Analysis for Dust Sebum Emulsion Cleaning Evaluations

Regardless of the substrate, wiping six times with HFE-7100/IPA azeotrope approaches the same cleanliness achieved with wiping three times with IPA. For some substrates, wiping three times with HFE-7100/IPA azeotrope is roughly equivalent to wiping three times with IPA. For some substrates, wiping six times with HFE-7100 approaches the same cleanliness achieved with wiping three times with IPA. Wiping six times with HFE-7100/IPA azeotrope is more effective than wiping six times with HFE-7100.

Conclusions

For the two alternative solvents evaluated, the HFE-7100/IPA azeotrope is much more effective than HFE-7100 for removing the three contaminants from the four substrates evaluated.

MESERAN data indicates that the HFE-7100/IPA azeotrope was able to remove all three of the contaminants from all of the substrates to a level below $1 \mu\text{g}/\text{cm}^2$ (the cleanliness level called out by most aerospace companies for metal cleaning operations). In some cases, the HFE-7100/IPA azeotrope was even able to remove the contaminants below $100 \text{ ng}/\text{cm}^2$ (the cleanliness level LLNL calls out for some of their NIF cleaning operations). However, no defined cleanliness levels are called out for the product being wipe cleaned at BWXT Pantex Plant. Therefore, these evaluations centered on determining if either HFE-7100 or HFE-7100/IPA azeotrope could clean as well as or better than the baseline solvent (IPA).

Generally, for Krytox 240-AD and dust sebum emulsion, wiping six times with HFE-7100/IPA azeotrope approached the same cleanliness achieved with wiping three times with IPA. For some substrates, wiping three times with HFE-7100/IPA azeotrope was roughly equivalent to wiping three times with IPA. However, for other substrates, wiping six times was necessary.

For Krytox 240-AD, three wipes with HFE-7100/IPA azeotrope was equivalent to three wipes with IPA.

For dust sebum emulsion, wiping six times with HFE-7100/IPA azeotrope approaches the same cleanliness achieved with wiping three times with IPA.

IPA is the most effective solvent evaluated for removing DC-4 silicone. In initial evaluations, wiping six times with HFE-7100 approaches the same cleanliness achieved with wiping three times with IPA; however, the results could not be repeated when the HFE-7100 evaluations were performed again. The HFE-7100/IPA azeotrope was not as effective as IPA even when six wipes were used.

DC-4 silicone was the hardest contaminant for the HFE-7100/IPA azeotrope to remove. Wiping six times with HFE-7100/IPA azeotrope did not remove the DC-4 silicone as well as IPA. Wiping six times with HFE-7100/IPA azeotrope was able to clean the DC-4 silicone from all of the substrates to a level below $1 \mu\text{g}/\text{cm}^2$. However, it was unable to clean it to submonolayer levels. It must be determined whether the cleanliness level achieved is good enough or if additional wipes are necessary.

HFE-7100 Premoistened Wipers were not effective in removing contamination. In addition, they appeared to leave large amounts of contamination on the samples. Either the HFE-7100 in the premoistened wipers is not very clean or the HFE-7100 is extracting something from the wipers or the bags they are stored in.

Wipe cleaning is an operator-dependent process and can lead to variability in cleaning results. Even when the same operator cleans all of the samples, significant variability will exist. Generally, wipe cleaning is good for removing gross amounts of contamination, but will not consistently remove low levels of contamination unless the contaminants being cleaned are soluble in the solvent used for wiping.

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Appendix

Detailed Information on MESERAN Analysis for Organic Contamination Detection

Appendix

Detailed Information on MESERAN Analysis for Organic Contamination Detection

Background

The principle of the MESERAN technique was discovered by John L. Anderson, President of ERA Systems, Inc., The MESERAN Company, in 1960. This analytical technique is used in a number of industrial and governmental facilities (within the United States and abroad) for research and development purposes as well as for quality and production control. The characterization of the surface being analyzed is carried out by depositing a chemical detector onto the test surface and observing the rate at which the chemical detector disappears from the surface. The MESERAN technique is routinely used for quantifying organic contamination on surfaces and the crosslink density (or degree of cure) in polymers. In addition, the MESERAN technique can be used for quantifying chemically active sites on surfaces.

The Kansas City Plant (KCP) has been using MESERAN Analyzers for approximately 30 years to detect and quantify organic contamination on parts and evaluate various cleaning processes for removing organic contamination. KCP has used MESERAN Analyzers extensively to evaluate the ability of alternate solvents and processes for removing specific organic contaminants to eliminate the use of chlorinated and fluorinated solvents.

Principle of MESERAN Technique

The standard microcomputer-based MESERAN technology involves deposition, using a “clean” precision microsyringe, onto a flat or concave surface of 18 μL of a test solution consisting of a low boiling solvent or solvent combination (for these evaluations - cyclopentane) and a high-boiling-but-volatile Carbon-14 labeled compound (in a ratio of approximately 60,000:1). Figure 1 shows the application of test solution. For example, the amount of tridecane-C14 radiochemical per single test ($< 0.06 \mu\text{Ci}$) corresponds to approximately 6×10^{14} molecules which equates to *one nanomole*, the *equivalent* of approximately one molecular layer over one square centimeter. Metered air or nitrogen gas is permitted to flow across the surface and between the surface and a Geiger Müller detector positioned directly above the surface. The evaporation of the low boiling solvent and then the radiochemical is observed as a function of time by recording the detected emissions per second arising from the radiochemical molecules remaining on, or retained by, the surface--the vapor-phase, already-evaporated molecules having been swept out from under the detector by the metered gas (see Figure 2)

Figure 1. Application of Test Solution

Figure 2. Measurement of Emissions

Each test takes less than 3 minutes and the amount of radiochemical employed is EXEMPT from U S Nuclear Regulatory Commission and/or 'Agreement State' licensing regulations due to the very low level of C-14 involved.

For the measurement of microorganic residues, the MESERAN method may be used:

- (1) Directly on a flat or concave surface and any microorganic residue thereon which is chemically compatible with the particular radiochemical employed, or
- (2) Indirectly using an extracting solvent followed by depositing and evaporating an aliquot amount onto a "clean" reference surface. Subsequent deposition and evaporation of the radiochemical solution permits measurement of the amount of deposited residue by comparing the results with previously obtained standards similarly deposited from volumetric dilutions.

For non-polar and/or hydrocarbon type residues, tridecane-C14 in cyclopentane (designated **BK**) is employed. For more polar residues, tetrabromoethane-C14 in cyclopentane (designated **AK**) is used. In order to provide a high number of detected emissions for the minimal amount of radiochemical deposited, the tridecane-C14 has a specific activity of approximately 57 $\mu\text{Ci}/\mu\text{mole}$ (one tridecane atom is essentially pure C14 isotope) while the tetrabromoethane has both carbon atoms labeled (approximately 114 $\mu\text{Ci}/\mu\text{mole}$). Approximately 200 ng of radiochemical are deposited in each test with similar levels of radioactivity.

The MESERAN method assumes that the particular radiochemical employed is chemically compatible with the residue, that the test solution droplet covers all of the residue, and that the test solution solvent substantially dissolves the residue within the time period of the solvent evaporation. *Attention to the avoidance of inadvertent contamination and the maintenance of reasonably constant temperature and pressure are required for optimal reproducibility from test to test.*

Mechanism of the MESERAN Technique for Quantifying Organic Residues

When a homogeneous chemical is permitted to evaporate, the classical mechanism of the process (normally measured by monitoring the already evaporated portion) follows first order kinetics, i.e., the plot of log concentration vs. time is a straight line. This mechanism applies to pure materials as well as to solutions of chemicals in which the components are chemically compatible and in which the second component is non-volatile under the conditions of the process. In the presence of the second component, the rate of evaporation is slowed.

In the MESERAN technology, however, the amount of radiochemical retained by the surface as a function of time is measured by counting the emissions arising from the radiochemical molecules remaining on the surface. In this discussion, the temperature and pressure are assumed constant and the concentration of already evaporated molecules in the adjacent gaseous phase approaches zero due to the flowing air or nitrogen referred to above. The molecular weight of each evaporating molecule and the intermolecular forces among the near-neighbor molecules are thus the primary factors in determining the tendency of each molecule to remain in solution or conversely to escape from the liquid portion of the air/liquid (or semisolid) interface. In the MESERAN technology, which employs only a monolayer *equivalent* of the radiochemical, the observed rate of evaporation is thus a function of the residual concentration of the non-evaporated molecules of the Carbon-14 radiochemical. Figure 3 illustrates the typical evaporation of the radiochemical solution from a clean surface. The A-B line represents the evaporation of the low boiling solvent (e.g., cyclopentane). The rationale for the initial increase in counts/second is that the C-14 soft beta emissions are partially absorbed by the solvent molecules. B represents the point at which substantially all of the low boiling solvent has evaporated and the maximal amount of residual radiation reaches the GM detector. The B-C line represents the evaporation of the radiochemical from the surface under the conditions of the test. C represents a level where the GM detector can no longer adequately differentiate the residual radiation from background.

A solution of the high-boiling-but-volatile tridecane-C14 in higher boiling hydrocarbons (i.e., contamination) follows a similar but slower path than does the evaporation of the labeled tridecane itself since the non-volatile “residue” molecules occupy increasing portions of the liquid (or semi-solid) interface. The rate at which the solvent evaporates is slowed somewhat and the rate at which the radiochemical evaporates is slowed considerably with the observed rate of evaporation being a function of the amount of residue on the surface. The observed rate of evaporation of the radiochemical (*the slope expressed as a positive integer*) thus is an inverse measure of the amount of non-evaporating residue. The lower the slope, the more the residue and vice versa. Figure 4 illustrates typical evaporations of the radiochemical solution with increasing amounts of residue. ABC is repeated from Figure 3 and illustrates a typical evaporation of the radiochemical solution with no interactions from residue (i.e., a clean substrate). A*B*C* illustrates a typical evaporation of the radiochemical solution with some contamination present. A**B**C** illustrates a typical evaporation of the radiochemical solution with a larger amount of contamination present.

Figure 3. Typical Evaporation of Radiochemical Solution from a Clean Surface

Figure 4. Typical Evaporations of Radiochemical Solution with Increasing Amounts of Residue

Methods of Analyzing MESERAN Data

Historical Method (Total Counts)

Historically, KCP only used the Total Counts method of analysis for analyzing MESERAN data. This involved summing the radioactive counts detected by the GM detector for a period of 112 seconds. The Total Counts method has been used for approximately 30 years at KCP by testing a surface with the MESERAN Analyzer and comparing the results to those obtained from known clean standards for that particular surface. The total counts of the clean standard are subtracted from the total counts obtained on the surface being tested to give a net total counts representing the contamination amount. Lower total counts represent low levels of organic residue and high total counts represent high levels of organic contamination. This result can be compared to previously performed calibrations of contamination to obtain a quantitative result for the contamination amount.

In many cases, quantitative contamination data are not needed. For instance, if one is performing process control work to determine if the cleaning process is performing as designed, quantitative data on the actual amount of contamination may not be necessary. Often times, as long as the parts being cleaned are less than a certain level of contamination, they are clean enough. Therefore, one only has to establish the MESERAN total counts that correspond to that level of contamination and relate the tests as being in compliance or not. KCP has used this technique for years to control cleanliness and compare the abilities of different cleaners and cleaning processes to remove various contaminants. KCP related net total counts MESERAN data to electrical and catastrophic adhesive failures. They noted that electrical failures on products occurred when the net total counts exceeded 100 and adhesive failures occurred when net total counts exceeded 200. As long as the MESERAN net total counts were below these levels, no cleaning related failures occurred.

The 112 second test time period was used because the original MESERAN units in the 1960s and 1970s operated by recording counts in 14 second increments and KCP used to total of eight of the 14 second increments for their test time. These units also only recorded every 8th count that was detected due to the counting mechanism used at the time. When microcomputers became available in the 1980s, automatic MESERAN units were manufactured and equipped with computer interfaces. These units recorded counts every ½ second and the test time could be set to any length desired; however, to make everything equivalent to historical data, the program was written for KCP to base the calculations on 112 seconds with only every 8th count reported.

Newly Developed Method (Slope of Radiochemical Evaporation)

A new Slope Method of analyzing the MESERAN data has recently been developed and improved. The Slope Method is the slope of the evaporation of radiochemical (the post-peak portion of the curve shown in Figure 3 as the B-C line). Even though this method of analysis was known as a viable method since the 1960s and 1970s, it was not a practical method to use until microcomputers became widely available so the data could be manipulated easily. Some initial slope calculations were available with the automatic MESERAN units in the mid-to-late 1980s and early 1990s; however, KCP found them of limited use at the time. This project further developed the slope technology to its current state.

Based upon raw data minus background, Figure 5 illustrates three typical experimentally derived curves of natural logarithm (Ln or ln) counts per second minus background vs. time in seconds. Figure 5 is similar to Figure 4 except raw data from actual tests are shown.

In Figure 5, the upper curve represents a high level of organic residue, the middle curve represents a medium level of organic residue, and the lower curve represents a low level of organic residue. The scatter, particularly at the lower values, is due to the inherent randomness of radiation (the Poisson distribution in which the square root of each count total is the best estimate of one standard deviation). In an effort to reduce the effect of the randomness of radiation, the data (ln (counts – background)) are “smoothed” from six seconds through 115 seconds (for 120 second length tests) and replotted. The smoothing is accomplished by summing the ln (counts – background) at the sixth second with the previous five seconds and the subsequent five seconds and dividing this number by 10. A divisor of 10 is used instead of 11 because it is statistically sound to take the number of items being smoothed and subtract one from it because a degree of freedom is lost. This process is carried out through the 115th second and the subsequent data are replotted as smoothed (ln (counts – background)) versus time. Figure 6 represents the same data as in Figure 5 except that the data in Figure 6 are logarithmically smoothed to increase the reliability of the individual points. The plotted smoothed curve is then analyzed via linear regression to determine the slope of the post-peak line (down to near background) which best fits the data representing the evaporative process. The determined slope is multiplied by –10,000 to convert it to a positive integer; this becomes the reported MESERAN slope value with units of smoothed (ln (counts – background))/sec x (–10,000).

Figure 5. Plot of Raw Data Showing Low, Medium, and High Levels of Organic Contamination

Comparison of Methods

The Slope Method is more sensitive than the Total Counts Method, especially at low levels of contamination. For measuring microorganic residues, the Total Counts Method of analysis (i.e., the area under each overall curve based on actual counts) is valid from somewhat less than 200 ng to approximately 100,000 ng (100 μ g). Higher total counts are indicative of higher organic residue levels and vice versa. The Slope Method of data analysis, normally based on statistically smoothed data and based on the log count vs. time relationship, increases the sensitivity of the lower limit markedly (to less than 1 ng) since the total counts method (the total area under each curve) approaches statistical insignificance somewhat below 200 ng. Expressed as a positive integer, the higher (or steeper) the slope, the cleaner the surface and conversely, the lower (or more flat) the slope, the higher the residue.

Figure 6. Logarithm Plot of Smoothed Data Showing Low, Medium, and High Levels of Organic Contamination

Both the Total Counts and Slope Methods of analysis can be used qualitatively or quantitatively. Qualitative measurements can be made by comparing the results obtained on the surface being tested to the results obtained from known clean standards for that particular surface. Quantitative measurements can be made by comparing the data to previously performed calibrations of contamination to obtain a result for the contamination amount.

Formulation of Calibration Solutions

Calibrations of various contaminants (oils, greases, mold releases, rosin fluxes, etc) typically encountered in KCP production operations have been performed to develop calibration curves for these contaminants on substrates of interest. MESERAN total counts and slope results obtained can now be compared to the calibration curves to determine quantitative amounts of contamination detected.

The volumetric dilution process for making calibration solutions is shown in Figure 7.

Figure 7. Calibration Solution Formulation

Volumetric dilutions were used to make contamination solutions for depositing known amounts of the organic contaminant on reference substrates (e.g., aluminum panels, stainless steel disks, glass cones, etc.). These calibrations were performed in the following manner. A master calibration solution was prepared in a 10-mL volumetric flask by dissolving 100 mg of the organic contaminant in 10 mL of solvent (e.g., cyclopentane, methylene chloride, or hexane that has been double distilled in an all-glass still with no grease in the joints – NVOR of these solvents are approximately 10 ppb). The master calibration solution was thoroughly mixed and 1 mL of this solution was placed in another 10-mL volumetric flask. The second volumetric flask was then diluted with the double distilled solvent until the solution level was at 10 mL and this solution was thoroughly mixed. Subsequent dilutions were carried out in a similar fashion. Ten microliters (μL) of each calibration solution were deposited on the precleaned substrates and allowed to evaporate. This resulted in the following amounts of contamination on the substrates: 1 ng, 10 ng, 100 ng, 1 μg , 10 μg , and 100 μg . In some cases, intermediate levels were obtained by depositing 3 μL and 5 μL of the calibration solutions.