

Cleaning Urethane, Ink and Paint Manufacturing Vessels: A Toxics Use Reduction Case Study

**Raffi and Swanson Inc.
Wilmington, MA 01887**

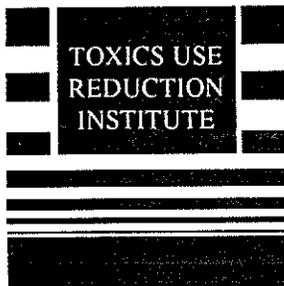
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The Toxics Use Reduction Institute FY 97 Matching Grants Program

**The Toxics Use Reduction Institute
University of Massachusetts Lowell**

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Toxics Use Reduction Institute Matching Grants Program

The Institute annually provides direct funding to Massachusetts industries on a matching basis for toxics use reduction (TUR) feasibility and technology studies. The Matching Grants Program was initiated in FY 93 to facilitate the development and use of innovative techniques that reduce the use of toxic chemicals or the generation of toxic by-products in Massachusetts businesses. Grants are awarded on a competitive basis for companies to conduct TUR studies at their facilities. Recipients prepare project reports which assist in transferring toxics use reduction technologies and methods to other companies.

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Table of Contents

| | |
|---|----|
| 1. Summary..... | 1 |
| 2. Raffi and Swanson, Inc. | 1 |
| 3. Project Management..... | 1 |
| 4. Evaluating Alternative Urethane Cleaning Chemistry..... | 2 |
| 4.1 Lab Testing | 3 |
| 4.1.1 Lab Test 1: Screening Chemistries | 3 |
| 4.1.2 Lab Test 2: Semi-aqueous and NMP at Different Soil Loadings | 4 |
| 4.1.3 Lab Test 3: Aqueous Cleaner Evaluation | 4 |
| 4.1.4 Lab Test 4: Semi-aqueous and NMP Temperature Dependence | 5 |
| 4.1.5 Lab Test 5: 69MC Rinsability Study | 5 |
| 4.1.6 Lab Test 6: 69MC Temperature and Foam Study | 6 |
| 4.2 Bench Scale Testing | 6 |
| 4.2.1 Bench Test 1: 69MC | 7 |
| 4.2.2 Bench Test 2: HTF 85B versus NMP | 7 |
| 4.3 Summary of Lab and Bench Test Results | 7 |
| 4.4 Economic Evaluation | 8 |
| 4.5 Other Improvements to Urethane Cleaning System..... | 8 |
| 5. Reducing Plant Cleaner Use..... | 9 |
| 5.1 Changes in Procedures..... | 9 |
| 5.2 Data Collection Changes | 10 |
| 5.3 Equipment Modifications | 10 |
| 5.4 Summary of Plant Cleaner Reductions | 11 |
| 5.5 Financial Evaluation..... | 12 |
| 5.6 Future Efforts..... | 12 |
| 6. Appendix A: TURI Lab Test Results..... | 1A |

1. Summary

In 1995, Raffi and Swanson, Inc. received a \$20,000 grant from the Massachusetts Toxics Use Reduction Institute (TURI) to examine alternatives to toxic solvents in the company's cleaning operations. Raffi and Swanson set a project goal of reducing chemical use and byproducts from these cleaning operations by 50%. Raffi and Swanson met this goal, reducing chemical use and emissions by roughly 50% (roughly 46,000 lb.) and chemical byproducts 50% (roughly 200,000 lb.). These reductions were achieved through the implementation of over thirty worker-identified changes to cleaning practices. These changes included redesigning equipment and procedures to eliminate the need for solvent cleaning, methods to increase solvent reuse, and more careful management of solvent distribution and application throughout the site. These changes have saved Raffi and Swanson approximately \$18,000/yr.

During the course of the project, Raffi and Swanson worked closely with the TURI Surface Cleaning Lab to evaluate alternatives to N-methyl pyrrolidone in the company's urethane reactor vessel cleaning operation. The Surface Cleaning Lab evaluated seven aqueous cleaners and four semi-aqueous cleaners as replacements for N-methyl pyrrolidone. Two semi-aqueous cleaners were the most promising substitutes but the inability to recycle the cleaners makes them cost-prohibitive.

Despite having exceeded the 50% reduction goal for solvents used in cleaning operations, Raffi and Swanson is looking to go further. The company has received several bids that would eliminate nearly all solvent-cleaning of portable containers. Other semi-aqueous chemistries that hold promise for eliminating N-methyl pyrrolidone are also being examined. Lastly, the highly successful worker-management team continues to meet and work towards fulfilling its goal of reducing wash solvent use to the extent economically and technically feasible.

2. Raffi and Swanson, Inc.

Located in Wilmington, Massachusetts, Raffi & Swanson, Inc. (R&S) manufactures specialty coatings, inks, and adhesives for use on textiles, plastics, and metals. The company sells its products to manufacturers for use in final products or as intermediate materials. Raffi and Swanson operates under Standard Industrial Classification (SIC) code 2851 (paint and allied products) and reports on 23 chemicals in three production units under the Massachusetts Toxics Use Reduction Act (TURA). Raffi and Swanson's chief production processes include loading, mixing, reacting, filtering, transferring and packaging. The company's cleaning operations are conducted in on-line and off-line systems using a combination of pressure spraying and mechanical brushing.

3. Project Management

At the start of the project, Raffi and Swanson recognized the need for both management and worker participation in the project. Two project teams were created as forums for involvement: the TURA Team and the Solvent Wash Elimination and Alternative Techniques Team (SWEATT). The TURA Team was comprised of management, supervisory, research and development personnel and a general practice TUR planner (consultant). The team acted both as a steering committee for the project and as a forum to discuss project activities and assign work tasks. SWEATT was comprised of workers from each solvent-using department. Workers volunteered for SWEATT and

were responsible for developing and implementing TUR options on the plant floor. Both the TURA Team and SWEATT met every other week and posted minutes for the meetings.

In addition to setting up teams, Raffi and Swanson saw the need for a tracking system that could measure solvents used in cleaning operations on a regular basis. Raffi and Swanson wanted to establish a baseline of solvent use and emissions from which to measure TUR progress. The TURA and SWEATT teams developed a tracking system that monitored all solvents used for the purpose of cleaning by each manufacturing department. The tracking system also measured all cleaning solvent waste. Results were summarized weekly, graphed, and posted on the Raffi and Swanson employee bulletin board. To ensure that all solvents used in cleaning would be tracked by the system, the company made important changes to its solvent handling and distribution system. Raffi and Swanson eliminated distribution lines of plant cleaner (a solvent mixture). Prior to this change, any operator could go to solvent manifolds located in several places in the plant, and pump their own solvent. Now employees are required to sign-out solvent from a centralized distribution area. Rather than workers getting their own solvents, all solvent requests must go through a single employee that signs out solvent for each department.

The TURA Team and SWEATT split the project into two sub-projects. One focused on evaluating aqueous and semi-aqueous chemistries to clean urethane vessels. The second focused on making procedural, equipment, and formulation changes to reduce the need to clean and the use of cleaning solvents in the company's centralized container cleaning operation. Each of these sub-projects is reviewed in detail in the following sections of this report.

4. Evaluating Alternative Urethane Cleaning Chemistry

One of Raffi and Swanson's main product lines involves the manufacture of urethane products for the textile coating industry. Making urethane products involves mixing and reacting isocyanate-terminated species with hydroxyl-terminated species. This operation results in long urethane polymer chains. Compounding additives are then added to the urethane polymer in the same mixing vessel to adjust the batch physical properties to the customer's desired specification. Following the compounding operation, the batch is packaged into 55-gallon product drums and shipped to customers.

Before making another batch, residues left on the interior reaction vessel walls must be cleaned. Residues which are not completely removed from the reaction vessel will interfere with future polymerization reactions by pre-maturely "capping" the molecular chain being formed. This premature ending of the chain formation results in a low molecular weight polymers and corresponding poor physical properties. Therefore, the cleaning operations of the tanks are critical to the end-product's quality.

In 1993, Raffi and Swanson changed its urethane vessel cleaning process. The company shifted from using a solvent mixture known as "plant cleaner" (composed of methyl ethyl ketone (MEK), methyl isobutyl ketone, toluene, ethyl acetate, and xylene) to N-methyl-pyrrolidone (NMP). The switch to NMP was an improvement over the plant cleaner process since NMP cleans better, has a lower flash point, poses a lower occupational hazard, and contains fewer Volatile Organic Compounds (VOCs) per gallon. In the cleaning process, 15 gallons of NMP are sprayed onto the 1,500 gal tank walls at 400 psi, drained at the bottom of the vessel, filtered, and recirculated. An MEK rinse follows the NMP wash and is also sprayed, drained, filtered, and

recirculated. Process wastes and emissions include dirty cleaning solvents, fugitive emissions, and sludge that accumulated in 400 micron filter bags.

In 1995 Raffi and Swanson began considering alternatives to NMP when the chemical became a listed TURA substance because of its developmental and reproductive hazards. Cost considerations were also an issue since N-methyl-pyrrolidone wash solvent must be distilled in a costly process involving a contractor that brings mobile vacuum distillation equipment to the plant site. Raffi and Swanson was also interested in getting away from using an MEK rinse. The rinse results in a VOC emission and has a low flash point.

The intent of this task was to test aqueous and semi-aqueous chemistries to further improve the urethane vessel cleaning process. Aqueous and semi-aqueous chemistries typically have lower flammability, lower VOCs, and fewer worker health concerns

4.1 Lab Testing

To evaluate the efficacy of aqueous and semi-aqueous solutions in cleaning urethane residues, the Toxics Use Reduction Institute Surface Cleaning Lab performed a series of bench scale tests. Candidate aqueous and semi-aqueous cleaning solutions were identified via a literature search, an Internet search, a review of TURI vendor files, and recommendations from colleagues. Once the candidate aqueous and semi-aqueous chemistries were identified, the Surface Cleaning Lab performed a series of tests examining their cleaning efficacy. These tests were performed on two Raffi and Swanson coatings, a hydrophobic product (base-coat #51144) and a hydrophilic product (top coat #51072). The following sections review each of the six lab tests. Complete results for each test are presented in Appendix A¹.

4.1.1 Lab Test 1: Screening Chemistries

February 5, 1996

The Surface Cleaning Lab conducted tests on eight chemistries -- four water-based cleaners and four semi-aqueous cleaners (terpenes). The tests were conducted on metal samples covered with hydrophilic and hydrophobic coatings. Samples were placed into beakers with the solution and agitated with a stir rod for 30 minutes at 160°F.

Test results showed that two aqueous cleaners (69MC and PolySpray 790P) and two semi-aqueous cleaners (HTF 85B and EP 921) had the highest performance (see Table 1). All chemistries had a more difficult time removing the base-coat #51144 verses the #51072. Since base-coat #51144 was more difficult to remove, all subsequent tests were conducted only on this urethane product

¹ Material safety data sheets for all chemistries are on file at the TURI Surface Cleaning Lab.

Table 1: Screening Results

| | Chemistry | Percent Removal | Standard Deviation | Qualitative Results (#51072)* | Qualitative Results (#51144)* | Recommendation |
|------------------------------|-------------------|-----------------|--------------------|-------------------------------|-------------------------------|------------------|
| Aqueous-based chemistry | PolySpray Jet 790 | 44.82 | 2.45 | 4 | 6 | optimize further |
| | PolySpray Jet 790 | 37.04 | 31.71 | 6 | 7 | do not test more |
| | PolySpray Jet 790 | 41.51 | 30.19 | 6 | 8 | do not test more |
| | PolySpray 69 MC | 50.79 | 6.29 | 1 | 4 | optimize further |
| Semi-aqueous based chemistry | Cleppo 288-D | 48.54 | 36.42 | 8 | 3 | do not test more |
| | Safe Strip | 94.81 | 3.43 | 4 | 4 | do not test more |
| | HTF 85B | 98.61 | 2.09 | 2 | 2 | optimize further |
| | EP 921 | 99.84 | .08 | 3 | 1 | optimize further |

*based on a comparative rating of 1 to 8 with 1 being the best.

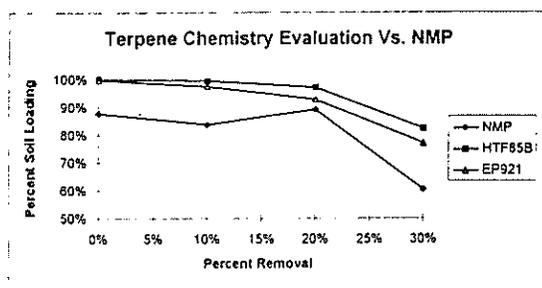
4.1.2 Lab Test 2: Semi-aqueous and NMP @ Different Soil Loadings

February 20, 1996

The next test examined the two leading semi-aqueous chemistries (based on Lab Test 1 results) and N-methyl pyrrolidone (the current Raffi & Swanson urethane cleaning chemical). In this test, the cleaners were loaded with the urethane at different levels. Loading the cleaners was done to determine at what soil levels cleaning efficacy begins to decline. Lab Test 2 was conducted on metal samples covered with #51144 coatings. Samples were placed into beakers with solution and agitated with stir rod for 30 minutes at 160°F.

Test results showed that both semi-aqueous chemistries out-performed NMP at all percent soil loadings (see Figure 1). The HTF-85B performed better than EP921 and N-methyl pyrrolidone at all loading levels.

Figure 1: Soil Loading Results



| Loading | NMP | HTF85B | EP921 |
|---------|-----|--------|-------|
| 0% | 88% | 100% | 100% |
| 10% | 84% | 99% | 98% |
| 20% | 89% | 97% | 93% |
| 30% | 60% | 82% | 77% |

4.1.3 Lab Test 3: Aqueous Cleaner Evaluation

March 4, 1996

Lab Test 3 took a closer look at the performance of six aqueous cleaners. Since aqueous cleaners use a surfactant process -- as opposed to NMP and semi-aqueous that dissolve the urethane coating -- the cleaners were evaluated by how easily the urethane coating peeled off following immersion. The tests were conducted on metal samples covered with #51144 coating. Samples were placed into beakers with solution at different concentrations and agitated with stir rod for 30 minutes.

One aqueous cleaner (PolySpray 69MC) did an excellent job lifting off the urethane coating when compared with the other cleaners. Urethanes were peeled off easily at concentrations as low as 20%. (see Table 2 - test results indicated by POE (Peeled Off Easily) and DNP (Did Not Peel)).

Table 2: Aqueous Cleaner Test Results

| Conc. | Aluminex 5781 | Aluminex 4874 | PolySpray P | 69MC | PolySpray XS | PolySpray C | Aluminex 5834 |
|-------|---------------|---------------|-------------|------|--------------|-------------|---------------|
| 50% | POE | | POE | POE | POE | POE | DNP |
| 40% | DNP | | DNP | POE | DNP | DNP | |
| 30% | | | | POE | | | |
| 20% | | | | POE | | | |
| 10% | | | | DNP | | | |

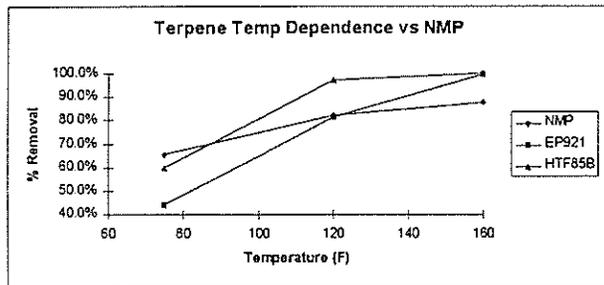
4.1.4 Lab Test 4: Semi-aqueous and NMP Temperature Dependence

March 8, 1996

This test examined the cleaning efficacy of two semi-aqueous chemistries and NMP at different temperatures. Raffi and Swanson preferred a process that would clean at lower temperatures since lower temperature processes present fewer hazards and use less energy. As in prior tests, Lab Test 4 was conducted on metal samples covered with #51144 coating. Samples were placed into beakers with solution at different temperatures and agitated with stir rod for 30 minutes.

Tests were conducted at 75°F, 120°F, and 160°F. Cleaning performance improved at elevated temperatures for all materials although performance began to plateau for two materials (HT-85B and NMP) at 120°F (see Figure 2).

Figure 2: Temperature Test Results



| Temperature | NMP | EP921 | HTF85B |
|-------------|-------|-------|--------|
| 75 | 65.6% | 44.0% | 59.7% |
| 120 | 82.3% | 81.2% | 97.4% |
| 160 | 87.6% | 99.6% | 99.9% |

4.1.5 Lab Test 5: 69MC Rinsability Study

April 4, 1996

This test was performed to determine what percentage of drag-out of 69MC could be tolerated in deionized (DI) rinse water. Investigators then extrapolated the number of times rinse water could be reused before leaving visible residue on metal coupons to the number of times the rinse could be reused in a urethane reactor. To perform the test, different concentrations of 69MC in DI water (0% to 5%) were rinsed on 304 stainless steel coupons. The coupons were inspected with a microscope and pictures were taken of rinsing spots.

DI rinse water with 1% 69MC showed no spotting. The 2% 69MC rinse showed good results with exception of a single streak. Rinse water with 3% 69 MC, 4% 69 MC and 5% 69 MC all exhibited heavy streaking. Using 2% as the maximum permissible amount of 69 MC in rinse water, a fifteen gallon DI rinse could contain no more than 0.3 gallons of concentrated 69 MC. Therefore the number of rinses that could be reused (assuming a 15 gallon charge) equals:

$$No. Rinses = \frac{0.3}{D \cdot x}$$

where: D = amount of 69MC residue left in tank after cleaning
x = volume fraction of 69 MC in cleaning solution

Lab Test 6: 69MC Temperature and Foam Study

April 8, 1996

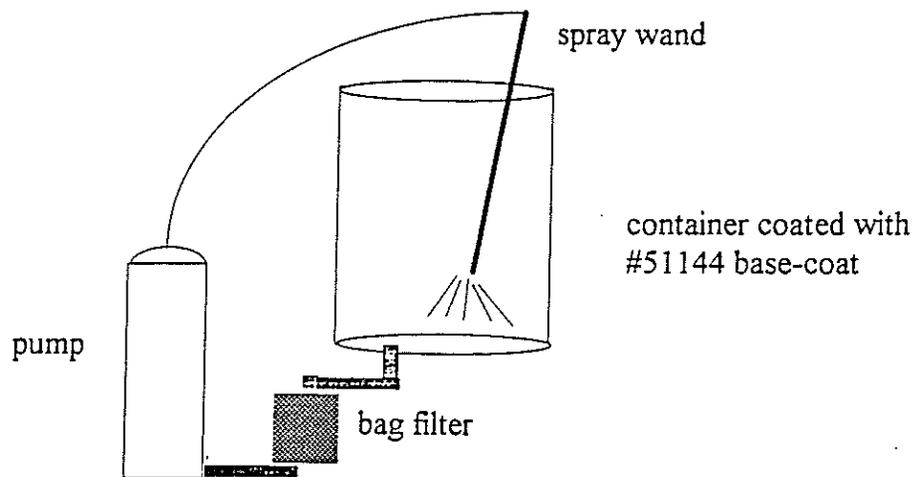
This test was performed to determine the effect of temperature on 69 MC cleaning and to determine the amount of foam generated at various temperatures. Coupons were coated using the same methods used in previous experiments. Cleaning was performed in the laboratory's Miele pressure washer at temperatures 60°F, 120°F, and 160°F with a 15% solution of 69 MC.

Although the process did not remove all of the base-coat #51144, some of the urethane was removed and the rest of the coating could be easily peeled of the coupon. The 69 MC posed no foaming problems at all temperatures.

Bench Scale Testing

The Surface Cleaning Lab tests indicated that one aqueous chemistry (PolySpray 69 MC) and one semi-aqueous chemistry (HTF 85B) had the greatest potential as a substitute for N-methyl pyrrolidone. Therefore, following the TURI Surface Cleaning Lab tests, Raffi and Swanson conducted in-plant bench scale tests on both chemistries. To perform these tests, the sidewalls of a ~50 gal. stainless steel container was coated with base-coat #51444. The drum was then allowed to dry for a specified time period at ambient temperatures -- rendering the urethane semi-cured. Raffi and Swanson's pilot reactor was used to heat the cleaning solutions to ~180°F. A recirculating pump delivered cleaning solution to a nozzle inserted in the top of the container. Cleaning solution was sprayed out of the nozzle at ~400 psi and piped from the bottom of the container through a filter bag back into the pump. Figure 3 depicts the bench scale set-up.

Figure 3: Bench Testing Set-up



4.2.1 Bench Test 1: 69MC

May 31, 1996

This test was performed to examine the cleaning efficacy of 69 MC at a bench scale level. A stainless container was coated with #51144 urethane, allowed to sit 4 hours, and cleaned for 17 minutes using 10 gal of 100% 69 MC heated to 200 F.

The 69 MC removed the urethane coating wherever the spray directly impinged the container walls and bottom. Where there was no impingement, the coating swelled but was not removed. The cloth filter bags filled up with material that had the consistency of melted marshmallows. The filter bag filled during the test requiring the test to be aborted. Results of this test were poor in two respects. First, where there was only indirect impingement, the 69 MC did not remove coating. Second, the filter bag collected a significant amount of undissolved coating -- making filtering of 69 MC a difficult task.

4.2.2 Bench Test 2: HTF 85B versus NMP

May 31, 1996

This test was performed to compare the cleaning efficacy of HTF 85B and NMP at a bench scale level. Stainless containers was coated with #51144 urethane, allowed to sit 4 hours, and cleaned for roughly 15 minutes using 15 gal of 100% HTF 85 B and NMP heated to 200 F.

Both the HTF 85B and the NMP dissolved the coating in areas where there was direct impingement and in areas where there was no direct impingement. Both chemistries dissolved the urethane base-coat #51144. HTF 85B appeared to remove the urethane somewhat better than NMP. HTF 85B's odor was not as strong as that of NMP's.

4.3 Summary of Lab and Bench Test Results

Lab tests at the TURI Surface Cleaning Lab found that PolySpray 69 MC was the best aqueous cleaner of the seven chemistries test. However, when tests were performed at the bench scale, 69 MC results were not acceptable. The 69MC cleaner did not remove the urethane unless it directly impinged the container walls. Since there are numerous recessed areas inside the urethane reactors, cleaning with 69MC would leave water-entrapped residues. Since hydroxyl molecules terminate the polymer reaction, these residues would ruin subsequent batches of urethane product. It is possible that expensive upgrades to Raffi and Swanson's high-pressure washing system could remove all of the urethane coating but it is equally likely that no matter how thorough a power wash, some residue would be left on difficult to clean areas such as the underside of the mixing blades or in recessed areas around the shaft collar. The 69 MC results were also problematic from the standpoint of filtering and separation. Cleaning with 69 MC would require a new settling and separation system to remove the urethane marshmallow-like residue cleaned off the reactor walls.

Lab tests found that HTF 85B cleaned Raffi and Swanson's base-coat urethane better than all of the aqueous cleaners, better than the other semi-aqueous, and better than NMP. HTF 85B contains terpene organic solvents and dibasic ester. HTF 85B has a high vapor pressure (<0.3654 mmHg), has low VOC potential, and is less toxic than NMP.

4.4 Economic Evaluation

An cost comparison of NMP versus HTF 85B was performed to examine how switching to HTF 85B would affect operating costs. Annual purchase, disposal and recycling costs and amounts were used. The comparison shows that NMP has significantly lower operation costs than HTF 85B. NMP can be recycled twice before being too contaminated for further recycling at which time it is disposed of as hazardous waste. HTF 85B is not recyclable.

Table 3: Economic Comparison

| | NMP | | | HTF 85B | | |
|--------------|----------|---------------------|-----------------|----------|---------------------|-----------------|
| | Cost/lb. | Pounds | Cost | Cost/lb. | No. lb. | Cost |
| Purchase | 2.1 | 16,500 | \$34,650 | 2.12 | 30,493 ^a | \$64,645 |
| Recycle | 0.419 | 13,993 | \$5,858 | | 0 | \$0. |
| Disposal | 0.114 | 21,450 ^b | \$2,438 | 0.114 | 39,641 ^b | \$4,505 |
| Total | | | \$43,946 | | | \$69,150 |

a. The estimated amount of HTF 85B purchased per year is equal to the amount of NMP purchased plus the amount of NMP recycled.

b. The estimated amount of NMP-containing and HTF 85B-containing material disposed of is equal to 1.3 times the amount purchased -- this assumes that both cleaning chemistries become loaded with 30% solids over the course of their use.

Qualitatively, HTF 85B is a better cleaning material than NMP, is less hazardous to workers, and smells better. However these qualitative factors do not outweigh the increased operation costs that would occur with a switch from NMP to HTF 85B.

Raffi and Swanson has not discontinued its efforts to find suitable substitutes for NMP. At the time this report was written the company had contacted vendors selling a recyclable semi-aqueous chemistry. Raffi and Swanson hopes that through continued testing and evaluation, the company will identify a less-hazardous material for use in its urethane reactor vessel cleaning operations.

4.5 Other Improvements to Urethane Cleaning System

In addition to the chemistry work described in the preceding sections, Raffi and Swanson made two equipment-related changes to improve the reactor cleaning process. First, Raffi and Swanson performed tests to evaluate whether surface polishing of the urethane reactor inside surfaces would make cleaning easier. The inside surface of the reactors was extremely rough and covered with baked-on coatings from years of urethane processing. The rougher the surface, the higher the coefficient of friction and the more difficult it is to clean the reactor sidewalls. The tests were performed on three different surface finishes (unpolished coupons, mirror #4 finish, and mirror #8 finish) in the same bench set-up depicted in Figure 3. This set-up was used to simulate conditions in the company's 1,500 gallon reactors. The tests showed that polishing the reactor to a mirror #8 finish considerably improved the rate at which uncured urethanes were removed from the reactor sidewalls. Based on these results, Raffi and Swanson hired a contractor to mechanically

polish one of the company's reactor vessels. The polished reactor is easier to clean. Raffi and Swanson has seen improvements in product quality from the reactor as well.

The second equipment change was the installation of a pressure gauge on the high-pressure pump used to deliver wash solvent (~ 400 psi) to the urethane vessels. By installing a pressure gauge, Raffi and Swanson can monitor the pump's performance and determine when pump performance begins to degrade. By properly maintaining the pump, Raffi and Swanson can make sure the process delivers sufficient pressure to blast residue off the reactor side-walls.

5. Reducing Plant Cleaner Use

The second grant-related effort was work by the TURA and SWEATT teams to reduce the amount of plant cleaner used to clean production containers. Plant cleaner is solvent mixture composed of toluene, methyl ethyl ketone, ethyl acetate, methyl isobutyl ketone, and xylene. Plant cleaner is used in centralized container cleaning operation. The insides of the containers, which range in size from 5 gal to 200 gal, become covered with coatings during mixing and blending operations. The containers are brought by workers to the container cleaning operation where they are cleaned manually or with the use of an automated unit.

The TURA and SWEATT teams' first efforts were to track the use of cleaning solvent throughout the plant (described in detail in section 3). Tracking solvent use and discharge *by department* gave the TURA and SWEATT teams the ability to measure improvement from an objective baseline.

The SWEATT team was instrumental in identifying opportunities to reduce wash solvent use. Since the team was staffed by representatives from Raffi and Swanson's five main production departments, the team was able to review each area for opportunities. Highlights of changes made as a result of SWEATT efforts follow in sections 5.1, 5.2 and 5.3.

5.1 Changes in Procedures

| | |
|-----------------------|---|
| Reuse Plant Cleaner | Workers switched from using clean plant cleaner to keep the bottom of mixing containers moist to using dirty plant cleaner. The small amount of dirty plant cleaner (~1 gal) keeps the coating from hardening. Hardened coatings are more difficult to clean than uncured ones. Plant cleaner is also reused in this operation as many times as possible to reduce the amount of dirty plant cleaner generated. Production departments such as the grind room are saving solvent washes in drums for reincorporation into subsequent production runs. |
| Revise Grind Formulas | SWEATT carried out a study comparing waste generation and production rates of ball mills with horizontal mills. The study showed that horizontal mills generated far less waste per batch, processed material nearly an order of magnitude faster, and improved product quality. Recommendations were developed to begin reformulating high-volume products from the ball mills to the premier mills. |
| Restrict Plant | In the past, workers could get their own plant cleaner. Delivery of drums |

Cleaner Delivery of plant cleaner now requires sign-off by department personnel. As a result, plant cleaner is used with greater discrimination.

5.2 Data Collection Changes

Survey Plant Cleaner Usage and Discharge SWEATT instituted a survey to monitor all wash solvent use and discharge. Monitoring made it possible to track reduction trends and provided the team with information on the major operations in the plant using plant cleaner.

Container Survey SWEATT instituted a survey to determine the size and number of container being cleaned. The survey highlighted the production department generating the greatest number of containers needing cleaning -- prompting SWEATT to concentrate its TUR efforts in those areas.

5.3 Equipment Modifications

Modify Containers and Procedures to Eliminate the Need for Solvent Cleaning A SWEATT team member developed a packing method that reduced the number of containers needing cleaning by roughly 50%. Under the old process, after a 100 or 200 gal job was packaged, the dirty container was sent to the solvent washing area where it was cleaned using plant cleaner.

Under the new process, the water-phase part of the coating was poured first into the batch by running down the sidewalls of the container. Coating the container sidewalls with the water-phase, made cleaning the vessel with water possible. To increase the effectiveness of water-cleaning, a small nozzle on flexible hose spraying ~0.25 gal per minute was installed and used to spray-down the sidewalls of containers during the packaging operation. Raffi and Swanson also designated that specific containers be used in the operation. These containers were polished to make them easier to clean. The valves on these containers were relocated and switched from gate valves to molasses valves. The change made it much easier and quicker to clean the valves.

Add Site Glass to Automatic Unit A SWEATT team member recommended installing a site glass to give greater control over the use and discharge of solvent from the automatic solvent washing system used to clean 100 gal and 200 gal containers. The site glass makes it possible to see when new solvent needs to be added to the system and when dirty plant cleaner should be replaced with clean plant cleaner.

Eliminate Direct Plant Cleaner Pumping Raffi and Swanson made plumbing changes so that plant cleaner can no longer be pumped directly from a solvent manifold into a drum or cleaning operation on the plant floor. Workers now sign-out drums. The sign-out procedure helps track usage and reinforces SWEATT's goal of reducing plant cleaner use wherever possible.

5.4 Summary of Plant Cleaner Reductions

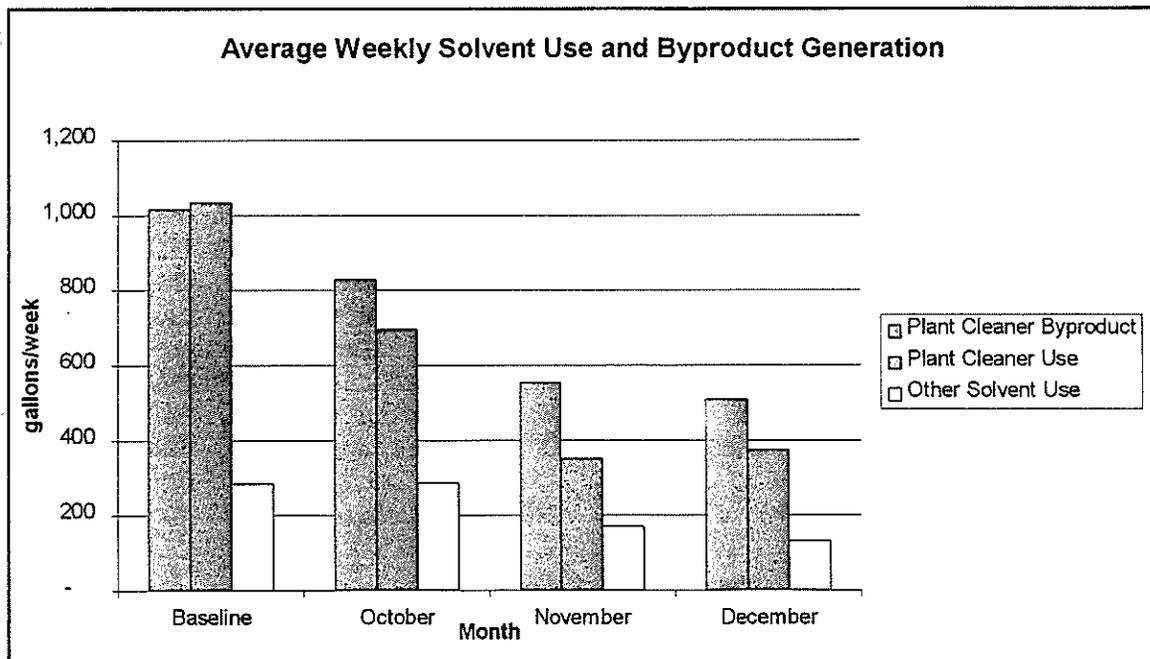
Table 4 presents plant chemical reduction data. Before the start of the project, Raffi and Swanson decided to track all solvents used in cleaning. This gave the company a good baseline to track progress. As Table 4 indicates, the changes outlined previously have yielded considerable annual reductions in both use and byproduct.

Table 4: Annual Plant Cleaner

| Chemical | Baseline Use/Emissions (lb.) | Use/Emissions Reduction (lb.) | Baseline Byproduct (lb.) | Byproduct Reduction (lb.) |
|------------------------|------------------------------|-------------------------------|--------------------------|---------------------------|
| Toluene | 47,736 | 23,868 | 205,889 | 102,945 |
| Methyl Ethyl Ketone | 27,769 | 13,885 | 116,270 | 58,135 |
| Ethyl Acetate | 11,108 | 5,554 | 51,697 | 25,848 |
| Xylene | 3,333 | 1,666 | 14,342 | 7,171 |
| Methyl Isobutyl Ketone | 3,333 | 1,666 | 13,852 | 6,926 |
| <i>Total</i> | <i>93,279</i> | <i>46,639</i> | <i>402,050</i> | <i>201,025</i> |

Figure 4 shows this progress has occurred steadily since SWEATT and the TURA Team began working on reduction projects in September 1995.

Figure 4: Average Weekly Solvent Use and Byproduct Generation



5.5 Financial Evaluation

Table 5 presents a rough financial analysis of the changes implemented under the TURI grant to date. Only costs that have a measurable effect on cash flow have been included in the analysis.

Table 5: Financial Analysis

| RCRA Waste Savings | <i>Change/ Wk (gal)</i> | <i>Annual Change (gal)</i> | <i>% Reject</i> | <i>cost/gal</i> | <i>RCRA Savings</i> |
|---------------------|-----------------------------|--------------------------------|---------------------------------|---|--------------------------|
| | 481 | 24,050 | 17% | 0.75 | \$ 3,066 |
| Still Elec. Savings | <i>Still hr./mo.</i> | <i>Still kW/hr</i> | <i>Reduced Use of Still</i> | <i>Electricity Rate (\$/kW- hr)</i> | <i>Elec. Savings</i> |
| | 107 | 78.5 | 50% | 0.04811 | \$ 2,424 |
| Still Labor Savings | | | <i>labor cost/yr.</i> | <i>% still red</i> | <i>Labor savings</i> |
| | | | 25,000 | 50% | \$ 12,500 |
| TOTAL | | | | | \$ 17,991 |

5.6 Future Efforts

With more than 50% of solvent use reduced in the container cleaning operation, Raffi and Swanson has turned its sights on further reductions. Raffi and Swanson prepared and mailed requests for proposal to over 10 vendors of alternative cleaning systems. These vendor systems include carbon dioxide blasting, automated terpene and water-based cleaning, plastic media cleaning, and high-pressure water blasting. Raffi and Swanson received bids from several firms and is in the process of evaluating the technologies.

APPENDIX

TURI
Surface Cleaning Laboratory
Experimental Log

DATE OF TEST: February 2, 1996

PURPOSE OF TEST: Phase I preliminary 'lift' study of solvent action on two Durane coatings

SUBSTRATE MATERIAL: 304 stainless steel coupons

CONTAMINANTS: Durane base coatings #51144 and #51072

CONTAMINATING PROCESS: Coatings applied by swab

ANALYTICAL METHOD: Visual inspection of coating lift after cleaning

LABORATORY PROCEDURES: Five cleaning chemistries were tested at concentrated (i.e., full-strength) levels. Ten 2" x 4" ss coupons were contaminated, five with Basecoat #51072 and five with Basecoat #51144. Contaminated coupons were allowed to set for 15 minutes followed by application of the cleaners for a contact/reaction time of 30 minutes.

RESULTS: Visual observations were made and recorded on the chemistries' effectiveness. Cleaners were rated on a scale of 1 to 5 for each coating, with a rank of 1 being the best performer.

| Chemistry | #51072 Coating | #51144 Coating | Notes and Observations |
|-------------------------|----------------|----------------|----------------------------|
| Terpene Tech 85B | 2 | 4 | Dissolving mechanism |
| U.S. Polychem 69 MC | 3 | 1 | Lift & Dissolve mechanisms |
| Frederick Gumm 228-D | 1 | 3 | Lift & Dissolve mechanisms |
| Sentry Chem. Safe Strip | 4 | 2 | Dissolving mechanism |
| Inland Tech EP-921 | 4 | 5 | Dissolving mechanism |

CONCLUSIONS: In this brief test, the U.S. Polychem and the Frederick Gumm chemicals performed best. More testing with these and other chemistries will be required.

DATE OF TEST: February 5, 1996

PURPOSE OF TEST: Completion of Phase I 'lift' study in preparation for in-depth testing

SUBSTRATE MATERIAL: 304 stainless steel coupons

CONTAMINANTS: Durane base coatings #51072 and #51144

CONTAMINATING PROCESS: Coatings applied by swab and allowed to cure overnight

ANALYTICAL METHODS: Gravimetric and scrape tests after cleaning

LABORATORY PROCEDURES: Twenty-four 2" x 4" ss coupons were precleaned in a 20% solution of ND-Supreme in a Crest 40 KHz ultrasonic console for 20 minutes at 140°F followed by rinsing in DI (deionized) water for 2 minutes at 120°F. The coupons were then placed under ambient air knives for 2 minutes, dried for 30 minutes in a convection oven and allowed to cool for 30 minutes. After cooling, the coupons were measured for a clean weight on an analytical balance.

Half of each coupon was contaminated with Basecoat #51144 and the other half with Basecoat #51072. After overnight curing, the coupons were weighed for a contaminated weight.

Eight cleaning chemistries were tested at appropriate operating conditions for time, same-source agitation, concentration and temperature. All cleaning trials were performed for 30 minutes at 160°F (+/-5°F) in 600 mL beakers with stirbar agitation. The stirbar setting was maintained constant for each cleaner tested. Water-based chemistries were diluted to 50% while the rest of the chemistries were used at full-strength. After cleaning, the coupons were immersed in tap water for 2 minutes at 100°F (municipal water rinsing is not recommended for some cleaning chemistries; rinsing will be more carefully evaluated in Phases II and III). After rinsing, the coupons were dried at 140°F in a convection oven for 60 minutes, allowed to cool overnight and weighed.

Four of the chemistries, the U.S. Polychemical formulations, showed a lifting action on the coatings. To evaluate this mechanism for coating removal, a scrape test was performed on the coupons' remaining basecoat after cleaning. The other four cleaners use a dissolving mechanism on the coatings. Gravimetric analysis was successfully employed instead to determine the cleaning efficiencies of these chemistries.

RESULTS:

U.S. Polychem Polyspray Jet 790P (50% solution) - #51072 Coating totally removed on all 3 coupons. Remaining #51144 Coating scraped and pulled off with little effort.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 4 | 60.4205 | 61.4891 | 60.9933 | 0.4958 | 46.40% |
| 5 | 59.7666 | 60.8911 | 60.4188 | 0.4723 | 42.00% |
| 6 | 60.484 | 61.4893 | 61.0261 | 0.4632 | 46.08% |
| Average | | | | | 44.82% |
| StDev. | | | | | 2.45% |

U.S. Polychem Polyspray Jet 790 XS (50% solution) - Effective on the #51072 Coating (except coupon #7). Scrape test showed that the #51144 Coating could be pulled off with quite a bit of effort.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 7 | 59.9058 | 61.0715 | 61.042 | 0.0295 | 2.53% |
| 8 | 60.11 | 61.2037 | 60.7258 | 0.4779 | 43.70% |
| 9 | 60.6564 | 61.5477 | 60.9692 | 0.5785 | 64.91% |
| Average | | | | | 37.04% |
| StDev. | | | | | 31.71% |

U.S. Polychem Polyspray Jet 790C (50% solution) - Not effective on the #51144 Coating.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 10 | 60.0143 | 61.09 | 60.4747 | 0.6153 | 57.20% |
| 11 | 60.7083 | 61.8142 | 61.1438 | 0.6704 | 60.62% |
| 12 | 60.278 | 61.3615 | 61.2888 | 0.0727 | 6.71% |
| Average | | | | | 41.51% |
| StDev. | | | | | 30.19% |

Frederick Gumm Cleppo 288-D (50% solution) - Effective on the #51144 Coating but a longer cleaning time will be necessary to remove the #51072 Coating.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 13 | 60.3352 | 61.8657 | 61.2669 | 0.5988 | 39.12% |
| 14 | 60.5393 | 61.6671 | 60.6663 | 1.0008 | 88.74% |
| 15 | 60.7022 | 62.1118 | 61.8616 | 0.2502 | 17.75% |
| Average | | | | | 48.54% |
| StDev. | | | | | 36.42% |

Ecolink Safe Strip (non-diluted) - Excellent removal of the #51072 Coating but a longer cleaning time will be necessary to remove the #51144 Coating.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 16 | 60.5761 | 61.6049 | 60.656 | 0.9489 | 92.23% |
| 17 | 60.3589 | 61.4997 | 60.3737 | 1.126 | 98.70% |
| 18 | 59.9049 | 61.1859 | 59.9882 | 1.1977 | 93.50% |
| Average | | | | | 94.81% |
| StDev. | | | | | 3.43% |

U.S. Polychem 69MC(non-diluted) - Lifts off the #51072 Coating in 10 minutes. Scrape test showed that the remaining #51144 Coating could be removed easily after cleaning.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 19 | 60.2195 | 61.3182 | 60.8249 | 0.4933 | 44.90% |
| 20 | 60.4775 | 61.4268 | 60.9516 | 0.4752 | 50.06% |
| 21 | 60.2207 | 61.2342 | 60.6523 | 0.5819 | 57.41% |
| Average | | | | | 50.79% |
| StDev. | | | | | 6.29% |

Terpene Technologies HTF-85B (non-diluted) -Almost as effective as EP-921(below); lower %-Coating removal on coupon #22 due to high contaminant loading.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 22 | 60.0098 | 62.058 | 60.0875 | 1.9705 | 96.21% |
| 23 | 60.106 | 61.169 | 60.1068 | 1.0622 | 99.92% |
| 24 | 60.2287 | 61.1451 | 60.2314 | 0.9137 | 99.71% |
| Average | | | | | 98.61% |
| StDev. | | | | | 2.09% |

Inland Technologies EP-921(non-diluted) - Most effective chemistry tested. Total Coating removal in approx. 20 minutes.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 25 | 60.5203 | 61.7187 | 60.5213 | 1.1974 | 99.92% |
| 26 | 59.3011 | 60.1552 | 59.3024 | 0.8528 | 99.85% |
| 27 | 60.242 | 61.0863 | 60.244 | 0.8423 | 99.76% |
| Average | | | | | 99.84% |
| StDev. | | | | | 0.08% |

CONCLUSIONS: The Safestrip, Cleppo-228C, PolySpray Jet 790C & 790XS were ineffective in this coating removal application and will not be considered for further testing. The 69MC and the Polyspray Jet 790P will need longer cleaning cycles to remove all of the urethane.

The EP-921 and HTF-85B were excellent performers. Two concerns remain: the low flash point of EP-921 (146°F) and rinsing configurations.

DATE OF TEST: February 20, 1996

PURPOSE OF TEST: Phase II determination the soil loading characteristics of cleaners EP-921 and HTF-85B, as compared to NMP (n-methyl-2-pyrrolidone) currently in use.

SUBSTRATE MATERIAL: 304 stainless steel coupons

CONTAMINANT: Durane base coating #51144

CONTAMINATING PROCESS: Coating applied by swab and allowed to cure overnight

ANALYTICAL METHOD: Gravimetric analysis after cleaning

LABORATORY PROCEDURES: Five coupons in each chemistry at each percentage of soil loading, described below, were cleaned. By observing how efficient the chemicals are at various levels of dissolved urethanes, the best performing terpene (i.e., semi-aqueous) cleaner can be ascertained. The soil loading will be done as a weight percentage of Basecoat #51144 to the total weight of contaminated cleaning solution. Soil loadings were increased in increments of 10% from 0% to 50% for a total of 30 coupons to be cleaned for each chemical.

Previous testing had determined that Basecoat #51144 was more difficult to remove so soil loading tests were performed with this coating. All coupons were contaminated in an identical fashion to the Phase I testing.

Three 400 mL beakers were filled with 350 mL of each chemical. Cleaning was conducted at 160°F for 30 minutes with stirbar agitation. Rinsing commenced with a 60-second tap water rinse at 130°F followed by a brief acetone rinse (to eliminate residual water/cleaner prior to gravimetric analysis). The coupons were dried under a UV light for 10 minutes and then allowed to cool overnight.

To determine the amount of Basecoat #51144 needed to achieve a particular percentage, the specific gravities of the cleaning solutions were obtained from Material Safety Data Sheets (MSDSs): NMP = 1.025, EP-921 = 0.9800, HTF-85B = 0.9932. The chart below shows the grams of Basecoat #51144 needed to be added to each chemistry to achieve a specified loading:

| Cleaner | 10% | 20% | 30% | Concentrations |
|---------|--------|--------|---------|----------------|
| NMP | 40.00g | 89.69g | 153.75g | |
| EP-921 | 38.11g | 85.75g | 147.00g | |
| HTF-85B | 38.86g | 86.91g | 148.98g | |

The amount of urethane removed during cleaning was taken into consideration when increasing the soil loading. All urethane added to increase soil loading was uncured to reduce the time taken to dissolve.

It was assumed that there were no evaporative or dragout losses with respect to chemical volume. Therefore, a solvent volume of 350 mL was used for all soil loading calculations.

RESULTS:

NMP - No soil loading.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 1 | 60.42 | 61.2718 | 60.4887 | 0.7831 | 91.93% |
| 2 | 59.7665 | 60.4789 | 59.8633 | 0.6156 | 86.41% |
| 3 | 60.4839 | 61.3083 | 60.6515 | 0.6568 | 79.67% |
| 4 | 59.9056 | 60.663 | 59.9399 | 0.7231 | 95.47% |
| 5 | 60.0143 | 60.9037 | 60.1513 | 0.7524 | 84.60% |
| | | | | 3.531 | 87.62% |
| | | | | | 6.21% |

Inland Tech EP-921 - No soil loading.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 1 | 60.6562 | 61.3097 | 60.6566 | 0.6531 | 99.94% |
| 2 | 60.7081 | 61.3835 | 60.7082 | 0.6753 | 99.99% |
| 3 | 60.3344 | 61.3955 | 60.335 | 1.0605 | 99.94% |
| 4 | 60.7016 | 61.6404 | 60.717 | 0.9234 | 98.36% |
| 5 | 60.359 | 61.2314 | 60.3592 | 0.8722 | 99.98% |
| | | | | 4.1845 | 99.64% |
| | | | | | 0.72% |

Terpene Tech HTF-85B - No soil loading.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 1 | 59.9047 | 60.8014 | 59.9049 | 0.8965 | 99.98% |
| 2 | 60.2195 | 60.822 | 60.2197 | 0.6023 | 99.97% |
| 3 | 60.477 | 61.1145 | 60.4773 | 0.6372 | 99.95% |
| 4 | 60.2211 | 60.8589 | 60.2211 | 0.6378 | 100.00% |
| 5 | 60.0083 | 60.729 | 60.009 | 0.72 | 99.90% |
| | | | | 3.4938 | 99.96% |
| | | | | | 0.04% |

NMP - 10% soil loading.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 6 | 60.4197 | 61.2909 | 60.5188 | 0.7721 | 88.62% |
| 7 | 59.7668 | 60.5674 | 59.8698 | 0.6976 | 87.13% |
| 8 | 60.4838 | 61.4613 | 60.6831 | 0.7782 | 79.61% |
| 9 | 59.9053 | 60.9938 | 60.0204 | 0.9734 | 89.43% |
| 10 | 60.1101 | 60.9607 | 60.3303 | 0.6304 | 74.11% |
| | | | | 3.8517 | 83.78% |
| | | | | | 6.66% |

Inland Tech EF-921 - 10% soil loading.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 6 | 60.6562 | 61.3019 | 60.6708 | 0.6311 | 97.74% |
| 7 | 60.0137 | 60.6603 | 60.0164 | 0.6439 | 99.58% |
| 8 | 60.7082 | 61.6846 | 60.7346 | 0.95 | 97.30% |
| 9 | 60.278 | 61.0849 | 60.2823 | 0.8026 | 99.47% |
| 10 | 60.3344 | 61.4548 | 60.4056 | 1.0492 | 93.65% |
| | | | | 4.0768 | 97.55% |
| | | | | | 2.41% |

Terpene Tech HTF-85B - 10% soil loading.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 6 | 60.539 | 61.409 | 60.5428 | 0.8662 | 99.56% |
| 7 | 60.7019 | 61.8941 | 60.7044 | 1.1897 | 99.79% |
| 8 | 60.5755 | 61.2914 | 60.5788 | 0.7126 | 99.54% |
| 9 | 60.3591 | 61.2216 | 60.3643 | 0.8573 | 99.40% |
| 10 | 59.3007 | 60.1078 | 59.3208 | 0.787 | 97.51% |
| | | | | 4.4128 | 99.16% |
| | | | | | 0.93% |

NMP - 20% soil loading.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 11 | 60.6562 | 61.4064 | 60.6783 | 0.7281 | 97.05% |
| 12 | 60.0137 | 60.7303 | 60.0804 | 0.6499 | 90.69% |
| 13 | 60.7082 | 61.2288 | 60.7426 | 0.4862 | 93.39% |
| 14 | 60.278 | 60.9753 | 60.4015 | 0.5738 | 82.29% |
| 15 | 60.3344 | 61.1885 | 60.4885 | 0.7 | 81.96% |
| | | | | 3.138 | 89.08% |
| | | | | | 6.74% |

Inland Tech EP-921 - 20% soil loading.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 11 | 60.1058 | 60.6858 | 60.1275 | 0.5583 | 96.26% |
| 12 | 60.2287 | 61.0412 | 60.2767 | 0.7645 | 94.09% |
| 13 | 60.5201 | 61.2172 | 60.5646 | 0.6526 | 93.62% |
| 14 | 60.2423 | 60.969 | 60.3341 | 0.6349 | 87.37% |
| 15 | 59.9511 | 60.6721 | 60.011 | 0.6611 | 91.69% |
| | | | | 3.2714 | 92.61% |
| | | | | | 3.35% |

Terpene Tech HTF85B - 20% soil loading.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 11 | 59.9047 | 60.7873 | 59.9083 | 0.879 | 99.59% |
| 12 | 60.2195 | 60.921 | 60.2228 | 0.6982 | 99.53% |
| 13 | 60.477 | 61.0192 | 60.499 | 0.5202 | 95.94% |
| 14 | 60.2211 | 61.0052 | 60.2619 | 0.7433 | 94.80% |
| 15 | 60.0083 | 60.7387 | 60.0424 | 0.6963 | 95.33% |
| | | | | 3.537 | 97.04% |
| | | | | | 2.34% |

NMP - 30% soil loading.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 16 | 60.5386 | 61.7741 | 60.7543 | 1.0198 | 82.54% |
| 17 | 60.7016 | 61.9505 | 61.2649 | 0.6856 | 54.90% |
| 18 | 60.5752 | 61.7028 | 60.9753 | 0.7275 | 64.52% |
| 19 | 60.359 | 61.2255 | 60.7621 | 0.4634 | 53.48% |
| 20 | 59.9047 | 60.993 | 60.4955 | 0.4975 | 45.71% |
| | | | | 3.3938 | 60.23% |
| | | | | | 14.15% |

Inland Tech EP-921 - 30% soil loading.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 16 | 60.6562 | 61.6654 | 60.8742 | 0.7912 | 78.40% |
| 17 | 60.0137 | 61.105 | 60.2685 | 0.8365 | 76.65% |
| 18 | 60.7082 | 61.8099 | 60.9082 | 0.9017 | 81.85% |
| 19 | 60.278 | 61.4399 | 60.5666 | 0.8733 | 75.16% |
| 20 | 60.3344 | 61.2438 | 60.5804 | 0.6634 | 72.95% |
| | | | | 4.0661 | 77.00% |
| | | | | | 3.37% |

Terpene Tech HTF-85B - 30% soil loading.

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 16 | 60.4197 | 61.4393 | 60.5603 | 0.879 | 86.21% |
| 17 | 59.7668 | 60.663 | 59.9172 | 0.7458 | 83.22% |
| 18 | 60.4839 | 61.5339 | 60.5702 | 0.9637 | 91.78% |
| 19 | 60.1101 | 61.3927 | 60.4882 | 0.9045 | 70.52% |
| 20 | | | | | |
| | | | | 3.493 | 82.93% |
| | | | | | 9.00% |

Estimated dragout and evaporative losses were calculated by adding the original solvent volume to the volume of Basecoat #51144 added (density of 8.2 lb/gal) and subtracting the final volume of contaminated solvent. Losses were:

- EP-921 - 42 mL (7.8%)
- HTF-85B - 15 mL (2.63%)
- NMP - 3 mL (0.55%)

CONCLUSIONS: Data obtained reveals that HTF-85B is the best semi-aqueous solvent tested and should be trialed on a pilot scale level. Phase III testing will focus on determining the most effective cleaner from U.S. Polychem & General Chemical products.

DATE OF TEST: March 4, 1996

PURPOSE OF TEST: Phase III determination of the most effective aqueous cleaner for removing Durane coating

SUBSTRATE MATERIAL: 304 stainless steel coupons

CONTAMINANT: Durane base coating #51144

CONTAMINATING PROCESS: Coating applied by swab and allowed to cure overnight

ANALYTICAL METHOD: Peel test after cleaning

LABORATORY PROCEDURES: Seven chemistries were tested under identical conditions at 160°F for 30 minutes with stirbar agitation. Cleaner concentrations started at 50% with incremental decreases of 10 %.

Cleanliness levels achieved were determined by a peel test after cleaning. If an aqueous chemistry is effective, the remaining urethane should peel easily from the surface of the coupon. Chemistries tested include:

| Company | Tradename | Ph | Ingredients |
|------------------|----------------|------|---|
| U.S. Polychem | Polyspray C | 12.5 | 20-30% Potassium Hydroxide |
| U.S. Polychem | Polyspray P | 11.5 | 5-15% Tetra Potassium Hydroxide |
| U.S. Polychem | Polyspray XS | 11.5 | 2-8% Sodium Silicate 2-10% Tetra Potassium Pyrophosphate |
| U.S. Polychem | 69 MC | | 20-30% Amino Ethyl Alcohol |
| General Chemical | Aluminex 5761 | 13 | 1-2% Potassium Hydroxide 5-6% Sodium Metasilicate 2-4% Borax |
| General Chemical | Aluminex 5834 | 14 | 5% Nonyl Phenyl Ethoxylate <10% Gluconic Acid, Potassium Salt <5% Ethoxylated Alcohol <5% Octylphenoxypolyethoxyethanol <12% Silicates |
| General Chemical | Aquaclean 4784 | 12 | <2% Potassium Hydroxide <15% Ethylene Glycol Monophenyl Ether <15% Diethylene Glycol Monobutyl Ether <15% Diethylene Glycol <5% Triethanolamine |

RESULTS:

X - Indicates Basecoat #51144 peeled off coupon after cleaning.

| Conc. | Aluminex 5761 | Aluminex 5834 | Aquaclean 4874 | Polyspray P | 69MC | Polyspray XS | Polyspray C |
|-------|---------------|---------------|----------------|-------------|------|--------------|-------------|
| 50% | X | X | * | X | X | X | |
| 40% | | | * | X | X | | |
| 30% | | | | | X | | |
| 20% | | | | | X | | |
| 10% | | | | | | | |

*The Aquaclean worked on a dissolving mechanism (due to high glycol ether content) performed a 100% removal.

CONCLUSIONS: U.S. Polychem's 69MC outperformed all other aqueous cleaners tested to date.

DATE OF TEST: March 8, 1996

PURPOSE OF TEST: Determine the temperature dependence of two terpene cleaners, as compared to NMP

SUBSTRATE MATERIAL: 304 stainless steel coupons

CONTAMINANT: Durane base coating #51144

CONTAMINATING PROCESS: Coating applied by swab and allowed to cure overnight

ANALYTICAL METHOD: Gravimetric analysis after cleaning trials

LABORATORY PROCEDURES: Three coupons were cleaned in 500 mL of each solution at temperature levels of 75°F, 120°F and 160°F. Cleaning cycle duration was 30 minutes followed by an acetone rinse as previously described (all coupons were prepared and contaminated using the same methods in prior experiments).

Data for cleaning efficiency at 160°F was imported from Phase II.

RESULTS:

NMP - Ambient °F

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 19 | 60.2196 | 61.7264 | 60.8123 | 0.9141 | 60.67% |
| 20 | 60.4776 | 62.0680 | 61.0376 | 1.0304 | 64.79% |
| 27 | 60.2424 | 61.4824 | 60.5981 | 0.8843 | 71.31% |
| | | | | 2.8288 | 65.59% |
| | | | | | 5.37% |

Inland Tech EP-921- Ambient °F

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 11 | 60.7079 | 62.2201 | 61.5401 | 0.68 | 44.97% |
| 12 | 60.2775 | 61.6108 | 60.8862 | 0.7246 | 54.35% |
| 14 | 60.5386 | 62.3071 | 61.7279 | 0.5792 | 32.75% |
| | | | | 1.9838 | 44.02% |
| | | | | | 10.83% |

Terpene Tech HTF-85B - Ambient °F

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 6 | 60.4836 | 62.0351 | 61.0971 | 0.938 | 60.46% |
| 7 | 59.9048 | 61.4770 | 60.4385 | 1.0385 | 66.05% |
| 10 | 60.0137 | 61.6361 | 60.7846 | 0.8515 | 52.48% |
| | | | | 2.828 | 59.67% |
| | | | | | 6.82% |

NMP - 120°F

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 21 | 60.2213 | 61.9631 | 60.8190 | 1.1441 | 65.68% |
| 22 | 60.0081 | 61.6345 | 60.2803 | 1.3542 | 83.26% |
| 23 | 60.1057 | 61.5124 | 60.1346 | 1.3778 | 97.95% |
| | | | | 3.8761 | 82.30% |
| | | | | | 16.15% |

Terpene Tech. HTF-85B - 120°F

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 16 | 60.5752 | 62.4356 | 60.6127 | 1.8229 | 97.98% |
| 25 | 60.5201 | 62.1252 | 60.6090 | 1.5162 | 94.46% |
| 28 | 59.9508 | 61.3642 | 59.9562 | 1.408 | 99.62% |
| | | | | 4.7471 | 97.35% |
| | | | | | 2.64% |

Inland Tech EP-921 - 120°F

| sample # | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 8 | 60.1098 | 61.4076 | 60.3152 | 1.0924 | 84.17% |
| 24 | 60.2288 | 61.8040 | 60.6116 | 1.1924 | 75.70% |
| 26 | 59.3009 | 60.6873 | 59.5260 | 1.1613 | 83.76% |
| | | | | 3.4461 | 81.21% |
| | | | | | 4.78% |

DATE OF TEST: April 4, 1996

PURPOSE OF TEST: Ascertain the limits of cleaner drag-out tolerated in DI rinsing

SUBSTRATE MATERIAL: 304 stainless steel coupons

CONTAMINANTS: Durane base coating #51144

CONTAMINATING PROCESS: Coating applied by swab and allowed to cure overnight

ANALYTICAL METHODS: Microscopic and visual observation after cleaning

LABORATORY PROCEDURES: In the first part of this experiment, the rinsing effects of seven different solutions were evaluated:

- 1) 0% 69MC / 100% 16.4 M-ohm-cm DI water
- 2) 1% 69MC / 99% 16.4 M-ohm-cm DI water

- 3) 2% 69MC / 98% 16.4 M-ohm-cm DI water
- 4) 3% 69MC / 97% 16.4 M-ohm-cm DI water
- 5) 4% 69MC / 96% 16.4 M-ohm-cm DI water
- 6) 5% 69MC / 95% 16.4 M-ohm-cm DI water
- 7) 100% 69MC / 0% 16.4 M-ohm-cm DI water

Seven ss coupons were precleaned in the same manner as previous trials. One coupon was immersed in each rinsing solution for 10 minutes at ambient temperature. After immersion, the coupons were dried under a UV light for 10 minutes and allowed to cool for 10 minutes.

Since the non-volatile residues on the test coupons contained no organic material, FT-IR measurements were not taken. Instead, the coupons were inspected under a microscope and photomicrographs taken with a Polaroid Microcam at low magnification ranges.

In the second part of this experiment, a parts washer was used to establish cleaning efficiencies and cleaner foaming at different temperatures. Cleaning was performed in a Miele low-pressure, cabinet-style spray washer. The Miele recirculates 4.5 gallons of solution with a discharge pressure of 13 psi. The ss pieces were arranged in the washer so that the face of the sheets were directly facing the spray jets.

Cleaning was performed at 90°F, 120°F and 160°F. The parts were cleaned for 30 minutes at the desired temperatures and rinsed with tap water from a hand-held spray system at room temperature. In addition to cleanliness determinations by visual observation, foam levels in the Miele were also noted for each temperature.

RESULTS: In part one of this experiment, rinse spots appear as dark streaks on the coupons' surfaces. The 1% contamination of 69MC showed no streaks (the dark spots were polished spots on the coupons). The 2% rinse was acceptable except for the one streak on the coupon. All specimens >3% cleaner showed heavy streaking.

In part two of this experiment, the 69MC was low-foaming at all temperatures tested. The percent-coating removal at the different temperatures was as follows:

- 1) 90°F - The edges of the plates had some hardened urethane - 0% Removal
- 2) 120°F - All of the urethane had hardened and was starting to lift off - 5% Removal
- 3) 160°F - A few contaminated spots remained, but the majority of urethane was removed - 80% Removal

CONCLUSIONS: This data would seem to suggest that the maximum amount of 69MC tolerated would be 2%. For a 15 gal. DI rinse this would be 0.3 gal. concentrated 69MC. Number of rinses that could be accomplished with the same charge should be:

$$Rinses = \frac{.3}{D * x}$$

D = amount of Dragout cleaning solution from tank
 x = volume fraction of 69MC in cleaning solution

DATE OF TEST: April 8, 1996

PURPOSE OF TEST: Phase III to determine (1) the effect of temperature on the cleaning effectiveness of Cleaner 69 MC and (2) the amount of foam generated at various temperatures under pressure spray agitation

SUBSTRATE MATERIAL: 304 stainless steel samples

CONTAMINANT: Durane base coating #51144

CONTAMINATING PROCESS: Coating applied by swab and allowed to cure overnight

ANALYTICAL METHOD: Gravimetric analysis after cleaning

LABORATORY PROCEDURES: Three 10" x 16" 304 stainless steel pieces were contaminated with Basecoat #51144. Cleaning was performed in a Miele low-pressure, cabinet-style spray washer. The Miele recirculates 4.5 gallons of solution with a discharge pressure of 13 psi. The ss pieces were arranged in the washer so that the face of the sheets were directly facing the spray jets.

Trials were conducted at 90°F, 120°F and 160°F with solutions of 15% 69MC. This concentration was used due to operating problems with the Miele washer, otherwise a concentration of 30% would have been tested. The parts were cleaned for 30 minutes at the desired temperatures and rinsed with tap water from a hand-held spray system at room temperature. In addition to cleanliness determinations by gravimetric analysis, foam levels in the Miele were also noted for each temperature.

RESULTS:

| Temperature °F | clean mass (g) | mass with contamination (g) | mass after cleaning (g) | contaminant removed (g) | Percent Removal |
|----------------|----------------|-----------------------------|-------------------------|-------------------------|-----------------|
| 90 | 1121.2 | 1206.0 | 1200.6 | 5.4 | 6.37% |
| 120 | 1124.9 | 1190.7 | 1186.9 | 3.8 | 5.78% |
| 160 | 1452.2 | 1504.4 | 1483.9 | 20.5 | 39.27% |

Although the Miele did not remove all of Basecoat #51144, this could be attributed to the low cleaner concentration. The plates exhibited several clean spots and the urethane could be peeled off the surface.

U.S. Polychem's 69MC posed no foaming problems at all temperatures tested.

15A

Summary of Phase I testing for Raffi & Swanson-

Phase I evaluation of eight chemistries was completed on 2-8-96. The following results were obtained.

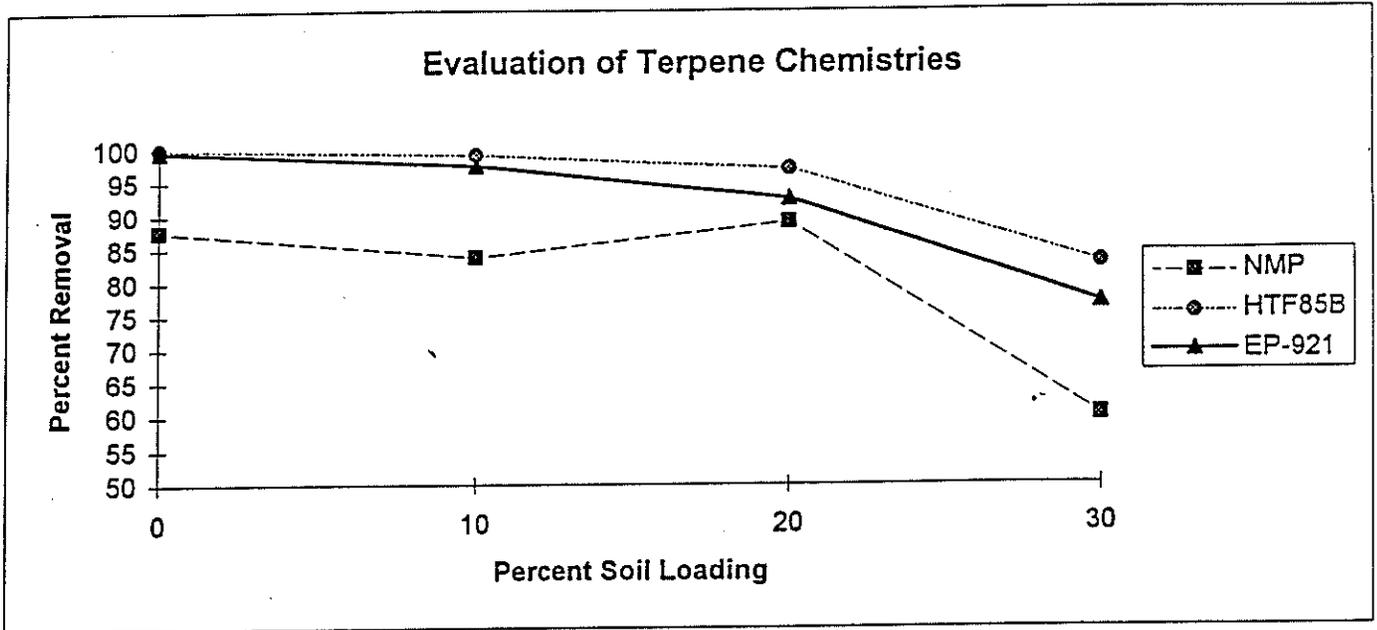
| Chemistry | % Removal | StdDev | Effectiveness on Coatings* | | Reccomendations for future testing |
|-----------------------------------|-----------|--------|----------------------------|--------|--|
| | | | #51072 | #51144 | |
| PolySpray Jet 790 P ^w | 44.82 | 2.45 | 4 | 6 | Optimize cleaning time and concentration |
| PolySpray Jet 790 XS ^w | 37.04 | 31.71 | 6 | 7 | Should not be tested further |
| PolySpray Jet 790 C ^w | 41.51 | 30.19 | 6 | 8 | Should not be tested further |
| 69 MC | 50.79 | 6.29 | 1 | 4 | Optimize cleaning time and evaluate soil loading |
| Cleppo 288-D ^w | 48.54 | 36.42 | 8 | 3 | Should not be tested further |
| Safe Strip | 94.81 | 3.43 | 4 | 4 | Should not be tested further |
| HTF 85B | 98.61 | 2.09 | 2 | 2 | Optimize Rinse and evaluate soil loading |
| EP 921 | 99.84 | .08 | 3 | 1 | Optimize Rinse and evaluate soil loading |

*based on a comparative rating of 1 to 8 with 1 being the best

^wIndicates a chemistry that was diluted with water to 50%

All chemistries had a tougher time removing the basecoat #51144 as compared to the #51072 (except for the Cleppo 288-D). Due to this we might want to think of just testing with the #51144 to expedite testing results.

Phase II Testing Results for Raffi & Swanson



| Loading | NMP Efficiency | | HTF85B Efficiency | | Inland Tech EP-921 | |
|---------|----------------|--------|-------------------|-------|--------------------|-------|
| | Ave. removal | STD | Ave. Removal | STD | Ave. Removal | STD |
| 0.00% | 87.62% | 6.21% | 99.96% | 0.04% | 99.64% | 0.72% |
| 10.00% | 83.78% | 6.66% | 99.16% | 0.93% | 97.47% | 2.56% |
| 20.00% | 89.08% | 6.74% | 97.04% | 2.34% | 92.61% | 3.25% |
| 30.00% | 60.23% | 14.15% | 82.93% | 9.00% | 77.00% | 3.37% |

Breakdown of Chemistries tested for Raffi & Swanson as of March 8, 1996:

| Company name | Tradename | Positive | Negative |
|----------------|----------------|---|--|
| Ecolink | Safe-Strip | | High VOC content (995 g/l). Not as effective as terpenes and 69MC |
| U.S. Polychem | Polyspray C | | Least effective of the Polyspray series. |
| U.S. Polychem | Polyspray P | Low environmental impact, could be effective with spray system | Will require a longer cleaning time than other chemistries and quite a bit of agitation |
| U.S. Polychem | Polyspray XS | | Not as effective as Polyspray P. |
| U.S. Polychem | 69MC | Performed excellent in lab testing. Filtration should be easy. Low environmental impact | Experimental chemical. |
| Frederick Gumm | Cleppo 228-D | | Highly caustic, had better results in lab with other chemistries |
| Terpene Tech. | HTF 85B | Outperformed NMP in lab tests, highest Solvency of all chemicals tested | Will have to be used at elevated temperature, still some questions about filtration Can also be made into a water based chemical. |
| Inland Tech. | EP-921 | Good solvency in lab tests. | Low flash point and needs to be solvent rinsed |
| ISP Tech | NMP | | Health, safety and regulatory concerns. |
| General Chem. | Aquaclean 4784 | Good Removal of Basecoat #51144 | Contains <45% Glycol Ethers (e-series), Would have problems in filtration |

17A

Chemical Properties of Chemistries tested for Raffi & Swanson:

| Company Name | Tradename | Classification | pH | Flashpoint | Listed Chemicals |
|----------------|----------------|--------------------|------|------------|--|
| Ecolink | Safe-Strip | NMP Based Solvent | N/A | 197 F | Gamma-Butyrolactone (96-48-0) N-Methylpyrrolidone (872-50-4) |
| U.S. Polychem | Polyspray C | Alkaline Aqueous | 12.5 | None | 20-30% Sodium Hydroxide(1310-58-3) |
| U.S. Polychem | Polyspray P | Alkaline Aqueous | 11.5 | None | 5-15% Tetra Potassium Diphosphate(7320-34-5) |
| U.S. Polychem | Polyspray XS | Alkaline Aqueous | 11.5 | None | 2-8% Sodium Silicate(10213-76-3) 2-10% Tetra Potassium Pyrophosphate(7320-34-5) |
| U.S. Polychem | 69MC | Alkaline Aqueous | 12 | None | 20-30% Amino Ethyl Alcohol (141-43-5) |
| Frederick Gumm | Cleppo 228-D | Caustic Aqueous | 14 | None | 13.5% Potassium Hydroxide (1310-58-3) 9% Ethylene Glycol Phenyl Ether(122-99-6) 1% Diethylene Glycol Phenyl Ether(104-68-7) 10% Diethylene Glycol Monomethyl Ether (111-77-3) 5.94% Tetrahydrofurfural Alcohol (97-99-4) |
| Terpene Tech. | HTF 85B | Tarksol 97 Solvent | N/A | >200 F | Tarksol 97 Solvent |
| Inland Tech | EP-921 | D-limonene Terpene | N/A | 147 F | Propylene Carbonate (108-32-7) d-Limonene (5989-27-5) |
| ISP Tech | NMP | Cyclic Amine | N/A | 199 F | 1-Methyl-2-Pyrrolidinone (872-50-4) |
| General Chem. | Aquaclean 4784 | Alkaline Aqueous | >12 | >212 F | <2% Potassium Hydroxide (1310-58-3) <15% Ethylene Glycol Monophenyl Ether (122-99-6) <15% Diethylene Glycol Monobutyl Ether (112-34-5) <15% Diethylene Glycol (111-46-4) <5% Triethanolamine (102-71-6) |