

**AQUEOUS CLEANING AND VERIFICATION PROCESSES FOR  
PRECISION CLEANING OF SMALL PARTS**

**By**

**Gale J. Allen**

**National Aeronautics and Space Administration  
Materials Science Laboratory (DM-MSL-1)  
Kennedy Space Center, Florida 32899**

**Kenneth A. Fishell**

**Wiltech of Florida Corp., Inc.  
Kennedy Space Center, Florida 32899**

**ABSTRACT**

The National Aeronautics and Space Administration (NASA), Kennedy Space Center (KSC) Materials Science Laboratory (MSL) has developed a totally aqueous process for precision cleaning and verification of small components. In 1990 the Precision Cleaning Facility at KSC used approximately 228,000 kg (500,000 lbs) of chlorofluorocarbon (CFC) 113 in the cleaning operations. It is estimated that current CFC 113 usage has been reduced by 75% and it is projected that a 90% reduction will be achieved by the end of calendar year 1994.

The cleaning process developed utilizes aqueous degreasers, aqueous surfactants, and ultrasonics in the cleaning operation and an aqueous surfactant, ultrasonics, and Total Organic Carbon Analyzer (TOCA) in the nonvolatile residue (NVR) and particulate analysis for verification of cleanliness. The cleaning and verification process will be presented in its entirety, with comparison to the CFC 113 cleaning and verification process, including economic and labor costs/savings.

**INTRODUCTION**

In 1991, NASA set forth a mandate to begin the reduction and eventual phase-out of all use of CFCs at their centers by the year 1995. Precision cleaning and surface wipe down accounts for approximately 80% of CFC use at KSC, Florida and is considered an integral player in the processing of shuttle hardware and ground support equipment.

The Component Refurbishment and Chemical Analysis Laboratory (CRCA) located at KSC supplies decontamination, cleaning, and refurbishment services to most all the direct NASA contractors at the center. The CRCA is a high volume precision cleaning facility which receives approximately 120 work tasks per day. These work tasks represent configurations ranging from the smallest nut or screw to the largest 18" pneumatically controlled butterfly valve. The components, as they process through the facility, are reduced to their smallest piece part and represent over 11,000 individual pieces per day or approximately 250,000 piece parts a month.

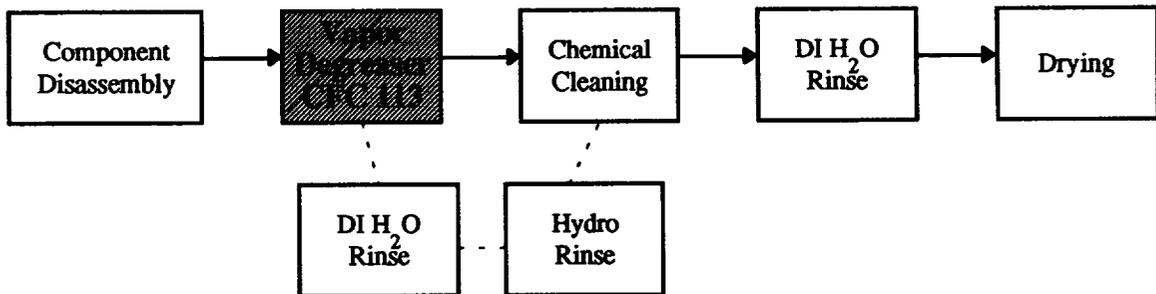
The NASA Materials Science Laboratory and Wiltech of Florida Corp., Inc., the contractor which operates the KSC CRCA facility, have been working together to implement a total aqueous process for precision cleaning and verification of small parts as an alternative to the current CFC 113 precision cleaning process used to clean aerospace hardware. It is believed that aqueous cleaning, if controlled and managed correctly, is the most desirable process to use. It is non-toxic, non-flammable, recyclable, and inexpensive.

## ROUGH CLEANING

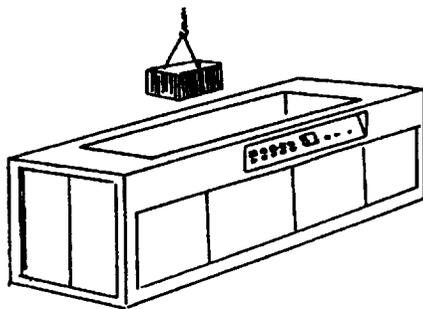
Components or hardware to be precision cleaned are brought to the CRCA by the many contractor customers at KSC in support of an array of aerospace support and launch activities. The components, when received from the customer, are routed to the rough clean and disassembly areas. They are reduced to the smallest piece part possible and readied for surface degreasing. Degreasing is the first step of the cleaning process. It is considered by many to be the most important use of CFC 113 in the precision cleaning process at CRCA. All fluorocarbon and hydrocarbon greases and oil films must be removed prior to chemical conditioning. Any oil or grease contamination could block the chemical surface conditioning and ultimately be suspect as the source of failure during the NVR analysis, (Kennedy, 1994).

The degreasing step of the CRCA rough cleaning process is shown in the flow process diagram of Figure 1. The degreasing process lowers parts into a typical vapor degreaser using CFC 113 as the degreasing medium as shown in Figure 2.

**Figure 1**  
**Rough Cleaning Flow Process**



**Figure 2**  
**Equipment and Solvent Specifications are:**



Tank Size:	132" x 6"
Solvent Volume:	210 Gallons
Solvent:	CFC 113
Work Area:	125" x 30"
Temperature of Solvent:	110 ± 5 °F
Condensing Coil Medium:	Chilled Water

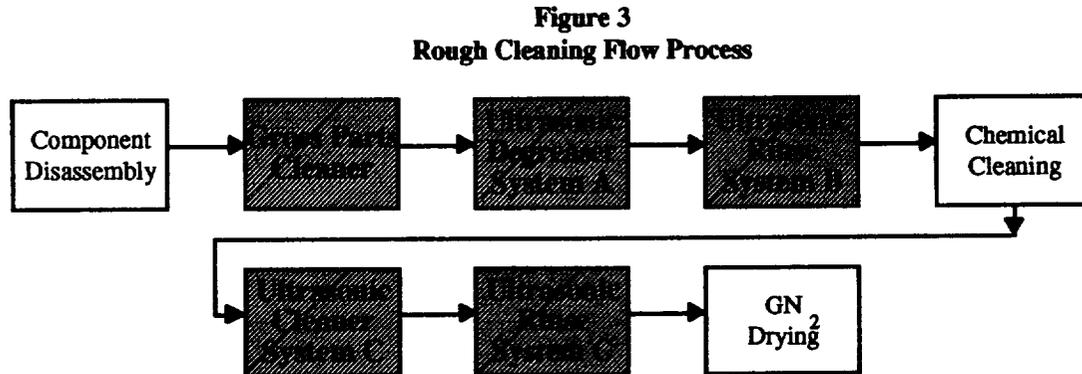
The parts remain in the solvent vapor until their surface temperature stabilizes at 110 ± 5 °F, or approximately ten minutes. Any stubborn contaminants are removed by dispersing the solvent through a pumping system and directing it to the part.

Often, after the parts were removed from the vapor degreaser, inspection revealed that fluorocarbon grease contamination remained. To remove this contamination, the parts are either brushed using a soft bristle brush with a surfactant and water or washed using a hand held pressurized hydro-wash

wand. The removal of all visual contaminants on the parts prior to entry into the chemical cleaning process generally warrants a smooth flow through the remaining cleaning process.

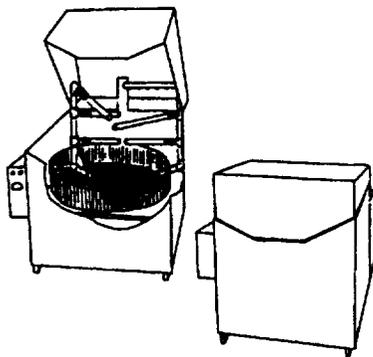
To eliminate vapor degreasing and CFC 113 during the rough cleaning process, a system of equipment and nonionic aqueous surfactants has been developed and is currently being phased into daily production. This system employs several pieces of equipment using ultrasonics, high pressure, high volume aqueous agitation, GN<sub>2</sub> agitation and hand wand spraying, (Kennedy, 1994).

The aqueous degreasing steps of the rough cleaning process are shown in the flow process diagram of Figure 3.



Aqueous degreasing begins with gross cleaning parts washing shown in Figure 4. This parts washer is required to remove gross contamination prior to entering the primary process degreaser, System A. This equipment is top loading to provide the most efficient access possible for the various size parts and production personnel. An overhead hoist provides the lifting capability for parts weighing up to 200 lbs. Three tiers of spray manifolds are available for the many different sized parts. These various levels of spray manifolds are adjustable to accommodate the many different heights. In addition, a rotating turntable rotates the parts during the wash and rinse cycles at a rate of three revolutions per minute resulting in approximately 95-100% contaminate removal efficiency.

**Figure 4**  
**Equipment and Solution Specifications**

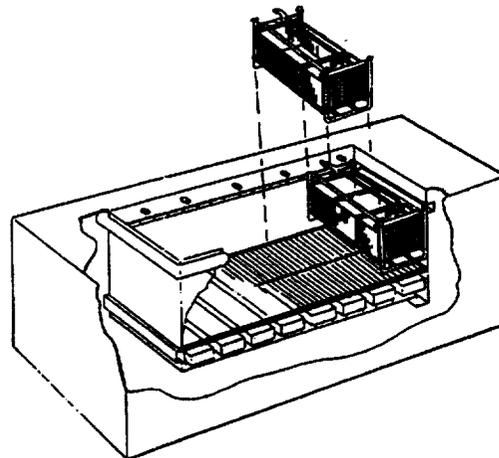


Equipment Foot Print:	4' x 4' x 6'
Work Access:	Top Loading
Tank Capacity:	30 Gallons
Surfactant:	Brulin 1990 GD @ 3%
Operating Temperature:	150 ± 10 °F
Functions:	Timer
Cycles:	Wash and Rinse
Operating Pressure:	400 ± 50 PSIG
Working Area:	3' x 3' x 3'

If inspection of the parts reveals a visual absence of contaminants, the parts are placed in the primary process degreaser, System A, shown in Figure 5. This degreaser is the main workhorse in removing contaminants that play a negative part in achieving precision clean hardware. It is a single tank

cabinetized ultrasonic cleaning system. The primary functions of the unit include: ultrasonic cleaning, agitation of parts, filtration of chemical solution, heating of cleaning solution, hand spraying of parts and GN<sub>2</sub> filter element processing. Ultrasonics, parts agitation and the nitrogen processing can be operated in an automated mode as well as manually. Parts processing for this unit is accomplished by the use of removable cradle fixtures which are supported inside the tank. The degreaser unit also contains a remote generator station for the ultrasonics.

**Figure 5  
Equipment and Solvent Specifications**



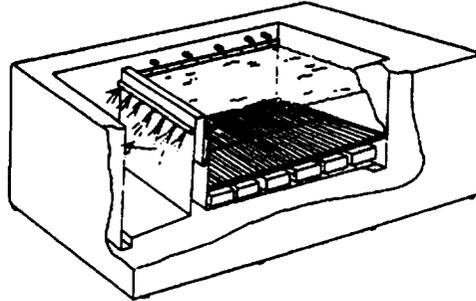
Equipment Foot Print:	113" x 54" x 54"	Electrical:	480 VAC, 30, 60 Hz, 90 AMPS
Work Area:	65" x 36"	Filtration:	<ul style="list-style-type: none"> <li>• 5 Micron Absolute</li> <li>• Cotton String</li> <li>• Porous Bag</li> </ul>
Work Access:	Top Loading	Ultrasonic Power:	9600 Watts @ 27 KHz
Tank Capacity:	260 Gallons	Average Ultrasonic Process Time:	10 Minutes
Surfactant:	Brulin 815 GD @		
Operating Temperature:	150 ± 10 °F		
Functions:	<ul style="list-style-type: none"> <li>• Agitation</li> <li>• Recirculation</li> <li>• GN<sub>2</sub> Injection</li> <li>• Ultrasonics</li> </ul>		

In this equipment, the parts are placed into special agitation racks. These racks are support fixtures that position the parts approximately eight to ten inches from the ultrasonic devices. They provide upper and lower agitation spray manifolds to assist the removal of remaining contaminants. Once the contaminants are removed and into solution, they are circulated through the filtration system where they are removed to help maintain clean solutions. The control panel consists of four separate timed systems: 1) the agitation system, 2) the ultrasonic system, 3) the GN<sub>2</sub> injection system, and 4) the recirculation system.

At the end of the primary degreasing effort, the parts are immediately removed and placed into the primary process rinse, System B, shown in Figure 6. The primary process rinse is a two tank cabinetized ultrasonic rinse system. The first tank is a manually operated rinse spray water deluge tank. The second tank is a status rinse tank with the following functions: ultrasonic rinsing, agitation of parts, filtration of rinse water and hand spraying of parts. The ultrasonics as well as the agitation can be

operated manually or automatically. Parts processing for this unit is accomplished by the use of removable cradles fixtures which are supported inside the tank. The rinse unit also contains a remote generator station for the ultrasonics.

**Figure 6  
Equipment and Solution Specifications**



Equipment Foot Print:	126" x 54" x 54"	Electrical:	480 VAC, 30, 60 Hz, 45 Amps
Work Area:	54" x 36" x 20"	Filtration:	• 5 Micron Absolute • Cotton String
Work Access:	Top Loading	Ultrasonic Power:	7200 Watts @ 40 KHz
Tank Capacity:	205 Gallons	Average Ultrasonic Processing Time:	5 Minutes
Solution:	DI Water		
Operating Temperature:	140 ± 10 °F		
Functions:	• Agitation • Recirculation • Ultrasonics		

As the parts are removed from the primary process degreaser, System A, they are immediately placed in a multi-manifold DI water spray rinse for approximately 30 seconds to remove any surfactant solutions from basket and parts. This is done to prevent any fast drying of contaminants when parts are removed from the previous 150 °F degreaser bath.

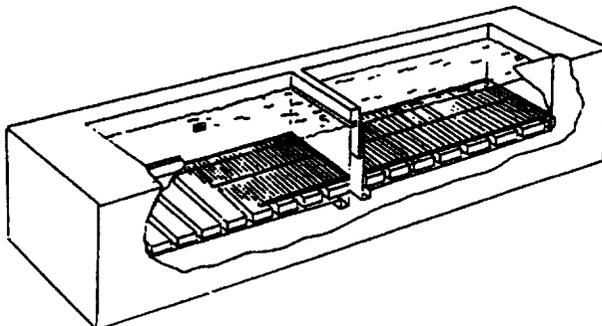
The parts are then placed in agitation racks like those installed in the primary process degreaser, System A. The parts are rinsed under agitation and ultrasonic conditions for five minutes.

Unlike the primary degreaser, fresh hot water enters the main tank through a five micron filter system and exits the tank over an open weir to drain.

The control panel is much like the primary process degreaser, System A, in that it consists of three separate timed systems: 1) the agitation system, 2) the ultrasonic system, and 3) the recirculation system.

The parts are removed from the primary process rinse, System B, and placed into one of the various chemical baths required for the various materials and their surface conditioning for removal of oxides and surface passivation. They are then rinsed with copious amounts of hot deionized water until a neutral pH between 6.0 - 8.0 is reached. Before the parts are allowed to dry, they are placed into the secondary process degreaser, System C, shown in Figure 7. This removes any contaminants received during the chemical cleaning baths.

**Figure 7  
Equipment and Solution Specifications**



<b>Equipment Foot Print:</b>	138" x 44" x 48"	<b>Electrical:</b>	• 480 VAC, 30, 60 Hz, 70 Amps
<b>Working Area:</b>		<b>Filtration:</b>	5 Micron Absolute
• <b>Wash Tank:</b>	78" x 28"		• Cotton String
• <b>Rinse Tank:</b>	60" x 28"		• Porous Bag
<b>Work Access:</b>	Top Loading	<b>Ultrasonic Power:</b>	
<b>Tank Capacity:</b>		• <b>Wash Tank:</b>	4800 Watts @ 40 KHz
• <b>Wash Tank</b>	160 Gallons	• <b>Rinse Tank:</b>	3600 Watts @ 40 KHz
• <b>Rinse Tank</b>	120 Gallons	<b>Average Ultrasonic</b>	
<b>Operating Temperature:</b>	140 ± 10 °F	<b>Process Time:</b>	
<b>Functions:</b>	• Recirculation	• <b>Wash Tank:</b>	5 Minutes
	• Ultrasonics	• <b>Rinse Tank:</b>	5 Minutes
<b>Surfactant:</b>	Dupont Zonyl FSN		

The secondary process degreaser is a two tank cabinetized ultrasonic clean and rinse system. The cleaning tank functions include: ultrasonic cleaning, heating of solution, filtration of wash solution and hand spraying of parts. The rinse tank functions include: ultrasonic rinsing and an overflow rinse to drain. Ultrasonics in the wash rinse tanks can be operated in a timed cycle or in a manual mode.

In this final degreasing process, the parts are placed into a surfactant and deionized water bath. They are ultrasonicated for a period of five minutes and then placed immediately into the rinse tank and ultrasonically processed for five minutes. After the final rinse, the parts are removed and prepared for cleanroom entry for particulate validation and NVR verification.

#### **VERIFICATION**

The current methods for verifying cleanliness are a gravimetric procedure using CFC 113 to measure nonvolatile residue and microscopic particulate measurement using a gridded filter. Alternatives include other organic solvents such as HCFCs, spectroscopic surface examination, and water. Water was chosen as the desirable verification solvent. The major drawback to water has been the lack of an established procedure for NVR and the issue of drying components after completion of the cleaning and

verification process. KSC has concentrated on developing an aqueous NVR procedure that can be used for small component verification. The two major issues to resolve if water were to be used in a verification process was the method of removal of any remaining NVR and particulate from the components the technique to use to measure the NVR and particulate removed.

Ultrasonics has been well documented as a successful method for the removal of surface contamination, (Kuttruff, 1991). Extensive evaluations were conducted to determine its applicability to NVR removal. These included the effectiveness of removal of low level surface contamination by selected materials, optimum frequencies and temperatures to be used, and the potential effects of ultrasonic cavitation on metallic and nonmetallic surfaces, (Mehta, 1992). Extensive experimentation concluded that 25 kilohertz frequencies and 52 degree Celcius water temperature were the best combination for maximum NVR removal, (Bryan and Gebert-Thompson, 1992).

Several analytical techniques were evaluated and the Total Organic Carbon Analyzer (TOCA) was selected as the technique of choice for detection of NVR. Through experimentation it was determined that the Total Organic Carbon Analyzer (TOCA) was the easiest and most repeatable and reliable of all analytical methods evaluated.

The cleaned components are rinsed with deionized water and ultrasonicated at 25 kilohertz for five minutes in 52 degree Celsius deionized water. Two hundred microliters of the water are injected into a high temperature (880<sup>o</sup> C) combustion chamber and any carbon is converted to carbon dioxide using high purity oxygen. Carbon dioxide is then measured using an infrared detector, (Allen, 1994).

A relationship between milligrams of NVR per 0.09 m<sup>2</sup> of surface area to total carbon was formulated using:

$$\frac{\text{mg NVR}}{0.09 \text{ m}^2} = \frac{(\text{TC}_s - \text{TC}_b)(\text{ml of water used})}{(\text{m}^2 \text{ in tank})(500 \text{ ml})(0.64 \text{ ppm/mg})}$$

where:

TC = Total carbon of sample

TC<sub>s</sub> = Total carbon in water blank

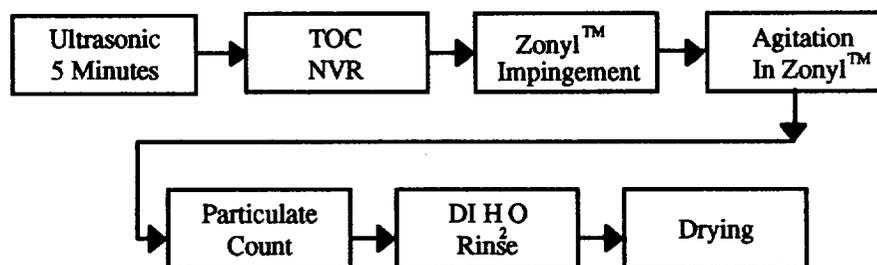
TC<sub>b</sub> = normalized value for TOCA sensitivity factor

0.64 ppm/mg = weighted average TOCA response to materials evaluated

The components are then analyzed for particulate using a 25 ppm mixture of Zonyl™ FSN and deionized water, (Clausen, 1994). Zonyl™ FSN is a nonionizing surfactant manufactured by Dupont. It is liquid oxygen (LOX) compatible and simply reduces the surface tension of water from 71 dynes per centimeter to approximately 34 dynes per centimeter. This allows complete wetting of the component surfaces and results in particulate analyses using the Zonyl™/water mixture which correlate well with the CFC 113 particulate analysis. The final step in the aqueous method of measuring particulate is similar to the CFC 113 method since the samples are agitated in the Zonyl™ water mixture and the mixture is then filtered through a gridded filter where the particulate retained on the filter pad is counted microscopically.

This aqueous system is in use at Kennedy Space Center and is working well. It has been noted that handling of the components through out the new process is much more critical than the CFC 113 process. The components must be completely clean prior to entering the clean room for verification and handled very carefully to avoid recontamination. In the prior process there was a final cleaning step after the components had been moved into the clean room. Figure 8 shows the sequence of the aqueous verification process.

**Figure 8  
Aqueous Verification Process**



It has been estimated that the process time will be lengthened by about 35 percent to 45 percent. This is not contact time since most of the process time is in ultrasonics which does not have to be attended. It is felt that some of the ultrasonic times may be decreased as the process is further evaluated and improved. The process itself is extremely economical. Table 1 summarizes the costs involving the use CFC 113 as a cleaning and verification solvent and Table 2 shows the costs associated with the solvents utilized new aqueous process.

**Table 1. CFC 113 Economic Analysis**

Year	Cost per Pound	Usage (pounds)	Annual Costs
1984	\$ 1.82	500,000	\$ 910,000
1994	\$ 8.75	500,000	\$ 4,375,000
Current Consumption		50,000	\$ 437,500

**Table 2. Aqueous Process Economic Analysis**

Product	Unit Cost	Annual Usage	Cost
Bruhin	\$ 12.00/gal	600 gal	\$ 7,200
Zonyl™ FSN	\$ 25.00/lb	16 lb	\$ 400
Breathing Air	\$ 11.25/1000 SCF	300,000 SCF	\$ 3,375
Water	\$ 0.08/gal	2,000,000 gal	\$ 160,000

## CONCLUSION

The NASA, Kennedy Space Center and Wiltech Corporation have developed a totally aqueous based process for the cleaning and verification of small components. These components can be either metallic or nonmetallic but are limited in size to and mass to 0.09<sup>2</sup> meters or approximately 1500 grams respectively. The new aqueous method does extend the overall process time but is very economical from a cost of materials standpoint. The verification technology is currently under evaluation for large components utilizing impingement via an air/water supersonic nozzle as the mechanical means for NVR removal.

## ACKNOWLEDGEMENTS

The authors wish to recognize the Kennedy Integrated Team (KIT), I-NET Incorporated, Wiltech Corporation, the University of Central Florida, and the Kennedy Space Center Materials Science

Laboratory for the hours of work put into this project and to congratulate them on a successful, environmentally safe process.

## REFERENCES

- <sup>1</sup> Allen, G. "An Aqueous Nonvolatile Residue Method for Cleanliness Verification" Precision Cleaning Vol II, No. 4, April, 1994, pp. 43-45.
- <sup>2</sup> Bryan, C. and Gebert-Thompson, K. "Alternatives to Chlorofluorocarbons", ASTM, STP 1181, 1992.
- <sup>3</sup> Clausen, C. "Annual Report on NASA CFC 113 NVR Project", NASA Grant No. NAG10-0094, July, 1994.
- <sup>4</sup> Kennedy Space Center "Chemical Cleaning and Testing Manual" No. 4-0-385, App. 8.9.23, 8.35.10 and 8.35.13 - 8.35.15, 1994.
- <sup>5</sup> Kuttruff, H. Ultrasonic Fundamentals and Applications, Elsevier Applied Sciences, London, New York, 1991.
- <sup>6</sup> Mehta, N. H. "Evaluation of Ultrasonic Cavitation of Metallic and Nonmetallic Surfaces", NASA/ASEE Summer Faculty Fellowship Program, NASA CR-191004, September, 1992, pp. 258 - 315.

