Laboratory Scale Evaluation of Hydra-Tone Graff-Off™ Coconut Oil-Based Degreaser

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NOTICE

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Abstract

This technical and economic assessment evaluated the effectiveness of a biodegradable, coconut oil-based degreaser called Graff-Off™. In immersion ("cold") cleaning and rinse tests, Graff-Off™ was compared to a conventional chlorinated solvent 1,1,1 trichloroethane (TCA) and to an alkaline cleaner Aeroclean DN-30 (DN-30). The cleaning process for Graff-Off™ and TCA was at room temperature and that of DN-30 was at 71 C. Both alternatives were found to be technically superior to TCA. Both alternative degreasers had lower cleaner costs and allowed a greater surface area to be cleaned per unit volume of degreaser than the TCA. Estimated savings were significant and capital requirements were modest. An economic assessment based on net present value, internal rate of return, and payback period indicated that Graff-Off™ and the DN-30 alkaline cleaner were extremely attractive alternatives to TCA (TCA assessment was based on immersion "cold" cleaning without vaporization and without TCA recycling).

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ACRONYMS AND ABBREVIATIONS

ASTM	American Society for Testing and Materials
CAA	Clean Air Act
CFC	Chlorofluoro Carbons
COCF	Chain of Custody Form
DN-30	Aeroclean DN-30 – an alkaline cleaner
EPA	(United States) Environmental Protection Agency
FTIR	Fourier Transform Infrared Spectroscopy
HBFC	Hydrogen bromine fluoro carbons
HCFC	Hydrogen chloro fluoro carbons
HTCI	Hydra-tone Chemicals, Inc.
IRR	internal rate of return
NPV	net present value
NRMRL	National Risk Management Research Laboratory
NVR	non-volatile residue
PCE	perchloroethylene
PE	purchased equipment
P2	Pollution Prevention
QC	quality control
TCA	1,1,1 trichloroethane – a solvent cleaner
TCE	trichloroethylene
TO	technical order
U.S. EPA	United States Environmental Protection Agency
VOCs	volatile organic compounds

1.0 Introduction

1.1 Background

The Pollution Prevention Act of 1990 made pollution prevention (P2) the national environmental policy of the United States. The U.S. EPA uses this policy to drive source reduction programs by preventing or minimizing wastes where they originate. P2 is also a vehicle for "reinventing" traditional Agency programs and devising innovative alternative strategies to protect public health and the environment. [1] The P2 Act of 1990 also covers the policy of the Clean Air Act (CAA). The policy of the CAA is to identify alternatives and replace to the maximum extent practicable, Class I and Class II substances with chemicals, product substitutes, or alternative manufacturing processes that reduce overall risks to human health and the environment. Class I substances include CFCs, halons, carbon tetrachloride, methyl chloroform, methyl bromide, and HBFCs. Class II substances include HCFCs and ozone-depleting substances [1].

Within the EPA, the National Risk Management Research Laboratory (NRMRL) works with industry to provide technical and economic information about new technologies for potential users so that they can achieve voluntary reductions in the use and release of hazardous substances. The NRMRL interests include industrial cleaning operations (e.g., parts degreasing, paints stripping, etc) where traditional solvent cleaning is being replaced with alternative methods. Traditionally, chlorinated solvents such as 1,1,1-trichloroethane (TCA), or trichloroethylene, or tetrachloroethane were used for removing contaminants from metal surfaces. Non-chlorinated solvents such as methyl ethyl ketone and toluene were also used for cleaning metallic parts. These chlorinated and non-chlorinated solvents have been found to be environmentally unfriendly due to groundwater contamination or volatile organic compound (VOC) emissions and belong to the Class I and/or Class II compounds under the CAA. Most of these solvents are also ozone-depleting substances and/or are targeted for reduced usage. It is, therefore, important to replace these organic solvents with more environmentally friendly cleaners such as aqueous-based cleaners.

1.2 Industrial Opportunity

Hydra-tone Chemicals, Inc. (HTCI) has introduced Graff-Off™ as an environmentally friendly degreasing agent that is designed to provide the same degreasing effect as conventional alkaline cleaners and solvent cleaning agents used in the parts cleaning industry. As claimed by HTCI's product literature, Graff-Off™ is 100% biodegradeable, non-hazardous, non-flammable, non-toxic, non-corrosive, and safe to use.[2] The Graff-Off™ product is a naturally derived product based on coconut oil and represents very low volatile organic compounds (VOCs). It is a ready-to-use liquid degreaser designed with a special microemulsion formulation. This unique technology is used for the removal of markings such as graffiti and other contaminants from hard surfaces while leaving the paint undamaged. Graff-Off™ has a flash point of > 93 C (> 200 F) and is considered non-flammable. This coconut-based degreaser does not have any SARA 313 list extremely hazardous substances and no California Proposition 65 components. Because of its degreasing capabilities, HTCI is confident that Graff-Off™ will be an effective parts cleaner.

1.3 Objective

The objective of this project was to assess the effectiveness of Graff-Off™ as a substitute for conventional alkaline cleaners and solvent cleaning agents. Generally three main issues are addressed when evaluating a replacement technology or a replacement product to reduce pollution. First, the proposed new technology or product must prove to be effective in performing the process function that it is intended to replace. Second, there must be a significant, measurable reduction in the quantity of waste hazard (pollutant) produced and in the level of hazards produced. Third, the economics of the alternative technology must be quantified and compared to the economics of the existing technology. The consideration of each issue is needed to enable the recommendation of a new technology/product as a feasible alternative to an existing technology/product.

The first goal in this project was to compare the cleaning efficiency of Graff-Off™ to that of an alkaline cleaner (DN-30) and a solvent cleaner (TCA). The second goal was to perform an economic assessment of Graff-Off™ compared to the DN-30 alkaline cleaner and the TCA solvent cleaner. The effect of the reduction of hazardous wastes was factored into the economic assessment.

2.0 Conclusions

A series of immersion cleaning experiments for the removal of jet engine oil were conducted. Four techniques for measuring the effective cleaning performance were evaluated. The techniques were the weight-change method, the non-volatile residue method (NVR), the Fourier transform infrared (FTIR) analysis and the water-break test. The weight-change, NVR and FTIR data did not show any significant trends with bath contamination levels and were not useful indicators for bath cleanliness performance measurements for these experiments. Only the water-break test was found to be an effective, consistent measure of degreased-surface cleanliness. The results from the water-break test were used as a basis for deciding bath exhaustion levels and as a basis for the economic assessment performed. Based on the experimental results and the economic assessment performed, the following conclusions were drawn:

1. Graff-Off™ provides an effective alternative to TCA. It degreases more area per gallon (881 vs. 831 ft^2/gal) and has a lower degreaser cost ($25.70 vs. $113.00/gal).
2. The Aeroclean alkaline cleaner, DN-30, can be used to clean even a greater surface area (2,060 vs. 881 ft^2/gal) compared to Graff-Off™. Because DN-30

is diluted 10 to 1 prior to use, the $5.00/gal (full strength) degreaser has a dramatically lower cleaner cost ($0.50 vs. $25.70/effective gal).

3. But, because of the elevated temperatures and more corrosive environment, the DN-30 retrofit cost is greater. In addition, it has been projected that the DN-30 tanks will require more frequent replacement. Thus, the capital, maintenance, and depreciation costs will be greater for DN-30 than for Graff-Off™.
4. Based on net present value (NPV), internal rate of return (IRR) and payback period, both Graff-Off™ and DN-30 are extremely attractive alternatives to TCA.
5. The NPV for substitution of Graff-Off™ and DN-30 for TCA indicates that over a 5, 10, or 15-year period, DN-30 is the superior investment.
6. However, the same assessment, when based on IRR indicates Graff-Off™ is the preferred investment.
7. Only the water-break test was found to be an effective, consistent measure of degreased-surface cleanliness. Other techniques studied, weight-change, non-volatile residue (NVR), and Fourier transform infrared (FTIR) analysis, failed to provide a clear measure of cleaning performance.
8. A beaker test was developed to allow rapid assessment of cleaning performance. The beaker test can be used to assess the effectiveness of Graff-Off™ for removal of other contaminants.

3.0 Recommendations

The following recommendations are made based on the technical and economic assessment performed:

1. The effectiveness of Graff-Off™ for the removal of other contaminants, such as lube oil, cutting oils, grease, dirt, buffing material etc. should be evaluated. The availability of an effective, rapid assessment procedure like the beaker test can expedite these tests.
2. The effectiveness of Graff-Off™ versus other cleaners should be assessed. Many alternative aqueous cleaners exist and their relative effectiveness should be assessed in order to provide a more complete evaluation of Graff-Off™.
3. A comparative evaluation of Graff-Off™ against other solvent cleaners like trichloroethylene (TCE) or perchloroethylene (PCE) should also be helpful. Also, a comparative study of Graff-Off™ against TCA in the vapor degreaser mode should also be helpful.
4. The economic assessment procedures developed should be extended to the different contaminants and alternative cleaners.
5. In subsequent or “Follow-on” work, metric units should be included in parentheses.

4.0 Methods and Materials

A simple, inexpensive method to analyze the industrial applicability of Graff-Off™ as an alternative cleaner was sought. An immersion process was chosen because it satisfied all these categories and it is widely used in industry. The test plan was to compare the performance of Graff-Off™ to DN-30 an alkaline cleaner, and to 1,1,1 trichloroethane (TCA) a solvent cleaner. Standard steel panels were contaminated and cleaned with each of these agents. Data on water break, weight change, NVR , FTIR, and other observational information were collected. The results were analyzed comparatively and an engineering/economic assessment was carried out. To accomplish the project objectives of evaluating the Graff-Off™, it was necessary to divide the effort into five subtasks. The five subtasks were:

1. Test system definition and selection
2. Experimental set-up
3. Cleaning and sampling procedures
4. Performance testing, and
5. Economic assessment.

4.1 Test System Definition and Selection

4.1.1 Cleaning Process Selection

The performance of Graff-Off™ and DN-30 was determined by an immersion cleaning process. This process was selected because immersion cleaning is simple, does not require sophisticated equipment, it is widely used in industry, and it is easy to set-up on a laboratory scale. The immersion cleaning process primarily involves a soak cleaning step, a rinse step, and then a drying step.

4.1.2 Alternate Alkaline Cleaner Selection

Aeroclean DN-30, an alkaline cleaner already being used in industry, was selected to be used in this study. This product had been used in an earlier EPA study and it was chosen to maintain some measure of consistency in the EPA programs. It was also chosen because it has been used in industry for parts cleaning.[3]

4.1.3 Solvent Cleaner Selection

For comparison purposes, TCA, a conventional, high performance chlorinated degreaser was chosen. TCA has been found to have serious negative environmental impacts. It is a toxic chemical and its use as a common degreaser has been mostly phased out. Its inclusion here was to provide a base case for comparison with the two alternative degreasers. To reduce its VOCs and other negative impacts, TCA was used as a liquid cleaner instead of a vapor degreaser and no recycling of TCA was done.

4.1.4 Cleaning Object Selection

Standard 1010 steel panels were chosen as the metallic object to be cleaned. These panels are inexpensive and were easily coated with oil in a uniform manner to enable performance testing. Steel, in general, is a good representative of metals used in industries for equipment and parts manufacture. The test panels had nominal dimensions of $3 \times 5 \times 1/32$ inches and were obtained from Q-Panel, Inc. The steel test panels were ordered and delivered to the project site pre-degreased to “water-break free” level by Q-Panel.

4.1.5 Contaminant Selection

Jet engine oil was the contaminant selected because it had properties that allowed for consistent coating of the panel surfaces. The jet engine oil contaminant stayed on the metal surface without forming strong chemical bonds on the surface. It was inexpensive and it could be seen when coated on the surface. Jet engine oil was also chosen because it was similar to the special formulations that manufacturers apply to metallic surfaces to extend shelf life.

4.1.6 Pre-Contamination of Degreaser Bath

Based on previous experience with cleaning solvents, several thousand contaminated panels were cleaned before the bath exhaustion level was reached. To decrease the quantity of panels that would be used in this project, it was decided to pre-contaminate the degreaser bath with jet engine oil. The goal of the pre-contamination was to decrease the actual number of panels processed through the cleaning operation to approximately 500 panels for each degreaser tested. It was important that the pre-contamination did not affect the performance assessment conclusions, but rather shorten the time between test initiation and bath exhaustion.

4.2 *Experimental Set-up*

4.2.1 Materials

The materials and equipment used are noted below:

1. Graff-Off™ (Hydra-Tone Chemicals, Inc., 7785 Foundation Drive, Florence, KY 41042)
2. 1,1,1 trichloroethane, TCA, (Cat. #402877, Sigma Aldrich)
3. Aeroclean DN-30 (Aerocote Corporation, P.O. Box 15849, Houston, TX 77220)
4. Standard 1010 steel panels 3 x 5 x1/32 inches (cat. #R-35, Q-Panel Lab Products, P.O. Box 75548, Cleveland, OH 44101-4755)
5. Jet engine oil (Mobil Oil II, Synthetic Jet Engine Oil, NSN9150-00-985-7099 Class C/I, MIL-L-23699)

6. Degreasing baths (Cat. #EW-07405-00, 316 SS 1-gallon tank's, Cole Palmer)
7. 8-L polypropylene rinse baths
8. 4-L stainless steel tank (7.5 x 5x 7 inches)
9. Ethyl acetate (Cat. #27,052, Sigma Aldrich).

4.2.2 Beaker Test for Bath Exhaustion Determination

To accelerate the testing process and decrease the quantity of panels being cleaned, it was decided that the degreaser baths would be pre-contaminated with the jet engine oil. The amount of pre-contamination was estimated based on small-scale experiments done in beakers. In this experiment, 350 mL of degreasing solution was placed in a 1-L beaker. The degreasing solution was contaminated with a known amount of jet engine oil. Fresh steel panels were immersed into the contaminated degreasing solution for three minutes, then pulled from the solution and rinsed under running distilled water for three minutes. These panels were then subjected to the water-break test and a pass/fail determination was made. If these steel panels passed the water-break test, then more jet engine oil was added to the solution and the test of fresh steel panels run again. This sequence was repeated until a failure point was accomplished. The lowest failure point among the three degreasers was used as the pre-contamination level for all the degreasing baths.

This beaker test provided a rapid method to estimate bath exhaustion levels. It could be used to rapidly evaluate or screen the effectiveness of multiple degreasers and contaminants.

4.2.3 Panel Contamination

4.2.3.1 Panel Holding Racks. Rack hangers were designed to allow a group of 20 panels to be processed at one time (Figure 1). Use of this rack drastically reduced the time it would have taken to individually process panels. It also allowed consistent processing of panels during oil contamination or panel cleaning.

The rack was designed with ¼-inch spaced notches in the bars. These notches prevented panels from sliding off the bars, prevented the panels from touching each other during processing, and also allowed the panels to be equally spaced throughout handling and processing.

4.2.3.2 Panel Labeling. All panels were scribed with a number for tracking and identification purposes. An electronic scribe was used to number the panels.

4.2.3.3 Automated Immersion Unit. An automatic immersion unit was used to process a single rack containing 20 panels at a time. The immersion unit allowed for smooth up and down movement of the panels. This allowed the panels to be uniformly coated with the jet engine oil. The automated immersion unit was

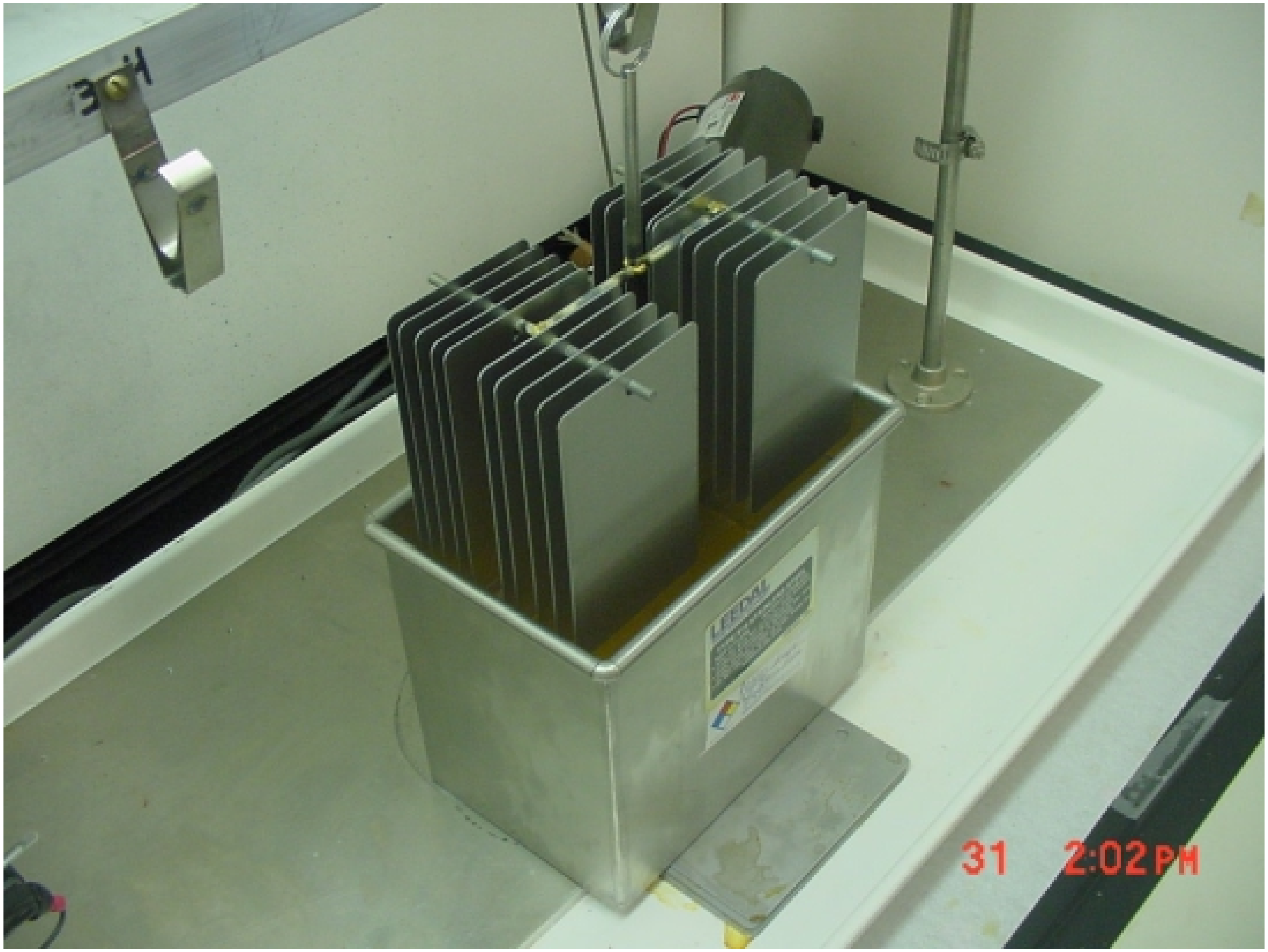


Figure 1. Panel Rack

operated at a dipping or removal rate of 17 cm (6.7-inches) per minute. Panels were immersed for 1 minute in the contaminating bath before they were removed. Figure 2 shows the immersion unit, the bath, a rack of 20 panels for processing, the motor, the speed controller and the pulley system.

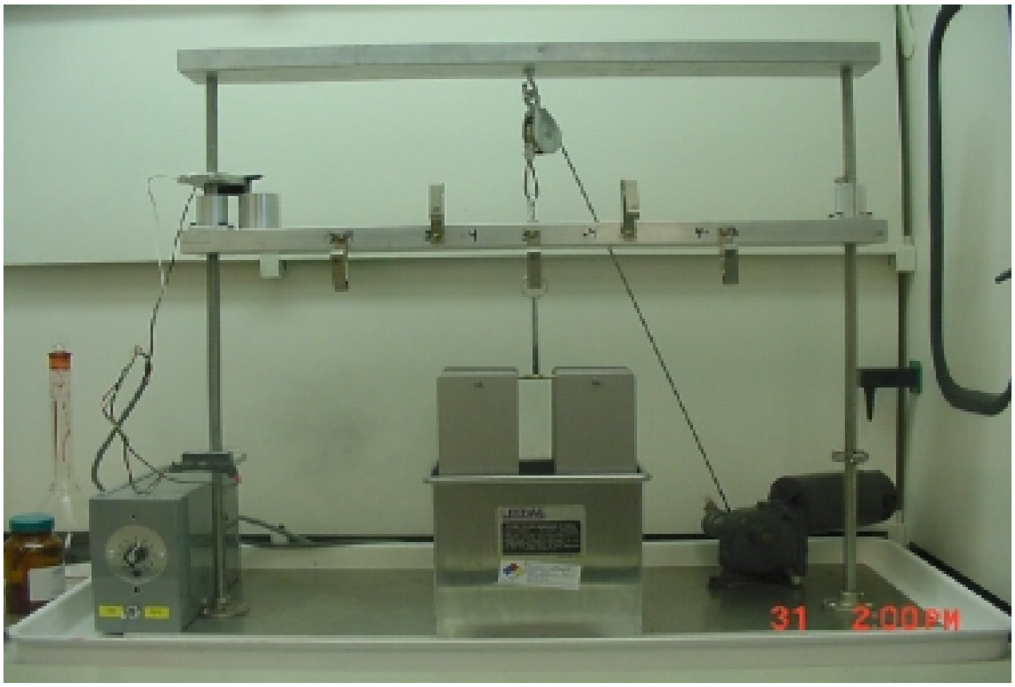


Figure 2. Automated Test Panel Immersion System

4.2.3.4 Panel Contamination Process. Panels were contaminated by controlled immersion in a 4-L stainless steel tank (19 x 13 x 18 cm, 7.5 x 5x 7 inches, L x W x D) containing 3.5 L of the contaminant solution. The contaminant solution was 2% jet engine oil in ethyl acetate. Ethyl acetate is a quick evaporative solvent and was used as a thinner for the jet engine oil. The thinning enabled the steel panels to be contaminated with a thin and uniform layer of jet engine oil and the quick evaporative property of ethyl acetate enabled rapid processing of panels.

The solution in the dip tank was weighed at the beginning and end of each series of racks to determine changes due to oil coated on the panel surfaces and or ethyl acetate evaporation. Adjustments were made to the bath by adding oil or ethyl acetate to maintain jet engine oil concentration and the liquid level in the bath. One rack of 20 panels was processed at a time. Panels were lowered into the bath and allowed to stay completely immersed in the bath for 1 minute. After this time, the panels were taken out with a removal rate of 17 cm per minute. This rate allowed the ethyl acetate to evaporate off the panel surface evenly. The panels were allowed to drip dry over the tank for a couple of minutes or until the drop rate was slower than 30 seconds per drop, then the panels were transported to a drying station. Panels were ready for testing with an hour or two at the drying station. The panel removal rate and the solution concentration are important in achieving this short cycle for panel contamination. This procedure ensured each panel was coated with a uniform and reproducible coating of jet engine oil. Typically, 0.01 g (0.003 g standard deviations) of oil was deposited on both sides of the 0.21 ft^2 panel.

4.3 Cleaning Process

The cleaning process involved the set-up and the operation of the cleaning system. Each cleaning system contained one cleaning bath, two rinse baths, and accessories. A separate cleaning system was prepared for each degreaser and was operated separately. All operations were carried out in chemical hoods.

4.3.1 Cleaning Bath

The cleaning bath consisted of a 4-L capacity 316 stainless steel tank. This tank was filled with 3.5 L of degreaser, which allowed for total immersion of the panels and unrestricted stir bar operation. The 316 stainless steel was compatible with TCA, DN-30 and Graff-Off™. There was no evidence of chemical attack on the steel in the duration of the project. Figure 3 shows a cleaning bath with panels completely immersed.

4.3.2 Rinse Tanks

The rinse station consisted of two 8-L polypropylene tanks for each degreaser evaluated. The tanks were fitted with air spargers to provide agitation and some scrubbing effect. Figure 4 shows the two tanks of a rinsing station. The US Air Force technical order (TO) 42C2-1-7 "Process Instructions for Metal Treatment"(4) was used as a guide in setting up the rinse baths. Rinse water was fed to the two rinse tanks continuously via separate widespread nozzles placed such that surface-floating contaminants would be swept away.

4.3.3 Cleaning System Accessories

Magnetic stir bars of 4 x 0.6-cm diameter size were used in each degreaser bath. Each cleaning bath was set on a stir plate, and in the case of DN-30, a hot plate/stir plate was used as well. Air spargers, thermometers, and pH probes were mounted onto the cleaning system. A hook on a cable was used to create a pulley system for moving the rack of panels in and out of the cleaning and rinsing baths.

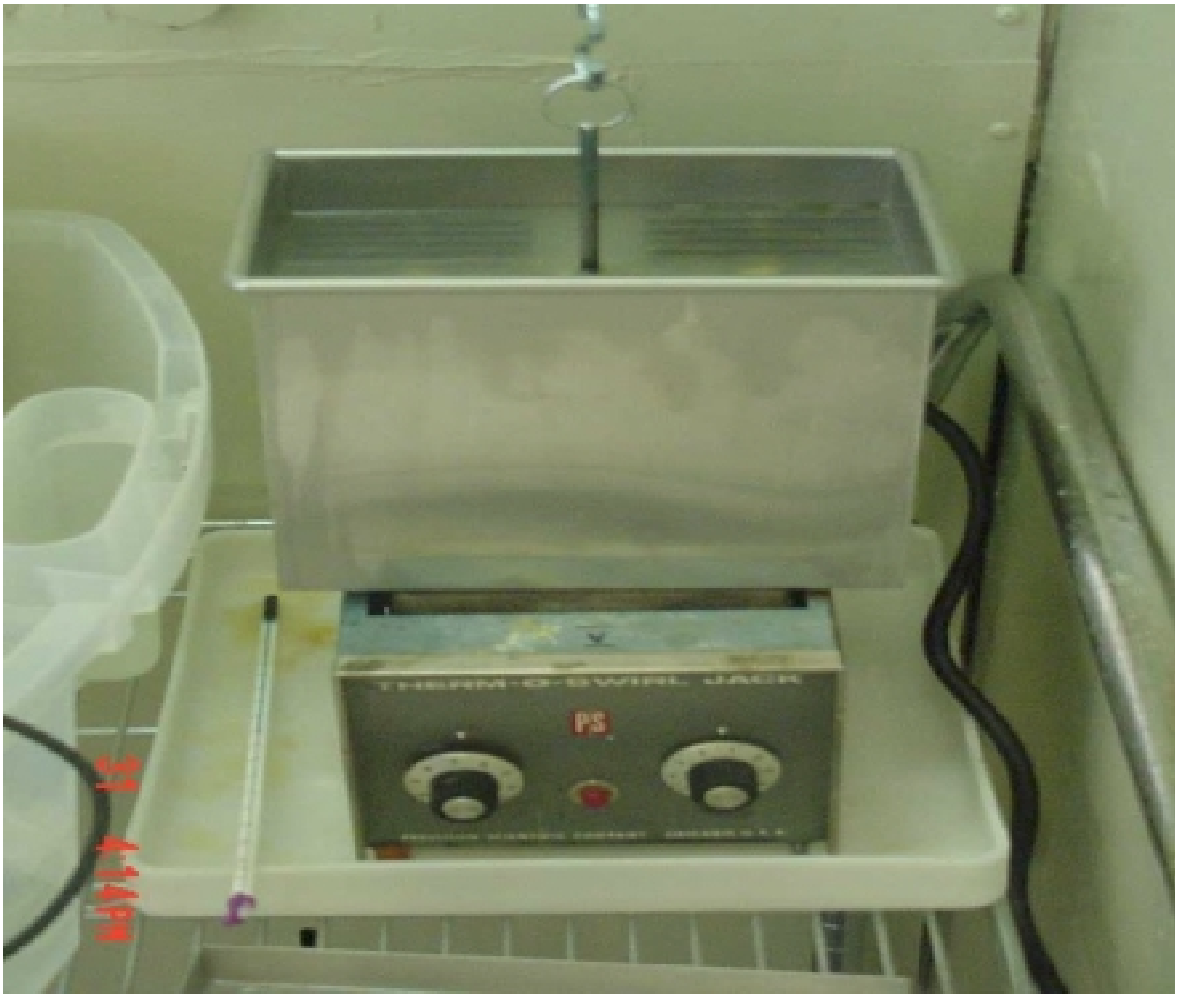


Figure 3. Cleaner Bath with Submerged Rack and Panels

4.3.4 Cleaning Operation

The cleaning operation involved preparation of the clean bath and rinse stations prior to cleaning the contaminated panels. The baths were brought to temperature and agitated before the panels were completely immersed for cleaning. The degreaser bath was agitated throughout the cleaning operation. To simulate an actual operation in industry, agitation was turned off over the weekends, but process heat was left on. The total immersion time in the

Figure 4. Rinse Tanks with Rinse Water Addition Nozzles

degreaser bath was 3 minutes. Afterwards, the panels were removed and rinsed sequentially in the two rinse baths. The rinsed panels were allowed to drip dry and then stored for further analysis. The aqueous degreaser baths and the rinse baths were monitored for changes in temperature and pH.

4.3.4.1 Preparation for Contaminated Panel Cleaning. The preparation procedure involved the following steps:

- The degreaser bath was pre-contaminated with jet engine oil. This was done only once at the start of the process.
- The baths were agitated with stir bars.
- Fluid levels were adjusted as necessary.
- Air supply to spargers located in rinse baths were turned on and adjusted for vigorous agitation.
- Water flow to rinse baths was turned on and adjusted.

- Degreaser bath temperature was recorded.
- pH of aqueous degreaser baths were recorded.
- pH and temperature of rinse tanks were recorded.
- Turbidity samples from the rinse tanks were taken for later analysis.

4.3.4.2 *Panel Cleaning Procedure.* The panel cleaning procedure included the following steps:

- Totally immerse contaminated panels in degreaser bath for 3 minutes.
- Remove rack/panels from degreaser bath and immerse in Rinse Bath #1 for 3 minutes.
- Remove rack/panels from Rinse Bath #1 and immerse into Rinse Bath #2 for 3 minutes.
- Remove rack/panels from Rinse Bath #2 and hang to air dry.
- Shut off water supply to rinse tanks.
- Shut off air supply to rinse tanks.
- While panels were still wet from the rinse station, remove sample panels and perform water-break test (See Section 4.4.2.1 for description of water-break test).
- Repeat cleaning procedure on the same set of panels if sampled panels failed the water-break test.
- The bath was considered exhausted if upon repeating the cleaning procedure the sampled panels from the group failed the water-break test again.
- DN-30 was forced to exhaustion by addition of 10 g of oil (equivalent to 1,000 panels) followed by cleaning a set of 20 panels and testing them. The addition of 10 g of oil was continued until sampled panels from a cleaned set of 20 panels failed the water-break test and the exhaustion point was established as illustrated above.
- Rinsed panels were hung to air dry, excess water at the bottom of panels was dabbed with a Kimwipe.
- Cleaned and dried panels were analyzed later for weight change, NVR, and FTIR.

4.3.4.3 Rejuvenation of Exhausted Baths

The concept that the degreaser baths could be rejuvenated after reaching exhaustion was very intriguing and was tested when the baths became exhausted by first removing all visible residues. The volume of liquid removed was replaced with water and the baths tested for cleaning potency. Contaminated panels were passed through one cleaning cycle and subjected to the water-break test. If the panels passed, then the bath was considered rejuvenated, if they failed, then the bath could not be rejuvenated.

4.4 Performance Measurements

Two types of samples were handled: (1) the steel panels and (2) the solutions resulting from the degreasing or the rinsing process baths. Laboratory performance tests and visual observations were carried out on these samples as detailed below. Critical performance measurements and bath and rinse tank measurements are noted in Tables 1 and 2. The bath capacity was reported in

Table 1. List of Critical and Non-Critical Performance Measurements

Matrix	Measurement	Critical
Graff-Off™ efficacy- steel panels contaminated with jet engine oil	- Weight change	Yes
	- NVR	Yes
	- FTIR	No
	- Water-break test	Yes
	- Clean appearance	No
	(Visual)	No
	- Residue residual	No
	(Visual)	No
	- pH and temperature	
	- Worker friendly usage	
Economic assessment	- Net present value	Yes
	- Internal rate of return	No
	- Payback period	No

units of square feet of panel surface treated per gallon of degreaser bath (ft^2/gal). Non-metric units were used to provide compatibility with U.S. industry practice. All selected and analyzed specimens are archived at Battelle (for a maximum period of one year).

4.4.1 Panel Sampling

All panels were scribed with a number for tracking and identification purposes. An electronic scribe was used to number the panels. A standard U.S. EPA chain-of-custody form (COCF) was generated for each sample that was transferred to the FTIR laboratory for analysis.

The stock (fresh) panels removed directly from the shipping package were subjected to the water-break test. This was a control test for the contaminated and cleaned panels and it validated the water-break test for identification of a failure point. Three representative panels out of every 100 pack were used in the control test.

Table 2. Summary of Non-Critical Surface Finishing Bath and Rinse Water Analyses

Measurement[a]	Solution(s) Tested	Sample Collection Time	Sample Handling, Preservation, and Holding	Sample Volume (mL)
pH	All degreaser baths and rinse baths except TCA degreaser bath	Beginning and end of each test	Direct in-tank measurement	Direct in-tank measurement
Turbidity	All rinse waters	Beginning and end of each test	Removed from tank and analyzed	10
Temperature	Only heated baths and heated rinse tanks	Just prior to each test system run	Direct in-tank measurement	Direct in-tank measurement
Volume	All tanks	Just prior to each test system run, at the end of day, and before the next run	Direct in-tank measurement.	Direct in-tank measurement

The panels were contaminated and subjected to the cleaning process in groups of 20 each. Three panels from each group were selected at random, weighed, and tested for water break. At specified panel loads, panels from a degreaser group were selected and subjected to NVR analysis and FTIR spectroscopy. Samples were always selected and analyzed in triplicates.

4.4.2 Bath Exhaustion Indicators

The bath exhaustion point was defined as the point where the degreased and rinse tanks produced cleaned panels that failed the water-break test. If a bath produced a set of failed panels (an apparent bath exhaustion), the tested panels were taken through another cleaning cycle in the same bath; if they still showed water-break test failure, the bath was declared exhausted. In the special situation where one or two panels disagreed, another set of 3 panels from the same group of 20 are tested and if the same situation of discrepancy exists then the majority finding would be reported. In addition, another group of twenty contaminated panels are passed through the cleaning cycle and 3 panels are randomly selected and tested in the water-break test to confirm bath exhaustion.

This allowed the bath exhaustion level as determined by the water-break test to be compared between the different cleaning baths.

4.4.2.1 Water-Break Test. The water-break test detects the presence of grease or oil on a cleaned surface. The procedure is described in MIL-STD 1359B and ASTM A 380. Basically, the panels are immersed in a vertical position into a container overflowing with water and then withdrawn with water draining off the surface. When the test passed, the draining water remained as an adherent film over the surface. When the test failed, the draining water broke up into a discontinuous coating of channels and droplets within 1 minute. The water-break test is generally a go/no go subjective test. As noted in Table 1, the water-break test is one of several critical measurements obtained.

4.4.2.2 Weight Change Test. The weight of the test panel after degreasing and air drying was compared to the test panel containing the jet engine oil. The amount of residue left on the sample was an indication of the cleaning effectiveness.

4.4.2.3 Non-Volatile Residue Measurements (NVR). The NVR procedure is described in MIL-STD 1359B and ASTM F 331-72. Cleaned panels were rinsed with a mixed solvent containing 75% TCA and 25% ethanol by volume. The mixed solvent was then collected and evaporated to dryness. The weight of the residue was measured and the results reported in mg residue/ft^2.

4.4.2.4 Fourier Transform Infrared Spectroscopy (FTIR). The FTIR analysis was performed with a Digilab FTS-60A system using the grazing angle reflectance method. FTIR is sensitive to low levels of contaminants and is a rapid detection method. As noted in Table 1, it is not considered a critical measurement. Three readings were obtained for each panel tested. A calibration curve was prepared using a cleaned and a fully contaminated panel. The reflectance of cleaned panels was compared to the curve and a value of percent of full contamination was reported.

4.4.3 Rinse Bath Monitoring

The rinse baths were monitored by measuring pH and temperature. The temperatures of the baths were recorded only at the beginning and at the end of the cleaning process to verify ambient conditions. Turbidity levels in the rinse baths were also monitored.

4.4.4 Other Observational Methods

Other physical observations were made to monitor the cleaning process. They included color, odor, formation of oil droplets or oil films, formation of sediments or coagulates in the baths and the appearance of the panels during the entire cleaning process. The volume levels of the baths were also observed. These visually monitored parameters are not critical to determining the effectiveness of any cleaning agent, but could affect the "friendliness" of the product to customer use.

4.5 Economics Assessment

The life cycle cost of implementing Graff-Off™ was determined. Capital costs for replacement degreasing lines were included. The primary measurement driving the analysis was the ft^2 of surface cleaned per gal of degreaser. Operating temperature and rinse water requirements were also considered. Cost for disposal of the contaminated rinse water and disposal of spent degreasing baths were also included. The capital and operating costs for Graff-Off™ and the DN-30 alkaline degreaser were compared to the TCA cleaning costs. Financial measures including NPV, IRR, and payback period were calculated for Graff-Off™ and DN-30 based on the estimated annual savings and the required capital inputs.

5.0 Results and Discussion

Each fabricating or processing step determines the degree of cleaning that should precede it. Cleanliness is a variable rather than a fixed criterion and is quite dependent on the immediate intended use of the surface. An adequately degreased surface for one application may be unacceptable or excessively cleaned for another. For example, the acceptable levels of cleanliness for galvanizing or anodizing operations will be different than the levels of cleanliness required for metal plating operations. There are many ways of measuring the cleaning effectiveness of cleaners. In this project, the four techniques evaluated were the weight-change method, the non-volatile residue method (NVR), the Fourier transform infrared (FTIR) analysis and the water-break test. The weight-change, NVR, and FTIR data did not show any significant trends with bath contamination levels and were not useful indicators for bath cleanliness performance for these experiments. Only the water-break test was found to be an effective, consistent measure of degreased-surface cleanliness. The water-break test is used for "high" level of cleanliness detection and in this project it was used as the primary cleaning performance indicator. The degreaser cleaning effectiveness was measured in terms of ft^2 of surface that can be successfully cleaned/gal of degreaser. Data and results collected from the four test methods are detailed in this section and the appendices. Section 6.0 describes how the cleanliness results based on the water-break test results were used in assessing the economic effectiveness of Graff-Off™ compared to DN-30 and TCA.

5.1 Load of Jet Engine Oil on Contaminated Panels

The panels were contaminated with jet oil using the automated immersion process described in Section 4.2.3.2 The average weight gain after contamination for a group of 20 panels was 0.2 g or 0.01 g per panel (See Appendix A - Section 1 for details). The surface area for each panel was 0.21 ft^2.

5.2 Beaker Test for Predicting Bath Exhaustion Level and Bath Pre-Contamination Level

The degreaser baths were precontaminated with jet engine oil to decrease the quantity of panels that would be cleaned before bath exhaustion and to accelerate the testing process (Section 4.1.6). To do this effectively, an estimate for the amount of oil that would completely exhaust a degreaser was required. A beaker test was set-up (Section 4.2.2) to provide a rough estimate of the exhaustion level for each degreaser. Jet oil was sequentially added to the degreaser solution and fresh panels were immersed in the degreaser and then checked for failure in the water-break test. The jet oil load level that produced failure on the water-break test was the estimated bath exhaustion level. The results in Table 3 show that TCA has the lowest exhaustion level at 43.46 g of jet oil per gallon of TCA. Graff-Off™ was better than the TCA, and DN-30 was the best with 93.12 g/gal as the exhaustion level. From this quick beaker test, it can be concluded that TCA will not perform as well as either cleaner, and that DN-30 would be the best performer. It was decided that the pre-contamination level for all the degreasers would be 90% of the exhaustion level of the worst performer in the beaker test (i.e., TCA). From Table 3 the pre-contamination level was 36.17 g of jet oil for TCA, which when extrapolated is equivalent to 3,617 coated panels (assuming all the jet oil is removed).

A verification test was conducted to ensure that this pre-contamination level was not above the exhaustion point for any degreaser bath. Fresh panels were immersed in the pre-contaminated baths for each degreaser and then subjected to the water-break test. Panels from all three degreaser baths passed the water-break test verifying the pre-contamination level to be below the exhaustion level for each degreaser bath.

Table 3 Results of Beaker Test[(a)]

Description	Units	TCA	Graff-Off™	DN-30
Total volume in beaker	L	0.70	0.70	0.70
Exhaustion oil level	mL	9.683	13.833	20.750
Estimated surface area exhaustion rate	ft^2/gal	918	1312	1968
Estimated panel exhaustion rate of 3.5L bath	panels/ bath	4018	5741	8611
Estimated weight of oil to exhaust 3.5L bath	g	40.18	57.41	86.11
Pre-contamination level	g	36.17	51.67	77.50
Equivalent panels at pre-contamination level	# of panels	3617	5167	7750

(a) See Appendix A, Section 1, for details.

Figure 5 shows the estimated exhaustion levels via the beaker test compared to the exhaustion levels found after cleaning contaminated panels. Within a 10% margin of error, the beaker test estimate for bath exhaustion was equal to the actual exhaustion levels determined for TCA and DN-30. For the Graff-Off™, the beaker test predicted a slightly higher level for bath exhaustion than the actual exhaustion level determined by the water-break test. The beaker test has proven to be a quick estimator of cleaner performance.

The exhaustion levels were equal for TCA because the first set of contaminated panels that passed through the pre-contaminated TCA bath failed the water-break test. Obviously, the pre-contamination level for TCA was very close to the exhaustion point. Verification tests were less sensitive because they were conducted using fresh panels instead of contaminated panels.

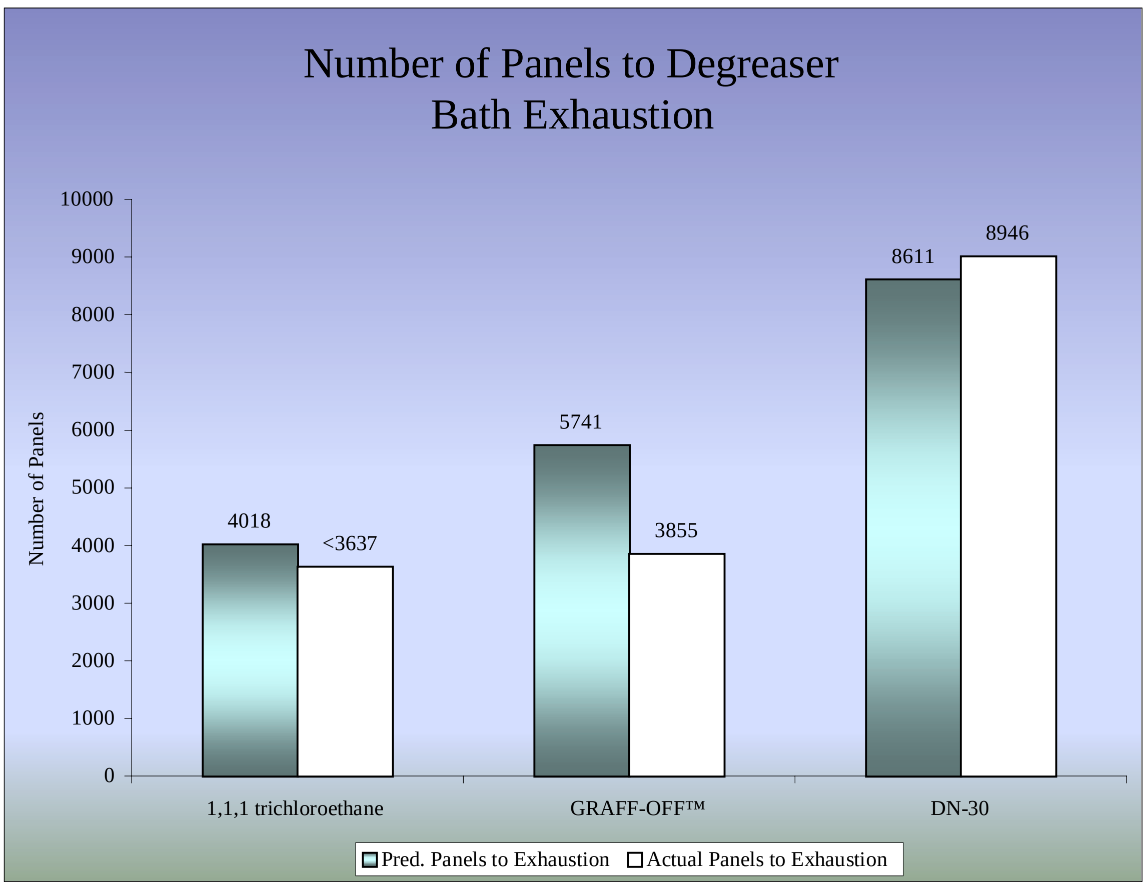


Figure 5. Beaker Test Results Compared to Actual Bath Cleaning Results

5.3 Cleaning Performance by Water-Break Test

The primary cleanliness performance measure was the water-break test and it was used in predicting bath exhaustion levels. Panels from the cleaning process were given a "pass" or "fail" code when subjected to the water-break test (see Section 4.4.2.1). All degreaser baths were pre-contaminated to a load equivalent to 3617 loaded panels. The cleaning process was conducted using a set of 20 panels each time. From Figure 5, the TCA cleaning did not produce water-break free cleaned panels when the first set of panels (3637 equivalent loaded panels) was tested. This suggests that the pre-contaminated level was on the verge of the TCA bath exhaustion level. An equivalent of 3855 and 8946 panels for Graff-Off™ and DN-30, respectively, were cleaned before they failed the water-break test. Figure 6 shows the total surface area that was cleaned for these panel counts. Appendix A - Section 4 presents the details of the water-break tests. From these results, the water-break test serves as a good indicator of cleanliness performance.

5.4 Alternative Performance Measure Test

Three alternative performance techniques were described in Section 4.4.2.1. The weight-change, NVR, and FTIR results did not show any correlation with bath contamination. As each degreaser bath became progressively more contaminated, there was no corresponding increase in the residual weight, non-volatile residue weight, or FTIR reflectance. Even at the bath exhaustion points, there was not a noticeable change in residual weight, NVR, or FTIR results. Thus, none of the techniques were found to be a useful performance measure.

Details of the tests and the results are presented in Appendix B.

5.5 Cleaning Performance by Other Observations

Graff-Off™ was compared to DN-30 and TCA using process parameters like pH, temperature, and bath volume. Other subjective observations like color, odor, particle settling, and others were also used. The details are given below.

5.5.1 Physical Properties of the Cleaners

Graff-Off™ degreaser looked opaque to milky white in color, with a more than mild fruity aroma that could be overpowering in a confined space. The DN-30 solution was clear and became cloudy when oil was added to it. TCA had a strong organic scent and was light yellow in color. It was light and handled smoothly.

5.5.2 Cleaning Process Observations

In the Graff-Off™ process a milky mixture containing oil settled to the bottom of the tank when agitation ceased. It dispersed evenly with little agitation. In the

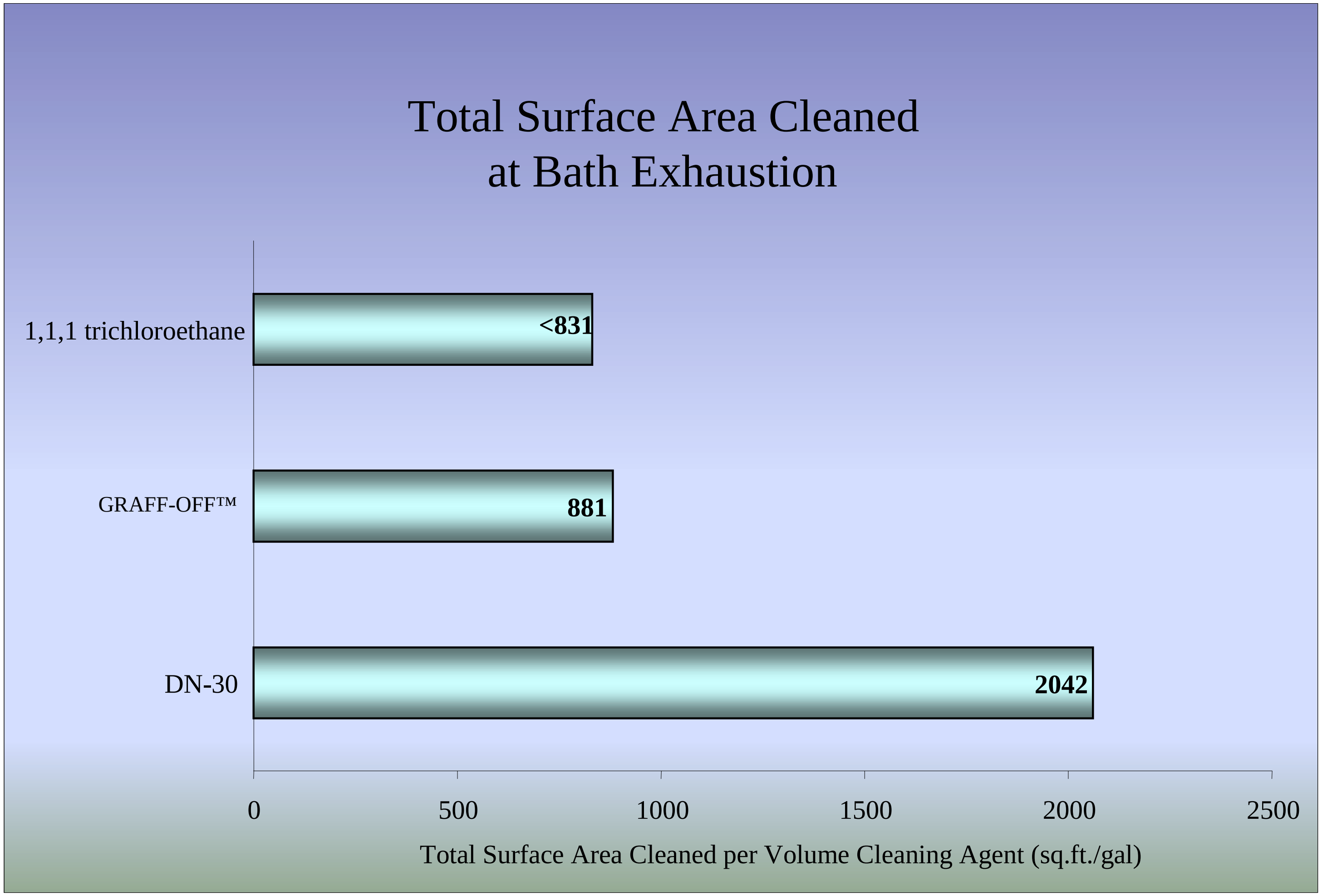


Figure 6. Total Surface Area Cleaned at Bath Exhaustion

case of DN-30, the oil contaminant floated on the liquid surface and required vigorous mixing in order to disperse it evenly into solution. The oil residue observation implies that Graff-Off™ baths might be used over a long time by simply draining the coagulated residue from the bottom of the tank and replenishing with water and Graff-Off™ cleaner. For DN-30, a skimmer would be used to remove oil from the surface. TCA baths did not have any residue settling or floating. The oil was completely miscible in the solvent at all times. DN-30 was used at a 10% concentration in distilled water whereas the Graff-Off™ was used at full strength.

The quantity of Graff-Off™ required for filling the bath would be 10 times more than the DN-30. This would put a demand on shipping and handling and other related costs like container disposal etc.

5.5.3 pH and Temperature of Baths

The pH and temperature of the Graff-Off™ and the DN-30 baths were measured at the beginning and end of a day's operation. DN-30 was used at 71 C (160°F) during operation. The pH and temperature recordings in Appendix A-Section 1 do not show any trends related to cleaning performance. The pH of the TCA bath was not measured because it was an organic solvent.

5.5.4 Particulates in the Cleaning Baths

Particles were seen on the magnetic stir bars used in the degreasing tanks. The Graff-Off™ tanks showed the most particles followed by DN-30 baths. Very little, if any, particulates were seen in the TCA tanks.

5.5.5 Panel Corrosion

The panels cleaned with TCA showed visible corrosion. It was more noticeable than with the Graff-Off™ or DN-30 panels. This suggests that TCA did not remove only the jet oil contamination on the surface, but attacked the metal and rendered it susceptible to easy corrosion. The Graff-Off™ and DN-30 cleaning action was gentler to the standard 1010 steel-metal surfaces than TCA.

5.6 Rejuvenation of Exhausted Degreaser Baths

The degreaser baths were considered exhausted when cleaned panels failed the water-break test. As described in Section 4.3.4.3, the oil residues formed were removed from the baths. Water was used to readjust liquid volume to the operating level in the hope that the baths could be rejuvenated. In the case of Graff-Off™, the residue settled to the bottom of the tank and was drained off. For DN-30 the oil film was skimmed off the surface. After this was accomplished, contaminated panels were re-cleaned through the process and subjected to the water-break test. The Graff-Off™ and DN-30 panels failed the test. The Graff-Off™ and DN-30 baths were not rejuvenated. This suggests that the remaining solution was exhausted. It also suggests that all or most of the active cleaning components were exhausted during the cleaning process.

6.0. Economic Analysis

The life cycle costs for Graff-Off™ and a standard alkaline cleaner DN-30 were compared with a base case where TCA is used for degreasing. Two scenarios were considered:

- Retrofit into a commercial job shop
- Retrofit into a large commercial or DoD maintenance operation.

The basic assumptions associated with each case are described below.

6.1 Assumptions

A "high" level of cleanliness, as determined by the water-break test, was used as the baseline for the job shop performance level. The effectiveness was measured in terms of ft^2 of surface that can be successfully cleaned/gal of degreaser.

6.1.1 Commercial Job Shop

Small parts are brought into the shop for degreasing prior to other shop operations. Each part is immersed in the cleaning bath for 3 minutes, removed, and allowed to drain into the bath, and then rinsed in two subsequent rinse tanks. Water is constantly added to the rinse tanks during the rinse operations. It is assumed that the cleaning tank is 3-ft by 3-ft by 3-ft deep (9 ft^2 of surface area and 27 ft^3 or 200-gal capacity). Note, non-metric units were used to be consistent with U.S. job shop operations.

Based on the performance testing, as discussed in the results section, and the assumption that over the course of that year 111,000 ft^2 of parts would be cleaned in the immersion-cleaning bath, <1,000 gal of TCA, 1,000 gal of Graff Off, and 40 gal of DN-30 degreaser would be required each year. Details are provided in Table 4.

Waste rinse water generation is estimated at 200 K gal/year based on 0.07 gpm/ft^2 rinse water rate and 2 dumps/year.

Due to evaporation, the tank level will drop with time. The tank level is checked weekly and water is added to maintain the level. Lost TCA is made up with fresh TCA. Experimental testing with Graff Off™ and DN-30 indicated that simple water makeup is adequate. Therefore, for the purposes of this analysis, it is assumed that no make up Graff Off™ or DN-30 is added between tank filling and tank exhaustion.

Table 4. Estimated Degreaser Requirements

Parameter	Commercial Scale Degreasing Operations		
	Base Case: TCA[(a)]	Graff Off™	DN-30
Cleaning capacity, ft^2/gal	<831	881	2,042
Part surface area cleaned, ft^2	<111,000	111,000	111,000
Degreaser make up due to evaporative losses, K gal/year[(b)]	<0.79	0	0
No. of bath changes per year[(c)]	<0.7	0.6	0.3
Volume of degreaser solution required per year for 200 gal baths, K gal[(d)]	<0.14	0.12	0.06
Total losses, K gal	<0.93	0.12	0.06
Proportion of degreaser utilized, %	<100	100	10
Volume of 100% degreaser required, K gal	<0.93	0.12	0.006

[a] The water-break test conducted on TCA samples failed with the first set of 20 panels tested. This implies that the true values could be less than the calculated values.

[b] The [0.01 gal/hr/ft^2 evaporation rate at 25 C (77 F) x 9 ft^2 x 365 day/year x 24 hr/day/1000 gal/K gal]

[c] The No. of bath changes per year = [111,000 ft^2/ (Cleaning capacity x 200 gal/bath)]

[d] The volume of degreaser solution required per year for 200 gal baths = [No. of bath changes per year x 200 gal/bath x K gal/ 1000 gal]

The TCA and the Graff Off™ cleaners are operated at room temperature while the DN-30 alkaline cleaner is maintained at 71 C (160 F). Heat losses of 10% per day were used to estimate heating requirements for the DN-30.

Utility requirements including water for evaporative losses, rinse water, and steam to maintain the operational temperature of the DN-30 bath are summarized in Table 5.

Rinse water is sent to an oil water separator where the residual oil level is maintained below 15 ppm prior to discharge to a municipal sewer for treatment. Most municipal sewer systems allow an oily-water input as long as the effluent has oil and grease levels below 200 ppm.

When the tank no longer allows water-break free performance, the tanks are dumped. The TCA is drummed and disposed as hazardous waste. Note that in industry, the TCA will be distilled and recycled after bath exhaustion. This practice will reduce the cost and amount of TCA used. The pH in the alkaline cleaner batch is adjusted from a pH of 10 to the 6.5 to 9 level with waste acid assumed available in the shop and sent to the oil/water separator prior to being discharged to the municipal sewer for treatment. When the Graff Off™ becomes spent, it is also discharged to the sewer.

Cost for chlorinated solvent disposal is ~$90/55-gal drum (based on methylene chloride incineration costs at Hill AFB). The cost for oil and grease treatment typically ranges from $0.41 to $1.10/1000 gal.(5) A cost of $1.00/1000 gal was arbitrarily chosen for this cost assessment.

Table 5. Utility Requirements

Parameter	Commercial Scale Degreasing Operations		
	Base Case: TCA[(a)]	**Graff Off™**	**DN-30**
Water evaporation rate gal/hr/ft^2	0	0.002 at 25 C or (77 F)	0.06 at 75 C or (167 F)
Water makeup for evaporative losses from 9 ft^2 surface area bath, K gal/year[(b)]	0	0.2	5
Rinse water, K gal/year[(c)]	200	200 same as base case	200 same as base case
Total water, K gal/year	~200	~200	~205
Steam use for bath heating, K lb steam/year[(d)]	0 [no heating required]	0 [no heating required]	5

[a] The water-break test conducted on TCA samples failed with the first set of 20 panels tested. This implies that the true values could be less than the calculated values.
[b] Water make-up for evaporative losses from 9 ft^2 surface area bath = water evaporation rate x 9 x 8760 hr/year x K gal/1000gal
[c] The rinse water K gal/year = [(0.07 gpm/ft^2 rinse tank surface area x 9 ft^2 x 16 hr/day x 365 day/year x 90% usage factor x 60 min/hr + 2 dumps/year x 200 gal/dump)/1000 gal/K gal]
[d] Steam use for bath heating, K lb steam/year = [15,000 Btu loss/day x 365 day/year x 1 K lb steam/million Btu]

6.1.2 Large Shop Operation

Large parts are brought to the shop for degreasing prior to operations such as anodizing, conversion coating, galvanizing, and/or plating. In the alkaline cleaner operations at Hill Air Force Base (prior to the phosphoric acid anodizing operations), each part is immersed in a 4-ft x 12- ft x 5-ft deep (1,800 gal) degreasing bath for 3 minutes, removed, and allowed to drain into the bath. If the TCA or Graff Off™ cleaner were used for this service, they would be operated at room temperature whereas the DN-30 alkaline cleaner bath would be operated at 71 C. The tank level would be checked weekly and TCA or water would be added to make up for evaporative losses. For the purposes of this analysis, it is assumed that TCA is added in the base case, but only water is added to the aqueous degreaser tanks to make up for evaporative losses (i.e., no fresh Graff Off™ or DN-30 is added between tank filling and tank exhaustion).

The baths become spent over time and are drained. In the base case, the waste TCA is dumped less than five times per year; the waste chlorinated solvent is drummed for off site disposal. In the case of Graff Off™ or the alkaline cleaner, this would occur 5 and 2 times per year, respectively. The concentrated waste would be transferred to the industrial wastewater-treatment plant (IWTP) for pH adjustment and treatment with the other base wastewater. Note TCA may be distilled and recycled after the bath is spent. This practice would reduce costs and the amount of TCA waste.

The degreased parts (regardless of the type of degreasing agent) are next immersed in a water dip tank. The 1,800-gal rinse tank is constantly fed with 3.5 gpm of water during rinsing operations. The wastewater is sent to the drain and then to the IWTP. The rinse tank is also emptied twice per year.

The exact quantity of parts cleaned cannot be determined. However, using the 2,042 ft^2/gal figure established in the testing reported above, the potential surface-cleaned area is estimated at 7.4 million ft^2/year (1,800 gal/bath x 2 baths/year x 2,042 ft^2/gal).

Based on the performance testing results and the assumption of 7.4 millions ft^2/year, the volumes of the degreasers required are noted in Table 6.

Utility requirements including water for evaporative losses and rinse water and steam to maintain the operational temperature of the DN-30 bath (calculated following similar assumptions as noted above) are summarized in Table 7.

The rinse water rate is 3.5 gpm for the 4-ft x 12-ft x 5-ft deep rinse tank (1,800 gal), or 0.07 gpm/ft^2 of tank surface area. The waste rinse water generation is estimated at 1,063 K gal/year.

Table 6. Large Scale Operations Degreaser Requirements

Parameter	Large Scale Degreasing Operations		
	Base Case: TCA[(a)]	Graff Off™	DN-30
Cleaning capacity, ft^2/gal	<831	881	2,042
Parts surface area cleaned, million ft^2	7.4	7.4	7.4
Degreaser make up due to evaporative losses, K gal/year[(b)]	<4	0	0
No. of bath changes per year[(c)]	<5	5	2
Volume of degreaser solution required per year for 1,800-gal baths, K gal[(d)]	<9.0	9.0	3.6
Total losses, K gal	<13.0	9.0	3.6
Dilution factor, proportion of degreaser utilized, %	100	100	10
Volume of 100% degreaser required, K gal	<13.0	9.0	0.36

[a] The water-break test conducted on TCA samples failed with the first set of 20 panels tested. This implies that the true values could be less than the calculated values.

[b] The [0.01 gal/hr/ft^2 evaporation rate at 25 C (77 F) x 48 ft^2 x 365 day/year x 24 hr/day/1000 gal/K gal]

[c] The No. of bath changes per year = [7.4E6 ft^2/ (Cleaning capacity x 1800 gal/bath)]

[d] The volume of degreaser solution required per year for 1800 gal baths = [No. of bath changes per year x 1800 gal/bath x K gal/ 1000 gal]

Table 7. Large Scale Operations Utility Requirements

Parameter	Large Scale Degreasing Operations		
	Base Case: TCA[a]	Graff Off™:	DN-30
Water evaporation rate gal/hr/ft^2	0	0.002 at 25 C or (77 F)	0.06 at 71 C or (160 F)
Water makeup for evaporative losses from 48 ft^2 surface area bath, K gal/year[b]	0	0.8	25
Rinse water, K gal/year[c]	1,063	1,063 [same as for base case]	1,063 [same as for base case]
Total water, K gal/year	1,063	~1,064	~1,088
Steam use for bath heating, K lb steam/year[d]	0 [no heating required]	0 [no heating required]	49

[a] The water-break test conducted on TCA samples failed with the first set of 20 panels tested. This implies that the true values could be less than the calculated values.

[b] Water make-up for evaporative losses from 48 ft^2 surface area bath = water evaporation rate x 48 x 8760 hr/year x K gal/1000gal

[c] The rinse water K gal/year = [(0.07 gpm/ft^2 rinse tank surface area x 48 ft^2 x 16 hr/day x 365 day/year x 90% usage factor x 60 min/hr x 2 dumps/year x 1800 gal/dump)/1000 gal/K gal]

[d] Steam use for bath heating, K lb steam/year = [135,000 Btu loss/day x 365 day/year x 1 K lb steam/million Btu]

Treatment costs vary from installation to installation, but are typically between \$1.00 and \$12.00/K gal[6,7]. A figure of \$1/K gal was used because it was more typical of large industrial users. In the IWTP, the alkaline solution is used to adjust the pH of waste acid solutions. The solution is treated for metals removal via Cr^{VI} reduction, metals precipitation, clarification and sludge disposal, final effluent pH adjustment, and activated carbon treatment.

The operational assumptions are summarized in Table 8

Table 8. Estimated Values Used in the Cost Assessment

Assumptions or Estimated Quantity	Base Case: TCA[(a)]		Graff Off™		DN-30	
	Commercial Scale Plating Shop	Large Commercial or DoD Plating Shop	Commercial Scale Plating Shop	Large Commercial or DoD Plating Shop	Commercial Scale Plating Shop	Large Commercial or DoD Plating Shop
Degreaser usage, K gal/year	<0.93	<12.8	0.12	8.5	0.006	0.36
Total water required, K gal/year	199	1,063	199	1,064	204	1,088
Steam required, K lb/year	0	0	0	0	5	49
Spent degreaser, K gal/year	<0.14	<8.8	0 Combined with wastewater	0 Same as commercial case	0 Same as Graff Off™	0 Same as Graff Off™
Wastewater, K gal/year	<199	<1,072	199	1,064	204	1,088
Combined spent degreaser and rinse water, K gal/year	NA	NA	199	1,073	204	1,089

(a) The water-break test conducted on TCA samples failed with the first set of 20 panels tested. This implies that the true values could be less than the calculated values.

6.2 Life Cycle Analysis

There are three steps in the financial evaluation of an investment opportunity. They include:

1. Estimating the relevant cash flows
2. Calculating a financial performance measure
3. Comparing the measure with the acceptance criterion.

6.2.1 Financial Performance Measure

The financial measures require an understanding of the time value of money and an accurate estimate of relevant cash flows. Three measures were evaluated for this assessment: net present value (NPV), internal rate of return (IRR), and payback period. NPV and IRR are the preferred ranking measures because they both consider the time value of money. Payback period does not take into account the time value of money and can overstate benefits of an investment. It was included here for completeness, and because it is a simple to understand and common measure of an investment's value.

The investment criteria selected for this analysis are summarized in Table 9.

Table 9. Summary of Investment Criteria[(a)]

Criteria	Recommendation
NPV > 0	Investment return acceptable
NPV < 0	Investment return not acceptable
Highest NPV	Maximum value
IRR > discount rate	Project return acceptable
IRR < discount rate	Project return not acceptable
Shortest payback period	Fastest investment recoup and lowest risk

(a) Reference: Environmental Cost Analysis Methodology Handbook, ESTCP, March 29, 1999.

6.2.2 Capital Costs

The initial capital cost for commercial- and large-scale degreasing and rinse tanks were estimated as shown in Table 10.

6.2.3 Retrofit Cost

It is assumed the TCA dip tanks and two rinse tanks are in place. The cost to retrofit the facility and retrain the workers for use of the alternative degreasers is estimated in Table 11.

6.2.4 Capital Replacement

Over the 15-year assessment period, it is assumed that the TCA and the Graff Off™ tanks will have to be replaced once. Because of the higher temperature DN-30 degreasing tanks, they are assumed to be replaced twice. The results are summarized in Table 12. The net change in capital costs includes the sum of the retrofit costs (see Table 11) and the net increase in the replacement capital costs summarized in Table 12.

Table 10. Estimated Initial Capital Investment

Estimated Quantity	Base Case: TCA(a)		Graff Off™		DN-30	
	Commercial Scale Plating Shop	Large Commercial or DoD Plating Shop	Commercial Scale Plating Shop	Large Commercial or DoD Plating Shop	Commercial Scale Plating Shop	Large Commercial or DoD Plating Shop
Purchased equipment costs, $K						
Degreaser tank	1	<12	1	12	1	15
Rinse tanks (2)	2	<24	2	24	2	27
Vapor control	1	5	0	0	1	4
Agitator	1	3	1	3	1	4
Heat exchanger	0	0	0	0	1	3
Total PE Cost	<5	<44	4	39	6	53
Multiplier for installation, instrumentation, piping, electrical, engineering and fees(b)	2.4	2.8	2.4	2.8	3.1	3.4
Total installed cost, $K	<12	<123	10	109	19	180

(a) The water-break test conducted on TCA samples failed with the first set of 20 panels tested. This implies that the true values could be less than the calculated values.
(b) Reference: Peters and Timmerhaus, Plant Design and Economics for Chemical Engineers, 4th ed., McGraw-Hill, 1991, p 183. Cost factors for specific activities were added as appropriate to derive the multiplication factor for each case under review.

Table 11. Retrofit and Training Costs

Cost Parameter, $K	Graff Off™		DN-30	
	Commercial Scale Plating Shop	Large Commercial or DoD Plating Shop	Commercial Scale Plating Shop	Large Commercial or DoD Plating Shop
Retrofit cost(a)	3	27	5	45
Training	1	3	1	3
Total	4	30	6	48

(a) Retrofit costs were estimated as 25% of the initial capital investment.

Table 12. Estimated Net Change in Capital Investment

Estimated Quantity	Base Case: TCA[(a)]		Graff Off™		DN-30	
	Commercial Scale Plating Shop	Large Commercial or DoD Plating Shop	Commercial Scale Plating Shop	Large Commercial or DoD Plating Shop	Commercial Scale Plating Shop	Large Commercial or DoD Plating Shop
Replacement #1 installed costs	<12	<123	10	109	19	180
Replacement #2 installed costs	NA	NA	NA	NA	19	180
Total replacement capital cost	<12	<123	10	109	38	360
Retrofit cost	NA	NA	4	30	6	48
Net change in capital costs	NA	NA	2[(b)]	16	32	285

(a) The water-break test conducted on TCA samples failed with the first set of 20 panels tested. This implies that the true values could be less than the calculated values.

(b) Net change is total replacement costs and retrofit costs for the commercial or large-scale system minus the replacement cost for the base case system. For the commercial Graff Off™ case, this is ($10K + $4K) – $12K = $2K.

6.2.5 Annual Operating Costs

The estimated annual costs are summarized in Table 13. Annual savings compared to the TCA base case are:

- Graff Off™: ~$102K/year (commercial case) and $1,251K (large scale case) and
- DN-30: ~$101K/year (commercial case) and $1,421K (large-scale case).

Table 13. Estimated Annual Costs

Estimated Cost, $K/year	Basis	Base Case: TCA[a] Commercial Scale Plating Shop	Base Case: TCA[a] Large Commercial or DoD Plating Shop	Graff Off™ Commercial Scale Plating Shop	Graff Off™ Large Commercial or DoD Plating Shop	DN-30 Commercial Scale Plating Shop	DN-30 Large Commercial or DoD Plating Shop
Direct Costs, $K/year[b]							
TotalDegreaser	Usage see Tables 4 & 6	<105 (@$113/gal)	<1,449 (@$113/gal)	4 (@$25.7/gal)	217 (@$25.7/gal)	~0 (@$5/gal)	1.8 (@$5/gal)
Water @ $1.00/K gal	Usage see Tables 5 & 8	<0.2	<1.1	0.2	1.1	0.2	1.1
Steam @ $5.00/K lb	Usage see Tables 5 & 7	0	0	0	0	0	0.2
Electricity	Assumed insignificant						
Wastewater treatment	Waste rate see Table 8, cost is $1.00/1000 gal	<0.2	<1.1	0.2	1.1	0.2	1.1
Waste solvent disposal	Usage see Table 8, cost is $90/55 gal drum	<0.3	<14.4	NA	NA	NA	NA
Environmental compliance costs	$2 K/site plus $18/drum	<2.1	<8.9	2	6	2	6
Operating supplies	10% of maintenance costs[c]	~0	~0	~0	~0	~0	1

Table 13. Estimated Annual Costs

Estimated Cost, $K/year	Basis	Base Case: TCA[a] Commercial Scale Plating Shop	Base Case: TCA[a] Large Commercial or DoD Plating Shop	Graff Off™ Commercial Scale Plating Shop	Graff Off™ Large Commercial or DoD Plating Shop	DN-30 Commercial Scale Plating Shop	DN-30 Large Commercial or DoD Plating Shop
Maintenance and repair	3% of capital investment[c]	<0.4	<3.7	0.3	3.3	0.6	5.4
Fixed Charges, $K/year							
Depreciation	Annual charge to cover replacement capital	<1.0	<8.0	1.0	7.0	3.0	24.0
Plant overhead	10% of capital investment[c]	<1.2	<12.3	1.0	10.9	3.8	36.0
Total annual cost, $K/year		<110	<1,498	8	274	10	77
Total annual cost, $/1000 ft^2 surface degreased per year		<1	<0.20	0.08	0.03	0.09	0.01
Savings, $K/year versus Base Case		NA	NA	102	1,251	101	1,421

(a) The water-break test conducted on TCA samples failed with the first set of 20 panels tested. This implies that the true values could be less than the calculated values.

(b) Assumes no change in labor charges for the various alternative degreasers.

(c) Reference: Factors from Peters and Timmerhaus, Plant Design and Economics for Chemical Engineers, 4th ed., McGraw-Hill, 1991, p 210.

6.3 Investment Analysis and Conclusions

The capital cost and operating cost savings were assessed. Three performance factors, NPV, IRR, and payback period were calculated for each case investigated, see Table 14.

Table 14. Investment Assessment

Investment Measure	Basis	Graff Off™		DN-30	
		Commercial Scale Plating Shop	Large Commercial or DoD Plating Shop	Commercial Scale Plating Shop	Large Commercial or DoD Plating Shop
Retrofit Costs, $K		4	30	6	48
Savings vs. base case, $K/year		102[a]	1,251 [b]	101[c]	1,421[d]
Charge for tanks for each replacement, $K		10	109	19	180
NPV, $K	15 year investment period and 8% discount rate	867	10,646	838	11,896
IRR, %	15 year investment period	2,553	4,171	1,677	2,961
Payback period, years		0.04	0.02	0.06	0.03

(a) Except in year 15 where the tanks are replaced and savings are reduced to $92K/year (e.g., $102K -$10K = $92K/year).
(b) Except in year 15 where the tanks are replaced and savings are reduced to $1,142K/year.
(c) Except in years 7 and 15 where the tanks are replaced and savings are reduced to $82K/year.
(d) Except in years 7 and 15 where the tanks are replaced and savings are reduced to $1,241K/year.

The investment criteria were presented earlier in Table 9. The analysis indicates that both Graff-Off™ and DN-30 are very attractive degreasing substitutes for removing oil from contaminated steel surfaces as compared to TCA. The NPV is positive and very large, the IRR is far above the 8% discount rate, and the payback is much less than the desired 3-year level.

Comparison of the two alternatives is less clear. The payback periods are similar. But, the NPV for the Graff-Off™ and the IRR are slightly higher. The higher cleaning efficiency (ft^2/gal) and lower degreaser costs of DN-30 are offset by the higher capital requirements.

Financially, both appear to be excellent choices as a replacement for the base-case TCA degreaser.

7.0 References

1. Literature reference from EPA website at www.EPA.gov/opptintr/p2home
2. Graff-Off™ literature at www.hydra-tone.com/Graff-OffFrame/Source.htm
3. Monzyk, Bruce, Hindin, Barry, and Chen, Abraham, "Use of Picklex® as a Cost Effective Metal Treatment", Final Report to U.S. EPA, National Risk Management Research Laboratory, by Battelle Memorial Institute, June 14, 2000, p. 22.
4. Technical Order (TO) 42C2-1-7. Process Instructions. Metal Treatments to Meet Air Force Maintenance Requirements.
5. Yi, Ye, "Removal of Oil and AFFF Foam from Wastewater by Air Sparged Hydrocyclone Technology", Air Force Research Laboratory, Report No. AFRL-ML-TY-1998, Reference No. 4537, Tyndale Air Force base, Panama City, FL, August 1997.
6. Private Communication, Jon Owens, Hill Air Force Base, November 12, 1999. (Rate for IWIP was $10.72/K gal).
7. Metcalf & Eddy "Investigation of Industrial and Sanitary Wastewater Treatment Plant, at Robins AFB, Georgia", March 1992, Submitted to HAZWRAP Office of Martin Marietta Energy Systems for the U.S. Department of Energy. (Rate of IWIP was $1.67/K gal for biological treatment).

8.0 Appendices

There are three appendices. Appendix A includes data from the degreasing tests. Appendix B includes information on alternative performance measure tests. Appendix C includes QA and Technical reviews from the EPA with Battelle's responses inserted. Battelle's responses have been incorporated into the main report where appropriate.

Appendix A: Data

This Appendix contains the following sections:

Appendix A - Section 1. Beaker Test Data

Table A-1 Results of Beaker Test for Predicting Bath Exhaustion Level and Bath Pre-Contamination Level

Description[(a,b)]	Units	TCA	Graff-Off™	DN-30
Total volume in beaker	L	0.70	0.70	0.70
Exhaustion oil level[(b)]	ml	9.68	13.83	20.75
Estimated weight exhaustion rate[(1)]	g/gal	43.46	62.08	93.12
Estimated surface area exhaustion rate[(2)]	ft^2/gal	918	1312	1968
Estimated panel exhaustion rate of 3.5L bath[(3)]	Panels/bath	4018	5741	8611
Estimated weight of oil to exhaust 3.5L bath[(4)]	g	40.18	57.41	86.11
Pre-contamination level[(5)]	g	36.17	51.67	77.50
Equivalent panels at pre-contamination level[(6)]	No. of panels	3617	5167	7750

[a] See Appendix A Section 2 and Section 3 (1 through 6) for details on the constants and equations used.

[b] The beaker test was carried out by placing 350 mL of degreasing solution (cleaner) in a 1-L beaker and adding known amounts of jet engine oil. After good mixing panels were immersed into the contaminated degreasing solution and then tested in the water-break test. The amount of contamination needed to cause panels to fail the water-break test noted as the cleaner exhaustion oil level.

Appendix A – Section 2. Constants Used in the Calculations

- Panel Size = 2.9989 X 4.9879 X 0.0315 inches (measured)
- Total panel surface area = 0.2113 ft^2/panel
- Average load on a panel = 0.01 g/panel
- Density of oil = 0.83 g/ml
- 1 gal = 3.785 L
- Bath volume = 3.5 L

Appendix A-Section 3. Equations Used in the Calculations:

1 Estimated Weight Exhaustion Rate is g of exhaustion oil level per gallon of degreaser

= (oil volume* oil density/total volume tested)*(3.785 L/gal)

2 Estimated Surface Area Exhaustion Rate is surface area cleaned per gallon of degreaser

= Estimated Weight Exhaustion Rate *(0.2113 ft^2/panel)/(0.01g/panel)

3 Estimated Panel Exhaustion Rate of 3.5L Bath

= Estimated Weight Exhaustion Rate/(0.01 g/panel)*3.5L *(gal/3.785L)

4 Estimated Weight of oil to Exhaust 3.5L bath

= Estimated Weight Exhaustion Rate* Bath volume of 3.5L*(gal/3.785L)

5 Pre-contamination level is 90% of Estimated Weight of oil to Exhaust 3.5L bath

= 0.9*(Estimated Weight of oil to Exhaust 3.5L bath)

6 Equivalent panels at pre-contamination level

= Pre-contamination level/ (0.01 g/panel)

Appendix A-Section 4. Panel Cleaning Data

Data in Tables A-2, A-3, and A-4 are based on measurements for groups of 20 panels per cleaning cycle. These measurements were carried out before random sampling was done for the water-break test, NVR, and FTIR. The contamination load for 20 panels ranged from 0.1 to 0.3 g and the residual contamination after a cleaning cycle, but before bath exhaustion on 20 panels ranged from less than 0.01 to 0.1 g. Therefore, the contamination load on a single panel would be expected to range from 0.005 to 0.015 g and the residual contamination per panel to range from 0.0005 to 0.005 g (i.e., 0.5 to 5 mg). These contamination levels for individual panels border around the limits of instrumentation and accuracy.

Table A-2. TCA Process Data

						Rinse Bath #1		RinseBath #2					
	Panel Throughput	Equivalent Panel Throughput	Total Surface Area Cleaned per Volume Cleaning Agent, ft^2/gal	Net Applied Weight on 20 Panels, gms	Degreaser Temp, C	pH	Temp., C	pH	Temp., C	Residual Contam. Wt. on Panels, gms	Oil Mass Solubilized in Bath, gms	Total Oil Solubilized in Bath, gms	Water Break Test
		3617[(a)]	826									36.2	
	20	3637	831[(b)]	0.22	24.5	7.80	24.8	7.85	24.8	0.03	0.19	36.4	Fail
	20	3657	835	0.14	24.5	7.89	25.0	7.90	25.0	0.00	0.14	36.5	Fail
	20	3677	840	0.20	24.0	7.82	22.3	7.80	22.0	0.11	0.09	36.6	Fail
	20	3697	845	0.24	24.0	7.93	22.5	8.03	22.6	0.08	0.16	36.7	Fail
	20	3717	849	1.26	24.0	7.93	22.2	7.94	22.5	0.33	0.93	37.7	Fail
	19	3736	853	0.26	20.0	7.88	22.6	7.88	22.9	0.09	0.17	37.8	Fail
	20	3756	858	0.24	19.5	7.82	22.6	7.84	23.2	0.11	0.13	38.0	Fail
	20	3776	863	0.20	19.5	7.86	23.2	7.87	23.3	0.11	0.09	38.1	Fail
	20	3796	867	0.22	20.3	7.93	24.5	7.92	25.2	0.10	0.12	38.2	Fail
	20	3816	872	0.30	20.0	7.86	22.6	7.85	22.7	0.12	0.18	38.4	Fail
	20	3836	876	0.26	19.5	7.93	23.2	7.91	23.4	0.07	0.19	38.5	Fail
Ave.:	19.9			0.32						0.10	0.22		
Ave./Panel:				0.02						0.01	0.01		

(a) Corresponds to the number of panels equivalent to the gms of additional oil used to pre-contaminate the bath

(b) <3637 panels x 0.2113 ft^2/panel/3.5L degreaser x 3.785L/gal <831 ft^2/gal (This value was used for economic calculations)

Table A-3. Graff-Off™ Process Data

					Rinse Bath #1		Rinse Bath #2					
Panel Throughput	Equivalent Panel Throughput	Total Surface Area Cleaned per Volume Cleaning Agent, ft^2/gal	Net Applied Weight on 20 Panels, gms	Degreaser Temp, C	pH	Temp., C	pH	Temp., C	Residual Contam. Wt. on Panels, gms	Oil Mass Solubilized in Bath, gms	Total Oil Solubilized in Bath, gms	Water Break Test
	3617(a)	826									36.17	
19	3636	831	0.22	25.0	7.65	24.2	7.69	24.8	0.03	0.19	36.36	Pass
20	3656	835	0.25	24.0	7.90	24.4	7.92	24.3	0.01	0.24	36.60	Pass
20	3676	840	0.24	24.0	7.94	23.7	7.93	24.3	0.05	0.19	36.79	Pass
20	3696	844	0.22	20.1	7.34	22.6	7.4	22.6	0.00	0.22	37.01	Pass
20	3716	849	0.26	20.5	7.77	22.9	7.74	22.9	0.07	0.19	37.20	Pass
20	3736	853	0.26	20.5	7.78	22.6	7.76	22.9	0.04	0.22	37.42	Pass
20	3756	858	0.27	22.0	7.59	22.9	7.69	22.3	0.01	0.26	37.68	Pass
20	3776	863	0.23	22.0	7.69	23.3	7.77	23.2	0.01	0.22	37.90	Pass
20	3796	867	0.21	22.6	7.62	25.5	7.69	25.4	0.00	0.21	38.11	Pass
20	3816	872	0.24	22.6	7.88	25.8	7.89	25.7	0.01	0.23	38.34	Pass
19	3835	876	0.25	24.0	7.90	24.9	7.89	24.9	0.04	0.21	38.55	Pass
20	3855	881(b)	0.25	24.0	8.22	24.0	8.17	24.0	0.06	0.19	38.74	Fail
Ave.: 19.8			0.24						0.03	0.21		
Ave./Panel:			0.01						0.00	0.01		

(a) Corresponds to the number of panels equivalent to the gms of additional oil used to pre-contaminate the bath

(b) 3855 panels x 0.2113 ft^2/panel/3.5L degreaser x 3.785L/gal = 881 ft^2/gal

Table A-4. DN-30 Process Data

				Degreaser		Rinse Bath #1		Rinse Bath #2					
Panel Throughput	Equivalent Panel Throughput	Total Surface Area Cleaned per Volume Cleaning Agent, ft^2/gal	Net Applied Weight on 20 Panels, gms	Temp, C	pH	pH	Temp., C	pH	Temp., C	Residual Contam. Wt. on Panels, gms	Oil Mass Solubilized in Bath, gms	Total Oil Solubilized in Bath, gms	Water Break Test
	3617[a]	826										36.17	
20	3637	831	0.20	71.5		7.86	24.3	7.88	24.5	0.01	0.19	36.36	Pass
20	3657	835	0.22	68.0		7.82	23.7	7.82	24.3	0.02	0.20	36.56	Pass
20	3677	840	0.20	69.0		7.91	24.2	7.90	24.5	0.01	0.19	36.75	Pass
20	3697	845	0.20	62.5	10.3	8.24	22.6	8.24	22.7	0.00	0.20	36.95	Pass
20	3717	849	0.20	58.4	10.4	8.43	22.9	8.49	22.9	0.02	0.18	37.13	Pass
20	3737	854	0.26	66.3	10.5	8.32	23.4	8.20	23.0	0.05	0.21	37.34	Pass
20	3757	858	0.23	55.9	10.5	7.87	23.0	7.76	23.0	0.04	0.19	37.53	Pass
20	3777	863	0.26	61.8	10.7	7.99	23.4	7.99	23.3	0.02	0.24	37.77	Pass
20	3797	867	0.21	57.7	10.4	7.94	25.6	7.97	25.2	0.02	0.19	37.96	Pass
20	3817	872	0.20	54.1	10.5	7.97	25.8	8.01	25.5	0.02	0.18	38.14	Pass
19	3836	876	0.22	62.1	10.1	8.28	23.1	8.21	24.8	0.04	0.18	38.32	Pass
20	3856	881	0.23	71.0	9.83	8.42	20.9	8.30	20.1	0.07	0.16	38.48	Pass
20	3876	885	0.31	64.0	9.94					0.01	0.30	38.78	Pass
20	3896	890	0.30	65.6	10.0					0.02	0.28	39.06	Pass
20	3916	895	0.25	62.5	10.0					0.00	0.25	39.31	Pass
6[c]	4922[c]	1124[c]	0.06	59.6	10.0	8.11	20.9	7.60	21.1	0.01	10.05[c]	49.36[c]	Pass
6[c]	5928[c]	1353[c]	0.05	65.6	9.9	8.28	22.1	8.26	22.1	0.04	10.01[c]	59.37[c]	Pass
6[c]	6934[c]	1583[c]	0.06	64.0	9.9	8.47	22.9	8.43	22.8	0.02	10.04[c]	69.41[c]	Pass
6[c]	7940[c]	1813[c]	0.04	60.3	9.9	8.26	22.5	8.32	22.6	0.01	10.03[c]	79.44[c]	Pass
6[c]	8946[c]	2042[b] [c]	0.05	57.6	10.0	8.79	18.5	8.35	18.6	0.05	10.00[c]	89.44[c]	Fail

(a) Corresponds to the number of panels equivalent to the gms of additional oil used to pre-contaminate the bath

(b) 8946 panels x 0.2113 ft^2/panel/3.5L degreaser x 3.785L/gal = 2042 ft^2/gal

(c) 6 contaminated panels were tested after additional 10 g of contamination added. Results reflect number of panels equivalent to the additional contamination and the oil solubilized in bath.

Appendix B. Alternative Performance Measure Test Results

Three alternative performance measures were examined to quantify bath contamination and the approach to bath exhaustion. None of the three methods provided an effective alternative to the water-break tests. Results for each technique are presented in this appendix. The appendix consists of the following sections:

Appendix B-Section 1. Weight Change Analysis

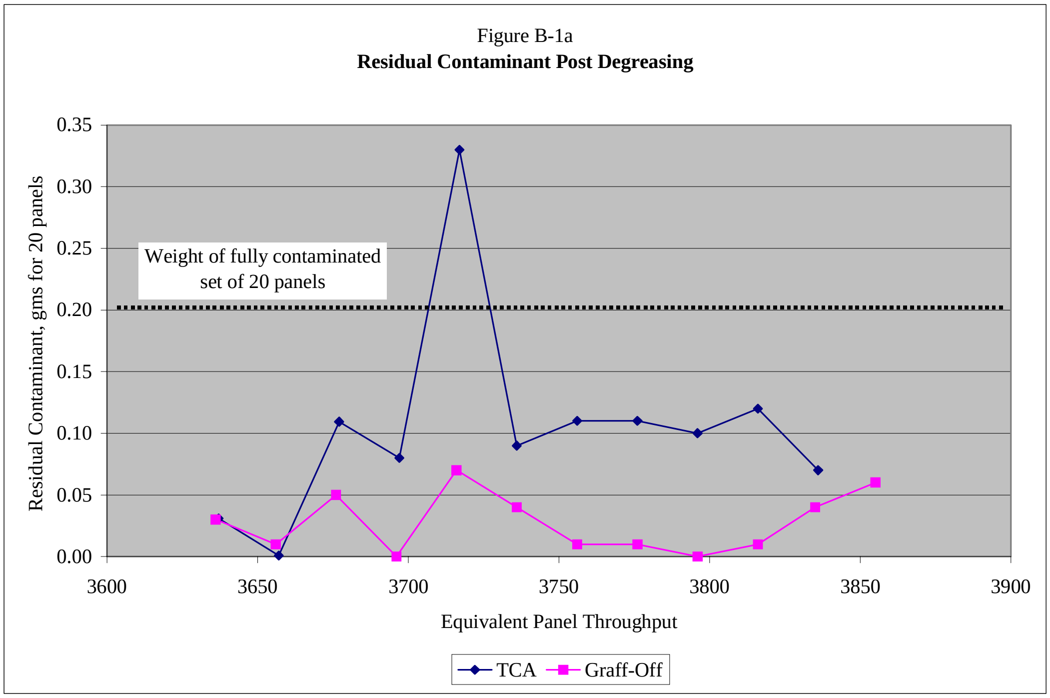
Appendix B-Section 2. NVR Analysis

Appendix B-Section 3. FTIR Analysis

Appendix B - Section 1. Weight Change Analysis

The weight of the test panel after degreasing and air-drying was compared to the test panel containing the jet engine oil prior to degreasing. Results were reported in mg of contaminant/ft^2 of panel. The samples were weighed before contamination and weighed after contamination to establish the contaminant load. After the cleaning process, the samples were dried in a clean atmosphere and weighed again to find out how much material was retained on the sample. The amount of residue left on the sample was an indication of how much cleaning had taken place. Test data were included in the TCA, Graff-Off™, and DN-30 tables in Appendix A.

Weight reduction analysis has been found to be an accurate and sensitive measure of cleanliness. It, however, has an inherent problem with detection of very small amounts of soils or residues. Figures B-1a and B-1b show the results of the weight reduction analysis. In general, panels coming out of any degreaser process had greater than 70% of the contaminant load removed. The weight reduction results showed significant scatter and did not show a trend useful for predicting the level of cleanliness. This could be due to the inherent problem of measuring very small amounts of contaminants. Another reason could be that as the bath approached exhaustion, more residual deposits (e.g., phosphate deposits from the alkaline cleaners) adhered to the panels leading to scatter in the weight differences. The weight reduction analysis did not compliment the water-break test and was not a good performance measurement for panel cleaning in this project.



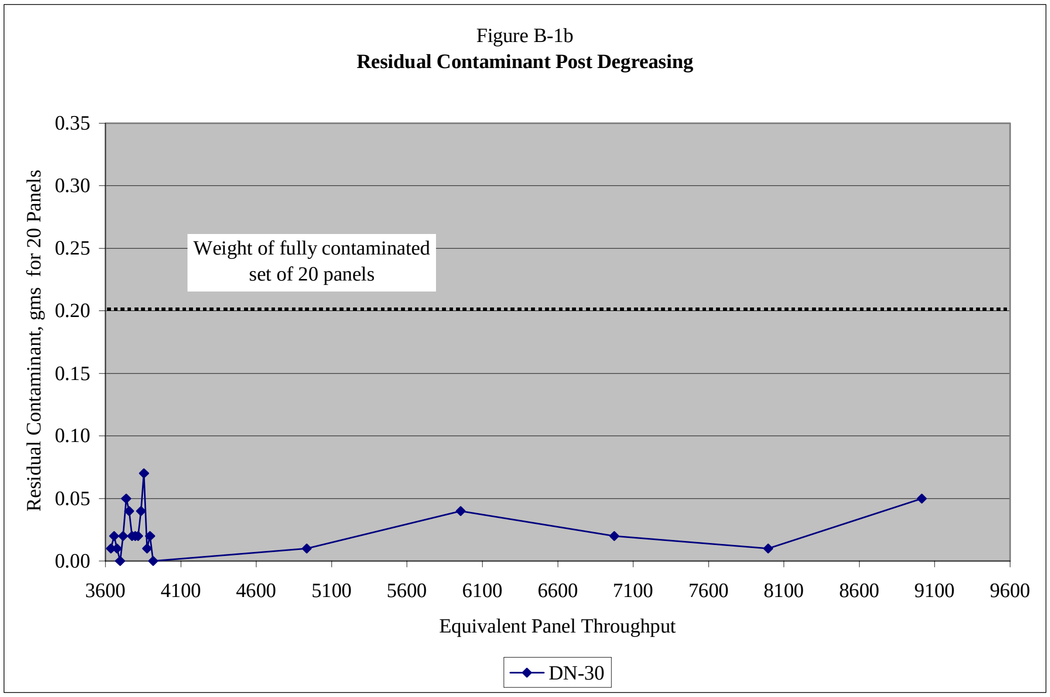


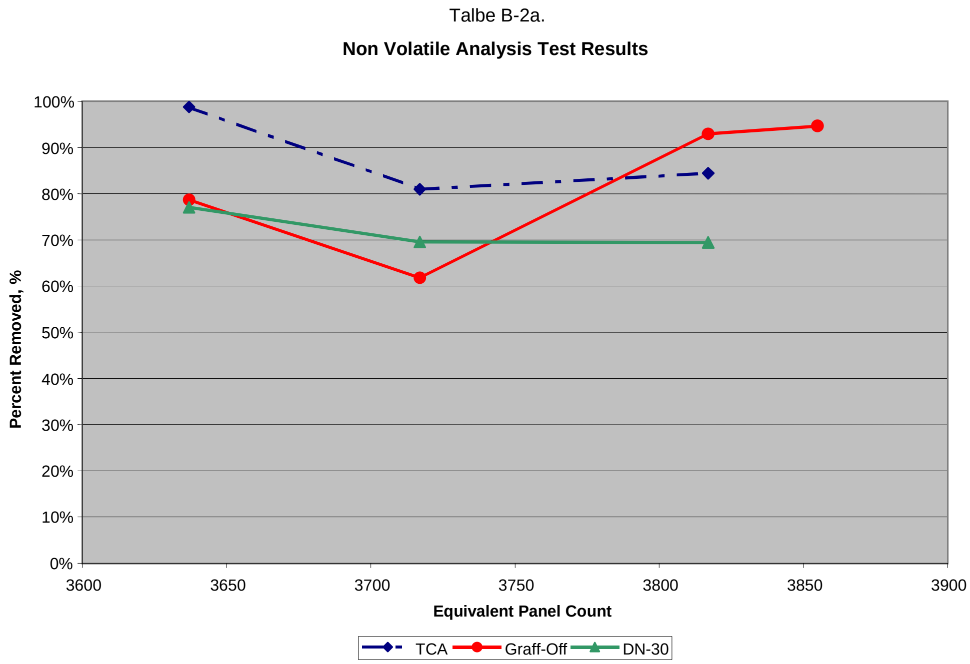
Figure B-1. Weight Reduction for Degreasing Baths

Appendix B - Section 2. NVR Analysis

The NVR provides a means to assess the amount of non-volatile contaminants left on a cleaned surface after degreasing. The procedure is described in MIL-STD 1359B and ASTM F 331-72 (Standard Test Methods for Nonvolatile Residue Halogenated Solvent Extract for Aerospace Components). One change implemented was making ethyl acetate the solvent choice. Three panels out of the group of 20 panels coming out of a cleaning cycle were randomly selected and individually prepared for the NVR analysis. The degreased panels were rinsed with ethyl acetate until no residual oil was visible.

The solvent is collected and mixed with an equal amount of clean solvent. The weight of the residue is determined by evaporating to dryness at a maximum temperature of 45 C (113 F). The results are reported in mg residue/ft^2. The NVR has been found to be insensitive to small amounts of contaminants adhering to the surface. But, in some cases, there is evidence that considerable amounts of contaminants can adhere to the surface after NVR testing.

The NVR measures the residue contaminants left on the surface of the panels following degreasing. It should provide information similar to weight reduction test. The contamination load for 20 panels ranged from 0.1 to 0.3 g and the residual contamination after a cleaning cycle, but before bath exhaustion on 20 panels ranged from less than 0.01 to 0.1 g. Therefore, the contamination load on a single panel would be expected to range from 0.005 to 0.015 g and the residual contamination per panel to range from 0.0005 to 0.005 g (0.5 to 5 mg). These contamination levels for individual panels border around the limits of instrumentation and accuracy. The data for the NVR tests are shown in Figures B-2a and B-2b. The results, reported in terms of percent oil removed, indicate that 70% or more of the contamination is removed by all 3 degreaser baths, but the non-volatile residual weight change does not vary significantly with the number of panels cleaned. This is expected since the residual contamination from an individual panel is in the 0.5 to 5 mg range. The water-break tests indicated that the TCA, Graff Off™, and DN-30 failed at 3,637, 3,855, and 9,016 panels, respectively, but the NVR data does not show any indication of changes in the residual removed from the panels at or near these exhaustion points. Thus, no correlation between the exhaustion levels could be identified. Therefore, the NVR was not found to be an effective performance measure.



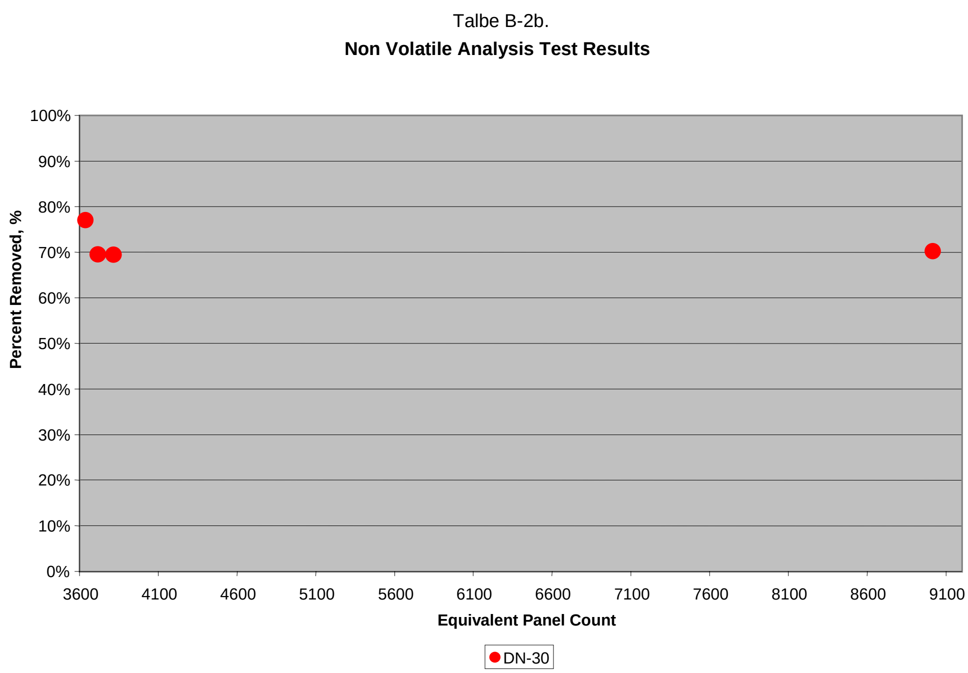


Figure B-2. NVR Data on Cleaned Panels

Appendix B - Section 3. FTIR Analysis

The FTIR analysis system used in this study was a Digilab FTS-60A. It is sensitive to low levels of contaminants. Grazing angle reflectance was used to provide rapid and precise information of organic contamination. A contaminant area of ¼-in by 3/8-in. to ½-in. were inspected. Contaminated areas were identified and quantified based on the relative peak height and peak area of absorption of infrared energy.

The FTIR system was calibrated by measuring the relative IR absorption of a test surface versus a totally clean and a fully contaminated surface.

The FTIR measured absorption was compared to this 2-point calibration curve and a relative cleanliness measure was determined as a percent of oil retained (i.e., contamination). As in the case of weight-change and NVR tests, there was no correlation between FTIR results and the water-break test results. As noted in Figure B-3a and B-3b, and Table B-1, as the bath became progressively more contaminated, there was no trend in predicted contamination level. Even at bath exhaustion points, there was no change in predicted contamination level.

Thus, FTIR was not found to be a useful performance measure.

Figure B-3a.
FTIR Results

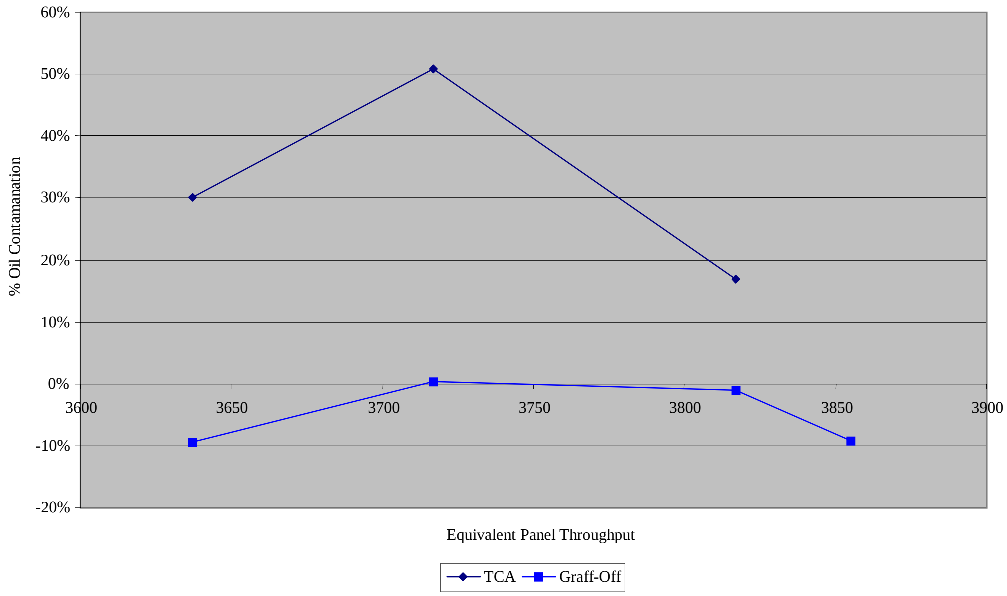


Figure B-3b.
FTIR Results

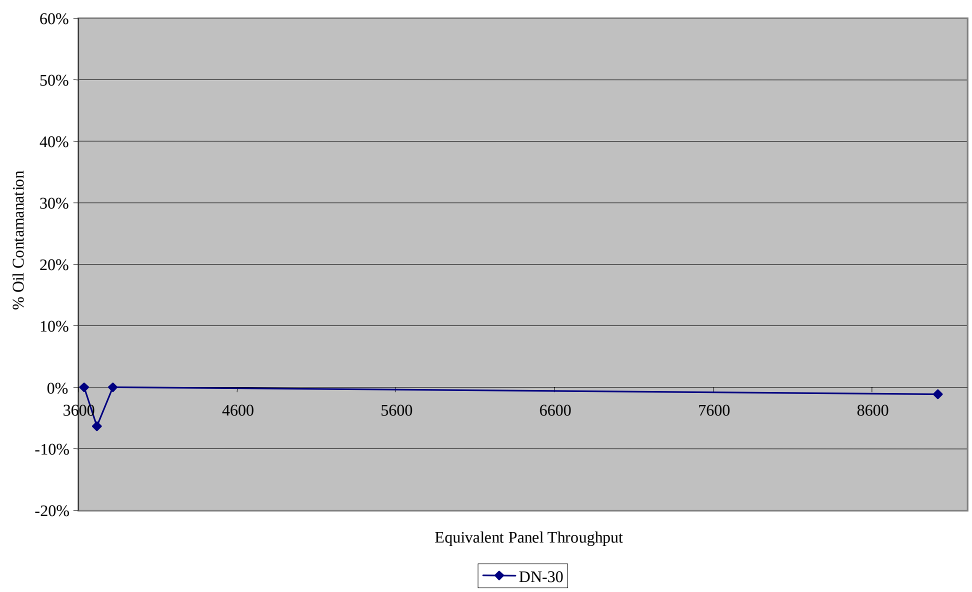


Figure B-3. FTIR Analysis on Cleaned Panels

Table B-1. FTIR Data

Description	Equivalent Panel Throughput	% of Oil Coating Retained, based on Peak Height	% of Oil Coating Retained, based on Peak Area	Notes
Blank steel panel	0	2%	5%	No Hydrocarbon
Blank steel panel	0	2%	4%	No Hydrocarbon
2% Jet engine oil	0	100%	100%	Hydrocarbon
2% Jet engine oil	0	100%	100%	Hydrocarbon
DN-30, 20 panel throughput	3637	5%	9%	Value due to poor spectrum
DN-30, 20 panel throughput	3637	13%	20%	Value due to poor spectrum
DN-30, 20 panel throughput	3637	13%	22%	Value due to poor spectrum
DN-30, 100 panel through put	3717	3%	3%	No Hydrocarbon
DN-30, 100 panel throughput	3717	0%	-1%	No Hydrocarbon
DN-30, 100 panel throughput	3717	16%	30%	Value due to poor spectrum
DN-30, 200 panel throughput	3817	-2%	-4%	
DN-30, Exhaustion	9016	7%	6%	
Graff-Off™, 20 panel throughput	3637	-1%	-8%	No Hydrocarbon
Graff-Off™, 20 panel throughput	3637	0%	-6%	No Hydrocarbon
Graff-Off™, 20 panel throughput	3637	-3%	-14%	No Hydrocarbon
Graff-Off™, 100 panel throughput	3717	7%	4%	Hydrocarbon
Graff-Off™, 100 panel throughput	3717	2%	-1%	No Hydrocarbon
Graff-Off™, 100 panel throughput	3717	-3%	-2%	No Hydrocarbon
Graff-Off™, 200 panel throughput	3817	0%	1%	No Hydrocarbon
Graff-Off™, 200 panel throughput	3817	2%	-2%	No Hydrocarbon
Graff-Off™, exhaustion	3855	-2%	-9%	
TCA, 20 panel throughput, exhaustion	3637	26%	28%	Hydrocarbon
TCA, 20 panel throughput	3637	28%	28%	Hydrocarbon
TCA, 20 panel throughput	3637	33%	34%	Hydrocarbon
TCA, 100 panel throughput	3717	57%	71%	Hydrocarbon
TCA, 100 panel throughput	3717	42%	43%	Hydrocarbon
TCA, 100 panel throughput	3717	38%	38%	Hydrocarbon
TCA, 200 panel throughput	3817	15%	17%	Hydrocarbon

Appendix C. Report Review: EPA Comments and Battelle's Responses

The report was reviewed by EPA for quality assurance (QA review) and for technical merit (Technical review). Battelle responded to both reviews and then received endorsements from the EPA on both responses. The reviews and Battelle's responses are presented in this appendix. References to page numbers may no longer correspond since the report has been revised. Battelle's responses have already been incorporated into the main report where appropriate. The appendix consists of the following sections:

Appendix C-Section 1. QA Review and Battelle's Response

Appendix C-Section 2. Endorsement of Battelle's Response to QA Review with some comments

Appendix C-Section 3. Technical Review and Battelle's Response

Appendix C-Section 1. QA Review and Battelle's Response

QA REVIEW OF RESEARCH RESULTS DOCUMENT

GENERAL INFORMATION

QA ID No.:	341-F1-0	Project QA Category:	Applied
EPA Technical Lead Person (TLP):		David Ferguson	
Document Type/Title:		Draft Report - Laboratory Scale Evaluation of Hydra-Tone Graff-Off Coconut Oil Based Degreaser	
Document Generator (Organization):		Battelle	
Document Date:	09/30/00	Date Rec'd in QA Office:	10/19/00

REVIEW SUMMARY

QA Review Distribution Date	11/09/00	Endorsement Status	**Not Endorsed**
NRMRL-Ci QA Reviewer	Lauren Drees	No. of Findings	1
Telephone No.	569-7087	No. of Observations	5

INSTRUCTIONS IF NOT ENDORSED:

Information needs to be provided to the QA office to address all cited findings (see attachment) before the document is finalized. You are also encouraged to address each observation and editorial comment identified. The method of communicating the information is left to your discretion. Suggested communication methods include: a) submission of a revised document with the changes marked; b)submission of a memorandum that addresses each issue which includes evidence of proposed document modification(s); or c) a meeting to discuss cited issues, with subsequent submittal of a revised document for review. Only after resolution of all findings will the research results document be endorsed.

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cc: Roger Wilmoth (w/attachment)

FINDING

1.0 **Performance Measurements.** Table 1 identifies three measurements as critical to demonstrating the performance of the Graff-Off™ technology - weight change, NVR, and water-break test. However, the report indicates that neither weight change nor NVR data could be used in assessing performance, due to the minimal quantity of contamination applied to the test panels. Therefore, all conclusions relative to performance are based on results for the qualitative and subjective (i.e., pass/fail) water-break tests. Additional information is needed to support the validity of these water-break tests. For example, the associated QAPP for this project stated that "control tests will be conducted to compare and contrast the qualitative performance measurements." How were these control tests conducted and what were the results? How was the comparability of water-break results ensured? What is the impact of invalid weight change and NVR data on project conclusions?

Battelle's Response:

A. The supporting information for the validity of the water-break tests:
   1. Controls: Fresh panels removed directly from the shipping package were tested for water-break as a control to those contaminated and cleaned. 3 panels out of every box of 100 were tested. They all passed the water-break test. (This is included in the report in Section 4.4.1)
   2. Comparability: The panels that failed the water-break test were passed through the cleaning process a second time. If they still failed the water-break test then the conclusion of the bath being at the point of exhaustion was validated. Another set of panels was then passed through the cleaning process again and 3 panels were tested to confirm bath exhaustion as determined by the water-break test. This ensured comparability of the water-break test between the different cleaning baths. (This is included in the report in Sections 4.4.1 and 4.4.2)
   3. Impact of invalid weight change and NVR data: There is no real impact of the invalid weight change and NVR data. It was the intention of this project work to have more than one indicator of bath (cleaner) performance. It was also intended that all measurements of performance would be in agreement with each other to make a stronger case for judgement. The results indicate that NVR and weight change were neutral indicators of performance. The weight change and NVR data did not indicate whether one cleaner performed better than the other one. Only the water-break test gave a clear indication of bath or cleaner performance. The fact that the water-break test was the only performance indicator does not jeopardize the results especially when none of the others indicated otherwise. (This is included in the report in Sections 2.0 and 3.0)

OBSERVATIONS

1.0 **Panel Sampling.** Section 4.4.1 indicates that three panels from each group of 20 were selected at random, weighed, and tested for water break. However, it is not clear what the data in Tables A-1 through A-3 represent, since each group of 20 panels lists only a single result for each measurement. Are the results presented averages for the three panels? If so, how were average results determined for the water-break test? Clarification is needed regarding the applicability of the results presented in these tables to the three panels which were randomly selected from each group of 20.

Battelle's Response:

1. Table A-1: This table is based on the results of the beaker test. The beaker test does not use panels for testing but rather physico-chemical observations such as deposits falling out of solution. The contamination (oil) level that causes the physico-chemical changes is then extrapolated to an equivalent panel loading or equivalent surface area cleaned per volume. (This is clarified in Appendix A of the report.)
2. Table A-2 through A-4: The single result listed for each measurement in these tables is for 20 panels (all 20 measured together). The only measurement in these tables done on 3 panels is the water-break test. (This is clarified in Appendix A of the report.)
3. Average Results for Water-Break Test: The results for 3 panels tested for the water-break test were always in agreement. They were either all passed panels or all failed panels. The plan for a situation where one out of the three was in disagreement was this: Three more panels would be tested from that batch (group of 20) and if the same situation arose then majority finding would be reported. If the opposite result occurred in the second set then it would be noted and the bath would be presumed approaching exhaustion. The next set of panels to be cleaned would then be evaluated to confirm. (This is clarified in Section 4.4.2 of the report.)
4. Applicability of Results: The results are applicable to the three panels that were randomly selected because the weight change data was collected for all 20 panels together before randomly selecting the 3 panels for different tests. However, the data is clear that small loads of 0.2 to 0.3 grams per 20 contaminated panels and 0.01 to 0.1 grams per 20 panels for residual contaminant were acheived. This clearly suggests that the NVR data for individual panels will be around 0.5 to 5 mg for the residual contamination on single panels. This weight range borders around the limits of instrumentation and accuracy. Therefore, the group data will not be directly applicable to the NVR data. (This is clarified in Appendix A and Appendix B of the report.)

2.0 **Beaker Test Results.** Section 5.2 states "The beaker test estimate for bath exhaustion was equal to or higher than the actual exhaustion levels determined for each degreaser." However, for the DN-30 alkaline cleaner, Figure 5 shows the estimated number of panels to be 8611 and the actual number to be 9016. Are these considered equal?

Battelle's Response:

1. Are these considered equal?: Yes, within a 10% margin of predictability this will be considered "equal".

2. Section 5.2 statement amended to read: "Within a 10% margin of error, the beaker test estimate for bath exhaustion was equal to the actual exhaustion levels determined by the water-break test for each degreaser except the Graff-Off™. For Graff-Off™, the beaker test predicted a higher level for bath exhaustion than the actual exhaustion level determined by the water-break test".

3.0 **TCA Performance.** Figure 6 lists the total surface area cleaned at bath exhaustion for TCA to be 831 ft^2/gal. This value is used throughout the report when determining degreaser requirements and related costs. However, for TCA, the first set of panels tested after pre-contamination failed the water-break test, meaning that the actual value is less than this reported value. The magnitude of the difference is not known. It is recommended that the report address this issue and clarify that the actual result is some number less than the reported value.

Battelle's Response:

1. The magnitude of the difference is not known for real, but using the beaker test as a point of reference, it would not be far off.
2. The report will be amended to clarify the values as less than the reported values. This could be done by reporting the calculation results as inequalities (i.e., <X).

4.0 **Estimated Degreaser Requirements.** Section 6.1.1 indicates that the volume of a typical cleaning tank in a commercial job shop is 27 ft^3, which is equivalent to 200 gal. However, in Table 4, when determining the number of bath changes per year, the report uses a value of 27 gal/bath. Should this be 200 gal/bath? If so, results for the number of bath changes per year and volume of degreaser required per year will be affected, as well as costs determined in Table 8. The volume used in Table 4 should be verified.

Battelle's Response:

The volume used in Table 4 was incorrect and changes have been made to reflect the error(s) in Table 4 and Table 8. Also, errors in Table 6 and A4 were found and corrected. In addition, Dave Ferguson found that the cost of Graff-Off™ in large quantities was $30.70/gal (in 55 gal drums) and $25.70 (in 250 gal totes). These values have been incorporated into the operating cost calculations. The updated NIV, IRR, and payback period data are summarized in Table 14. Overall these errors and changes mandated changes to Tables 4, 5, 6, 8, 13, 14, and A4..

5.0 **TCA Bath Change.** Section 6.1.2 states that waste TCA is dumped six times per year for large shop operations. However, Table 6 apparently indicates that the TCA bath is changed five times per year. Consistency is needed.

Battelle's Response:

1. Section 6.1.2 will be amended to be consistent with Table 6 and will read: "TCA is dumped less than five times per year for large shop operations."

EDITORIAL COMMENTS

1.0 Footnote b to Table A-2 should be reviewed for applicability.

2.0 In Table A-4, the total oil solubilized in the bath does not appear to account for the additional contamination added late in the testing.

Battelle’s Response:
A.

1. The footnote b to Table A-2 would be amended to read: “<3637 panels x 0.2113 ft(2)/panel/3.5L degreaser x 3.785L/gal <831 ft2/gal.”
2. The footnote reference “b” is missing in Table A-2 and will be appropriately positioned.
3. The footnote b to Table A-4 would be amended to read: “8946 panels x 0.2113 ft(2)/panel/3.5L degreaser x 3.785L/gal = 2042 ft2/gal.”
4. The footnote reference “b” is misplaced in Table A-4 and will be appropriately positioned.

B. Total oil solubilized in the bath: An adjustment will be made to the numbers and Table A-4 will be amended to reflect the additional contamination added in late testing. Also a footnote (c) will be included to indicate the use of 6 panels after additional contamination was added and also to show that the values in late testing reflect the additional contamination added and the number of panels are the equivalent number based on the amount of oil added.

Appendix C-Section 2. Endorsement of Battelle’s Response to the QA Review with some additional comments

QA REVIEW OF RESEARCH RESULTS DOCUMENT

GENERAL INFORMATION

QA ID No.:	341-F1-1	Project QA Category:	Applied
EPA Technical Lead Person (TLP):		David Ferguson	
Document Type/Title:		Draft Report - Laboratory Scale Evaluation of Hydra-Tone Graff-Off Coconut Oil Based Degreaser	
Document Generator (Organization):		Battelle	
Document Date:	None	Date Rec'd in QA Office:	01/23/00

REVIEW SUMMARY

QA Review Distribution Date	01/25/00	Endorsement Status	**Endorsed**
NRMRL-Ci QA Reviewer	Lauren Drees	No. of Findings	0 (see attached)
Telephone No.	569-7087	No. of Observations	0 (see attached)

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cc: Roger Wilmoth (w/attachment)

Note: While the initial comments have largely been addressed, the following should be considered prior to finalizing the report. The numerical identifiers below are used to track the previous comments. Also, since the revised report was not submitted, it will be necessary to verify that the proposed revisions were made before the report is finalized.

FINDING

1.0 **Performance Measurements.** The response provides information to support the validity of the water-break tests. However, it does not indicate how this information will be incorporated into the report. It is recommended that this information, along with any other applicable information, be included in a readily identifiable QA/QC section in the report.

EDITORIAL COMMENTS

2.0 The footnotes to the revised Table A-4 need additional corrections. Currently, there are two entries for footnote (a) and none for footnote (c). Also, the actual footnote (b) should specify 8946 panels, not 3946 as listed.

Appendix C-Section 3. Technical Review and Battelle's Response

Comments Reviewer 1

While the project provides useful information for general manufacturing facilities that may utilize cold cleaning operations (dunking oily parts in cold solvent), I have some concerns about the applicability (scope) of the project in the metal finishing field, where TCA is generally used in vapor degreasing operations. The concerns are:

1. The project compares Graff-off and DN-30 against TCA, which is no longer used in the industry. It is not clear to me that the same results would be obtained if the comparison was made against trichloroethylene (TCE) or Perchloroethylene (PCE). I would be especially interested in how the economics turned out when the comparison is against these presently used solvents.

Battelle's Response:
The Graff-Off™ evaluation project was designed on a limited scope and limited budget to quickly identify the benefits of Graff-Off™. Due to the limitations, no further work will be done in this work phase. The TCE and PCE comparison will be in the report for "Follow-on" work.

2. The project did not take into account that the TCA could be rejuvenated and re-used many times via distillation. This would alter the economic analysis vs. the alternate degreasers.

Battelle's Response:
This is a very good point. We will mention this option in the economic evaluation section of the report but do not plan to reevaluate the economics to quantity the effects of using TCA rejuvenation and re-use or determine how it impacts the economics findings.

3. The project compared the alternate degreaser products against TCA in a "cold cleaning" application, where the oil is removed via immersion. A more common utilization of TCA is in a vapor degreaser, where the part is both immersed and subjected to the pure vapors of the solvent. This normally produces a very clean, oil free surface, even when the solvent is heavily laden with oil. A comparison against TCA used in such a manner would be of greater use by the metal finishing industry, while the cold cleaning comparison has some use in the manufacturing industry and the aircraft maintenance industry as is indicated in the project (Hill AFB).

Battelle's Response:
Based on the limited scope, limited budget and the hazardous implications associated with using TCA in the vapor degreaser mode, it was decided in the project planning stage (including the proposal scope of work) to do the TCA cleaning using the "cold cleaning" method. The comment is well taken and will be included in the report as a possible "Follow-on" work.

4. One of the key benefits TCA provides is the ability to remove a broad range of soils (oils, greases, buffing material etc.). This project only covers one soil (jet engine oil). The comparison would again have a broader level of utility in the metal finishing industry, if it showed favorable results with a variety of soils, instead of one.

Battelle's Response:
Once again, the Graff-Off™ evaluation project was designed on a limited scope and limited budget as a first cut at identifying the benefits of Graff-Off™. Due to this limitation, no further work will be done in this phase of the evaluation but the comment is well taken and will be included in the report as a possible "Follow-on" work.

Technical Review: Laboratory Scale Evaluation of Hydra-Tone Graff-Off Coconut Oil Based Degreaser
Reviewer 2

This report provides a thorough evaluation of the Hydra-Tone Graff-Off Coconut Oil Based degreaser in comparison to 1,1,1-trichloroethane (TCA) and DN-30 alkaline cleaner. The report provides an excellent economic analysis required for use of Graff-Off or the alternatives. It also provides a clear analysis of the performance capabilities of each cleaner for the tests conducted in this study. The document is concisely written and makes excellent use of figures and tables to convey information.

Although this report is very informative, some minor revisions should be made to improve it. For example, though the report is written for an American audience, the report would be more accessible to the international scientific community if metric system units were included. Also, the report does not make clear why ethyl acetate was used in the contamination solution.

Battelle's Response:
i) The comment on the metric system for a broader international audience is a very good one and will be incorporated into any "Follow-on" work that is done. For now, due to the limited scope and limited budget for this first phase of the Graff-Off™ evaluation no changes will be made to the report.
ii) Clarification for the use of ethyl acetate in the contamination will be made in the report. Ethyl acetate was used as a quick evaporative thinner to allow for thin and uniform contamination of the steel panels.

Attention should be directed to specific areas of text in the report as well. On page 11, the water-break test evaluation method should be explained in more detail instead of waiting until later in the document. On page 23, the average cost for oil and grease treatment is reported at $ 1.00. How was this estimate arrived at? On the next page, why is the estimated treatment cost chosen as the lowest number in the estimate range?

Battelle's Response:
i) It is understood that the water-break test method should be explained in more detail. This change will be incorporated into the report on page 11.
ii) The $1.00 estimate for oil and grease treatment on page 23 was arrived at by a study conducted by a another contractor evaluating improved oil/water separators.
iii) On page 24 the lowest number for the estimated treatment cost was chosen because this figure the most conservative estimate of savings using the Graff Off degreaser. The range provided was very broad. Data from two Air Forces bases where we are familiar with the costs showed their costs were closer in agreement with the lower end of the range of the costs.

There are also some minor organization and presentation issues that need to be addressed. There are tables that reflect lengthy multiplication calculations (tables 4-7). These tables should be reworked to make them more visually appealing. For example, report the number with a footnote beneath the table that refers to an example calculation. Another approach might be to place the calculations in the appendices. On another issue, the discussions of weight change, NVR and FTIR would be more helpful to the reader if they were located in the main body of the report.

Battelle's Response:

i) The minor organizational and presentation issues will be addressed as best as we can without compromising the data or the reports by using footnotes and appendices as suggested.

ii) The discussions of weight change, NVR and FTIR were not added to the main body of the report because they distract from the conclusions that were drawn with the water-break test.

There are additional references to minor editorial corrections, misspelling, etc. dispersed throughout the draft copy of the report. These corrections should be made in addition to the previous comments. With these minor revisions, this document will be an excellent example of an EPA technology evaluation study that incorporates technical performance, economic analysis and life cycle analysis in a manner understandable by a broad audience

Battelle's Response:

The editorial corrections and misspellings noted in the report will be addressed as suggested.