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# Development of a Replacement for Trichloroethylene in the Two-Stage Cleaning Process

W. B. Harding

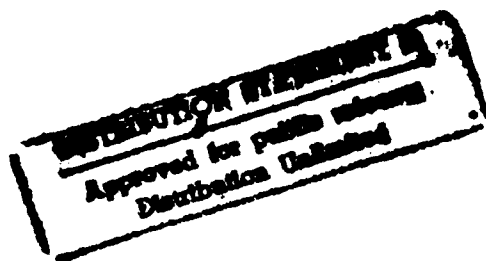
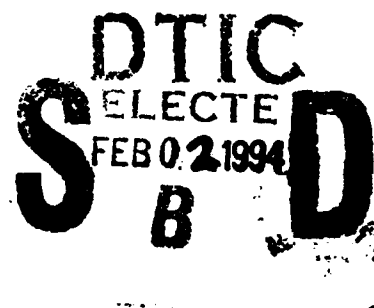
**AD-A275 245**



KCP-613-4941

Published December 1992

Topical Report



Prepared Under Contract Number DE-ACO4-76-DP00613 for the  
**United States Department of Energy**

**94-02930**



**Allied-Signal Aerospace Company**

**Kansas City Division**



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DE-ACO4-76-DP00613.

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P.O. Box 419159  
Kansas City, Missouri  
64141-6159

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Topical Report  
W. B. Harding, Project Leader

**Technical Communications  
Kansas City Division**

***Allied-Signal Aerospace Company***



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## Abstract

*Isopropyl alcohol, d-limonene, and a synthetic mineral spirits were compared for effectiveness as replacements for trichloroethylene in an ultrasonic cleaning process. All were found to be suitable. Isopropyl alcohol is recommended as the replacement.*

## Summary

A solvent was sought to replace trichloroethylene in the two-stage cleaning process that is used in the Allied-Signal Inc., Kansas City Division (KCD) Miniature Electro-Mechanical Assembly Department. The process is an ultrasonic cleaning process in which product is first cleaned in trichloroethylene and then in isopropyl alcohol. After a general review of the properties of available solvents, isopropyl alcohol, d-limonene, and a synthetic mineral spirits, were chosen to be evaluated as trichloroethylene replacements.

Stainless steel test panels were cleaned and then soiled with several different organic materials. Certain of the panels were cleaned by the two-stage process.

The others were cleaned by the two-stage process using one or another of the solvents under evaluation in the place of the trichloroethylene.

The cleanliness of the panels was determined by Auger and photoelectron spectroscopy. The panels cleaned with any of the three solvents under evaluation were found to be as clean as those cleaned by the standard two-stage process.

Because of simplicity and minimization of inventory, it is recommended that the two-stage process be changed to use isopropyl alcohol in both stages.

## Discussion

### Scope and Purpose

A cleaning process that has been used for many years on stronglinks and other mechanisms is the "two-stage process." It is an ultrasonic cleaning process that is carried out in either stainless steel or Pyrex beakers, typically of 1.5- or 2-liter volume. Two beakers are filled approximately two-thirds full, one with high-purity trichloroethylene and one with high-purity isopropyl alcohol. The beakers are partially immersed in water in an ultrasonic cleaner. Approximately 0.1 volume percent of a wetting agent such as a common dishwashing liquid is added to the water to enhance the transmission of the ultrasonic energy into the beakers.

The ultrasonic generator is turned on and allowed to run for ten minutes to drive dissolved gases, principally air, from the water and the solvents.

Product is ultrasonically cleaned for two to three minutes in trichloroethylene, blown off with nitrogen, cleaned for another two to three minutes in isopropyl alcohol, blown off with nitrogen, and vacuum baked.

The process is used at Allied-Signal Inc., Kansas City Division (KCD), in two locations in the Miniature Electro-Mechanical Assembly Department. The process is also required in the production of several purchased products. To comply with a policy requiring reducing the usage of halogenated solvents to the lowest practical level, a project was started to find acceptable alternate cleaning processes. Two approaches were followed. One was

to change to a water-based cleaning process. Prior research had shown it to be effective for most of the product that required cleaning, and conversion to it is proceeding in these cases.

The other cases were where the all-aqueous process was not available or where the product was an assembly with joints and other internal features wherein detergents could be trapped if the water-based process were used. Here the two-stage process was to be retained with the trichloroethylene replaced with a different and acceptable solvent.

This report covers the evaluation of solvents for this purpose.

### Activity

#### Selection of Candidate Solvents

All commercially available solvents, except clear health hazards such as aromatics, were considered. Although they were not counted, on the order of a hundred candidates were involved. In an initial screening several were tested for their abilities to dissolve a group of organic soils. Solubility is important because dissolution is the main mechanism by which a solvent removes soil. On this basis, the following were removed from further consideration: butyl lactate, dimethyl succinate, hexyl and decyl acetates, propylene and dipropylene glycol ethers, and propylene carbonate. Three solvents were selected for the first series

of tests. Others would be tested if these proved unacceptable.

One candidate solvent was isopropyl alcohol. Here the alcohol would be used in both stages, an approach that offers clear benefits in simplicity and familiarity.

Another candidate was d-limonene. It has been extensively evaluated as a trichloroethylene replacement in the spray cleaning of certain electronic assemblies. The solvent has a strong citrus odor that, to ensure operator comfort, can require modification of exhaust systems. At present, d-limonene is not provided in the same high levels of purity as are trichloroethylene and isopropyl alcohol.

The third candidate was a proprietary mixture of synthetically produced undecane, dodecane, and tridecane. It is essentially a highly refined mineral spirits with a mild odor. It is preferred to mineral spirits because the latter is a complex mixture whose composition varies with the supplier and the crude petroleum from which it is obtained.

### **Experimental Approach**

The two-stage process was jointly developed by the Sandia Corporation and the KCD in the early 1960s. No records of the cleanliness criteria used to qualify it and none of the persons involved remain. The cleanliness requirement placed by the cleaning specification on cleaned parts is, "Cleaned piece parts and subassemblies shall be free from particles, fibers, and films that are visible at up to 30 power magnification." This is a simple requirement that can be readily exceeded by many solvents to yield a level of cleanliness well beyond it.

Because the existing process had been used for many years, yielding this higher level of cleanliness, it was felt that any replacement process should be required to produce at least the same level of cleanliness, not merely the formally stated requirement. The approach selected, therefore, was to soil test specimens with representative soils, clean them by the standard two-stage process, clean equivalent specimens using the candidate solvents, and compare the levels of cleanliness by Auger spectrometry, a sensitive procedure capable of detecting traces of foreign material on surfaces.

### **Soils**

Two-stage cleaning is used to clean incoming and in-process product. In each case, the product already is relatively clean and the cleaning process is required only to remove light handling and environmental soils. The soils could be any of a large number of materials. The test soils selected were among many products found in a factory environment of the kind at the KCD. They all are organic and represent several types of organic materials. The soils are described in Table 1.

Particles and fibers were not included in the soils because they are mechanically removed by the ultrasonics, and removal is relatively unrelated to the solvent used.

Ten-g/L solutions of the test soils were prepared using convenient solvents: trichloroethylene, chloroform, methylene chloride, and hexane.

The test specimens were half-inch square panels of 302 stainless steel cut from 1/32-inch sheet and electropolished. (In other, unpublished, studies it was found



**Table 1. Test Soils**

Soil	Description
Neatsfoot Oil	Triglyceride; primarily of oleic acid, less of palmitic and stearic acids.
Lanolin, Anhydrous*	Esters and polyesters of 33 high molecular weight alcohols and 36 fatty acids.
Beeswax	Esters of straight chain alcohols with even carbons 24 to 36 and of straight chain acids with even carbons up to 36 and straight chain hydrocarbons of odd carbons 21 to 33.
Erucamide**	A fatty amide with average molecular weight of 335.
Dioctylphthalate	A widely used plasticizer.
Stearic Acid	A mixture of closely related fatty acids.
Kester 197 Flux	Rosin dissolved in isopropyl alcohol.
WD-40	A mixture of light hydrocarbon oils.
Apiezon H	Extensively branched and unsaturated petrolatum grease.
Apiezon L	Similar to Apiezon H but of higher molecular weight.
Sunicut 462-P	A cutting oil; mix of petroleum oils, sulfurized lard oil, ethylene/propylene copolymer, chlorinated olefins, carbon 4 through 8 sulfurized olefins.

\* Lanolin, as supplied, usually contains approximately 25 percent water. For these tests the lanolin was melted to drive off the water and produce a more difficult-to-remove material.

\*\* Erucamide is commonly added to polyethylene film to enhance slipping of films against each other. Erucamide will transfer to products that are packaged in polyethylene bags.

that organic soils clung more tenaciously to electropolished than to as-rolled stainless steel or to glass.) The specimens were cleaned by the aqueous process mentioned before and air-dried. Some cleaned specimens were set aside to serve as cleanliness standards and blanks. Each of the rest was dipped into one of the soil solutions and air-dried. (Specimens were contaminated with individual soils rather than a mixture of soils in the belief that removal of a mixture is an average property of the constituents allowing the removal of more difficult soils

that by themselves might be more obdurate.) Several weighed, larger specimens (1 in. x 1.5 in.) were immersed in erucamide, beeswax, Vaseline, and Apiezon H solutions and reweighed after drying. From the increase in weight the applied soil was estimated to be 100 to 300 nm thick.

Several cleaned specimens were immersed in soil solutions that were reduced in concentration to 1 g/L, a tenth of the standard solutions. These were analyzed in the soiled state to provide a

measure of the response from a lightly contaminated specimen.

The study was carried out in four sets of soiled panels. The panels were cleaned by the two-stage process using either trichloroethylene or a candidate solvent in the first stage. After air-drying, the specimens were packaged individually in clean glass vials with Teflon-lined caps.

The sets are described in Table 2.

In the Auger evaluations, spectra were obtained at two locations on each specimen. An initial spectrum was run at each location. Approximately 3 nm of material were sputtered away and another spectrum was run. Another 3 nm were sputtered away and a third spectrum was run. XPS of Set 2 also was run at two locations, but sputtering was not done. Figure 1 is a typical Auger spectrum. Figure 2 is a typical XPS spectrum.

In both techniques the response is from the upper 5 nm of the surface and, so, represents the material on the surface. The spectrum is a series of peaks, each characteristic of a specific chemical

element and of a size that is related to the amount of the element present.

The elements that were typically found were the major constituents of stainless steel (iron, chromium, and nickel, with a lesser amount of carbon), oxygen from the inherent passive film, and carbon from residual soils. Small amounts of silicon and phosphorous were common. No other elements were present in significant amounts.

The cleanliness of a specimen was estimated by comparing the sizes of the responses from carbon and from iron. Iron is a good benchmark because it is the major constituent of the steel and is present in the same amount. Its response is reduced by foreign material on the surface. Since the foreign material in these tests is organic, the carbon response is a function of the amount of foreign material present. In each spectrum the level of cleanliness was estimated by calculating the carbon/iron ratio, the lower the ratio, the cleaner the surface.

**Table 2. Description of Test Sets**

Set	Description
1	Compared isopropyl alcohol and trichloroethylene. Neatsfoot oil, Apiezon L, and dioctylphthalate were not included.
2	Compared d-limonene and synthetic mineral spirits. Apiezon L and H and erucamide were not included.
3	Compared trichloroethylene, isopropyl alcohol, and d-limonene. Soils were neatsfoot oil, lanolin, Sunicut 462-P, and erucamide.
4	Recompared trichloroethylene and isopropyl alcohol and, with two soils, aqueous-based cleaning. Soils were erucamide, beeswax, Sunicut 462-P, neatsfoot oil, and stearic acid. Aqueous-cleaned specimens were soiled with erucamide and Sunicut 462-P.

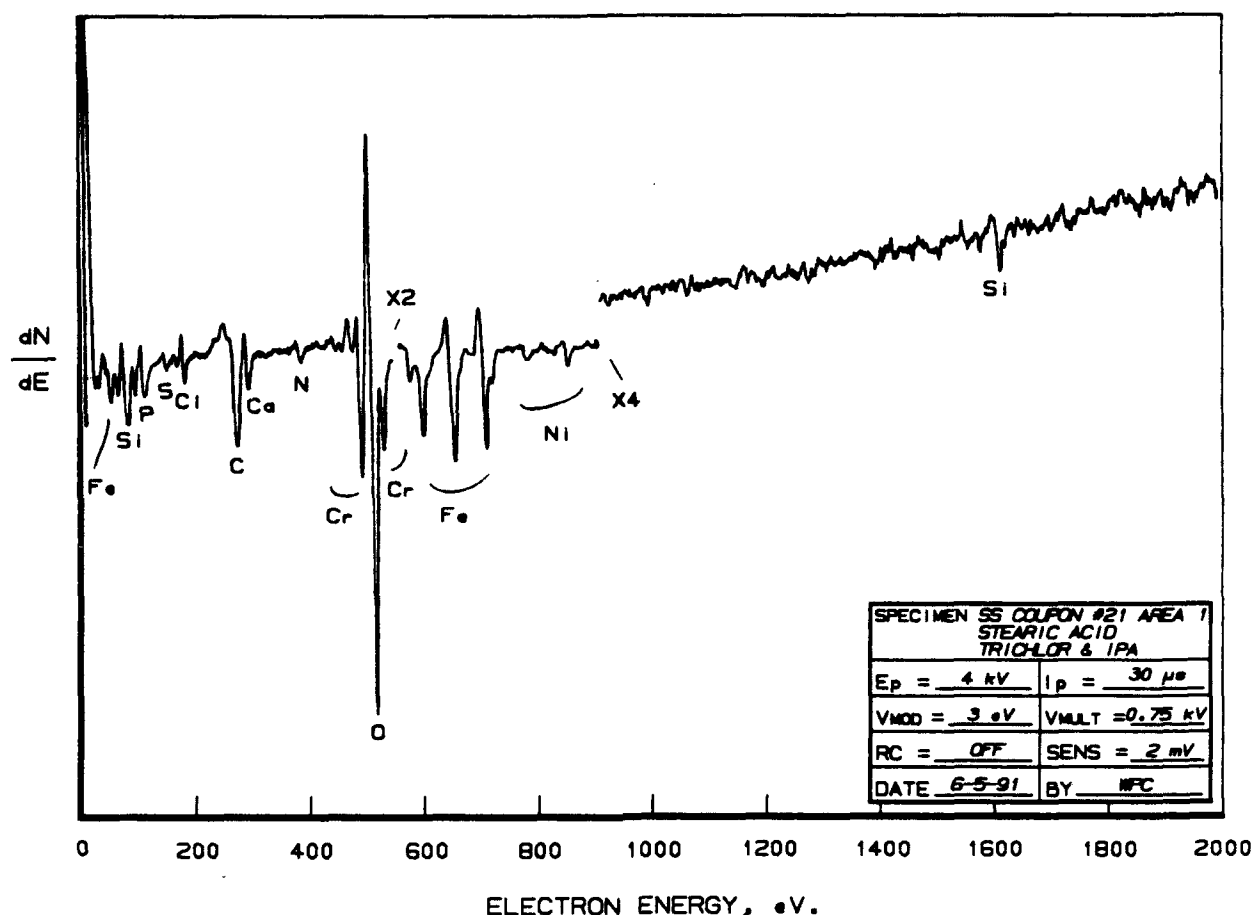


Figure 1. Auger Spectrum of Cleaned Stearic Acid-Soiled Panel, Trichloroethylene in First Stage

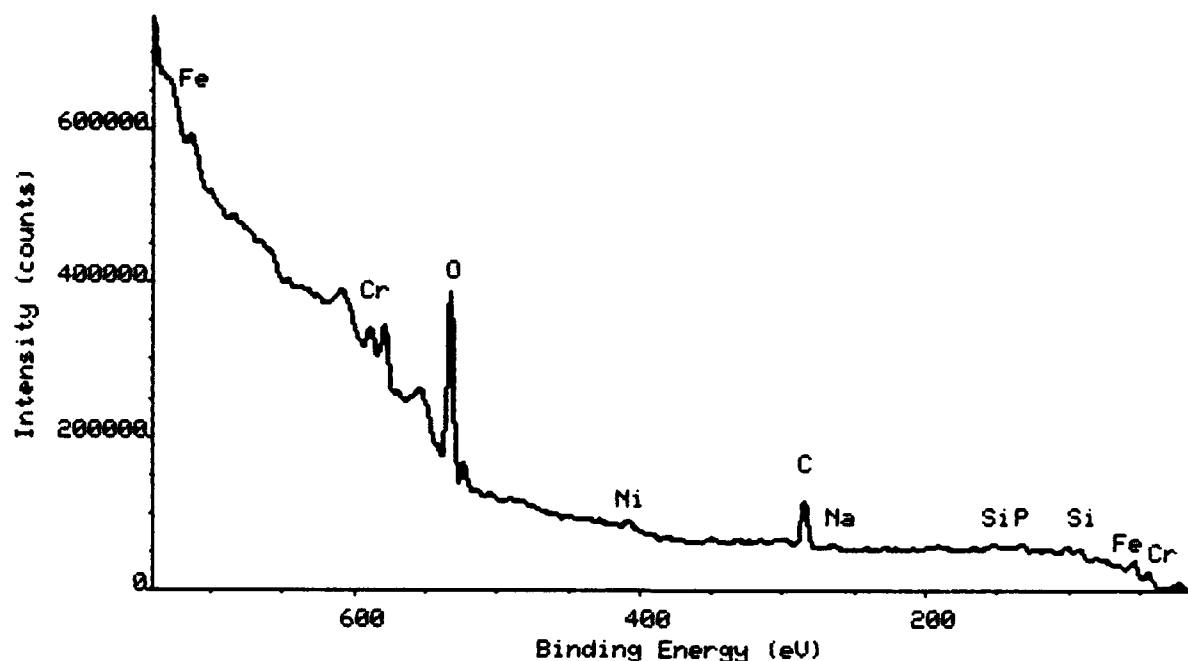
## Results and Discussion

### Standard Soil Specimens

The Auger analyses of the standard (uncleaned) soil specimens are given in Table 3. During sputtering, all of the panels except WD-40 approached a stable carbon/iron ratio of 0.2 to 0.6, which can be taken as the ratio of the carbon in the alloy to the iron and, thus, the sign of a foreign material-free surface. The soil films, which were produced with ten-to-one dilutions of the standard soil solutions, were mostly 3 to 6 nm thick.

### Set Number One

The Auger results for the first set of soiled and cleaned specimens are given in Table 4. The initial values are comparable to the values obtained for the clean blank (0.26 and 2.81, mean = 1.54), are substantially lower than those for the soiled standards (Table 3), and are of the same magnitude as the 3- and 6-nm sputtered soiled standards. On all of the cleaned specimens there is a trace of carbon that is removed in the first 3 nm of sputtering. The mean value and standard deviation for the trichloroethylene-cleaned specimens are 1.213 and 0.510, for the isopropyl



**Figure 2. XPS Spectrum of Cleaned Stearic Acid-Soiled Panel, Synthetic Mineral Spirits in First Stage**

alcohol specimens, 0.917 and 0.510. There is no difference between two-stage cleaning with trichloroethylene and isopropyl alcohol and two-stage cleaning with isopropyl alcohol alone. The two processes were effective for all of the soils.

### Set Number 2

Table 5 gives the results of the XPS analysis of Set 2. As before, two areas on each panel were evaluated.

The clean blank panel values were 0.33 and 0.26 with a mean of 0.29.

A dioctylphthalate-soiled panel cleaned with trichloroethylene had values of 0.30 and 0.27, mean 0.29.

A dioctylphthalate-soiled panel cleaned with isopropyl alcohol had values of 0.30 and 0.27, mean 0.29.

The means and standard deviations are 0.34 and 0.049 for d-limonene and 0.35 and 0.071 for the synthetic mineral spirits. There is no difference between the two solvents with the possible exceptions that d-limonene was slightly less effective for dioctylphthalate and the synthetic mineral spirits slightly less effective for Sunicut 462-P. Neither achieved the cleanliness of the blank although both were within a standard deviation of the blank value of 0.29.

### Set Number 3

The results of the Auger examination of Set 3 are given in Table 6. The means and standard deviations for trichloroethylene, isopropyl alcohol, and d-limonene are, respectively, 0.30 and 0.08, 1.28 and 1.72, and 0.42 and 0.10. The higher isopropyl alcohol mean is due to the erucamide specimen; without it the mean is 0.52 and

**Table 3. Auger Measurements of Carbon/Iron Peaks for Standard Soil Specimens**

Soil	Sputter Depth (nm)			
	0	3	6	9
<b>Apiezon H</b>				
Area #1	86	1.72	0.32	0.50
Area #2	104	9.52	1.29	0.47
<b>Beeswax</b>				
Area #1	93.8	8.09	0.59	0.11
Area #2	17.5	0.85	-	-
<b>Erucamide</b>				
Area #1	10.7	0.64	0.51	0.44
Area #2	49.0	3.36	0.32	0.36
<b>Kester 197</b>				
Area #1	23.9	0.18	0.51	0.57
Area #2	27.6	8.53	2.30	0.21
<b>Lanolin</b>				
Area #1	49.7	14.2	0.20	0.22
Area #2	89.2	7.4	0.56	0.40
<b>Stearic Acid</b>				
Area #1	11.0	0.33	0.37	0.24
Area #2	10.3	0.37	0.31	0.22
<b>Sunicut 462-P</b>				
Area #1		0.28	0.30	0.28
Area #2	3.12			
<b>WD-40</b>				
Area #1	58.9	29.9	3.03	2.48

standard deviation is 0.38. The value of 6.7 for Area #2 of the isopropyl alcohol-cleaned erucamide specimen cannot be explained. Possibly it was a local contaminate picked up in handling. Whatever the material, it was removed during the first 3-nm sputter; and the value after 3 nm was lower than for trichloroethylene and d-limonene.

#### **Set Number 4**

Sets 1 through 3 indicated that isopropyl alcohol can replace trichloroethylene. Set 4, the results of which are in Table 7, was designed to confirm this. Two other purposes were included. First, most of the spectra in the prior runs included small peaks for silicon. These could have been from adsorbed silicate left from the sodium metasilicate in the aqueous cleaner used

**Table 4. Auger-Determined Carbon/Iron Ratios of Set 1**

Soil	Trichloroethylene	Isopropyl Alcohol
Apiezon H		
1	2.30	0.65
2	0.73	1.46
Mean	1.56	1.06
Beeswax		
1	0.92	1.08
2	2.00	1.22
Mean	1.46	1.15
Erucamide		
1	0.60	1.86
2	0.30	2.07
Mean	0.45	1.96
Kester 197		
1	0.72	0.50
2	3.33	0.41
Mean	2.02	0.46
Lanolin		
1	1.69	0.81
2	0.19	0.56
Mean	0.94	0.64
Stearic Acid		
1	1.41	0.28
2	1.71	0.32
Mean	1.56	0.30
Sunicut 462-P		
1	1.06	0.53
2	0.80	1.13
Mean	0.93	0.83
WD-40		
1	0.76	0.73
2	0.87	1.06
Mean	0.82	0.90

to preclean the panels. The silicon/iron ratios were calculated as well as the carbon/iron.

**Table 5. XPS Measurements of Carbon/Iron Peaks of Set 2**

Soil	Synthetic Mineral Spirits	D-Limonene
Diethylphthalate		
1	0.29	0.48
2	0.29	0.45
Mean	0.29	0.46
Lanolin		
1	0.41	0.35
2	0.39	0.26
Mean	0.40	0.30
Stearic Acid		
1	0.37	0.33
2	0.37	0.36
Mean	0.37	0.35
WD-40		
1	0.33	0.33
2	0.33	0.31
Mean	0.33	0.32
Kester 197		
1	0.26	0.30
2	0.26	0.30
Mean	0.26	0.30
Erucamide		
1	0.32	0.35
2	0.42	0.32
Mean	0.37	0.33
Sunicut 462-P		
1	0.52	0.35
2	0.49	0.36
Mean	0.50	0.36
Beeswax		
1	0.30	0.35
2	0.30	0.34
Mean	0.30	0.34
Apiezon H		
1	0.35	0.32
2	0.32	0.33
Mean	0.33	0.32

**Table 6. Auger-Determined Carbon/Iron Ratios of Set 3**

Soil	Sputter Depth (nm)								
	Trichloroethylene			Isopropyl Alcohol			D-Limonene		
	0	30	60	0	30	60	0	30	60
<b>Sunicut 462-P</b>									
Area #1	0.50	0.25	0.15	1.35	0.15	0.10	0.12	0.15	0.11
Area #2	0.14	0.15	0.13	0.78	0.14	0.10	0.73	0.15	0.07
Mean	0.32	0.20	0.14	1.06	0.14	0.10	0.43	0.15	0.09
<b>Neatsfoot Oil</b>									
Area #1	0.16	0.29	0.35	0.20	0.30	0.09	0.21	0.37	0.18
Area #2	0.24	0.08	0.08	0.11	0.34	0.11	0.86	0.13	0.28
Mean	0.20	0.18	0.22	0.16	0.32	0.10	0.53	0.20	0.23
<b>Apiezon L</b>									
Area #1	0.52	0.23	0.10	0.56	0.14	0.11	0.50	0.15	0.10
Area #2	0.30	0.24	0.14	0.41	0.13	0.10	0.18	0.15	0.10
Mean	0.41	0.24	0.12	0.44	0.14	0.10	0.34	0.15	0.10
<b>Lanolin</b>									
Area #1	0.36	0.16	0.16	0.52	0.15	0.10	0.48	0.13	0.11
Area #2	0.20	0.18	0.18	0.32	0.14	0.10	0.10	0.18	0.10
Mean	0.28	0.17	0.17	0.42	0.14	0.10	0.29	0.16	0.10
<b>Erucamide</b>									
Area #1	0.34	0.33	0.14	1.92	0.13	0.09	0.60	0.28	0.27
Area #2	0.23	0.26	0.14	6.67	0.23	0.17	0.33	0.15	0.10
Mean	0.28	0.30	0.14	4.30	0.18	0.13	0.50	0.22	0.33

In all of the sets, a single quantity of solvent was used for each set rather than a fresh quantity for each panel because the amount of soil picked up by the solvent during the set amounted to only a few parts per million and the solvent was still within purity limits. To confirm this, blank panels were processed through the solvents at the beginning and ending of the Set 4 runs and compared.

The soils in Set 4 were those that appeared to be the more difficult to remove in the prior sets: erucamide, beeswax,

Sunicut 462-P, neatsfoot oil, and stearic acid. The first-stage solvents were trichloroethylene and isopropyl alcohol. Also, erucamide- and Sunicut 462-P-soiled panels were cleaned in the aqueous cleaner.

The mean value of the silicon/iron ratios for the pre-sputtered panels was 0.36. After 3 nm of sputtering, it was 0.07. Silicon, probably as silicate, is present and less than 3 nm thick. The mean value for the blank panel was 1.14.

**Table 7. Auger-Determined Carbon/Iron Ratios of Set 4**

Panel	Trichloro-ethylene	Isopropyl Alcohol	Aqueous Detergent
<b>Initial Solvent</b>			
1	0.28	0.26	
2	0.57	0.20	
Mean	0.43	0.23	
<b>Final Solvent</b>			
1	0.24	1.36	
2	0.29	0.22	
Mean	0.26	0.79	
<b>Erucamide</b>			
1	0.30	0.32	1.05
2	0.21	0.19	0.25
Mean	0.26	0.26	0.65
<b>Beeswax</b>			
1	0.50	0.32	
2	0.61	0.36	
Mean	0.56	0.34	
<b>Sunicut 462-P</b>			
1	0.68	1.22	1.19
2	0.90	1.89	1.00
Mean	0.79	1.56	1.10
<b>Neatsfoot Oil</b>			
1	15.2	0.22	
2	4.00	0.30	
Mean	9.60	0.26	
<b>Stearic Acid</b>			
1	0.97	0.26	
2	0.75	0.23	
Mean	0.86	0.28	

All panels except for the trichloroethylene-cleaned stearic acid and neatsfoot oil ones were clean after 3 nm sputtering. The latter still had a ratio of 0.47 after sputtering 12 nm; possibly, through error, the panel was not cleaned.

The final trichloroethylene blank panel was clean. The final isopropyl alcohol panel had a higher carbon/iron ratio, but this was due to one of the two readings.



## **Conclusions and Recommendations**

All but a few of the panels, regardless of the cleaning process used and including the standard clean panels that had not been soiled, had traces of organic materials on them that were less than 3 nm thick. All four solvents, with isolated exceptions, were equally effective as first-stage solvents. The exceptions were not associated with any one solvent.

Isopropyl alcohol is recommended as the replacement for trichloroethylene because of its availability in high-purity and its mild odor and because of the simplifications provided by using a single solvent rather than two different ones.

## **Accomplishments**

It was demonstrated that isopropyl alcohol, d-limonene, and synthetic mineral spirits, each, is an effective replacement for trichloroethylene in two-stage cleaning. This was done with cleaning tests that established the level of cleanliness achieved by the traditional two-stage cleaning process. Cleanliness was measured by Auger spectrometry. Additional studies demonstrated that the process is equally effective when trichloroethylene is replaced by isopropyl alcohol, d-limonene, or synthetic mineral spirits. Isopropyl alcohol was recommended over the others for simplicity and familiarity. The change will contribute to the desired reduction in use at the KCD of trichloroethylene.