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Demonstration Evaluation of Biodegradable Degreaser

by

Battelle
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Task Order Manager

David Ferguson
National Risk Management Research Laboratory
U.S. Environmental Protection Agency
Cincinnati, Ohio 45268

National Risk Management Research Laboratory
Office of Research and Development
U.S. Environmental Protection Agency
Cincinnati, OH 45268

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FOREWORD

The U.S. Environmental Protection Agency (EPA is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems yto support and nurture life. To meet this mandate, EPA's research program is providing data and technical support for solving environmental problems today and building a science knowledge base necessary to manage our ecological resources wisely, understand how pollutants affect our health, and prevent or reduce environmental risks in the future.

The National Risk Management Research Laboratory (NRMRL) is the Agency's center for investigation of technological and management approaches for preventing and reducing risks from pollution that threaten human health and the environment. The focus of the Laboratory's research program is on methods and their cost-effectiveness for prevention and control of pollution to air, land, water, and subsurface resources; protection of water quality in public water systems; air pollution; and restoration of ecosystems. NRMRL collaborates with both public and private sector partners to foster technologies that reduce the cost of compliance and to anticipate emerging problems. NRMRL's research provides solutions to environmental problems by developing and promoting technologies that protect and improve the environment; advancing scientific and engineering information to support regulatory and policy decisions; and providing the technical support and information transfer to ensure implementation of environmental regulations and strategies at the national, state, and community levels.

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Sally Gutierrez, Director
National Risk Management Research Laboratory

ABSTRACT

The objective of this project was to evaluate a bio-based parts-degreasing fluid called Eagle Kleen™ manufactured by Hydra-Tone Chemicals, Inc. (HTCI). Performance tests of this methyl-ester/surfactant, ready-to-use, micro-emulsion degreaser indicated that it was effective in removing oil and grease contamination from bare metal and painted surfaces, and its degreasing power is similar to alkaline and solvent cleaners. The project included the preparation of the Quality Assurance Project Plan (QAPP) and conducting the following three Tasks: 1 – Laboratory Testing, 2 – Site Testing (conducted at the Vehicle Shop at Robins Air Force Base [AFB], the Gas Turbine Engine [GTE] Shop at Hill AFB, and an equipment supplier), and 3 – Engineering Cost Assessment.

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ACRONYMS AND ABBREVIATIONS

AFB	Air Force Base
ASTM	American Society for Testing and Materials
CPVC	chloropolyvinylchloride
CFR	Code of Federal Regulations
DOT	Department of Transportation
ECA	Engineering Cost Assessment
EPA	(United States) Environmental Protection Agency
ESOH	Environmental, Safety, and Occupational Health
HDPE	high-density polyethylene
HTCI	Hydra-Tone Chemicals, Inc.
IVD	Ion Vapor Deposited
IWTP	industrial wastewater treatment plant
LD_{50}	lethal dose at which 50% of the species does not survive
MSDS	Material Safety Data Sheet(s)
NESHAP	National Emission Standards for Hazardous Air Pollutants
NRMRL	National Risk Management Research Laboratory
NPV	net present value
PPE	personal protective equipment
ppm	parts per million
PVC	polyvinylchloride
QAPP	Quality Assurance Project Plan
QA/QC	quality assurance/quality control
QPL	qualified product list
RTU	ready-to-use
SARA	Superfund Amendment and Reauthorization Act
SMI	Scientific Materials International, Inc.
TOM	Task Order Manager
USAF	United States Air Force
UN	United Nations
VOC	volatile organic compound

EXECUTIVE SUMMARY

The objective of this project was to evaluate a bio-based parts-degreasing fluid called Eagle Kleen™ manufactured by Hydra-Tone Chemicals, Inc. (HTCI). Performance tests of this methyl-ester/surfactant, ready-to-use, micro-emulsion degreaser indicated that it was effective in removing oil and grease contamination from bare metal and painted surfaces, and its degreasing power is similar to alkaline and solvent cleaners. The project included the preparation of the Quality Assurance Project Plan (QAPP) and conducting the following three Tasks: 1 – Laboratory Testing, 2 – Site Testing (conducted at the Vehicle Shop at Robins Air Force Base [AFB], the Gas Turbine Engine [GTE] Shop at Hill AFB, and an equipment supplier), and 3 – Engineering Cost Assessment.

Analytical Testing. In this task, the non-hazardous, non-flammable, non-corrosive degreaser was assessed against a military specification (MIL-PRF-87937D) for water dilutable aerospace cleaning compounds. It met biodegradable, flash point, cleaning performance, residue, and cold stability requirements, as well as a series of metal and painted surface corrosion tests. It was found acceptable for sealants, rubber, and insulated wire, as well as hydrogen embrittlement. This ability to meet the hydrogen embrittlement requirement is a significant achievement that sets it apart from most aqueous cleaners. Unfortunately, it was found to craze acrylics and polycarbonates. It also could not pass the heat stability tests designed for water dilutable cleaners, which is a test not totally appropriate for a ready-to-use formulation such as Eagle Kleen™. The fluid was found suitable for full-scale demonstration trials where simple degreasing of metal and/or painted surfaces was required. Independent material compatibility testing indicates that Eagle Kleen™ is a powerful degreaser and, in some cases, could dissolve polymeric materials used for gloves, seals, gaskets, tank construction plastics, and certain materials used for tubing and hoses. However, acceptable alternatives for these items are commonly available, such as neoprene gloves, Viton or Teflon gaskets and seals, and high-density polyethylene (HDPE) tanks and containers.

HTCI describes Eagle Kleen™ as "non-toxic." However, limited aquatic toxicity testing, as part of the MIL-PRF-87937D evaluation, indicated that Eagle Kleen™ was toxic to some aquatic life forms even at low concentrations. Additional toxicity tests, where Eagle Kleen™ was evaluated against a typical solvent and a typical alkaline cleaner, indicated that all three were toxic to aquatic life forms at typical use concentrations.

Site Testing. Full-scale demonstration trials were held at Robins AFB and at Hill AFB. Brief tests were also conducted at Ransohoff, Inc., an immersion parts washer manufacturer. Details of those demonstrations are summarized below.

Robins AFB. The Robins AFB vehicle shop demonstration included side-by-side testing of 3-inch-diameter steel wheel bearings cleaning with the shop's regular solvent, Safety Kleen PRF 680 Type II hydrocarbon degreaser, and with Eagle Kleen III™ degreaser. The initial assessment, using actual wheel bearings removed from base vehicles for routine maintenance, showed that there was little or no difference in the appearance of the cleaned parts, regardless of the degreaser used. The following characteristics were noted for Eagle Kleen™:

- In general, the level of cleaning was adequate, but on an individual part basis, the performance was either equal to or slightly inferior to the hydrocarbon degreaser.

- Cleaning times using Eagle Kleen™ were approximately 50% longer for a part of similar size, shape, and type of contamination than using Safety Kleen.

- The degreaser had an odor that was objectionable to some operators.
- Parts felt dramatically more slippery in gloved hands, until the parts were rinsed with water.

The Vehicle Shop manager indicated that Eagle Kleen™ may not be cost-effective due to the longer cleaning time that was required. For Eagle Kleen™ to be economically attractive, its base cost, handling cost, and disposal costs would have to be lower, and/or its lifetime would have to be much longer than that of a traditional solvent.

Hill AFB. The demonstration at the Hill AFB GTE shop included side-by-side testing of a variety of GTE parts cleaned in the shop's automated RAMCO system. The shop used hot alkaline cleaning (Turco 6849), water rinsing, and drying operations. Many of the parts that were degreased at the GTE shop had heavy carbon contamination. All had some degree of oil and/or grease contamination too. The initial assessment showed that Eagle Kleen™ was less effective than Turco 6849. The following characteristics were noted for Eagle Kleen™:

- The operators reported that Turco 6849 removed the oil, grease, and carbon deposits; in contrast, Eagle Kleen™ removed most of the oil and grease, but was not effective at removing heavy carbon contamination.
- Test results indicated that operating Eagle Kleen™ at an elevated temperature (90-120°F) did not significantly improve performance; however, the change in temperature dramatically increased complaints related to odor.
- The two-week testing schedule was stopped after three days due to these issues.

Ransohoff. Tests at the Ransohoff, Inc. company in Cincinnati, OH included testing of selected, condemned GTE parts cleaned using the Ransohoff, Inc. ultrasonics-enhanced system. A few of the parts showed some carbon contamination, and had some degree of oil contamination. The initial assessment showed that a 5% solution of Eagle Kleen™ with ultrasonics was extremely effective at removing the carbon deposits. The following characteristics were noted for Eagle Kleen™:

- A dilute solution of Eagle Kleen III can be used in an ultrasonically enhanced parts washer to remove a substantial portion of carbon deposited on typical GTE shop parts.
- Cleaning appeared good, but was not complete. A longer immersion time or hand cleaning might be required for complete carbon removal.
- The solution was not effective at removing grease/heavy oils.
- The Eagle Kleen III odor was detectable but not overpowering, even with the top off of the cleaning unit. It is anticipated that the odor problem would be reduced if the top is open for only a few minutes per hour. However, some type of ventilation would be required in most applications.

Engineering Cost Assessment. The assessment indicated that the installation of a new ultrasonic cleaning bath and the use of Eagle Kleen III results in an attractive payback of <3.3 years depending on the cost for handling, treatment and disposal of the spent alkaline cleaner and spent rinse.

1.0 PROJECT DESCRIPTION

1.1 General Overview

The purpose of this Task Order was to evaluate Hydra-Tone Chemicals, Inc.'s (HTCI's) Eagle Kleen™ biodegradable degreaser as a potential substitute for conventional alkaline cleaners and hydrocarbon cleaning solvents. Battelle conducted the evaluation under contract agreement with the National Risk Management Research Laboratory (NRMRL) of the United States Environmental Protection Agency (EPA). The approach of the NRMRL is to work 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 intent of EPA's approach is to encourage the use of less-polluting substances in industrial operations.

HTCI introduced Eagle Kleen™ as an environmentally friendly degreasing agent, designed to provide the same degreasing effect as conventional alkaline cleaners and non-chlorinated cleaning solvents used in immersion tanks and spray washers. HTCI's product literature indicates that Eagle Kleen™ is completely biodegradable, non-hazardous, non-flammable, non-toxic, non-corrosive, and safe-to-use. Eagle Kleen™ is a naturally derived product based on seed oil. It is a ready-to-use (RTU) liquid degreaser designed with a special methyl-ester micro-emulsion formulation. This unique solvent technology is intended to be used for the removal of grease, cutting fluids, motor and transmission oils, hydraulic fluids, and other surface contaminants. Eagle Kleen™ has a flash point greater than 200˚F (93˚C) and is considered non-flammable. Eagle Kleen does not contain Superfund Amendment and Reauthorization Act (SARA) 313-listed extremely hazardous substances or California Proposition 65 components. HTCI recommends that Eagle Kleen™ should be used at temperatures above 50˚F (10˚C) to provide adequate parts cleaning.

1.2 Goals

The goal of this NRMRL project is to validate the cleaning efficiency and economics of using HTCI's Eagle Kleen™ biodegradable degreaser as an alternative to (and potential substitute for) hydrocarbon-solvent degreasers and heated alkaline immersion cleaners. Such degreasers have been introduced in the parts cleaning industry as replacements for organic solvents, most of which are ozone-depleting substances and/or are targeted for reduced usage by EPA's 33/50 program (EPA, 1999).

Data were tracked on cleaning efficiency, bath performance, cleaning time, and working conditions when using each fluid. By comparing these data, the suitability of Eagle Kleen™ as a replacement fluid in these applications was assessed. The following three general issues were addressed:

1. The proposed new technology/methodology must be effective in performing the process function that it is intended to replace.

2. There must be a significant, measurable reduction in the quantity of waste hazard (pollutant) produced and in the level of hazard produced.

3. The economics of the alternative technology must be quantified and compared to the economics of the existing technology.

The consideration of each issue is critical to recommending the new technology as a feasible alternative to the existing technology. The site testing task was designed to address items 1 and 2, and gather information to evaluate item 3.

1.3 Demonstration/Report Organization.

The Battelle Program Manager for this project was Dr. Bruce Alleman. Dr. Alleman was responsible for all technical requirements and was supported by Dr. Bruce Sass who served as the Battelle Task Order Leader. Dr. Sass maintained regular telephone communication with the U.S. EPA Task Order Manager (TOM), Mr. David Ferguson. Ms. Sara Kuczek was responsible for preparing the QAPP, supervising the laboratory effort (Task 1), and data reporting. Mr. Nick Conkle was responsible for Site Testing (Task 2) and the Engineering Cost Assessment (Task 3). The Field Evaluation Integration (Task 4) was eliminated. Ms. Betsy Cutie was the Battelle QA Officer who monitored project performance with regard to the QAPP.

This report is organized in the following sections:

1. Task 1: Analytical Testing
2. Task 2: Site Testing
3. Task 3: Economic Cost Assessment
4. Conclusions
5. References
6. Appendices.

2.0 TASK 1 – ANALYTICAL TESTING

Prior to laboratory testing, Battelle consulted stakeholders at Robins Air Force Base (AFB) and Hill AFB to discuss immersion and spray degreaser requirements for military parts cleaning. The purpose of these discussions was to gain approval and learn about cleaning and materials compatibility requirements that could be used as criteria with which to measure the performance of the different cleaning materials and methods, and to form a direct way of comparing conventional cleaners with Eagle Kleen™. The appropriate cleaning and materials compatibility requirements are contained in MIL-PRF-87937D ("Performance Specification: Cleaning Compound, Aerospace Equipment"). It is attached as Appendix A. The purpose of Task 1 was to demonstrate Eagle Kleen's™ ability to meet the criteria listed in MIL-PRF-87937D. Testing was to proceed at Hill AFB only if Eagle Kleen™ was comparable to or surpassed the specification standards or requirements. No specific prequalification testing was required at Robins AFB's Vehicle Shop, but the corrosion testing results along with the other analytical tests provided by this specification provided valuable information.

Degreasers used by the United States Air Force (USAF) must conform to MIL-PRF-87937D and, in some cases, MIL-C-29602 or MIL-PRF-85570 to comply with the Process Orders and Technical Orders dictating repair and maintenance of F-15, F-16, C-5, C-17, C-130, and C-141 aircraft component parts. To facilitate the timely completion of this study, Battelle tested Eagle Kleen's™ performance on all requirements outlined in MIL-PRF-87937D with the exception of long-term storage stability. Due to the scheduling of the demonstration, this was not required because the material was not stored for longer than a few months. To meet MIL-PRF-87937D, Battelle completed the material qualification requirements tests listed for Type IV cleaners. Battelle managed the subcontracted analytical tests, performed quality assurance (QA)/quality control (QC) assessments, and performed pre-testing (QA/QC of sample) in its laboratories. Cleaning efficiency testing was part of the MIL-PRF-87937D series of tests, as described in Section 4.5.21 in MIL-PRF-87937D. Some qualifying cleaning tests were performed by Battelle prior to the on-site demonstration at Robins AFB in order to assess efficiency prior to final results being available from the certification testing. Table 1 provides details on the MIL-PRF 87937D testing performed by Scientific Materials International, Inc. (SMI).

The collection of test coupons/materials and conduct of laboratory testing required 2 months for each formulation tested. Per SMI's instructions, 3 gallons, supplied in plastic containers meeting DOT UN 1H1 as required by 49 CFR 178, were required to test per MIL-PRF-87937D. (Note: It is possible that Eagle Kleen™ could qualify as a Type II degreaser, a Water Dilutable Cleaning Compound, but it was felt that it would be appropriate to test it as a Type IV degreaser for these applications.)

Table 1. Subcontractor for MIL-PRF-87937D Testing

Laboratory	Address	Certifications
Scientific Materials International, Inc. (SMI)	12219 SW 131 Avenue Miami, Florida 33186-6401 Contact: Pat Viani (305) 971-7047	"Internationally recognized as an authorized facility by airframe and engine manufacturers throughout the world, including the U.S. Air Force and U.S. Navy." www.smiinc.com "We adhere to standard laboratory practices and utilize certified standards for our meters and instruments where applicable. We have military inspections which authorize our laboratory to perform testing of aerospace maintenance chemicals in accordance with military standards." Patricia Viani in an email to Sara Kuczek dated 20 February 2004.

The sample of material was to be collected from HTCI's readily available stock of Eagle Kleen™ to assure that a valid subset of the material was obtained and was not specialized for this test. Due to early failures, HTCI was required to alter Eagle Kleen's™ formulation and specialized batches were tested. This process of change is detailed later in the report. All material tested was sent to Battelle for quality assurance/
quality control (QA/QC) prior to sending the material on to SMI for certification.

By performing QA/QC on the sample at Battelle prior to shipment to SMI, it was possible to ensure that the product submitted for certification fell within the acceptable production ranges and appearances. When the product failed these tests, an additional sample was requested from HTCI that met the specifications prior to commencement of testing.

Per MIL-PRF-87937D, the tests described in performance specification were conducted (see Table 2). HTCI supplied data on the flash point, pH, toxicity, constituents, appearance, volatile organic compounds (VOCs), drying point, and total immersion corrosion. These data were used for comparison when evaluating the results from the SMI testing of MIL-PRF-87937D, but after the necessary formulation adjustments, it was found that the data previously supplied by HTCI were not reflective of the current formulation. HTCI certified that the composition of Eagle Kleen™ met the non-testable requirements (i.e., workmanship) outlined in MIL-PRF-87937D.

2.1 Initial Laboratory Evaluation

A laboratory experiment was conducted to test the original formulation of Eagle Kleen™ (designated Eagle Kleen I) for effectiveness as a degreasing agent. Contaminated bearings that were to be discarded were obtained through Robins AFB Vehicle Shop contacts.

Three contaminated wheel bearings (two ~3 inches in diameter and one ~2.5 inches in diameter) were obtained. One was placed in a beaker and submersed in Eagle Kleen™ for approximately 25 minutes. During that time there was little change to the solution color. Some grease may have been loosened in the inner bearings, but no apparent degreasing was observed (see Figure 1).

Because the part was not satisfactorily degreased by immersion, it was sprayed with virgin Eagle Kleen™ for 5 minutes at a very low rate (130 mL/min); this had some minor cleaning effect. The part was mechanically agitated by hand in the solution, which resulted in additional cleaning. The part then was sprayed with water and wiped with a paper towel, which left yellow to brown deposits on the towel. There was evidence of heavy grease deposits on the back side of the wheel bearing. After cleaning, the solution looked yellow.

The wheel bearing surface remained slippery and the cleaning results did not look impressive. The dried part was allowed to sit in a hood on a clean paper towel for later observation. Upon returning, the part had no rust, but was still very slippery due to either the residual grease or Eagle Kleen™ on the part.

A second wheel bearing initially was sprayed with virgin Eagle Kleen™ at a low flowrate, but this activity showed little grease removal. The part still showed evidence of grease deposits on the back side and the surface was slippery. It was determined that the part had not been cleaned adequately, so the bearing was returned to the cleaning solution and re-inspected after 30 minutes of soaking. After mechanically agitating the bearing in the bath, spraying with water, and drying with a paper towel, the part looked significantly better and nearly all the grease deposits were gone (see Figure 2). The surface was not slippery and in general the cleaning looked acceptable. The part was re-dipped in Eagle Kleen™, rinsed in water, and allowed to sit on a paper towel for an hour and then observed; there appeared to be some evidence of spot rusting.

Table 2. Analytical Testing Per MIL-PRF-87937D

Specification	Title		Test Method
MIL-PRF-87937D, Section 3.4	Compositional Assurance	Non-volatile Residue	Test described in MIL-PRF-87937D, Section 4.5.1
		pH	Test described in MIL-PRF-87937D, Section 4.5.3, ASTM E 70
		IR	Test described in MIL-PRF-87937D, Section 4.8.2
MIL-PRF-87937D, Section 3.5	Chemical Properties	Insoluble Matter	Test described in MIL-PRF-87937D, Section 4.5.2
		Flash Point	Test described in MIL-PRF-87937D, Section 4.5.7, ASTM D 56
		Emulsion	Test described in MIL-PRF-87937D, Section 4.5.8
		Wet Tape Adhesion	Test described in MIL-PRF-87937D, Section 4.5.27
		Cleaning Efficiency	Test described in MIL-PRF-87937D, Section 4.5.21
		Residue Rinsibility	Test described in MIL-PRF-87937D, Section 4.5.4
MIL-PRF-87937D, Section 3.6	Physical Properties	Heat Stability	Test described in MIL-PRF-87937D, Section 4.5.5
		Cold Stability	Test described in MIL-PRF-87937D, Section 4.5.6
MIL-PRF-87937D, Section 3.7	Effect on Metals	Hydrogen Embrittlement (Cadmium and Ion Vapor Deposited [IVD] Plated Bars)	Test described in MIL-PRF-87937D, Section 4.5.9, ASTM F 519
		Total Immersion Corrosion	Test described in MIL-PRF-87937D, Section 4.5.10, ASTM F 483
		Low-Embrittling Cadmium	Test described in MIL-PRF-87937D, Section 4.5.11, ASTM F 1111
		Effects on Unpainted Metals	Test described in MIL-PRF-87937D, Section 4.5.12, ASTM F 485
		Sandwich Corrosion	Test described in MIL-PRF-87937D, Section 4.5.16, ASTM F 1110
		Wet Tape Adhesion	Test described in MIL-PRF-87937D, Section 4.5.27
MIL-PRF-87937D, Section 3.8	Effect on Painted Surfaces		Test described in MIL-PRF-87937D, Section 4.5.13, ASTM F 502
MIL-PRF-87937D, Section 3.9	Stress crazing of MIL-PRF-5425 and MIL-PRF-25690 (Type A and C) acrylic plastics		Test described in MIL-PRF-87937D, Section 4.5.14, ASTM F 484
MIL-PRF-87937D, Section 3.10	Stress crazing of polycarbonate plastics		Test described in MIL-PRF-87937D, Section 4.5.15, ASTM F 484
MIL-PRF-87937D, Section 3.11[(b)]	Long term storage		**Test skipped.** Test described in MIL-PRF-87937D, Section 4.5.17, ASTM F 1104
MIL-PRF-87937D, Section 3.12	Hot dip galvanizing corrosion		Test described in MIL-PRF-87937D, Section 4.5.18, ASTM F 483

Table 2. Analytical Testing Per MIL-PRF-87937D (Continued)

Specification	Title	Test Method
MIL-PRF-87937D, Section 3.13	Workmanship	Certified by manufacturer
MIL-PRF-87937D, Section 3.14	Effect on polysulfide sealants	Test described in MIL-PRF-87937D, Section 4.5.19, ASTM D 2240
MIL-PRF-87937D, Section 3.15	Rubber compatibility	Test described in MIL-PRF-87937D, Section 4.5.20, ASTM D 2240
MIL-PRF-87937D, Section 3.16	Effect on polyimide insulated wire	Test described in MIL-PRF-87937D, Section 4.5.26
MIL-PRF-87937D, Section 3.3	Toxicity	Percent survival will be recorded for *Pimephales promelas* and *Ceriodaphnia dubia* at 1, 10, 50, and 100 ppm concentrations
MIL-PRF-87937D, Section 3.3.4	Biodegradability	Test described in MIL-PRF-87937D, Section 4.5.22, 40 CFR, Part 796.3100

(a) All listed analytical tests must be passed in order for the on-site demonstration to proceed. Due to this, all tests are considered critical in this phase of the program.

(b) Long-term storage was not tested.

Figure 1. Bearing Inspection after Initial Immersion

Figure 2. Bearing after Final Cleaning

At this stage, it appeared that Eagle Kleen™ would degrease a part effectively enough for a mechanic to determine if it had a major defect that would require replacement. It also appeared that the part would be clean enough for repacking with grease for reinstallation. It was determined that this cleaning ability was suitable for site testing to commence at Robins AFB, but because spot rusting was observed, the demonstration was delayed until corrosion data were obtained.

2.2 Analytical Laboratory Testing – Eagle Kleen I

After the initial laboratory evaluation, the original formulation (i.e., Eagle Kleen I) was submitted to SMI for MIL-PRF-87937D testing. However, there was not an exact category match for testing the fluid. Eagle Kleen™ solution is intended to replace dilutable fluids, but is supplied as a "ready-to-use" fluid not a water-dilutable concentrate. Therefore, it was determined to test it as a "Type IV – Heavy Duty Water-Dilutable Cleaning Compound" but to test it as ready-to-use (non-diluted) fluid when certain tests called for the dilution of the test material. The fluid was submitted on July 14, 2004 and test results were reported on September 20, 2004. Table 3 summarizes the results. The full results are included in Appendix B.

Eagle Kleen I did not pass MIL-PRF-87937D requirements. Specifically, it failed the heat stability and several metal corrosion tests. Because the material is intended to be used at room temperature, the team determined that failing the heat stability tests was not a reason to reformulate and retest alone, but that without passing the corrosion tests, the demonstrations could not proceed at either proposed location.

Table 3. Eagle Kleen I Analytical Test Results per MIL-PRF-87937D

MIL-PRF-87937D Section Number	Test Name	Results
3.3	Toxicity	Informational[(a)]
3.3.4	Biodegradability	Conforms
3.4	Compositional assurance	Informational
3.5	Chemical properties	
3.5.1	Chemical Requirements	
	Insoluble matter	Conforms
	Flash point	Conforms
	Emulsion characteristics	Conforms[(b)]
	Wet adhesion tape test	Conforms[(b)]
	% Cleaning efficiency	Conforms[(b)]
	Terpene hydrocarbons	Not applicable
3.5.2	Residue rinsibility	Conforms[(b)]
3.6	Physical properties	
3.6.1	Heat stability	Does not conform
3.6.2	Cold stability	Conforms
3.6.3	Rheology	
3.6.3.1	Consistency	Not applicable
3.6.3.2	Sprayability	Not applicable
3.7	Effect on metals	
3.7.1	Hydrogen embrittlement	Does not conform[(b)]
3.7.2	Total immersion corrosion	Does not conform[(b)]
3.7.3	Low-embrittling cadmium plate corrosion	Does not conform[(b)]
3.7.4	Effects on unpainted metal surfaces	Conforms[(b)]
3.7.5	Sandwich corrosion	Does not conform[(b)]
3.7.6	Wet adhesion tape test	Conforms[(b)]
3.8	Effect on painted surfaces	Conforms[(b)]
3.9	Stress crazing of MIL-PRF-5425 and MIL – PRF-25690 (Type A and C) acrylic plastics	Conforms[(b)(c)]
3.10	Stress crazing of polycarbonate plastic	Conforms[(b)(c)]
3.11	Long-term storage stability	Not performed
3.12	Hot dip galvanizing corrosion	Conforms[(b)]
3.13	Workmanship	Was "certified" by manufacturer to conform with requirements
3.14	Effect on polysulfide sealants	Conforms[(b)]
3.15	Rubber compatibility	Conforms[(b)]
3.16	Effect on polyimide insulated wire	Conforms[(b)]

(a) Toxicity data is reported, but not as the widely accepted LD_{50} values. Additional toxicity testing was conducted.
(b) Test performed using "as received" solution (ready-to-use) instead of dilution required by specification. Results were not considered for qualified product listing (QPL).
(c) Because of the high surfactant content of Eagle Kleen™, it should craze most stressed transparent plastics when subjected to extended exposure. Passing of these tests was unexpected and could not be duplicated in subsequent tests.

2.3 Analytical Laboratory Testing – Eagle Kleen II

HTCI, along with their manufacturing partner, decided to reformulate the Eagle Kleen I degreaser by adding a corrosion-controlling agent, and designated the resulting new formulation Eagle Kleen II. After HTCI submitted a sample of the newly revised formulation, Battelle performed QA/QC tests in addition to an additional cleaning test. It then was decided by the evaluation team that, in order to reduce testing time and conserve funds, only the tests that failed the initial analytical testing at SMI would be run on Eagle Kleen II. If all passed, the full set of tests would be continued. The sample was submitted to SMI on September 10, 2004 and the results report was received on September 30, 2004. Results are summarized in Table 4. The SMI Report is included in Appendix C.

Table 4. Eagle Kleen II Analytical Test Results per MIL-PRF-87937D

MIL-PRF-87937D Section Number	Test Name	Results[(a)]
3.7	Effect on metals	
3.7.1	Hydrogen embrittlement	Conforms
3.7.2	Total immersion corrosion	Does not conform
3.7.3	Low-embrittling cadmium plate corrosion	Does not conform
3.7.5	Sandwich corrosion	Conforms

(a) Test performed using "as received" solution (ready-to-use) instead of dilution required by specification. Results were not considered for QPL listing.

Although this reformulation was an improvement over Eagle Kleen I, Eagle Kleen II continued to fail some of the required tests. Of great significance, however, was the ability of this degreaser to pass the hydrogen embrittlement test. Most, if not all, near neutral pH aqueous cleaners fail this test. The ability of the reformulated fluid to not embrittle high-strength steels made the degreaser unique and opened the door for use in many aerospace applications.

2.4 Analytical Laboratory Testing – Eagle Kleen III

HTCI and their manufacturing partner decided to reformulate again, creating Eagle Kleen III using a different concentration of an alternative corrosion agent. After QA/QC testing by Battelle, a sample was submitted to SMI for testing. Initial testing at HTCI's manufacturing partner and by Battelle indicated that this formulation should pass the tests that were not passed by Eagle Kleen II. Therefore, the entire testing sequence at SMI was initiated. SMI received the sample of Eagle Kleen III on December 1, 2004 and the results report was issued on March 7, 2005. Results are summarized in Table 5. The SMI Report is included in Appendix D.

Again, because the material is intended to be used at room temperature, the team determined that failing the heat stability tests alone was not a reason to reformulate and retest. However, the corrosion testing passed and the fluid continued to meet the hydrogen embrittlement requirements, the changes to the formulation appeared to cause stress crazing failures on the acrylic and polycarbonate plastics. To confirm that the correct results were obtained, these tests were performed once more. Eagle Kleen III did not pass these repeat tests.

After consulting with the demonstration team, including representatives from Robins AFB and Hill AFB, it was determined that the parts that were processed in each of their shops did not require much, if any, processing of plastics, and the demonstrations could commence without passing these two tests.

Table 5. Eagle Kleen III Analytical Test Results per MIL-PRF-87937D

MIL-PRF-87937D Section Number	Test Name	Results
3.3	Toxicity	Informational[(a)]
3.3.4	Biodegradability	Conforms
3.4	Compositional assurance	Informational
3.5	Chemical properties	
3.5.1	Chemical Requirements	
	Insoluble matter	Conforms
	Flash point	Conforms
	Emulsion characteristics	Conforms[(b)]
	Wet adhesion tape test	Conforms[(b)]
	% Cleaning efficiency	Conforms[(b)]
	Terpene hydrocarbons	Not applicable
3.5.2	Residue rinsibility	Conforms[(b)]
3.6	Physical properties	
3.6.1	Heat stability	Does not conform
3.6.2	Cold stability	Conforms
3.6.3	Rheology	
3.6.3.1	Consistency	Not applicable
3.6.3.2	Sprayability	Not applicable
3.7	Effect on metals	
3.7.1	Hydrogen embrittlement	Conforms[(b)]
3.7.2	Total immersion corrosion	Conforms[(b)]
3.7.3	Low-embrittling cadmium plate corrosion	Conforms[(b)]
3.7.4	Effects on unpainted metal surfaces	Conforms[(b)]
3.7.5	Sandwich corrosion	Conforms[(b)]
3.7.6	Wet adhesion tape test	Conforms[(b)]
3.8	Effect on painted surfaces	Conforms[(b)]
3.9	Stress crazing of MIL-PRF-5425 and MIL – PRF-25690 (Type A and C) acrylic plastics	Does not conform[(b, c)]
3.10	Stress crazing of polycarbonate plastic	Does not conform[(b, c)]
3.11	Long-term storage stability	Not performed
3.12	Hot dip galvanizing corrosion	Conforms[(b)]
3.13	Workmanship	Was “certified” by manufacturer to conform with requirements
3.14	Effect on polysulfide sealants	Conforms[(b)]
3.15	Rubber compatibility	Conforms[(b)]
3.16	Effect on polyimide insulated wire	Conforms[(b)]

(a) Toxicity data is reported, but not as the widely accepted LD_{50} values. Additional toxicity testing was conducted.
(b) Test performed using “as received” solution (ready-to-use) instead of dilution required by specification. Results were not considered for QPL listing.
(c) Because of the high surfactant content of Eagle Kleen™, it should craze most stressed transparent plastics when subjected to extended exposure.

2.5 Analytical Laboratory Testing – Eagle Kleen III, Vapor Pressure

During laboratory evaluation of the Eagle Kleen™ formulations, it was noted that the odor from the fluid was strong, even at room temperature. In addition, the evaluation team wanted to confirm that the fluid was low to non-volatile, in order to avoid engineering control requirements and confirm that Eagle Kleen™ was below the 7-mm Hg limit set under the Aerospace National Environmental Standards for Hazardous Air Pollutants (NESHAP). The Air Force defines an aqueous cleaner as having greater than 80% water. As Eagle Kleen™ does not meet this criterion, it would be subject to the NESHAP restrictions.

SMI was contracted to perform the "Test Method for Vapor Pressure of Petroleum Products (Reid Method)", ASTM D 323. This test was performed on the same fluid lot of Eagle Kleen III used for the analytical testing described in Section 2.0, and the results are shown in Table 6. The Reid Vapor Pressure of the fluid was determined to be less that 0.2 mm Hg at 20°C, which is less than the 7-mm Hg limit. Therefore, Eagle Kleen™ was exempt from any VOC regulations set by the Air Force, and testing could proceed. The SMI test report on vapor pressure is included as Appendix E.

Table 6. Reid Vapor Pressure Results for Eagle Kleen III

Sample	Reid Vapor Pressure
Eagle Kleen III	<0.2 mm Hg at 20 °C

2.6 Material Compatibility Evaluation

While conducting the Robins AFB demonstration, it was determined that Eagle Kleen™ is not suitable for use with or on certain types of polymers. A scrub brush used during the hand cleaning at the Vehicle shop was left to lay in approximately ½-inch of Eagle Kleen III degreaser over the weekend. Upon return to the shop the following week, employees found that the scrub brush had "melted" (see Figure 3 for the "before" photograph and Figure 4 for the "after" photograph).

At this point, Russ Markesbery of HTCI supplied the team with a list of materials that should be compatible with Eagle Kleen™. This list was not comprehensive and additional testing was deemed necessary. Testing at Battelle found that the handle was composed of either a copolymer of polystyrene and methylene (copoly[styrene-methylene]) or a copolymer of styrene and polyethylene. Although the spectrum more closely matched the first copolymer listed, the second is a much more widely available material. Because this fluid would be used in conjunction with seals, hoses, pumps, and various personal protective equipment (PPE), Battelle completed a short, non-comprehensive study of material compatibility. Materials that may be used with, or cleaned by, this fluid were chosen for this study. Results are detailed in Table 7.

After reviewing this study, it became apparent that if Eagle Kleen™ were to be used in a large-scale setting, care would need to be taken to ensure the materials of construction for the equipment used in conjunction with the fluid were compatible with Eagle Kleen™. In addition, the facility would need to stress the importance of using the appropriate PPE for this task and set up a stringent replacement schedule of said PPE. However, from the preliminary compatibility testing performed, it appears that acceptable alternatives, such as neoprene gloves, Viton or Teflon gaskets and seals, and high-density polyethylene (HDPE) tanks and containers are readily available.

Figure 3. Handle Before Exposure to Eagle Kleen III

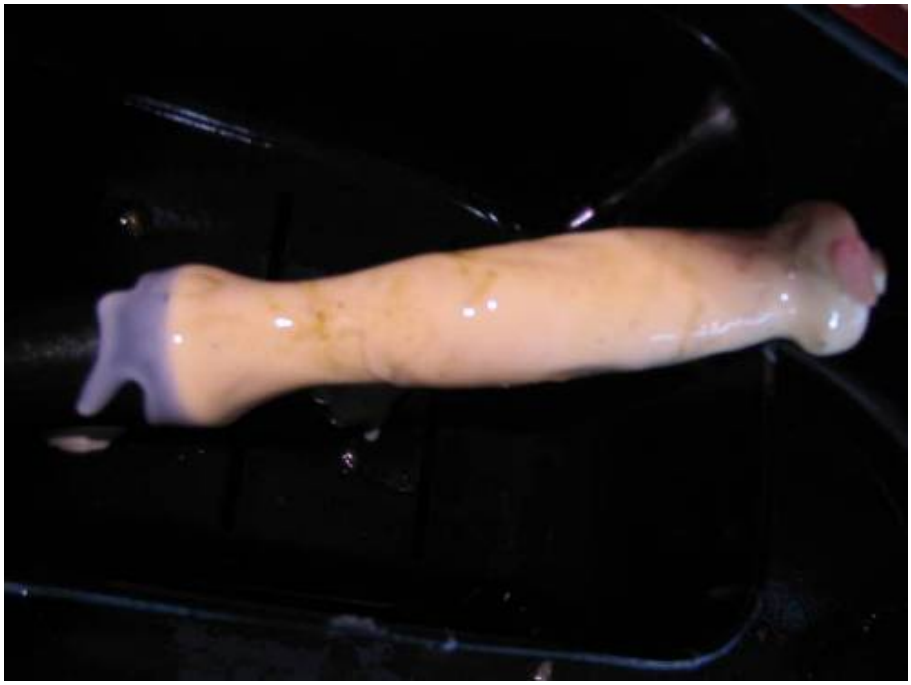

Figure 4. Handle After Exposure to Eagle Kleen III

Table 7. Material Compatibility Analysis Results

Materials	Disclosed by HTCI after Brush Incident as being Incompatible	Incompatible After an Hour of Exposure	Incompatible After 3 Weeks' of Exposure	Initial Testing Did Not Result in Signs of Incompatibility
Vinyl-acrylics	X			
PVC	X			
Tygon	X			
Polycarbonate	X			
SBR Rubber Sheet		X		
Neoprene		X[(a)]		
Viton		X[(a)]		
Buna-N		X		
EDPM		X		
PVC/Tygon		X		
Polyurethane			X	
Silicone			X	
Polycarbonate				X
Polyethylene (including HDpe0				X
CPVC			X	
Nylon				X
Phenolic				X
Fiberglass				X
Polyester				X
Polypropylene				X
PTFE (i.e., Teflon)				X
Delrin				X

(a) Minor discoloration of solution after one hour exposure but no evidence of performance deterioration.

2.7 Analytical Laboratory Toxicity Testing – Eagle Kleen III

The toxicity test run as part of the initial MIL-PRF-87937D screening indicated that Eagle Kleen™ may not be as "non-toxic" as originally indicated by the manufacturer. Specifically, after 24 hours at only 10 ppm, no fathead minnows remained alive in the Eagle Kleen solution (see Tables 8 and 9). This indicated that Eagle Kleen™ has some toxicity even at low levels.

A data search was conducted to determine the toxicity in relation to this information, but no results were found that were in a comparable format. Therefore, to make a fair comparison, LC_{50} data were collected for Eagle Kleen III and for the other cleaners used in this study, Safety Kleen PRF 680 Type II (hydrocarbon solvent) and Turco 6849 (alkaline cleaner). In general, LC_{50} values below 1,000 mg/L indicate a toxic substance; and, as noted in Table 10, all three cleaners have low LC_{50} figures, meaning they are toxic to aquatic life; see Appendix F for details.

2.8 Conclusions

After the third reformulation, it was determined that although Eagle Kleen™ still did not pass all laboratory certification tests specified in MIL-PRF-87937D, it passed all except (1) stress crazing of acrylic plastics and polycarbonate plastics, and (2) heat stability. These results were deemed sufficient by the evaluation team, Robins AFB, and Hill AFB to proceed with the demonstrations. In order to actually

certify Eagle Kleen™, a new category of degreaser must be added to MIL-PRF-87937D or another specification written for it because it did not fall into an established category.

Table 8. 96-Hour *Pimephales promelas* Bioassay

Percentage of Fathead minnows (*Pimephales promelas*) Surviving			
Concentration	**After 24 Hours**	**After 48 hours**	**After 96 Hours**
1 ppm	100	100	100
10 ppm	0	0	0
50 ppm	0	0	0
100 ppm	0	0	0

Table 9. 48-Hour *Ceriodaphnia dubia* Bioassay

Percentage of Water Fleas Cladoceran (*Ceriodaphania dubia*) Surviving		
Concentration	**After 24 Hours**	**After 48 hours**
1 ppm	90	50
10 ppm	50	20
50 ppm	0	0
100 ppm	0	0

Table 10. Aquatic Toxicity as Measured by Lethal Concentrations

Fluid	**LC_{50}, 48-hr *Daphnia magna* (mg/L)**	**LC_{50}, 96-hr *Pimephales promelas* (mg/L)**
Eagle Kleen III (100%)	25	30
Safety Kleen PRF 680 Type II (100%); hydrocarbon solvent	125	>70,000[(a)]
Turco 6849 (20%), alkaline cleaner	150	225
Turco Rust Bloc (4%), anti-rusting compound	79,200	33,500

(a) The lighter-than water solvent floated on the surface, and this may have biased the results.

From initial laboratory evaluations, Eagle Kleen™ did appear to degrease, but did not seem to degrease quickly or by immersion alone. From previous experiments, it did not immediately appear to be a significant improvement over traditional degreasers. Site demonstrations at Hill AFB and Robins AFB were conducted to gauge its actual cleaning ability and cost effectiveness.

Areas of improvement and development still remain. Eagle Kleen™ does not appear to be compatible with all materials used in the construction of hoses, pumps, seals, and gaskets. Eagle Kleen™ is a powerful degreaser and, in some cases, could dissolve polymeric materials used for gloves, seals, gaskets, tank construction plastics and certain materials used for tubing and hoses. But, acceptable alternatives for these items are commonly available, such as neoprene gloves, Viton or Teflon gaskets and seals, and HDPE tanks and containers.

Limited aquatic toxicity testing with 100% Eagle Kleen III, as part of the MIL-PRF-87937D evaluation, indicated that Eagle Kleen™ was toxic to some aquatic life forms even at low concentrations. Additional toxicity tests, where Eagle Kleen™ was compared to a typical solvent and a typical alkaline cleaner, indicated that all three were toxic to aquatic life forms at typical use concentrations.

3.0 TASK 2 – SITE TESTING

3.1 Background

The Task 2 site demonstration took place at two primary locations: the Vehicle Shop of Robins AFB, located at the Warner Robins Air Logistics Center of the U.S. Air Force; and the gas turbine engine (GTE) Shop at Hill AFB, located at the Ogden Air Logistics Center. Two parts washers were used at each demonstration location. One was filled with the conventional cleaner and operated at standard conditions, and the other was filled with Eagle Kleen™. By running two parts washers simultaneously, side-by-side results were obtained allowing a direct comparison of cleaning performance.

The tests included cleaning approximately the same type and number of parts, having a similar degree of contamination, through each bath under real world conditions. Cleaning fluids from the parts washers, and where applicable the rinse tanks, were sampled on a daily basis. At the Vehicle Shop the parts were restricted to wheel bearings. In the GTE Shop a variety of parts, representative of normal parts processed is the shop were evaluated. A limited number of condemned parts were used for off-site follow-on testing (see Section 3.4).

Operating parameters, such as cleaning efficiency and bath life, were evaluated. A qualitative cleaning evaluation score was assigned to each run for the conventional treatment and for Eagle Kleen™. In Table 11, scores in red (1 and 2) were ranked as "unacceptable" cleaning, whereas those in green (3, 4, and 5) indicated as acceptable. Critical measurements are listed in Table 12. Also, observations on bath cleanliness were made, and the pH was measured for the aqueous cleaner and rinse waters.

Table 11. Cleaning Score Description

Score	Grease Contamination Level After cleaning
1	Still very dirty
2	Not cleaned well; a lot of contamination still remains
3	Definitely contaminant present
4	Maybe some contaminant present, but fairly cleaned
5	Perfectly clean

Table 12. Critical Measurements for Site Testing

Critical Measurements	Qualitative/ Quantitative	Measurement Time Basis
Number of parts cleaned	Quantitative	At time of degreasing
Number of parts needing reprocessing	Quantitative	At time of degreasing
Fluid efficiency	Qualitative	At time of degreasing, daily, and weekly
Bath condition	Qualitative	Daily and weekly
Fluid addition and replacement	Qualitative	As needed
Cost of fluid	Quantitative	Once during project
Visual observation of bath sample for cleanliness	Qualitative	Weekly

3.2 Robins AFB Testing

The Vehicle Shop at Robins AFB processed a high throughput of 3-inch-diameter steel wheel bearings, which were degreased using a traditional hydrocarbon-based solvent in a parts washer. This demonstration study was structured for side-by-side testing of parts cleaning with the shop's regular solvent and with Eagle Kleen™ degreaser. This allowed the evaluation team, which consisted of personnel from Robins AFB and Battelle, to compare the performance of both fluids under controlled conditions. Information was tracked on cleaning efficiency, bath life (longevity), time for cleaning, and other noticeable effects that would be of concern to shop staff. By comparing these data, the suitability of Eagle Kleen™ as a replacement fluid in this application was assessed.

During the first week of testing (March 28-31, 2005), several different vehicle-shop mechanics were asked to assess the performance of both Eagle Kleen™ and Safety Kleen™ (PRF 680 Type II hydrocarbon degreaser) using three conventional parts washers set up for the demonstration (a parts washer and an aqueous rinse washer were used for Eagle Kleen™, and a single parts washer was used for Safety Kleen™), see Figures 5 and 6.

The initial assessment used actual wheel bearings removed from base vehicles for routine maintenance. Photographs of the bearings before and after cleaning with Eagle Kleen™ (Figure 7) showed that the degreaser was effective at removing grease and oil. However, this study also showed that there was little or no difference in the appearance of the cleaned parts, regardless of the degreaser used.

After conducting the side-by-side tests, the following characteristics were noted for Eagle Kleen™:

- In general, the level of cleaning was adequate, but on an individual part basis, the performance was either equal to or slightly inferior to the hydrocarbon degreaser;
- Cleaning times were approximately 50% longer for a part of similar size, shape, and type of contamination than using Safety Kleen;
- Eagle Kleen™ had an odor that was objectionable to some operators;
- Parts felt more slippery in gloved hands, until the parts were rinsed with water.

The Vehicle Shop manager indicated that he was interested in the potential degreasing ability of Eagle Kleen™, but that the product may not be cost-effective due to the longer cleaning time that was required (he was quoted as saying "time is money"). For Eagle Kleen™ to be economically attractive, according to the manager, its base cost, handling cost, and disposal costs would have to be lower, and/or its lifetime would have to be much longer than that of a traditional solvent.

HTCI, the supplier of Eagle Kleen™, stated that the degreaser could solubilize grease and oil up to 23% of its weight (e.g., 23 lb of grease or oil could be solubilized in 100 lb of Eagle Kleen™). This ratio of contaminant to degreaser would be equivalent to cleaning hundreds, or even thousands, of parts, making a true part-by-part test of this claim impractical in the time period allocated for the demonstration. To overcome these constraints, an accelerated contamination protocol was used, where both cleaning baths were contaminated with heavy grease and heavy-duty motor oil, and testing was done to determine cleaning effectiveness. Some of the same parts were contaminated and cleaned several times as part of the assessment. This portion of the demonstration was conducted during the week of April 18-22, 2005.

Figure 5. Degreaser and Rinse Tanks for Eagle Kleen™ and Safety Kleen™ Bath

Figure 6. Wheel Bearings Being Degreased with Eagle Kleen™

Figure 7. Wheel Bearings Before (left) and After (right) Degreasing with Eagle Kleen™

To rank the cleaning efficiency of the two degreasing fluids, the scoring system noted in Table 11 was employed. After cleaning each set of bearings, the cleaning performance was assessed and assigned a score. Before testing began, the team decided that if a part received an overall score below 3.0, the operator would need to perform additional cleaning for the part to be acceptable. After cleaning more than 1,000 equivalent parts, cleaning scores ranged from 4.0, when the degreasers were fresh, to 2.5. In general, both degreasers were effective in removing heavy oils, transmission and hydraulic fluids, regular lubricating oils, and grease. In general, it was found that Eagle Kleen™ has the ability to degrease a large number of parts and, under normal use, would tend to have a very long bath life. However, the hydrocarbon degreaser (Safety Kleen™) degreased a similar number of contaminated parts, and therefore also can be said to have performed well in this study. Based on results of the side-by-side comparison, it was concluded that Eagle Kleen™ would not have a longer life than the traditional solvent degreaser (see Figure 8).

At the conclusion of the testing program, the Vehicle Shop expressed interest in continuing to use Eagle Kleen™ and moved the three parts washers to a different building (Building 148) where the shop maintained powered equipment, such as portable generators and tug trucks. Testing was not supervised at the same level as was done in the Vehicle Shop. A preliminary assessment, based on a limited number of tests, indicated that the mechanics in this area did not find the degreaser suitable for this equipment. They cited longer degreasing times as the main deficiency in Eagle Kleen™ and noted that the degreaser's odor was a potential concern to shop staff.

A formal request for an Environmental Impact Analysis of Eagle Kleen™ was submitted to the Robins AFB Environmental Management Department. The approval criteria included environmentally friendliness and cost-effectiveness as compared to the current solvent degreaser. The application assumed that rinse water containing Eagle Kleen™ (drag-out) and dissolved contaminants could be discharged to the Robins AFB industrial wastewater treatment plant (IWTP). Discharge of spent Eagle Kleen™ to the IWTP appears to be impractical and probably not allowed by the base. This would require the spent Eagle Kleen™, like the spent hydrocarbon degreaser, to be drummed for off-site disposal. Approval for use at Robins AFB was not received.

A cost comparison between Eagle Kleen™ and the traditional solvent degreaser was not performed due to the longer cleaning time, odor issues, and the unfavorable review by the Building 148 shop.

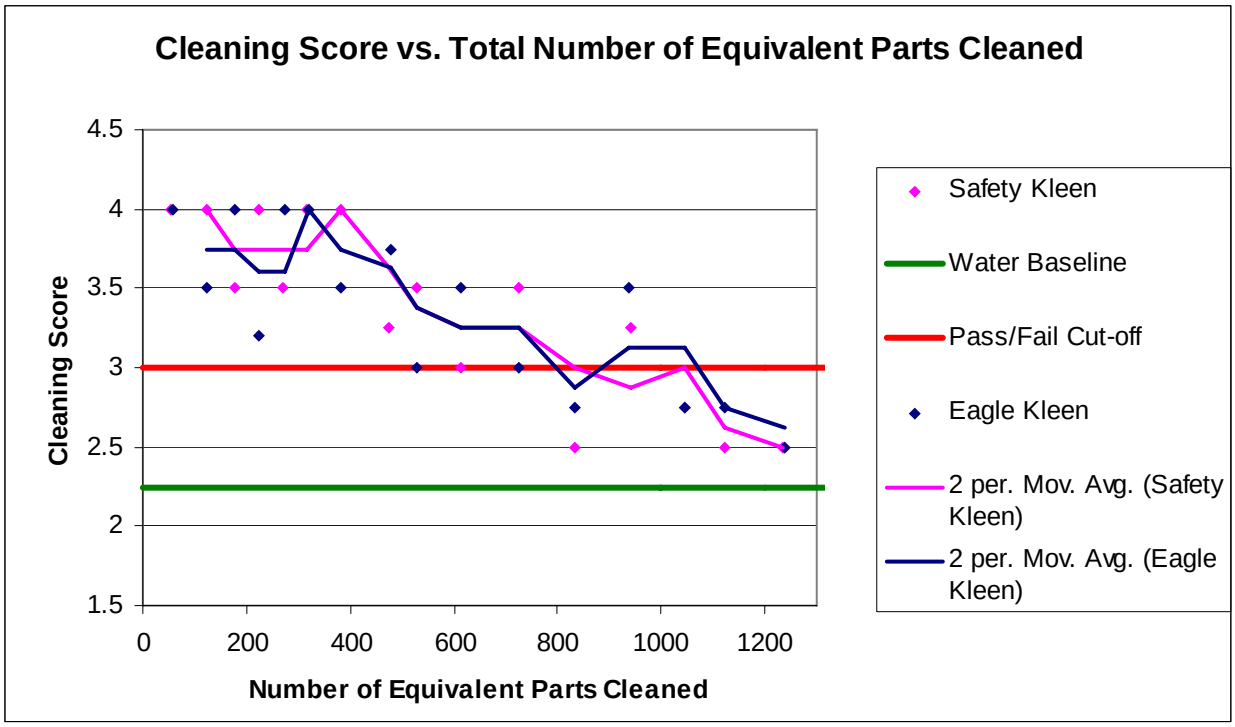

Figure 8. Cleaning Score for Equivalent Number of Parts Cleaned Using the Two Degreasers

3.3 Hill AFB Testing

The evaluation at Hill AFB was conducted for one week, over the period September 12-16, 2005. The GTE shop degreases parts on a routine basis and installed an automated RAMCO system for degreasing, rinsing, and drying operations (see Figure 9). The shop's RAMCO degreasing system was used for testing two types of degreasers. The normal cleaning tank was filled with Turco 6849 (prepared by diluting 10 gallons of Turco 6849 to approximately 65 gallons of water). Another tank was filled with Eagle Kleen™, which was prepared without dilution (see Figure 10).

Both tanks initially were heated to 145°F. However, the optimum temperature for Eagle Kleen™ was not known, so the bath temperature for Eagle Kleen™ was controlled at approximately 90°F in an initial series of tests and 120°F in a second series. Results indicated increasing the temperature did not significantly improve performance; however, the change in temperature dramatically increased complaints related to odor. A flow diagram illustrating the cleaning process during the demonstration is shown in Figure 11.

Many of the parts that were degreased in the GTE shop had heavy carbon contamination. All had some degree of oil contamination and some also had grease contamination. During the evaluation, parts were processed in the same manner as in normal operations. Similar types of parts with comparable levels of contamination were placed into steel baskets and prepared for cleaning. Each basket of parts was inserted into a cleaning tank for approximately 60 minutes, with mild agitation by circulating fluid. Ultrasonic energy was applied for approximately one minute in the Turco 6849 bath. No ultrasonic energy was used in the Eagle Kleen™ bath or its rinse tank. After the cleaning step, the baskets were transferred to a rinse tank for 30 minutes, and then dried for 30 minutes in air heated to 240°F.

Figure 9. RAMCO Cleaning Line at Hill AFB GTE Shop

Figure 10. Eagle Kleen™ Degreaser Bath

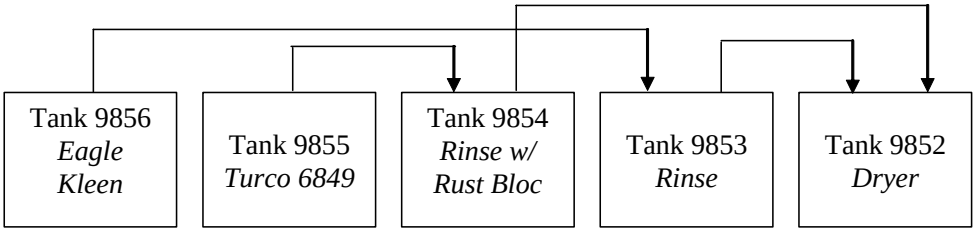

Figure 11. Process Flow Diagram for Cleaning in the Hill AFB Gas Turbine Engine Shop

The number of baskets processed through each bath was recorded and the shop work load was divided equally between the two degreasers. Cleaning times were staggered to allow use of only one dryer. After drying, the parts were allowed to cool and were examined side by side. Several cleaning operators in the GTE Shop were asked to evaluate the performance of Eagle Kleen™ against Turco 6849. The operators reported that Turco 6849 removed the oil and grease, and nearly all the carbon deposits. In contrast, Eagle Kleen™ removed part of the oil and grease, but was not effective at removing heavy carbon contamination. A comparison of heavily-carbon contaminated parts, after cleaning with the two degreasers, is shown in Figure 12.

Figure 12. Example of Air Inlet Housing Prior to Cleaning (left), After Degreasing with Turco 6849 (center), and After Degreasing with Eagle Kleen™ (right)

In general, tests at the GTE shop revealed the following about Eagle Kleen™:

- Removal of oil and grease tended to be equal to or slightly inferior to Turco 6849 degreaser for lightly soiled parts.
- Eagle Kleen™ was not effective at cleaning carbon-contaminated parts.
- Parts cleaned using Eagle Kleen™ required more manual cleaning than Turco 6849 after the 30-minute degreasing step for all types of contamination.
- The strong odor of Eagle Kleen™ was objectionable to the operators. Operation without a ventilation system caused some operators to complain of headaches, irritation of mucous membranes, and light headedness.

After each basket of parts was processed, the cleanliness of each part was ranked on a scale from 0 to 5, taking into account the appearance and feel (greasy/clean) (see Figure 13). Depending on the number of parts requiring reprocessing, the scores were reduced accordingly (see Figure 14). Parts that were degreased with Eagle Kleen™ required reprocessing on a consistent basis. In total, it was concluded based on these test results that Turco 6849 outperformed Eagle Kleen™.

Due to the odor issue and Eagle Kleen™'s ineffectiveness at cleaning heavy carbon-contaminated parts, the degreasing team decided to suspend the demonstration at the close of the first week (September 16, 2005). Better ventilation, or respirators, would be required for future use of Eagle Kleen™ in this operation.

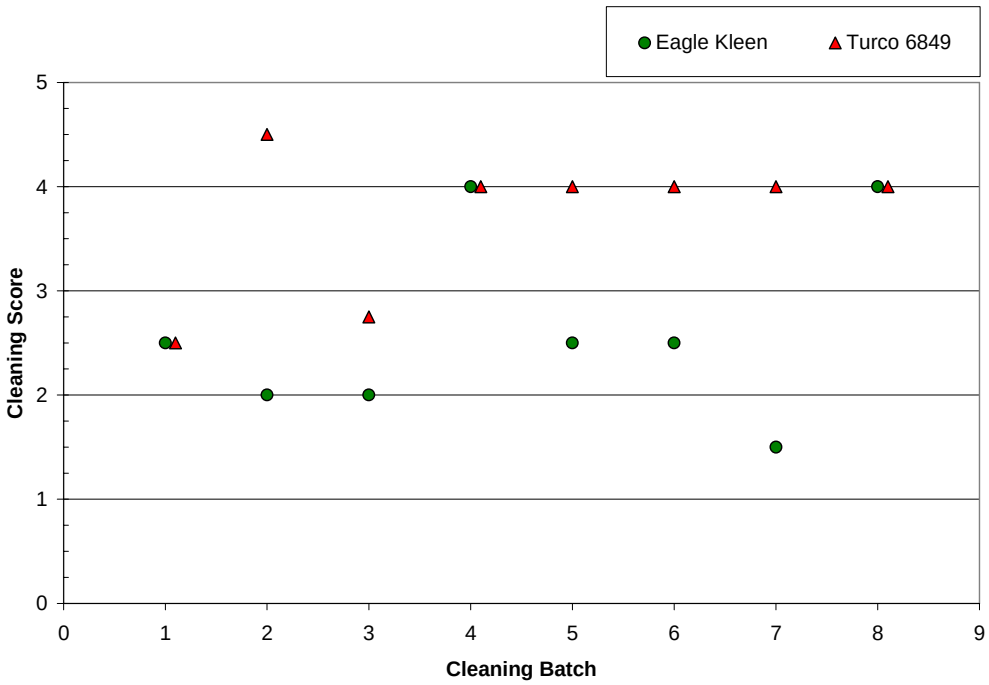

Figure 13. Overall Cleaning Score

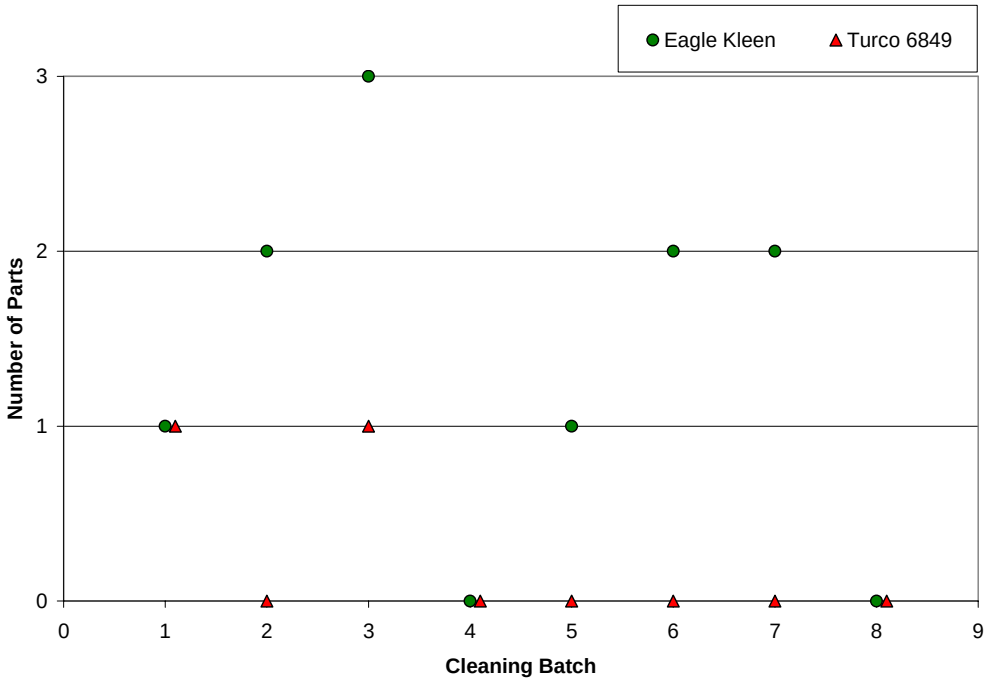

Figure 14. Parts Requiring Reprocessing

After the suspension of Eagle Kleen™ operations for the main degreasing area in the GTE shop, the cleaning engineer indicated that the shop may be interested in considering Eagle Kleen™ for non-carbon contaminated cleaning applications. He requested a quote form Hydra-Tone for a three-step cleaning system to replace a PD-680 solvent cleaning application. HTCI and the shop engineer independently pursued this option.

Additionally, the Plating Shop at Hill AFB expressed interested in considering Eagle Kleen™ for replacing certain alkaline cleaning baths. The SMI materials compatibility data (per MIL PRF 87937D) indicated that the fluid should be compatible with these application needs. The plating shop also explored this option with HTCI.

The tests in a standard parts washer, without ultrasonic energy, indicated that Eagle Kleen™ was effective in removing oil and grease but not for removing heavy carbon deposits.

Subsequent tests in November 2005 and March 2006 indicated that such carbon deposits could be removed when a diluted solution (5% Eagle Kleen™ and 95% water) were used in an appropriate cleaning bath.

After these tests, the cleaning engineer in the GTE Shop expressed renewed interest. A major driving force was economics. The conventional alkaline cleaner contains chelating agents. Because of the chelating agent's deleterious affect on metals precipitation at the Hill AFB IWTP plant, the IWTP staff required that all degreaser solutions and rinse water be drummed, and transported to their site for specialized treatment. The expense of pursuing this path made a non-chelating agent degreaser especially attractive, so an Engineering Cost Assessment was performed (see Section 4.0).

3.4 Ransohoff Testing

Once primary site demonstration testing was done, additional testing was conducted at Ransohoff, Inc., a manufacturer of parts cleaners, located in Cincinnati, OH, to determine whether a diluted solution of Eagle Kleen III in an ultrasonic-enhanced parts washer could remove carbon deposits and/or oil and grease from parts. First, on November 2, 2005, a series of cleaning tests were performed on condemned parts obtained from Hill AFB using a 100% solution of Eagle Kleen III. The tests were run using an unagitated 5.6-gal Ransohoff HT-1212 heated tank operated at 25 kHz with a 600-watt density. Initially, the results indicated showed poor de-carbonizing. However, after dilution to 5% and operation at 120ºF, good (in some cases dramatic) carbon removal was achieved with immersion times ranging form 5 to 15 minutes.

Then, on March 14, 2006, tests were conducted on parts obtained from Hill AFB to gather additional data operated at similar conditions. The parts were too large to fit in the small lab unit, so an available "Grease Monkey" CLASSIC 3523 Blackstone~Ney unit (sold by Ransohoff, Inc.) was adapted for the tests (see Figure 15). The unit dimensions were 35 inches long, 23 inches wide, and 13 inches deep, with built-in 40 kHz transducers, and was operated at 25 kHz and a 2000-watt density. However, operation at a more aggressive frequency (25 kHz) was desired, so two portable, rectangular transducers were inserted into the unit. The unit was filled with distilled with 120ºF water, and two gallons of Eagle Kleen III were added and allowed to warm up (see Figure 16). Results with 5% Eagle Kleen™ at 120ºF and 15 minutes immersion times indicated dramatic removal of encrusted grease and carbon for some parts; however, the dilute Eagle Kleen™ solution was not effective at removing oils and grease.

Figure 15. "Grease Monkey" CLASSIC 3523 Blackstone~Ney Parts Washer

Figure 16. Parts Washer after Eagle Kleen™ Added

In an attempt to allow a better evaluation of the effectiveness of Eagle Kleen™ a fan assembly along with a different part (some type of housing) were submerged and suspended over the solution (see Figure 17). The fan assembly after 15 minutes showed a dramatic removal of the black coating. On the front, there is a clear demarcation of where the fan was immersed in the cleaner, and in this area the surface looks very clean (see Figure 18). The area cleaned is free of the black deposit. Another view, showing the dramatic cleaning achieved, is shown in Figure 19. On the rear side, where the fins are observed, it shows the surface substantially cleaned, but not totally (see Figure 20). This photo also shows an area that was not submerged, and is still completely covered with the dark brown/black substance.

Figure 17. Parts Partially Suspended in Cleaning Bath

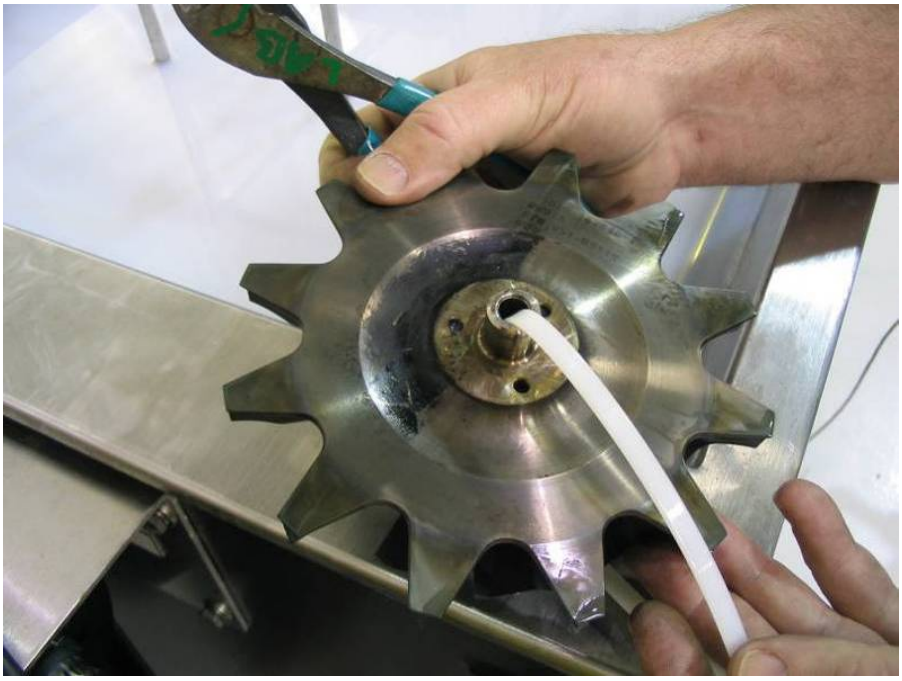

Figure 18. Top View of Fan Assembly After Cleaning

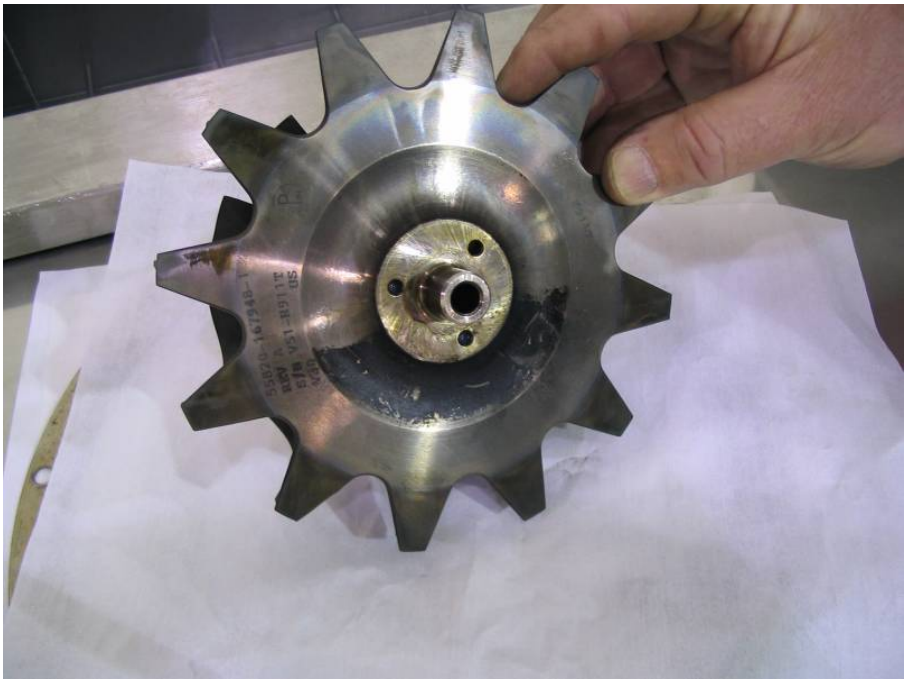

Figure 19. Front View of Fan Assembly after Cleaning

Figure 20. Rear View of Fan Assembly

In general, tests at the Ransohoff facility included the following:

- A dilute solution of Eagle Kleen III can be used in an ultrasonically enhanced parts washer to remove a substantial portion of carbon deposited on typical GTE parts.
- Cleaning appeared good, but was not complete. A longer immersion time or hand cleaning might be required for complete carbon removal.
- The solution was not effective in removing grease/heavy oils.
- Eagle Kleen III odor was detectable, but not overpowering even with the top off the cleaning unit. Odor when the top was open only a few minutes per hours would be less. However, some type of ventilation would probably be required in many applications.

A tank arrangement for Hill AFB GTE-parts cleaning was devised based on the test results. It consisted of the five-step degreasing and carbon removal set up presented in Table 13. Russ Markesbery indicated that he had proposed a similar tank configuration to Jeff Powell at Hill AFB. Photos of the Agisonic AG-30 are provided on Figures 21 and 22.

Table 13. Aqueous Cleaning Tank Operating Conditions

Step	Aqueous Concentration	Temperature (ºF)	Duration (minutes)	Other
1	100% Eagle Kleen III	120	30 – 60	Expect bath life: 4 to 8 weeks
2[(a)]	5% Eagle Kleen III in Water	120	15	Ultrasonics
3	4% Rust Bloc in Water[(b)]	150	30	
4	Water	150	30	
5	Dryer	200	60	

(a) All other units could employ the existing RAMCO line cleaning tanks. The ultrasonics would be conducted in a new 80-gallon, Blackstone~Ney Agisonic AG-30 agitated, parts cleaner.

(b) Rust Bloc may not be needed, but because Hill AFB is comfortable with its use, it was included in this process configuration.

Figure 21. Agisonic AG-30 Shown from Front
(Note: the lid is set off to the right of the tank)

Figure 22. Agisonic AG-30 Looking Down into Basket
(Note: the tray is in the parts-loading position)

4.0 TASK 3 – ENGINEERING COST ASSESSMENT

Task 3 is an Engineering Cost Assessment (ECA) designed to evaluate the functional, financial, and Environmental, Safety, and Occupational Health (ESOH) performance of Eagle Kleen™ based on information obtained at Hill AFB. The calculations were made using degreaser costs from Hill AFB and HTCI, equipment costs from Ransohoff Inc., and engineering estimates. (Because the Eagle Kleen™ degreaser was not found to be an acceptable replacement for the hydrocarbon solvent degreaser, no ECA was prepared for Robins AFB.)

The baseline ESOH and cost data for the conventional, alkaline immersion degreaser were collected by Battelle engineers during the Hill AFB demonstration task. Operating costs (labor, materials, energy) for industrial processes were difficult to collect, and were assumed to be the same for the conventional and Eagle Kleen™ degreasers.

Similarly, environmental treatment costs associated with a small operation or a process within a larger industrial facility also were difficult to obtain; engineering estimates were used to estimate wastewater collection, handling, and treatment for the conventional degreaser. (Note: the alkaline cleaner contains chelating agents and requires special collection and treatment, whereas the chelating-agent free Eagle Kleen™ can be discharged directly to the IWTP.)

Most industrial facilities track environmental costs at an aggregate level and rarely for a specific operation. The safety and occupational health profile of Eagle Kleen™ indicated ventilation was required to provide a similar profile as the alkaline cleaner. The ESOH evaluation included review of the material and safety data sheets (MSDS) and the physical and chemical properties of the degreasers. The assessment of their workplace impacts, based on aquatic toxicity, corrosivity, inhalation, skin contact, and flammability during operating conditions of the cleaning process, indicated similar impact should be expected once ventilation was installed.

The ECA included a list of assumptions, and appropriate extrapolations are documented for data gaps from the baseline data collection process. The assumptions were based on direct interviews with the shop floor workers and supervisors.

The ECA assumes that three of the four cleaning tanks, and the dryer, from the RAMCO system will be utilized in the new system. Only one new unit and ventilation for the two cleaning tanks, are included in the capitol costs estimate. The assessment also includes a payback period for transitioning from conventional degreasers to Eagle Kleen™.

The assessment factors are noted in Table 14. The cost for handling, treatment and disposal of the spent alkaline cleaner and spent rinse could not be determined and was estimated. Because this cost was a major contributor to the positive cash flow projected, this value should be determined. A sensitivity analysis was performed varying the drummed waste handling and disposal fee (see Table 15). Even with a relatively conservative $100/drum handling fee, the payback is only 3.2 years. Under the base case, where a rate of $150/drum was assumed, the payback is 2.2 years.

Table 14. ECA Assessment Factors

No.	Item	Information
1	Project Title	Replacement of Alkaline Cleaner with Chelating Agent-Free Biobased Cleaner
2	Project Description	This effort will qualify a new cleaning tank and alternative degreaser in the GTE shop. Implementation will improve degreasing and carbon removal performance, reduce waste generation, eliminate the discharge of chelating agents to the IWTP, and lower operating costs. Project duration is one-year and will start with the requirements definition phase that will include a 1-week on-site technology verification task that will help develop the design package for the new degreaser system. A new degreasing tank with an improved ultrasonic generator will be integrated into the existing RAMCO small-parts degrease line in the GTE shop. The equipment, installation in Building 238 at Hill AFB, training, and performance evaluation will be completed during FY07.
3	Justification	This project could provide significant reductions in waste generation: 21K gal/yr. of wastewater would be avoided. Also chemical and water usage would be reduced: 1100 gal/yr. of Turco 6849 alkaline cleaner, and 21K gal/yr. of fresh water. Waste-waters from the current operations contain chelating agents and require transport to the IWTP in carboys and special treatment. Substitution with Eagle Kleen III degreaser would eliminate the special handing requirements allowing the rinse water to be discharged directly to the industrial sewer. This will reduce handling labor and paperwork while ensuring un-interrupted metals precipitation in the IWTP. Additionally, the longer service life possible with Eagle Kleen™ will allow less frequent cleaning of the tanks, labor cost savings, and reduce degreaser chemical requirements and costs. Drivers: TRI, performance, and cost reduction.
4	Current Process Description	Small GTE parts are subjected to 1 hour of degreasing in two stages (30 minutes each) of hot degreasing (145ºF) using Turco 6849 alkaline cleaner. Each part is hand cleaned after the first stage. The parts are then rinsed for 1 hour in two hot (145ºF) aqueous rinse tanks (30 minutes each) fortified with Rustbloc rust inhibitor. The degreased parts are then sent to a dryer. In the current degreasing and rinsing operations, significant quantities of alkaline cleaner and inhibitor are consumed. Turco 6849 contains several compounds listed as hazardous materials.
5	Implementation Project Description and Budget	Total Project Cost: $200K Requirements Definition & Design Package: $15K Technology Verification (1 week) using Rented Equipment (on actual parts): $40K Technology Transfer: $100K (based on $68K of equipment and $22K for installation) Technology Validation 1-month (cleaning performance): $45K. Description of tasks: • The design package and specifications will be developed as part of the Re-quirements definition phase of the task. A test plan will be produced for 1-week onsite technology verification on serviceable parts. The results will help develop the design package for a full scale unit to be installed in the GTE shop. The 1-week technology verification will demonstrate the effectiveness of the integrated degreasing equipment and solvent. • Technology Transfer includes the purchase and installation of a new ultrasonic parts-cleaner, reconfiguration of the existing RAMCO cleaning and rinse tanks, and the addition of a ventilation system. It also includes training and integration of the new unit into the shop-floor cleaning operations. • Technology Validation will follow the operation of the equipment to ensure it is meeting the cleaning specifications. It will include a final report outlining the system performance.

Table 14. ECA Assessment Factors (Continued)

No.	Item	Information
6	Costs and Savings*	Current Annual Environmental Costs (~31K gal/yr. wastewater treated @ $15/Kgal; 570 drums/yr of drummed waste @ $150/drum) = $86K New Annual Environmental Costs (~10K gal/yr. wastewater treated @ $15/Kgal; 18 drums/yr of drummed waste @ $150/drum) = $3K Annual Environmental Savings = $83K Current Annual Operational Costs (29 Kgal/yr process makeup water @ $1.16/Kgal, 1100 gal Turco 6849 @ $13.75/gal, 8 gal of Rustbloc @ $17.40/gal) = $15,300 + solvent remaking time (4 hrs/week @$100/hr over 52 weeks/year) = $20,800 for a total of $36K. New Annual Operational Costs (8 Kgal/yr process makeup water @ $1.16/Kgal, 2300 gal Eagle Kleen III @ $9.98/gal, and 8 gal of Rustbloc @ $17.40/gal) = $23,100 + solvent remaking time (4 hrs every 4 weeks @ $100/hr over 52 weeks/yr) = $5,200 for a total of $28K Annual Operational Cost Savings = $8K Total Investment = $200K Total Savings = $83K + $8K = $91K/year Payback = Total Project Cost ($200K)/Savings ($91K) = 2.2 years Note: If Equipment is not needed, then payback is immediate

Table 15. ECA Sensitivity Analysis

Costs of Wastewater Handling and Treatment ($/drum)	Savings ($K/year)	Payback Period (years)
100	63	3.2
150	91	2.2
200	118	1.7
250	146	1.4

5.0 CONCLUSIONS AND RECOMENDATIONS

5.1 Conclusions

Based on the laboratory evaluation, field testing, and engineering cost assessment, the following conclusions were drawn:

1. Concentrated Eagle Kleen™:
   - The concentrated degreaser passed substantially all the MIL-PRF 87937D requirements for water dilatable degreasers, including physical property, corrosion, and the all-important hydrogen embrittlement test. However, it failed the heat stability and stressed-plastics crazing requirements, preventing it from being listed on the qualified fluids list.
   - In cleaning applications that do not involve acrylic or polycarbonates, the fluid may be used on a case-by-case basis. Eagle Kleen™ is a very aggressive degreaser and can dissolve certain plastics and elastomers. Attention must be paid to proper selection of construction material and PPE.
   - The fluid after degreasing leaves a slimy, slippery surface that must be rinsed with water.
   - The fluid can effectively remove oil and grease, but without ultrasonic energy is not effective at removing heavy carbon contamination.
   - The degreaser has a noticeable odor that some operators found offensive. Operation at elevated temperature (120ºF) dramatically increased complaints related to odor.
   - Ventilation during use is required.

2. Dilute Eagle Kleen™:
   - A dilute solution of Eagle Kleen III can be used in an ultrasonically enhanced parts washer to remove a substantial portion of carbon deposited on typical GTE parts. Cleaning appeared good, but was not complete. A longer immersion time or hand cleaning might be required for complete carbon removal.
   - The solution was not effective in removing grease/heavy oils.
   - The dilute degreaser in not slippery.
   - The odor of the dilute solution was detectable, but less severe an issues as with concentrated Eagle Kleen™.
   - Ventilation during use is required.

3. The economic cost assessment indicated a payback of 2.7 years with a positive NPV, indicating there are economic benefits to be gained when using Eagle Kleen™.

5.2 Recommendations

1. The degreaser should be evaluated in a real-world test using a suitable ultrasonically enhanced parts washer equipped with a suitable ventilation system. If cleaning results are verified, the degreaser should be considered for implementation.
2. The cost of handling, treatment, and disposal of spent alkaline cleaner and spent rinse should be verified, and the costs/savings/payback projections should be re-examined for specific applications.

6.0 REFERENCES

Anonymous. 1997. MIL-PRF-87937D “Cleaning Compound, Aerospace Equipment” (See Appendix A for a full copy.)

Tam, T.M., et al. 1993. “Evaluation Performance Test Methods for Aqueous Cleaner,” Plating and Shop Finishing, December, pp. 58-62.

Cohen, L. E. 1987. “How Clean is Your “Clean” Metal Surface?,” Plating and Surface Finishing, November, pp. 58- 61.

U. S. EPA. 1999. EPA-745-R-99-004, “33/50 Program The Final Record,” March, p. 2.

Appendix A

MIL-PRF-87937D “Cleaning Compound, Aerospace Equipment”

INCH-POUND

MIL-PRF-87937D
24 September 2001
SUPERSEDING
MIL-PRF-87937C
14 August 1997

PERFORMANCE SPECIFICATION

CLEANING COMPOUND, AEROSPACE EQUIPMENT

This specification is approved for use by all
Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification establishes the requirements for biodegradable, water dilutable, environmentally safe cleaning compounds for use on aerospace equipment to include aircraft, aerospace ground equipment (AGE) and AGE engines.

1.2 Classification. The cleaning compounds covered by this specification will be of the following types.

Type I -Terpene Based, Solvent Emulsion, Water Dilutable Cleaning Compound

Type II -Water Dilutable Cleaning Compound

Type III -Gel-Type Cleaning Compound

Type IV -Heavy Duty, Water Dilutable Cleaning Compound

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3 and 4 of this standard. This section does not include documents cited in other sections of this standard or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements documents cited in sections 4 and 5 of this standard, whether or not they are listed.

2.2 Government Documents.

2.2.1 Specifications, standards, and handbooks. The following specifications, standards and handbooks form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of these documents will be those listed in the issue of the Department of Defense Index of Specifications and Standards (DoDISS) and supplement thereto, cited in the solicitation (see 6.2).

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Code (68) DET 3, WR-ALC/AFTT, BUILDING 1621-K, 2261 HUGHES AVE STE 123, LACKLAND AFB TX 78236-9823, by using the Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A
FSC 6850

SPECIFICATIONS

FEDERAL

A-A-58054	Abrasive Mats, Non-Woven, Non-Metallic
TT-I-735	Isopropyl Alcohol
TT-P-2760	Primer Coating, Polyurethane, Elastomeric, High-Solids.
PPP-P-704	Pails, Metal: (Shipping, Steel, 1 through 12 gallons)

DEPARTMENT OF DEFENSE

MIL-PRF-2104	Lubricating Oil, Internal Combustion Engine, Combat/Tactical Service
MIL-PRF-5425	Plastic, Sheet, Acrylic, Heat Resistant
MIL-C-5541	Chemical Conversion Coatings on Aluminum and Aluminum Alloys.
MIL-A-8625	Anodic Coatings, For Aluminum and Aluminum Alloys
MIL-G-21164	Grease, Molybdenum Disulfide, For Low and High Temperatures, NATO Code Number G-353
MIL-PRF-22750	Coating, Epoxy, High-Solids
MIL-PRF-23377	Primer Coatings: Epoxy, High-Solids.
MIL-PRF-25690	Plastic, Sheets And Formed Parts, Modified Acrylic Base, Monolithic, Crack Propagation Resistant
MIL-DTL-81381	Wire, Electric, Polyimide-Insulated, Copper or Copper Alloy
MIL-PRF-81733	Sealing and Coating Compound, Corrosion Inhibitive
MIL-PRF-83282	Hydraulic Fluid, Fire Resistant, Synthetic Hydrocarbon Base, Aircraft, NATO Code Number H-537.
MIL-P-83310	Plastic Sheet, Polycarbonate, Transparent
MIL-DTL-83488	Coating, Aluminum, High Purity
MIL-PRF-85285	Coating: Polyurethane, High Solids
MIL-PRF-85582	Primer Coatings: Epoxy, Waterborne.

STANDARDS

FEDERAL

EPA-600-4-90-027	Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms
FED-STD-141	Paint, Varnish, Lacquer and Related Materials: Methods of Inspection, Sampling and Testing
FED-STD-313	Material Safety Data Sheets, Preparation and the Submission of
FED-STD-595	Colors Used In Government Procurement

(Unless otherwise indicated, copies of the above specifications, standards, and handbooks are available from the Standardization Document Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094.)

2.2.2 Other Government documents, drawings, and publications. The following other Government documents form a part of this specification to the extent specified herein. Unless otherwise specified, the issue should be that in effect on the date of the solicitation.

CODE OF FEDERAL REGULATIONS

40 CFR	-	Protection of Environment
49 CFR	-	Transportation

(Application for copies should be addressed to Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.)

2.3 Other publications. The following non-government documents form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of the documents which are DoD adopted will be those listed in the issue of the DoDISS specified in the solicitation. Unless otherwise specified, the issues of documents not listed in the DoDISS will be the issue of the non-government documents which is current on the date of the solicitation.

AMERICAN SOCIETY FOR TESTING AND MATERIALS

ASTM STANDARDS

A 153	Specification for Zinc Coating (Hot Dip) on Iron and Steel Hardware (DoD Adopted)
D 56	Test Method for Flash Point by Tag Closed Tester (DoD Adopted)
D 92	Test Method for Flash and Fire Points by Cleveland Open Cup (DoD Adopted)
D 93	Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester (DoD Adopted)
D 235	Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent) (DoD Adopted)
D 1193	Specification for Reagent Water (DoD Adopted)
D 2240	Test Method for Rubber Property - Durometer Hardness (DoD Adopted)
E 70	Test Method for pH of Aqueous Solutions with the Glass Electrode (DoD Adopted)
F 483	Test Method For Total Immersion Corrosion Test for Aircraft Maintenance Chemicals (DoD Adopted)
F 484	Test Method for Stress Crazing of Acrylic Plastics in Contact with Liquid or Semi-Liquid Compounds (DoD Adopted)
F 485	Test Method for Effects of Cleaners on Unpainted Aircraft Surfaces
F 502	Standard Test Method for Effects of Cleaning and Chemical Maintenance Materials on Painted Aircraft Surfaces.
F 519	Test Method for Mechanical Hydrogen Embrittlement Testing of Plating Processes and Aircraft Maintenance Chemicals
F 1104	Test Method for Preparing Aircraft Cleaning Compounds, Liquid Type, Water Base, for Storage Stability Testing
F 1110	Test Method for Sandwich Corrosion Test
F 1111	Test Method for Corrosion of Low-Embrittling Cadmium Plate by Aircraft Maintenance Chemicals

(Application for copies should be addressed to the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken PA 19428-2959.)

SOCIETY OF AUTOMOTIVE ENGINEERS

SAE STANDARDS

AMS QQ-A-250	Aluminum and Aluminum Alloy, Plate and Sheet
AMS 1640	Corrosion Removing Compound, Prepaint, For Aircraft Aluminum Surfaces.
AMS 2410	Plating, Silver, Nickel Strike, High Bake
AMS M-3171	Magnesium Alloy, Processes for Pretreatment and Prevention of Corrosion on
AMS 3204	Rubber, Synthetic Low-Temperature Resistant 25-35 (DoD Adopted)
AMS 3209	Chloroprene (CR) Rubber, Weather Resistant, 65-75 (DoD Adopted)
AMS 4377	Sheet and Plate, Magnesium Alloy, 3.01A-1.0Zn-0.20Mn (AZ31B-H24) Cold Rolled, Partially Annealed (DoD Adopted)
AMS 5046	Sheet, Strip, and Plate, Carbon Steel (SAE 1020 and 1025) Annealed
AMS S-8802	Sealing Compound, Temperature-Resistant, Integral Fuel Tanks and Fuel Cell Cavities, High-Adhesion
AMS T-9046	Titanium and Titanium Alloy, Sheet, Strip and Plate

(Application for copies should be addressed to the Society of Automotive Engineers, Inc., 400 Commonwealth Drive, Warrendale PA 15096.)

AMERICAN IRON AND STEEL INSTITUTE

AISI STANDARDS

AISI 4340 High Strength, Low Alloy Steel

(Application for copies should be addressed to American Iron and Steel Institute, 1133 15th St N.W. Suite 300, Washington DC 20005.)

(Industry association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

2.4 Order of precedence. In the event of a conflict between the text of this specification and a reference cited herein, the text of this specification will take precedence.

3. REQUIREMENTS

3.1 Qualification.

3.1.1 Qualification (Initial). The cleaning compound furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein and has been listed or approved for listing on the applicable Qualified Products List (QPL).

3.1.2 Re-Qualification (Periodic). The cleaning compound furnished under this specification shall be retested or recertified by the qualifying activity at least every three years for the product to remain listed on the QPL. Re-Qualification testing shall be accomplished on any qualified cleaning compound for which a using activity issues a valid deficiency report. The cleaning compound shall also be subject to re-qualification testing for any change in chemical formulation, material, process, or procedure in manufacturing the cleaning compound. Upon periodic re-qualification, any cleaning compound which does not conform to all the qualification tests specified herein shall be removed from the QPL.

3.1.3 Qualifying activity. The activity responsible for specification qualification and the QPL is the Air Force Petroleum Office, Product Engineering Branch, San Antonio TX. Activity mailing address is: DET 3 WR-ALC/AFTT, BUILDING 1621-K, 2261 HUGHES AVE STE 123, LACKLAND AFB TX 78236-9823.

3.2 Materials. The composition and formulation of the cleaning compound shall be optional with the manufacturer within the restrictions specified herein.

3.2.1 Acceptable materials.

3.2.1.1 Type I. Type I compounds shall contain terpene hydrocarbons as specified in Table I. Certification from the manufacturer is required on the percentage of total terpenes contained in the cleaning compound. The terpene hydrocarbons used shall be of a high grade with no extraneous materials.

3.2.1.2 Type II, Type III, and Type IV. Types II, III, and IV compounds shall consist of one or more of the following: Surfactants, adjuvant solubilizers for organic soils such as greases and oils, alkaline builders, water conditioning agents and corrosion inhibitors.

3.2.2 Unacceptable materials. The cleaning compound shall not contain any hazardous compounds as defined in 40 CFR 261, toxic pollutants in 40 CFR 301, nor hazardous air pollutants in 40 CFR 63 (see 4.6). The cleaning compound shall not contain any chemical listed by the current report of known carcinogens of the National Toxicology Program (NTP). The cleaning compound shall not contain detectable amounts of any of the following: abrasives, chromates, cadmium, lead, mercury, phenols,

cresols, ketones, chlorinated compounds or ozone depleting substances (ODS), except where specified within this specification. The following materials are unacceptable unless they are being used as an essential active ingredient in the cleaner: sodium chloride, urea, sodium sulfate, nitrites, nitrates, sucrose or any sugars. Types II, III, and IV compounds shall contain no terpene hydrocarbons or other hydrocarbon solvents.

3.3 Toxicity. The cleaning compound shall have no adverse effect on the health of personnel or the environment when used for its intended purpose and with proper personal protective equipment (when required). The product shall be evaluated for aquatic toxicity with a 96 hour Fathead minnow (pimephales promelas) bioassay and a 48 hour Ceriodaphnia dubia bioassay in accordance with Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, EPA/600/4-90/027. The percent survival at 1, 10, 50, and 100 ppm shall be reported for both organisms.

3.3.1 Formulation. The manufacturer shall submit to the qualifying activity a certified statement that provides the identity and percentage by weight of each ingredient in the cleaning compound, including solvent, using a readily recognizable chemical name and CAS number. Trade names alone shall not be considered satisfactory. All proprietary information shall be protected as such.

3.3.1.1 Type I. The manufacturer shall submit to the qualifying activity the chemical name of each terpene used in the formulation including its CAS number and range of values in percent by weight of the formulation. The manufacturer shall also submit test procedures used to verify the terpene percentages within these ranges. All procedures shall be subject to approval by the qualifying activity.

3.3.2 Material safety data sheet (MSDS). The manufacturer shall submit to the qualifying activity an MSDS for the finished product and for each component in the finished product. The MSDS shall be prepared in a 16 part format in accordance with the latest revision of FED-STD-313.

3.3.3 Toxicological data. The manufacturer shall submit to the qualifying activity a copy of pertinent toxicological data/information (see 4.6) for their product.

3.3.4 Biodegradability. The supplier of the cleaning compound shall furnish certification from the surfactant manufacturers that the surfactants are readily biodegradable in accordance with 40 CFR, Part 796, Subpart D. Biodegradability testing shall be accomplished as specified in paragraph 4.5.22 on the finished product by an independent laboratory approved by the qualifying activity. Biodegradability on the finished product shall be determined over 28 days by the Shake Flask Method monitored by analysis of Total Organic Carbon (TOC). The Type I compound shall meet the requirement of a minimum of 75% biodegradable and Types II, III, and IV compounds shall meet the requirement of a minimum of 85% biodegradable at the end of the 28 day period.

3.4 Compositional assurance. The cleaning compound shall be tested for nonvolatile matter as specified in paragraph 4.5.1. The concentrated cleaning compound and a 10% solution of the cleaning compound in distilled water shall be tested for pH as specified in paragraph 4.5.3. Results of these tests as well as an infrared spectrogram of the nonvolatile matter (see 4.8.2) and a gas chromatogram (see 4.8.1 for Type I only) shall be recorded by the qualifying activity for use in conformance inspections (see 4.3). Conformance inspection results for nonvolatile matter shall not differ by more than 2 percent absolute from the recorded value. Conformance inspection results for pH shall not differ by more than 1 pH unit from the recorded value. Conformance inspection infrared spectrograms and gas chromatograms shall show no significant difference when compared to the original qualifying spectrogram.

3.5 Chemical properties.

3.5.1 Chemical requirements. The cleaning compound shall meet the requirements listed in Table I.

3.5.2 Residue rinsibility. When a freshly prepared solution of the cleaning compound is tested in accordance with 4.5.4, it shall not leave any residue or stains. A freshly prepared solution is defined as one being prepared no longer than 30 minutes prior to testing. The weight change shall be not greater than that obtained with standard hard water tested under the same conditions.

3.6 Physical properties (All types unless otherwise noted).

3.6.1 Heat stability. The concentrated cleaning compound, when tested in accordance with 4.5.5, shall show no marked color change or precipitation and shall not corrode or stain the AMS 5046 (SAE 1020) steel strip (a slight darkening of the steel strip shall not be objectionable). Layering or separation shall constitute failure if it does not return to its original homogeneous state upon cooling.

3.6.2 Cold stability. The concentrated cleaning compound shall return to its original homogeneous condition when tested in accordance with 4.5.6.

3.6.3 Rheology (Type III only).

3.6.3.1 Consistency. When tested as specified in 4.5.24, the concentrated cleaning compound shall flow between 10 and 20 centimeters in 10 seconds. The product shall also exhibit rheology which enables it to meet the sprayability requirement.

3.6.3.2 Sprayability. The concentrated cleaning compound, when dispensed at 45 psig and tested in accordance with 4.5.25, shall give satisfactory spray characteristics and deposit a uniform layer on a vertical surface 3 feet away from the nozzle.

3.7 Effect on metals (All types unless otherwise noted).

3.7.1 Hydrogen embrittlement. When tested in accordance with 4.5.9, the concentrated cleaner (all types) and a 10% solution of the cleaner (Types I, II and IV only) in distilled water shall not cause hydrogen embrittlement of cadmium plated or IVD aluminum coated AISI 4340 steel.

3.7.2 Total immersion corrosion. When tested in accordance with 4.5.10 (ASTM F 483), the concentrated cleaning compound (all types) and a 10% solution of the cleaning compound (Types I, II and IV only) in distilled water shall not show any indication of staining, etching, pitting, or localized attack on any of the panels, or cause a weight change of an average of three (3) test panels greater than that shown in Table II. A slight discoloration of the panels shall not be objectionable. The cleaning compound shall not layer or separate for the duration of the test.

3.7.3 Low-embrittling cadmium plate corrosion. Steel panels coated with low-embrittling cadmium plate immersed in the concentrated cleaning compound (all types) and a 10% solution of the cleaning compound (Types I, II and IV only) in distilled water shall not show a weight change greater than 0.14 mg/cm^2 for 24 hours when tested in accordance with 4.5.11.

3.7.4 Effects on unpainted metal surfaces. The concentrated cleaning compound (Type III only) and a 10% solution (Types I, II and IV) of the cleaning compound in distilled water shall not cause streaking, stains or other deposits that cannot be easily removed with water when tested in accordance with 4.5.12.

3.7.5 Sandwich corrosion. When tested in accordance with 4.5.16, the concentrated cleaner (all types) and a 10% solution (Types I, II and IV only) shall show no corrosion in excess of that shown by control test coupons in ASTM D1193, Type IV, reagent water.

3.7.6 Wet adhesion tape test (Types II and IV). A ten (10) percent solution of the cleaning compound, when used as directed, shall remove soil from a painted surface in preparation for repainting such that paint applied after cleaning with the compound shall adhere to the surface when tested in accordance with 4.5.27.

3.8 Effect on painted surfaces. The concentrated cleaning compound (Type III only) and a 25% solution (Types I, II, and IV) of the cleaning compound in distilled water shall not cause streaking, blistering, discoloration or a permanent decrease in film hardness of more than one (1) pencil hardness level when

tested in accordance with 4.5.13. The Type I material shall be tested using only the Polyurethane Paint Systems (H).

3.9 Stress crazing of MIL-PRF-5425 and MIL-PRF-25690 (Type A and C) acrylic plastics. The concentrated product (Type III only) and a 10% solution (Types I, II and IV) in distilled water shall not cause stress crazing or staining of acrylic plastics when tested in accordance with 4.5.14.

3.10 Stress crazing of polycarbonate plastic. The concentrated product (Type III only) and a 10% solution (Types I, II and IV) in distilled water shall not cause stress crazing or staining of polycarbonate plastic conforming to MIL-P-83310 when tested in accordance with 4.5.15.

3.11 Long term storage stability. After being stored for a period of 12 months, in accordance with 4.5.17, the cleaning compound shall not layer, separate, precipitate or corrode the shipping container. Plastic containers shall not show leakage nor any cracking, crazing, or softening. All cleaning compounds shall meet the requirements of paragraphs 3.5.1, 3.7.1, 3.7.2, 3.15, and 3.16 of this specification.

3.12 Hot dip galvanizing corrosion. The concentrated product (Type III only) and a 10% solution of the cleaning compound (Types I, II and IV) in distilled water shall not show a weight change of an average of three (3) test panels greater than 0.14 mg/cm^2 when tested in accordance with 4.5.18.

3.13 Workmanship. The cleaning compound shall be a liquid having a uniform and homogenous appearance. The cleaning compound shall be manufactured from materials that shall produce a product harmless to metal surfaces and humans when used as directed.

3.14 Effect on polysulfide sealants. The concentrated cleaning compound (Type III only) and a 25% solution (Types I, II and IV) of the cleaning compound in distilled water shall not change the durometer hardness of the polysulfide sealant by more than 5 units when tested in accordance with 4.5.19.

3.15 Rubber compatibility. The concentrated cleaning compound (Type III only) and a 25% solution (Types I, II and IV) of the cleaning compound in distilled water shall not change the durometer hardness more than 5 units when tested in accordance with 4.5.20.

3.16 Effect on polyimide insulated wire. The cleaning compound, when tested according to 4.5.26, shall not cause dissolution, cracking, or dielectric breakdown (leakage) of the polyimide insulated wire in excess of that produced by distilled water.

4. VERIFICATION

4.1 Classification of tests. The inspection and testing of the cleaning compound shall be as follows.

a. Qualification inspection (4.2).

b. Conformance inspection (4.3).

4.2 Qualification inspection. Qualification inspection shall consist of all inspections and tests specified herein.

4.2.1 Qualification samples. The initial qualification samples shall consist of 12 liters (3 gallons) of the cleaning compound. The cleaning compound shall be furnished in containers of the type to be used in filling contract orders. Samples shall be identified as follows and forwarded to the laboratory responsible for testing, as designated in the letter of authorization from the qualifying activity (see 3.1.3):

- Samples for Qualification Tests.
- Cleaning Compound, Aerospace Equipment, (Types I, II, III, and IV).
- MIL-PRF-87937D.
- (Manufacturers Product and Code Number)

- (Name and Address of Contractor)
- Submitted by (Name), (Date) for Qualification Testing in Accordance with the Requirements of MIL-PRF-87937D Under Authorization (Reference Authority Letter).
- (Mixing and Other Important Instructions.)
- (Safety Information and Precautions.)

4.2.2 Test reports. The contractor shall provide certified test reports showing that the material conforms to all the requirements of this specification. The initial report consisting of all specification requirements except the storage stability tests shall be provided upon completion of those tests. The final report shall be provided after the completion of the storage stability tests and shall consist of those test results. Certified test reports shall include the gas chromatogram (4.8.1) or the infrared spectrogram (4.8.2) as required.

4.2.3 Qualification required. Prior to actual procurement, the cleaning compound shall pass the qualification inspections and requirements specified herein. If the product is later modified in any way, the modified form shall be subjected to and shall pass the same qualification inspections (see 3.1). Any changes or modifications from the formulation used at the initial qualification shall be approved by the qualifying activity and may require re-qualification. All initial qualifications shall be granted contingent upon compliance with the long term storage stability requirement specified in paragraph 3.11.

4.3 Conformance tests. Conformance tests (see 6.5) for acceptance of the cleaning compound shall consist of the following tests.

A. Workmanship
B. Cold Stability
C. Insoluble Matter
D. Consistency (Type III only)
E. Immersion Corrosion*
F. Emulsion Characteristics
G. Nonvolatile Matter
H. pH
I. Flash Point
J. Infrared Spectrogram (Types II, III and IV)
K. Gas Chromatogram (Type I only)

*Immersion Corrosion Conformance Test ran on Aluminum SAE AMS-QQ-A-250/4, Bare T3 alloy panel only.

If during conformance testing a lot fails any of the above acceptance tests, all tests required for qualification shall be reinstituted. These qualification tests shall be required until two successive lots meet all requirements of the specification, after which conformance testing shall again be authorized (see 4.3.5).

4.3.1 Sampling. Unless otherwise specified, not less than a 3.8 liter (1 gal) container of the cleaning compound shall be selected at random from each lot and subjected to the tests specified in 4.3. The contents of each selected container for sampling shall be thoroughly mixed by rolling and inverting immediately prior to sampling.

4.3.2 Lot. A lot shall consist of one of the following:

a. The cleaning compound produced in not more than 24 consecutive hours from a continuous process which is used to fill shipping containers directly from the process output. A continuous process shall be the production of product by continuous input of raw materials and output of finished product by one manufacturer in one plant with no change in manufacturing conditions or materials.

b. The cleaning compound from individual runs of a batch process which is used to fill shipping containers directly from the process output. A batch process shall be the production of product by runs from single additions of raw materials which are mixed, reacted, or purified forming the product.

c. The cleaning compound from either or both the continuous and batch processes which is held in a single storage tank and subsequently withdrawn to fill shipping containers. The product shall be homogeneous at the time of withdrawal and shall not be added to while being withdrawn. After each addition to the storage tank, the contents shall constitute a separate lot.

4.3.3 Sampling of product. Unless otherwise specified, conformance tests (4.3) shall be made on the sample of product taken directly from the filled containers. The number of filled containers selected for sampling from each lot shall be in accordance with Table III. The first and last containers to be filled within a given lot shall be sampled. Other containers shall be selected at random. The samples may be obtained in any convenient manner that does not compromise the integrity of the sample.

4.3.4 Inspection of materials. The contractor is responsible for ensuring that materials and components used are manufactured, tested and inspected in accordance with the requirements of referenced subsidiary specifications and standards to the extent specified, or, if none, in accordance with this specification. (see 2.3)

4.3.5 Rejection and retest. When any sample of the product examined and tested in accordance with this specification fails to conform to the requirements specified herein, the entire lot represented by the sample shall be rejected. Rejected material shall not be resubmitted for acceptance without prior approval of the qualifying activity. The application for resubmission shall contain full particulars concerning previous rejections and all measures taken to correct those defects. Samples for retest shall be taken only from a sealed container.

4.4 Testing standards. All laboratory tests shall be conducted at standard conditions unless otherwise specified herein. Standard conditions are defined by FED-STD-141, Section 9. Unless otherwise specified, all chemical tests shall be made with ACS specification reagent grade chemicals. Unless otherwise specified, all product dilutions shall be made with distilled water which conforms to the requirements of ASTM D 1193, Type IV, reagent water. The term "concentrated" cleaner or compound refers to that concentration of the cleaner/compound as received from the manufacturer. No further concentration shall be performed on the product.

4.5 Test methods.

4.5.1 Nonvolatile matter. Weigh 5.00 ± 0.01 g of the sample in a porcelain or glass dish about 6 to 8 cm in diameter and about 2 to 4 cm in depth. Dry to constant weight in an air oven at a temperature of 105 ± 2°C. Constant weight is attained when successive heating for 1-hour periods shows a loss (or gain) of not more than 0.1%. Nonvolatile matter determinations shall be made on a minimum of two samples and the average shall be reported. If the two weights differ by more than 0.5% (absolute) the procedure shall be repeated. The nonvolatile content of each sample shall be calculated as follows:

$$\% \ NVM = \left[\frac{A}{B}\right] 100$$

Where: A = Weight of residue
B = Weight of sample
$\% NVM$ = Percent nonvolatile matter

4.5.2 Insoluble matter. The concentrated cleaning compound shall be thoroughly agitated and a 200 ml test sample withdrawn. The insoluble matter shall be collected with the aid of a vacuum filtering apparatus consisting of a water tap filter pump, a 2,000 ml Erlenmeyer flask, a size 4 (126 mm ID) Buchner funnel and a piece of 126 mm diameter Whatman No 5 filter paper, or equivalent. The filter paper shall be dried at 60°C (140°F) for 30 minutes in a gravity convection oven, cooled for 3 minutes in a desiccator, and weighed to the nearest 0.1 mg. The filter paper shall be placed in the Buchner funnel so that its

circumference coincides with the circumference of the funnel. The vacuum shall be started and the filter paper wetted with approximately 10 ml of distilled water in order to secure it properly in place. The test sample shall be filtered. The sides of the beaker which contained the test sample shall be rinsed with 25 ml of distilled water from a wash bottle, and the rinse transferred to the funnel, insuring that any remaining insoluble matter is completely transferred with the rinse. When all the initial liquid and the rinse have been transferred through the filter, the sides of the funnel shall be washed with 25 ml of distilled water from a wash bottle and the rinse allowed to filter. The vacuum on the flask shall be relieved and the filter paper removed from the funnel. The filter paper shall be dried for 1 hour at 60°C (140°F) in a gravity convection oven, cooled for 3 minutes in a desiccator, and weighed to the nearest 0.1 mg. The percent insolubles shall be calculated as follows:

$$I = \left[\frac{A - B}{W} \right] 100$$

Where: A = Final filter paper weight
B = Initial filter paper weight
W = Weight of sample
I = % Weight insoluble matter

Care should be exercised throughout the final drying and weighing cycle to maintain the flat surface of the filter paper in a horizontal position so that none of the insoluble matter will be lost. Insoluble matter determinations shall be made on a minimum of two samples and the average shall be reported. If the two results differ by more than 0.5% (absolute) the procedure shall be repeated.

4.5.3 pH value. The pH value of the concentrated cleaning compound and a 10 percent solution of the cleaning compound in freshly boiled distilled water shall be measured in accordance with ASTM E 70.

4.5.4 Residue rinsibility. Six smooth aluminum dishes, containing no creases or crevices, shall be cleaned in a solution of Brite-Boy, (from 3D Inc., or equivalent), rinsed, and dried to constant weight. Ten ml of a 25% by volume solution of the cleaning compound in standard hard water (see 4.5.4.1) shall be placed in three of the precleaned dishes and tested according to the procedure in 4.5.4.2.

4.5.4.1 Preparation of standard hard water. A 20-grain (as $CaCO_3$) hard-water stock solution shall be prepared by dissolving 0.40 ± 0.005 g of reagent grade Calcium Acetate, $Ca(C_2H_3O_2)_2 \bullet 2H_2O$ and 0.28 ± 0.005 g of reagent grade Magnesium Sulfate, $MgSO_4 \bullet 7H_2O$, in 1 liter of boiled distilled water.

4.5.4.2 Procedure. Dry three dishes each containing 10.0 ml of a 25% cleaning solution for 7 1/2 hours in a circulating oven at 68 ± 2 °C with full draft. Cool in desiccator overnight and weigh. Rinse with running distilled water for 1 minute. Brush with a sash-type brush containing long-fiber bristles (2.5 cm diameter by 3.8 cm to 6.4 cm long) for 1 minute using distilled water. Rinse for 30 seconds with running distilled water. Dry in oven as before, cool and reweigh. Standard hard water (4.5.4.1) shall be tested as control for weight change comparison in the remaining three precleaned dishes, using the same procedure as above.

4.5.5 Heat stability. A 141.75 g sample of the well mixed concentrated cleaning compound shall be placed into each of two clean 255 ml (12 oz) clear glass bottles having approximate dimensions of 24 cm in height by 6.35 cm in diameter (9.5 in x 2.5 in). One bottle containing the concentrated cleaning compound shall be sealed with a screw type cap and stored in a dark place at standard conditions for 6 days (144 hrs) for reference purposes. Place into the second bottle of concentrated cleaning compound a strip of steel, 15.24 cm by 1.27 cm by 0.05 cm (6 in x 0.5 in x 0.02 in) conforming to AMS 5046 (SAE 1020). Clean the steel strip by abrasively polishing to remove surface scale and corrosion followed by immersion for one minute in ASTM D 235 Mineral Spirits or equivalent followed by immersion for one minute in isopropyl alcohol (TT-I-735, grade A) at standard conditions. Wipe test panels with an alcohol wetted lint free cloth and dry with a clean, lint free cloth. Oven drying is optional. Seal the bottle containing the concentrated cleaning compound and the cleaned steel strip with a screw type cover and shake thoroughly for 1 minute. Place the bottle in a bath maintained at 46 ± 2 °C (115 ± 3 °F) for 5 hours,

then remove and allow to cool to ambient conditions for 19 hours. This heating/cooling cycle shall be repeated 5 times. After completion of the test period, remove the test strip and inspect the portion of the strip which was immersed in the cleaning compound and the portion exposed to the vapor. Any corrosion, pitting or discoloration constitutes failure. The bottle is resealed and along with the control bottle that has been maintained in the dark is shaken thoroughly for 1 minute, then allowed to remain undisturbed for 1 hour at room temperature. The bottles are then examined. Any marked change in color, precipitation, layering or separation constitutes failure.

4.5.6 Cold stability. A 50 ml sample of the cleaning compound shall be poured into a test tube and cooled to 0°C. This temperature shall be maintained for one hour. The compound shall then be allowed to reach room temperature. After 5 (five) complete temperature inversion cycles of the test tube, the compound shall be examined for homogeneity. A slight turbidity shall not be objectionable provided no precipitation is present.

4.5.7 Flash point. The flash point of the concentrated cleaning compound (Type I, II, III, and IV) shall be determined in accordance with ASTM D 56 (Tag Closed Cup) and for materials that have a tendency to form a surface film under the test conditions, use ASTM D 93. The flash point of the 10% solution in distilled water (Type I only) shall be determined in accordance with ASTM D 92.

4.5.8 Emulsion characteristics. Twenty ml of a 25% by volume solution (Types I and II) of the cleaning compound (12.5% by volume solution for Types III and IV) shall be placed in a 50 ml glass stoppered graduated cylinder. Twenty ml of lubricating oil conforming to MIL-PRF-2104, grade 10W, shall be added. An emulsion shall be formed by 10 inversions of the graduated cylinder followed by a vigorous 15 second shake. After the emulsion has stood for 5 minutes, the 15 second shake shall be repeated. At 5 minutes and 8 hours for Type I and at 5 minutes and 24 hours for Types II, III and IV cleaners, the amount of free water and cleaner which separates from the lubricating oil shall conform to the requirements of Table I.

4.5.9 Hydrogen embrittlement. The hydrogen embrittlement properties of the cleaning compound shall be determined as passive chemicals in a service environment according to ASTM F 519 using two (2) sets of either Type 1a, 1c, or 2a AISI 4340 steel specimens. One set shall be plated per Table 2 Treatment B, ASTM F 519. The second set shall be coated with Ion Vapor Deposited (IVD) Aluminum per MIL-DTL-83488D, Class 2, Type I. Prior to coating, specimens for IVD Aluminum shall be prepared by grit blasting, including notched area, with size 180 virgin grain white aluminum oxide grit. The applied IVD coating shall not be peened or burnished in any manner. All specimens must be completely plated or coated except for the screw threads.

4.5.10 Total immersion corrosion. The total immersion corrosion effects of the cleaning compound on the new, unused metals and metal alloys listed in Table II shall be determined in accordance with ASTM F 483. After immersion for 24 hours and after 168 hours, panels shall be evaluated for appearance. Conformance to the requirements in Table II shall be for weight loss after 168 hours. In order to obtain the best results on test panels in this very low weight category, the panels shall be handled with gloves, cleaned in a very careful manner and dried in an oven. They are cooled and dried in a desiccator both before and after each weighing.

4.5.11 Low-embrittling cadmium plate corrosion. The cleaning compound shall be evaluated for corrosion on low-embrittling cadmium plate in accordance with ASTM F 1111.

4.5.12 Effects on unpainted metal surfaces. The cleaning compound shall be evaluated for effects on unpainted metal surfaces in accordance with ASTM F 485.

4.5.13 Effect on painted surfaces. The concentrated cleaning compound (Type III only) and a 25% solution (Types I, II and IV) with distilled water shall be tested in accordance with ASTM F 502 except that the panels used for testing shall be coated with the paint systems listed in Table IV. For all paint systems tested, a separate panel shall be required for both 25% solution and concentrate. For Types II, III and IV compounds, conduct the test on all paint systems listed in Table IV. For Type I compounds, conduct the test only on the polyurethane paint systems (H).

4.5.14 Stress crazing of MIL-PRF-5425 and MIL-PRF-25690 (Type A and C) acrylic plastics. The cleaning compound shall be evaluated for stress crazing of stretch (Type A and C) acrylic plastics in accordance with ASTM F 484.

4.5.15 Stress crazing of polycarbonate plastic. The cleaning compound shall be evaluated for stress crazing of polycarbonate plastics using the test procedure outlined in ASTM F 484 with the exception that the acrylic plastics called for in the procedure be replaced with polycarbonate plastic conforming to MIL-P-83310 of the same dimensions and the polycarbonate specimens shall be stressed for 30 ± 2 minutes to an outer fiber stress of 2000 psi.

4.5.16 Sandwich corrosion. The cleaning compound shall be tested in accordance with ASTM F 1110 as specified in paragraph 3.7.5.

4.5.17 Long term storage stability. The cleaning compound shall be prepared and stored for long term storage stability in accordance with ASTM F 1104 using one (1) 3.8 liter (one-gallon) can conforming to Federal Specification PPP-P-704 or DOT UN 1A1 steel container. Plastic containers shall conform to DOT UN 1H1 as required by 49 CFR 178. Manufacturers using both type materials in production packaging shall test each type container with their product.

4.5.18 Hot dip galvanizing corrosion. The total immersion corrosion effect of the cleaning compound shall be evaluated as specified in 3.12 after 24 hour immersion per ASTM F 483. Test coupons shall be AMS 5046 (SAE 1020) steel panels prepared by hot dip galvanizing per ASTM A 153.

4.5.19 Effects on polysulfide sealant.

4.5.19.1 Preparation of test specimens. MIL-PRF-81733, Type I, and SAE AMS S-8802 sealants shall be mixed as specified by their respective manufacturers and each pressed into a 1/8 inch thick sheet mold until cured (this shall be the sheet stock for each sealant). The sealants shall be cured for 7 days at 49°C. The specimens shall be cut from the sheet stock.

4.5.19.2 Test procedures. Immerse two specimens of each sealant in the concentrated product (Type III only) and a 25% solution of the cleaning compound (Types I, II and IV) at room temperature for 30 minutes. Remove from the solution, rinse with cool tap water, and test within 30 minutes for Shore A hardness in accordance with ASTM D 2240.

4.5.20 Test on rubber compatibility. Tests shall be conducted on AMS 3204 and AMS 3209 rubbers for compatibility with the cleaning compounds.

4.5.20.1 Preparation of test specimens. Three (3) test specimens shall be used for each type rubber specified. Test specimens shall be cut from 1/8 inch sheet stock.

4.5.20.2 Test procedure. Test and record the Shore A hardness of each test specimen in accordance with ASTM D 2240. Immerse each specimen in the concentrated product (Type III only) and a 25% solution of the cleaning compound (Types I, II and IV) at room temperature for 30 minutes. Remove from the solution, rinse with cool tap water, and test within 30 minutes for a Shore A hardness in accordance with ASTM D 2240.

4.5.21 Cleaning efficiency (all types). The cleaning efficiency of the cleaning compound shall be reported as the average of three test results and shall conform to the requirements of Table I.

4.5.21.1 Preparation of control formula. The control formula shall be prepared by the testing laboratory in accordance with Table V and subjected to the cleaning test (4.5.21.5) and evaluation (4.5.21.6). Valid control formula preparations shall produce denominator values greater than 0.95 during testing.

4.5.21.2 Panel preparation. Aluminum SAE AMS QQ-A-250/4, bare T3 panels, 40.6 x 12.7 x 0.05 cm (16 x 5 x .02 in) shall be used.

4.5.21.3 Soil preparation. Molybdenum disulfide grease soil shall be prepared by blending 50 grams of carbon black and 500 grams MIL-G-21164 grease with a mechanical grease worker for 15 minutes.

4.5.21.4 Application of grease soil. Panels shall be wiped with clean lint free cloths soaked in reagent grade acetone then dried to a constant weight. Record the weight to the nearest 0.1 mg. Apply approximately 200 mg grease soil using a soft bristle brush over an area approximately 2" x 7" in the center of the panel. Remove excess grease soil by covering the test panel with a folded absorbent tissue and exerting pressure by rolling a five pound rubber cylinder over the tissue. Repeat this blotting procedure twice. Each freshly soiled panel shall be baked at 105 ± 5 °C for 60 minutes then cooled to room temperature and weighed to the nearest 0.1 mg. Only use panels with more than 50 mg of grease soil. Panels shall be used within 4 hours.

4.5.21.5 Cleaning test. The test panels shall be cleaned using a Gardner heavy duty wear tester, or equivalent, fitted with a cellulose sponge. The sponge shall be cut such that the dimension parallel to the cleaning stroke is 9 cm (3.5 in) and the width is 7 cm (2.75 in). The cleaning head with the dry sponge attached shall be weighed to a mass of 495 to 505 grams. The cleaning stroke of the scrub tester shall be 12 inches. The cleaning compound (including Type III) and the control formula shall be diluted 1 part cleaner with 9 parts distilled water. After placing a soiled test panel in the template 100 ml of the cleaning solution shall be applied to the sponge then applied to the soiled test panel so that it is completely covered. After allowing a 30 seconds dwell time, the test panel shall be cleaned using 5 cycles of the wear tester. The panel shall then be rinsed with sufficient amounts of distilled water.

4.5.21.6 Evaluation. The rinsed panel shall be heated to 105°C ± 5°C for 10 minutes, cooled to room temperature, then weigh to the nearest 0.1 mg. Report the % Cleaning Efficiency as the average of three (3) tests using the following:

$$\%\ \text{Cleaning Efficiency} = \frac{\left[A-B_{A-C}\right]}{\left[X-Y_{X-Z}\right]} \times 100$$

where: A = Weight of the soiled panel before cleaning with product
B = Weight of the soiled panel after cleaning with product
C = Weight of the unsoiled panel used in the product cleaning test
X = Weight of the soiled panel before cleaning with the control formula
Y = Weight of the soiled panel after cleaning with the control formula
Z = Weight of the unsoiled panel used in the control formula cleaning test

4.5.22 Biodegradability. Biodegradation shall be determined by the "Shake Flask Biodegradation Tests" for measuring ultimate or ready degradation potential, as found in EPA Chemical Fate Test Guidelines 40 CFR Method 796.3100 (Aerobic Aquatic Biodegradation Test) or 40 CFR Method 796.3240 (OECD Screening Test for Ready Biodegradability). Biodegradability shall be shown as carbon transformation by both soluble organic carbon reduction and CO_2 evolution.

4.5.23 Terpene hydrocarbons (Type I only). An approved test procedure shall be used (see 3.3.1.1).

4.5.24 Consistency (Type III only). A consistometer (Central Scientific Company, Chicago, IL; Catalog No. 24925 or equivalent) shall be used as follows: Shake the container of cleaning compound by hand for 10 seconds. Pour the material into the well of the consistometer completely filling it. Release the gate and determine the extent of flow in ten seconds.

4.5.25 Sprayability (Type III only). Fill the reservoir of the application test equipment with Type III compound, as supplied. Release the compound flow valve and gradually increase the nozzle tip pressure to not more than 8 psi pressure observing the discharge spray characteristics. Report the following:

a. The maximum pressure at which no bubbles are released into the surrounding air.

b. The distance the gel can be satisfactorily projected.

4.5.25.1 Technique. Under these optimized conditions apply with a sideways sweeping motion the compound to a vertical surface and examine the deposited film and record assessment. The product should display uniformity with absence of large and entrained air bubbles or a consistency which would not inhibit effective cleaning.

4.5.25.2 Application test equipment.

a. Reservoir: Hand pump pressure sprayer (modified), or pressure pot with air pressure applied from external compressor.

b. Nozzle: Fan jet with an equivalent orifice diameter 1.1 mm and spray angle 65°. (Spraying Systems Co., Wheaton Illinois, Item Number H-VV 6503)

4.5.26 Effect on polyimide insulated wire. Coil two segments of MIL-DTL-81381/11-20 wire approximately 61 cm (24 in) and place into separate 118 ml (4 oz) wide mouth jars. To one jar add sufficient concentrate cleaning compound to completely cover the wire coil. To the other jar (control sample) add sufficient distilled water to cover the wire coil. Cap both jars and store at room temperature (20 - 25 °C) for 14 days. At the end of the storage period remove both coils, rinse thoroughly with distilled water and suspend to allow complete draining and drying. Uncoil the wires, examine each closely for dissolution, and report the results. The wire immersed in the cleaner shall perform as well as the wire immersed in distilled water. Both wires shall then be subjected to a double reverse wrap on a 0.3 cm (0.125 in) mandrel and examined for cracking. (Note: Failure of the control sample here voids the test and shall be repeated using new MIL-DTL-81381/11-20 material). Wire immersed in the cleaner shall then be examined for cracking. If cracking occurs results shall be reported and the test ended. Passing wire shall then withstand a one minute dielectric test of 2,500 volts (rms), using a Hypot model number 4045 or equivalent, and examined for breakdown or leakage. Wire immersed in the cleaner shall perform equally well as the control wire immersed in distilled water.

4.5.27 Wet adhesion tape test. This method tests the coating to metal and the intercoat adhesion of an organic coating system. This procedure is used to determine the cleanliness of the surface prior to coating.

4.5.27.1 Preparing test coupons. The test coupons shall consist of nine (9) 4 in x 6 in aluminum alloy coupons conforming to SAE AMS QQ-A-250/12. The coupons shall be cleaned with reagent grade acetone, then cleaned with the diluted cleaning compound (10% solution) agitated for 20 seconds with a Scotch Bright pad (A-A-58054, Type I Class 1, Grade B, maroon color) and thoroughly rinsed with water and allowed to dry. Pretreat the coupons with AMS 1640 and MIL-C-5541. The coupons shall be air dried and primed with MIL-PRF-23377, Type I Class C high solids epoxy primer. Topcoat the coupons according to Table VI as follows:

Set 1: Six coupons (Code A plus D)
Primer: MIL-PRF-23377, Type I, Class C High-Solids Epoxy
Topcoat: MIL-PRF-85285, Type I High Solids Polyurethane Color # 34092

Set 2: Three coupons (Code A plus D)
Primer: MIL-PRF-23377, Type I, Class C High-Solids Epoxy
Topcoat: MIL-PRF-85285, Type I High Solids Polyurethane Color # 17925.

The coatings should be allowed to cure for a minimum of seven (7) days before being validated by performing the wet tape test.

4.5.27.2 Coupon validation procedure. The nine coupons (two sets in 4.5.27.1) shall be validated using the following wet tape test.

a. Immerse the test coupons in distilled water for 24 hours.

b. Remove the test coupons from the water and wipe dry with a clean lint free cloth.

c. Immediately apply a 25.4 mm wide strip of Masking Tape (3M Co., Code No. 250) with the adhesive side down. Do not apply the tape within 1/2 inch of any edge.

d. Press the tape against the surface of the coating by passing a 2.0 kg rubber covered roller, having a surface Durometer hardness value of 70 to 80, across the tape eight times.

e. Remove the tape with one quick motion and examine for damage to the intercoat or surface adhesion.

f. If there is no damage to the surface, note and proceed to 4.5.27.3. If three or more coupons fail the wet tape test or there is any unusual or non-typical condition, investigate to determine if use of cleaner contributed to failure or unusual condition. Report findings. Failed coupons or coupons with unusual surface conditions shall not be validated nor used in the repaint testing of Section 4.5.27.3.

4.5.27.3 Test procedure. This test shall verify the cleaning compound's effectiveness to remove soil from a painted surface in preparation for repainting (touch up). Immerse the coupons validated in 4.5.27.2 in hydraulic fluid conforming to MIL-PRF-83282 for ten (10) minutes. Remove the panels from the fluid and blot excess fluid from the coupons with a paper napkin. Spray the diluted cleaning compound (10% solution) on the coupons, agitate for 20 seconds with a 3M Scotch Bright Pad (A-A-58054) and thoroughly rinse with clean water. After the coupons have air dried, recoat the panels from Table VI as follows:

Set 1: Three coupons (code B plus D)
Primer: MIL-PRF-85582, Type I, Class 1B Waterborne Epoxy
Topcoat: MIL-PRF-85285 Type I High Solids Polyurethane, Color #34092

Set 2: Three coupons (Code A plus D)
Primer: MIL-PRF-23377, Type I, Class C High Solids Epoxy
Topcoat: MIL-PRF-85285 Type I High Solids Polyurethane, Color # 34092

Set 3: Three coupons (Code C plus D)
Primer: TT-P-2760, Type I, Class C High Solids Elastomeric, Polyurethane
Topcoat: MIL-PRF-85285 Type I High Solids Polyurethane, Color # 34092

After the above coatings have air dried for seven (7) days, perform the Wet Tape Test in paragraph 4.5.27.2, steps (a) through (e). The coating system shall show no signs of damage.

4.6 Toxicity and waste disposal characteristics. The supplier shall provide the toxicological data and formulations required (see 3.3) to evaluate the safety of the material proposed for use. The manufacturer shall provide current procedures for disposal per federal EPA regulations.

4.7 Filler materials. The contractor shall furnish certification that the cleaning compound contains only the materials allowed and does not contain any filler materials disallowed per 3.2.

4.8 Qualitative identification of components (Types I, II, III, and IV).

4.8.1 Gas chromatogram (Type I only). A gas chromatogram of the Type I product shall be provided by a Government approved qualification laboratory (see 3.4). The chromatogram shall report all salient instrumental parameters (column type and dimensions, temperature(s), carrier gas and flow rate, detector type, sample dilution(s), etc. required to produce it.

4.8.2 Infrared spectrogram (Types II, III, and IV). Infrared spectrograms of the nonvolatile matter shall be prepared by a Government approved qualification laboratory (see 3.4). The spectrogram, including method for sample preparation, shall be provided to the qualifying activity by the qualification laboratory.

5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When actual packaging of material is to be performed by DoD personnel, these personnel need to contact the responsible packaging activity to ascertain requisite packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activity within the Military Department or Defense Agency, or within the Military Department's System Command. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity

6. NOTES

6.1 Intended use. The four types of cleaning compounds covered by this specification are intended to be used for cleaning Aerospace Equipment including aircraft, aerospace ground equipment (AGE) and AGE engines. These cleaners will be used in place of other cleaners when approved by the System Program Manager of the equipment being cleaned. Type I should be used only on polyurethane and enamel coatings as it may attack acrylic nitrocellulose lacquer coatings found in numerous aircraft. Types I and IV materials are intended for light to heavy duty removal of greases, oils, hydraulic fluid, and carbon. Type II is intended for light to medium cleaning and is not intended to remove heavy soils. Types II and IV cleaning compounds are also intended for cleaning aircraft and aerospace ground equipment surfaces of contaminants prior to coating or recoating with primers, topcoats, sealants and adhesives. Types I, II and IV must be diluted with water before use. Type III is intended for light to heavy duty removal of greases, oils, hydraulic fluid, and carbon in wheel wells, wing butts and other areas where complete rinsing with water can be tolerated. After cleaning, rinse off with water. These cleaners are not intended to be used as canopy cleaners. These products have not been tested for use at elevated temperatures.

6.2 Acquisition requirements. Acquisition documents should specify the following:

a. Title, number and date of this specification.
b. Type I, Type II, Type III or Type IV.
c. Size containers required.
d. QPL reference or test number.
e. Level of packing required.
f. Palletization, when applicable.

6.3 Material safety data sheets. Contracting officers should identify those activities requiring copies of completed Material Safety Data Sheets prepared in accordance with FED-STD-313.

6.4 Qualification. With respect to products requiring qualification, awards will be made only for products which are at the time set for opening of bids, qualified for inclusion in the applicable QPL whether or not such products have actually been so listed by that date. The attention of the contractors is called to this requirement, and contractors are urged to arrange to have their products that they propose to offer to the Federal Government tested for qualification in order that they may be eligible to be awarded contracts or orders for the products covered by this specification. Information pertaining to qualification of products may be obtained from the qualifying activity (see 3.1.3).

6.5 Conformance tests. Conformance inspection should consist of examinations and tests necessary to ensure that production items meet specification requirements. Conformance inspection should include a description of the inspection procedure, sequence of inspections, number of units to be inspected, and the criteria for determining conformance to the requirement specified. Conformance examinations and tests should not duplicate any long term or special tests that were used to justify inclusion of qualification in a specification.

6.6 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

6.7 Key words.

AGE

Biodegradable

Gel-type

QPL

Terpene

Custodians:
Air Force - 68
Navy - AS

Review activities:
Air Force - 11
DLA - GS

Preparing activity:
Air Force - 68
(Project 6850-1441)

Appendix A

TABLE I. Quantitative Requirements

REQUIREMENT	TYPE I		TYPE II		TYPE III		TYPE IV		TEST METHOD
	MIN	MAX	MIN	MAX	MIN	MAX	MIN	MAX	
Insoluble Matter (WT%)		0.05		0.05		0.05		0.05	4.5.2
Flash Point (°F)									
10% Solution	200								
Concentrated Solution	120		None 1/		None1/		None 1/		4.5.7
Emulsion Characteristics (ml free water)									
5 min		5.0		5.0		5.0		5.0	
8 hours	13.0								4.5.8
24 hours			13.0		8.0		11.0		
Wet Adhesion Tape Test			Pass				Pass		4.5.27
% Cleaning Efficiency	95		65		65		90		4.5.21
Terpene Hydrocarbons (% WT)	25	40	---	None	---	None	---	None	4.5.23

1/ No flash point should be observed up to the boiling point of the compound.

TABLE II. Total Immersion Corrosion Requirements

Alloy	Average of 3 Panels Weight Loss, Max ($mg/cm^2/168$ hrs)
Magnesium (AZ 31B-H24) AMS 4377 surface treatment per SAE AMS M-3171, Type III	0.50
Aluminum, SAE AMS QQ-A-250/4,T3 surface treatment per MIL-A-8625, Type I, Class I	0.15
Aluminum, SAE AMS QQ-A-250/4, Bare T3 Alloy	0.15
Aluminum, SAE AMS QQ-A-250/12, Bare T6 Alloy	0.15
Titanium, SAE AMS T-9046, 6AL-4V Class III, Composition C	0.10
Steel, AMS 5046, SAE 1020	0.25
Steel, 410 SS, Silver Plated per SAE AMS 2410	0.10

TABLE III. Sampling for Tests

Number of Containers in lot	Number of Containers to be sampled
2 to 15	2
16 to 25	3
26 to 90	5
91 to 150	8
151 to 280	13
281 to 500	20
501 to 1200	32
1201 to 3200	50
3201 to 10000	80
10001 to 35000	125
35001 to 150000	200
150001 to 500000	315
500001 and over	500

TABLE IV. Test Panel Finishes

Primer Coatings				
Panel Set No.	Primer Material Specification	Dry Film Thickness Per Coat/ mm (inches)	No. of Coats	Drying Time Before Topcoating
EH	MIL-PRF-23377, Type I, Class C High-Solids Epoxy Primer	0.0152-0.0229 (0.0006-0.0009)	1	2 - 8 hours

Top Coats, Color Number 17925 per FED-STD-595						
Panel Set	Topcoat Material	Dry Film Thickness Per Coat/ mm (inches)	No. of Coats	Drying Time Between Coats	Dry Film Thickness mm (inches)	Days to Dry Before Testing
E	MIL-PRF-22750 Coating, Epoxy Topcoat	0.0203 - 0.0305 (0.0008 - 0.0012)	2	1 hour	0.0406-0.0610 (0.0016 - 0.0024)	7
H	MIL-PRF-85285 Type I Coating: Polyurethane, High Solids	0.0203 - 0.0305 (0.0008 - 0.0012)	2	1 hour	0.0406-0.0610 (0.0016 - 0.0024)	7

TABLE V. Control Formula for the Cleaning Efficiency Test

Component	Control Formula Composition (% by weight) 1/
d-limonene	30.0
diethanolamine	5.0
nonionic surfactant (Triton X-100)	5.0
distilled water (ASTM D 1193, Type IV)	60.0

1/. This formulation is corrosive and intended solely for use as the control for the cleaning efficiency test. It will not qualify to the requirements in this specification.

TABLE VI. Test Panel Finishes

Primer Coatings				
Code	Primer Material Specification	Dry Film Thickness Per Coat/ mm (inches)	No. of Coats	Drying Time Before Topcoating
A	MIL-PRF-23377,Type I, Class C Primer Coatings; Epoxy, High-Solids	0.0152 - 0.0229 (0.0006 - 0.0009)	1	2 - 8 hours
B	MIL-PRF-85582, Type I, Class 1B Primer Coatings: Epoxy, Waterborne	0.0152 - 0.0229 (0.0006 - 0.0009)	1	2 - 8 hours
C	TT-P-2760, Type I, Class C Primer Coating: Polyurethane, Elastomeric, High-Solids	0.0380 - 0.0510 (0.0015 - 0.0020)	1	2 - 8 hours

Top Coats						
Code	Topcoat Material	Dry Film Thickness Per Coat/ mm (inches)	No. of Coats	Drying Time Between Coats	Dry Film Thickness mm (inches)	Time Before Testing (Days)
D	MIL-PRF-85285, Type I Coating: Polyurethane, High-Solids	0.0203 - 0.0305 (0.0008 - 0.0012)	2	1 hour	0.0406 - 0.0610 (0.0016 - 0.0024)	7

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

INSTRUCTIONS

1. The preparing activity must complete blocks 1, 2, 3, and 8. In block 1, both the document number and revision letter should be given.
2. The submitter of this form must complete blocks 4, 5, 6, and 7, and send to preparing activity.
3. The preparing activity must provide a reply within 30 days from receipt of the form.

NOTE: This form may not be used to request copies of documents, nor to request waivers, or clarification of requirements on current contracts. Comments submitted on this form do not constitute or imply authorization to waive any portion of the referenced document(s) or to amend contractual requirements.

I RECOMMEND A CHANGE:	1. DOCUMENT NUMBER MIL-PRF-87937D	2. DOCUMENT DATE (YYYYMMDD) 20010924

3. DOCUMENT TITLE CLEANING COMPOUND, AEROSPACE EQUIPMENT

4. NATURE OF CHANGE *(Identify paragraph number and include proposed rewrite, if possible. Attach extra sheets as needed.)*

5. REASON FOR RECOMMENDATION

6. SUBMITTER

a. NAME *(Last, First, Middle Initial)*	b. ORGANIZATION	
c. ADDRESS *(Include Zip Code)*	d. TELEPHONE (Include Area Code) (1) Commercial (2) AUTOVON *(if applicable)*	**7.DATE SUBMITTED** (YYYYMMDD)

8. PREPARING ACTIVITY

a. NAME Code (68) DET 3 WR-ALC/AFTT, Bldg. 1621-K	b. TELEPHONE *Include Area Code)* (1) Commercial (2) AUTOVON
c. ADDRESS *(Include Zip Code)* 2261 Hughes Ave. Ste 123 Lackland, AFB, TX 78236-9823	**IF YOU DO NOT RECEIVE A REPLY WITHIN 45 DAYS, CONTACT:** Defense Standardization Program Office (DLSC-LM) 8725 John J. Kingman road, Suite 2533 Ft. Belvoir, VA 22060-2533 Telephone (703) 767-6888 AUTOVON 427-6888

Appendix B

SMI Results from Eagle Kleen I Analytical Testing

SMI, Inc.

12219 SW 131 Avenue
Miami, Florida 33186-6401 USA

Phone: (305) 971-7047
Fax: (305) 971-7048

Attn: BATTELLE MEMORIAL INSTITUTE
505 King Avenue
Columbus, OH 43201

Date: 20-Sep-2004
SMI REF: 04JUL562

PRODUCT: **EAGLE KLEEN**
(received 14-Jul-2004)

40 CFR 796.3100: AEROBIC AQUATIC BIODEGRADATION
Code of Federal Regulations
Environmental Protection Agency
Title 40: Protection of Environment
Part 796: Chemical Fate Testing Guidelines
Shake Flask Method

Summary of Results:

Based on dissolved organic carbon analysis:

"EAGLE KLEEN" = 87.8 % Biodegradable in 28 days

See Appendix A for graphical representation of Biodegradability vs. Time .

PROCEDURE

I. Introduction

This procedure provides a way to determine the rate and extent of aerobic biodegradation that might occur when chemical substances are released to aquatic environments. A high biodegradability result in this test provides evidence that the test substance will be biodegradable in natural aerobic freshwater environments. A low biodegradability result may not necessarily indicate poor biodegradation, as other factors may interfere, such as inhibition of the microbial inoculum by the test material.

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II. Principle of the Test Method

The method consists of a 2-week inoculum buildup period during which the microbes are allowed to adapt to the test compound. The acclimated media containing a defined amount of test compound is added to specially equipped Erlenmeyer flasks. The test media is sampled periodically and analyzed for dissolved organic carbon (DOC). A reservoir filled with barium hydroxide is utilized to measure the amount of carbon dioxide evolved. The degree of biodegradation is determined by comparison of the extent of DOC disappearance and the amount of carbon dioxide liberated. Control flasks containing no test compounds are run simultaneously and are used to estimate the degree of ultimate biodegradation. Reference substances which will exhibit ultimate biodegradation may be run simultaneously to check the activity of the inoculum. If the reference samples do not exhibit at least 60 percent of theoretical maximum carbon dioxide, and at least 70 percent DOC removal within 28 days, the test will be regarded as invalid and shall be repeated using different inoculum.

This method is believed to be appropriate for a screening test which has solely an acceptance but no rejective function.

III. Test Procedure

The total organic carbon (TOC) of the test compound is first determined by analysis or calculation if the formulation is known. Determination of the minimum inhibitory concentration is useful to insure that the test compound will not be inhibitory to the microbes at the required concentration. The shake flask apparatus is assembled utilizing a 2-liter Erlenmeyer flask and a 50 ml centrifuge tube. The tube containing 10 mls of barium hydroxide will be suspended over the contents of the flask in such a way that liberated carbon dioxide may diffuse into the barium hydroxide, while allowing the contents of the tube to be removed for analysis without spilling into the test media. Glass tubing may be utilized as access into the flask for sparging, venting, and sampling.

Stock solutions I, II, and III are prepared (see Appendix B), along with 0.2 N barium hydroxide and 0.1 N HCl. Acclimation medium is prepared by adding 1 ml each of stock solutions I, II and III to 1 liter of distilled, deionized water (DIW). The microbial inoculum is obtained from sewage and soil or from Polyseed and is added to the acclimation medium. Test compounds are added incrementally during the acclimation period at concentrations equivalent to 4, 8, and 8 mg/L carbon on days 0, 7, and 11, respectively. On day 14, the medium is ready for use in the test.

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Biodegradability test flasks are prepared by adding 100 mls of acclimation medium to 900 mls of DIW along with 1 ml each of solutions I, II, and III to the 2-liter Erlenmeyers. Additional test compound equivalent to 10 mg/L carbon is added to the flasks. Ten mls of barium hydroxide are added to the suspended reservoirs in each flask and 10 mls are also saved for use as a titration blank. Flasks are sparged with carbon dioxide-free air, sealed and placed on a shaking table (approx. 125 rpm) at 20 - 25 deg C in the dark. Test flasks should be run in triplicate and sampling should occur at time zero and at least four other times to allow for a smooth plot of biodegradation. Each sample for DOC analysis is first centrifuged or filtered through a 0.45 micrometer or smaller pore diameter. On the day prior to terminating the test, 3 mls of 20 percent sulfuric acid are added to release carbonate bound carbon dioxide.

IV. ANALYTICAL MEASUREMENTS

The quantity of carbon dioxide evolved is measured by titration of the entire barium hydroxide sample with 0.1 N HCl to the phenolphthalein end point, blank subtracted. Theoretically, 10 mg of carbon is converted to 0.833 mmol of carbon dioxide. Absorbed carbon dioxide precipitates as barium carbonate, causing a reduction in alkalinity by the equivalent of 16.67 ml of 0.1 N HCl for complete conversion of the test compound carbon to carbon dioxide. Therefore, the percent theoretical carbon dioxide evolved from the test compound is calculated at any sampling time from the formula:

$$\% \, CO_2 \text{ evolution} = [(TF - CF)/16.67] \cdot 100$$

where:

TF = mls of 0.1 N HCl used in titration of test flask

CF = mls of 0.1 N HCl used in titration of control flask

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The DOC analysis is performed using a suitable organic carbon method. The percent DOC disappearance from the test compound is calculated from the formula:

$$\% \text{ DOC removal} = [1 - (DTF_x - DCF_x)/(DTF_o - DCF_o)] \cdot 100$$

where:

DTF = Dissolved organic carbon from test flask
DCF = Dissolved organic carbon from control flask

o = Day zero measurements
x = Day x measurements

V. REPORT OF RESULTS

Inoculum: Polyseed and Mixed inoculum

Date Received: July, 2004

Source: Fisher Scientific and Metro-Dade County Water & Sewer Authority

Storage: Ambient temperature, used within 24 hours

Minimum Inhibitory Concentration: MIC < 3.125 % (non-inhibitory to microbes at concentrations lower than 3.125%)

Percent Biodegradation based on DOC analysis:

EAGLE KLEEN:	**87.8 %** after 28 days (see Table 1)
Reference (Sodium citrate):	92.3 % after 28 days (see Table 1)

Percent Biodegradation based on carbon dioxide evolution:

EAGLE KLEEN:	34.5 % after 28 days (see Table 2)
Reference (Sodium citrate):	42.7 % after 28 days (see Table 2)

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Summary: Since the test compound was found to be over 70 % biodegradable based on the DOC analysis, it is reasonable to assume that the substance will undergo rapid and ultimate biodegradation in aerobic aquatic environments, also known as "ready biodegradability". The test is validated by the fact that the reference compound, sodium citrate, exhibited a biodegradability over 70%.

The percent biodegradability based on carbon dioxide evolution is typically lower than that of the DOC based numbers. In this case, the carbon dioxide evolution measured was significant, both on the test compound and on the reference, and the results generally agree.

Respectfully submitted,

Patricia D. Viani
SMI, Inc.

EPA 796.3100: AEROBIC AQUATIC BIODEGRADATION

TABLE I - DISSOLVED ORGANIC CARBON (DOC) VALUES

Sample: **EAGLE KLEEN**

		DAY 0	DAY 7	DAY 14	DAY 21	DAY 28
	A	38.2	12.7	9.5	8.1	8.1
	B	35.7	8.2	7.5	7.2	7.2
	C	37.9	7.1	7.0	7.1	7.2
AVERAGE		37.3	9.3	8.0	7.5	7.5
CORRECTED AV		34.1	6.0	4.7	4.2	4.2
% BIODEGRADED		**N/A**	**82.5%**	**86.3%**	**87.8%**	**87.8%**

Reference: Sodium Citrate

	A	38.8	9.5	5.9	5.7	5.8
	B	37.1	12.9	8.2	6.3	6.1
	C	36.2	10.7	7.1	5.9	6.0
AVERAGE		37.4	11.0	7.1	6.0	6.0
CORRECTED AV		34.2	7.7	3.7	2.7	2.6
% BIODEGRADED		N/A	77.6%	89.1%	92.2%	92.3%

BLANK	A	3.3	3.5	3.2	3.1	3.6
	B	3.2	3.2	3.5	3.6	3.0
	C	3.0	3.4	3.3	3.2	3.4
AVERAGE		3.2	3.4	3.3	3.3	3.3

EPA 796.3100: AEROBIC AQUATIC BIODEGRADATION

Table II - Titration Data for CO2 Evolution

Sample: **EAGLE KLEEN**

			DAY 7	DAY 14	DAY 21	DAY 28	
	A		4.2	11.8	17.4	17.6	
	B		5.2	13.8	17.4	18.2	
	C		4.6	10.2	17.2	18.2	
AVERAGE			4.7	11.9	17.3	18.0	
CORRECTED AVG			12.9	5.9	0.7	0.1	
% BIODEGRADED			22.6%	10.4%	1.3%	0.2%	
mls theoretical:		56.9				**TOTAL=**	**34.5%**

Reference: Sodium Citrate

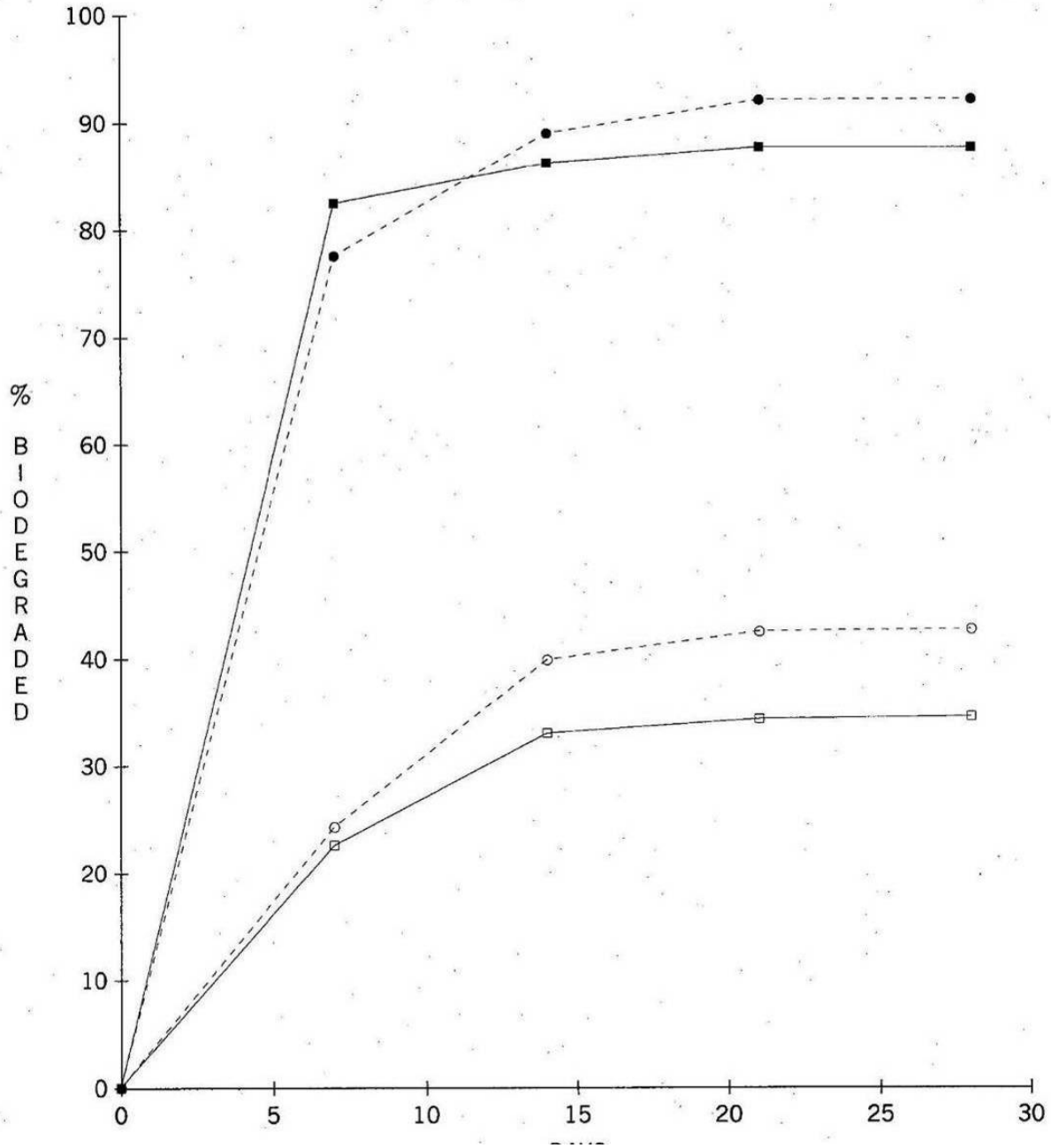
	A		3.4	11.8	16.8	18.2	
	B		3.8	7.8	17.2	18.0	
	C		3.8	7.4	15.8	17.8	
AVERAGE			3.7	9.0	16.6	18.0	
CORRECTED AVG			13.9	8.9	1.5	0.1	
% BIODEGRADED			24.3%	15.6%	2.6%	0.2%	
mls theoretical:		57.0				TOTAL =	42.7%
BLANK	A		17.8	18.2	18.0	18.2	
	B		17.0	17.6	18.2	17.8	
	C		17.8	17.8	18.0	18.4	
AVERAGE			17.5	17.9	18.1	18.1	

EPA 796.3100: AEROBIC AQUATIC BIODEGRADATION

Appendix A

BIODEGRADABILITY VS. TIME

EAGLE KLEEN



Appendix B

STOCK SOLUTIONS I, II, AND III

SOLUTION I:	35	g/L	NH_4Cl
	15	g/L	KNO_3
	75	g/L	$K_2HPO_4 \cdot 3H_2O$
SOLUTION II:	25	g/L	$NaH_2PO_4 \cdot H_2O$
	10	g/L	KCl
	20	g/L	$MgSO_4$
	1	g/L	$FeSO_4 \cdot 7H_2O$
	adjust pH of Soln II to 3.0		
SOLUTION III:	5	g/L	$CaCl_2$
	0.05	g/L	$ZnCl_2$
	0.5	g/L	$MnCl_2 \cdot 4H_20$
	0.05	g/L	$CuCl_2$
	0.001	g/L	$CoCl_2$
	0.001	g/L	H_3BO_3
	0.0004	g/L	MoO_3

Appendix C

SMI Results from Eagle Kleen II Analytical Testing

SMI, Inc.

12219 SW 131 Avenue
Miami, Florida 33186-6401 USA

Phone: (305) 971-7047
Fax: (305) 971-7048

Attn: Sara F. Kuczek
Battelle Memorial Inst.
505 King Avenue
Columbus, OH 43201

Date: 30-Sep-2004

SMI/REF: 04AUG682

Product: **EAGLE KLEEN II** *(received 10-Sep-2004)*

Dilution: Ready to Use

Modified partial testing of section 3.7, Effect on Metals
(product was tested neat - [undiluted])
MIL-PRF-87937D (24 Sep 2001)
CLEANING COMPOUND, AEROSPACE EQUIPMENT
Type IV - Heavy Duty, Water Dilutable Cleaning Compound

3.7	Effect on metals	
3.7.1	Hydrogen embrittlement	Conforms*
3.7.2	Total immersion corrosion	Does not conform*
3.7.3	Low-embrittling cadmium plate corrosion	Does not conform*
3.7.4	Effects on unpainted metal surfaces	Not performed
3.7.5	Sandwich corrosion	Conforms*
3.7.6	Wet adhesion tape test	Not performed

** Test performed using "as received" solution (ready to use) instead of dilution required by specification. Results should not be considered for QPL listing.*

Respectfully submitted,

Patricia D. Viani, SMI Inc.

3.7 Effect on metals

3.7.1 Hydrogen embrittlement: When tested in accordance with 4.5.9, the concentrated cleaner (all types) and a 10% solution of the cleaner (Types I, II, and IV only) in distilled water shall not cause hydrogen embrittlement of cadmium plated or IVD aluminum coated AISI 4340 steel.

Test temperature: 21 - 23°C (69 - 73°F)
Specimens: Type 1c, ***cadmium*** *plated in accordance with Treatment B of ASTM F519*

As received: ***No failures within 150 hours.***
Dilute (10 %): ***Not performed***

Result Conforms*

Test temperature: 21 - 23°C (69 - 73°F)
Specimens: Type 1c, grit blasted, ***IVD Aluminum*** *plated per MIL-DTL-83488D, Cl 2, Ty I.*

As received: ***No failures within 150 hours.***
Dilute (10 %): ***Not performed***

Result Conforms*

** Test performed using "as received" solution (ready to use) instead of dilution required by specification.*

3.7.2 Total immersion corrosion: When tested in accordance with 4.5.10 (ASTM F 483), the concentrated cleaning compound (all types) and a 10% solution of the cleaning compound (Types I, II and IV only) in distilled water shall not show any indication of staining, etching, pitting, or localized attack on any of the panels, or cause a weight change of an average of three (3) test panels greater than that shown in Table II. A slight discoloration of the panels shall not be objectionable. The cleaning compound shall not layer or separate for the duration of the test.

Table II Total Immersion Corrosion Requirements

Alloy	Weight Loss ($mg/cm^2/168hrs$)		
	Maximum allowed	**As received**	**10 %**
Magnesium (AZ 31B-H24) AMS 4377 surface treatment per SAE AMS-M-3171, Ty III	0.50	**0.14**	**NOT PERFORMED**
Aluminum, SAE AMS-QQ-A-250/4, T3 surface treatment per MIL-A-8625, Type I, Class I	0.15	**0.01**	
Aluminum, SAE AMS-QQ-A-250/4, Bare T3 Alloy	0.15	**0.01**	
Aluminum, SAE AMS-QQ-A-250/12, Bare T6 Alloy	0.15	**0.01**	
Titanium, SAE AMS-T-9046, 6Al-4V Cl III, Comp. C	0.10	**0.01**	
Steel, AMS 5046, Grade 1020	0.25	**0.57**[1]	
Steel, 410 SS, Silver Plated per SAE AMS 2410	0.10	**0.01**	

[1]Exceeds allowable weight change; significant discoloration/darkening

Result Does not conform*

3.7.3 Low-embrittling cadmium plate corrosion: Steel panels coated with low-embrittling cadmium plate immersed in the concentrated cleaning compound (all types) and a 10% solution of the cleaning compound (Types I, II and IV only) in distilled water shall not show a weight change greater than 0.14 mg/cm^2 for 24 hours when tested in accordance with 4.5.11.

As received: ***0.20* $mg/cm^2/24hrs$***
Dilute (10 %): *Not performed*

Result Does not conform*

** Test performed using "as received" solution (ready to use) instead of dilution required by specification.*

3.7.4 Effects on unpainted metal surfaces: The concentrated cleaning compound (Type III only) and a 10% solution (Types I, II and IV only) of the cleaning compound in distilled water shall not cause streaking, stains or other deposits that cannot be easily removed with water when tested in accordance with 4.5.12.

Result Not performed

3.7.5 Sandwich corrosion: When tested in accordance with 4.5.16, the concentrated cleaner (all types) and a 10% solution (Types I, II and IV only) shall show no corrosion in excess of that shown by control test coupons in ASTM D 1193, Type IV, reagent water.

	2024-T3 Bare Anodized	2024-T3 Alclad	7075-T6 Bare Anodized	7075-T6 Alclad
As received	**1**	**1**	**1**	**1**
Dilute (10%)	**Not performed**			
Control	1	1	1	1

Result Conforms*

3.7.6 Wet adhesion tape test (Types II and IV): A ten (10) percent solution of the cleaning compound, when used as directed, shall remove soil from a painted surface in preparation fro repainting such that paint applied after cleaning with the compound shall adhere to the surface when tested in accordance with 4.5.27.

Result Not performed

** Test performed using "as received" solution (ready to use) instead of dilution required by specification.*

Appendix D

SMI Results from Eagle Kleen III Analytical Testing

SMI, Inc.

12219 SW 131 Avenue
Miami, Florida 33186-6401 USA

Phone: (305) 971-7047
Fax: (305) 971-7048

Attn: Sara Kuczek
Battelle Memorial Institute
505 King Ave
Columbus, OH 43201

Date: 07-Mar-2005

SMI/REF: 04DEC086
Final Report

Product: **EAGLE KLEEN III** (Lot C531-84-1)
(received 01-Dec-2004)

Dilution: As received

Modified testing (product was tested "as received" [undiluted])
MIL-PRF-87937D (24 Sep 2001)
CLEANING COMPOUND, AEROSPACE EQUIPMENT
Type IV - Heavy Duty, Water Dilutable Cleaning Compound

3.3	Toxicity	Informational
3.3.4	Biodegradability	Conforms
3.4	Compositional assurance	Informational
3.5	Chemical properties	
3.5.1	Chemical requirements	
	Insoluble matter	Conforms
	Flash point	Conforms
	Emulsion characteristics	Conforms*
	Wet adhesion tape test	Conforms*
	% Cleaning efficiency	Conforms*
	Terpene hydrocarbons	Not applicable
3.5.2	Residue rinsibility	Conforms*
3.6	Physical properties	
	3.6.1 Heat stability	Does not conform*
	3.6.2 Cold stability	Conforms
	3.6.3 Rheology	
	3.6.3.1 Consistency	Not applicable
	3.6.3.2 Sprayability	Not applicable

** Test performed using "as received" solution (ready to use) instead of dilution required by specification. Results should not be considered for QPL listing.*

3.7	Effect on metals	
	3.7.1 Hydrogen embrittlement	Conforms*
	3.7.2 Total immersion corrosion	Conforms*
	3.7.3 Low-embrittling cadmium plate corrosion	Conforms*
	3.7.4 Effects on unpainted metal surfaces	Conforms*
	3.7.5 Sandwich corrosion	Conforms*
	3.7.6 Wet adhesion tape test	Conforms*
3.8	Effect on painted surfaces	Conforms*
3.9	Stress crazing of MIL-PRF-5425 and MIL-PRF-25690 (Type A and C) acrylic plastics	Does not conform*
3.10	Stress crazing of polycarbonate plastic	Does not conform*
3.11	Long-term storage stability	Not performed
3.12	Hot dip galvanizing corrosion	Conforms*
3.13	Workmanship	To be Cert. by Mfr.
3.14	Effect on polysulfide sealants	Conforms*
3.15	Rubber compatibility	Conforms*
3.16	Effect on polyimide insulated wire	Conforms

** Test performed using "as received" solution (ready to use) instead of dilution required by specification. Results should not be considered for QPL listing.*

Respectfully submitted,

Patricia D. Viani, SMI Inc.

3.1.1 Qualification (Initial): The cleaning compound furnished under this specification shall be a product which has been tested and has passed the qualification tests specified herein and has been listed or approved for listing on the applicable Qualified Products List (QPL).

3.3 Toxicity: The cleaning compound shall have no adverse effect on the health of personnel or the environment when used for its intended purpose and with proper personal protective equipment (when required). The product shall be evaluated for aquatic toxicity with a 96-hour Fathead minnow (*Pimephales promelas*) bioassay and a 48-hour *Ceriodaphnia dubia* bioassay in accordance with Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, EPA/600/4-90/027. The percent survival at 1, 10, 50, and 100 ppm shall be reported for both organisms.

% of Fathead Minnows *(Pimephales promelas)* Surviving			
Concentration	After 24 hours	After 48 hours	After 96 hours
1 ppm	**100%**	**100%**	**100%**
10 ppm	**0%**	**0%**	**0%**
50 ppm	**0%**	**0%**	**0%**
100 ppm	**0%**	**0%**	**0%**

% of Cladoceran (*Ceriodaphnia dubia*) Surviving		
Concentration	After 24 hours	After 48 hours
1 ppm	**90%**	**50%**
10 ppm	**50%**	**20%**
50 ppm	**0%**	**0%**
100 ppm	**0%**	**0%**

Result Informational

3.3.4 Biodegradability: The supplier of the cleaning compound shall furnish certification from the surfactant manufacturers that the surfactants are readily biodegradable in accordance with 40 CFR, Part 796, Subpart D. Biodegradability testing shall be accomplished as specified in paragraph 4.5.22 on the finished product by an independent laboratory approved by the qualifying activity. Biodegradability on the finished product shall be determined over 28 days by the Shake Flask Method monitored by analysis of Total Organic Carbon (TOC). The Type I compound shall meet the requirement of a minimum of 75% biodegradable and Types II, III, and IV compounds shall meet the requirement of a minimum of 85 % biodegradable at the end of the 28-day period.

Biodegradability after 28 days: 87.1 %

Result Conforms

3.4 Compositional assurance: The cleaning compound shall be tested for nonvolatile matter as specified in paragraph 4.5.1. The concentrated cleaning compound and a 10% solution of the cleaning compound in distilled water shall be tested for pH as specified in paragraph 4.5.3. Results of these tests as well as an infrared spectrogram of the nonvolatile matter (see 4.8.2) and a gas chromatogram (see 4.8.1 for Type I only) shall be recorded by the qualifying activity for use in conformance inspections (see 4.3). Conformance inspection results for nonvolatile matter shall not differ by more than 2 percent absolute from the recorded value. Conformance inspection results for pH shall not differ by more than 1 pH unit from the recorded value. Conformance inspection infrared spectrograms and gas chromatograms shall show no significant difference when compared to the original qualifying spectrogram.

PROPERTY	RESULT
Nonvolatile matter	**13.1 %**
pH (undiluted)	**6.9**
pH (10%)	**N/A (RTU)**
Infrared spectrogram	**Attached**

Result Informational

3.5 Chemical properties.

3.5.1 Chemical requirements: The cleaning compound shall meet the requirements listed in Table I.

3.5.1 Chemical requirements (continued):

TABLE I

Requirement	Type IV		Test Method
	Min.	Max.	
Insoluble Matter (WT%)	—	0.05	4.5.2
Flash Point (°F) 10 % solution concentrate	 None[1] None[1]	 — ---	4.5.7
Emulsion Characteristics (mls free water) 5 minutes 8 hours 24 hours	 — — 11.0	 5.0 — —	4.5.8
Wet Adhesion Tape Test	Pass		4.5.27
% Cleaning Efficiency	90 %	---	4.5.21
Terpene Hydrocarbons (% WT)	—	None	4.5.23

1/ No flash point should be observed up to the boiling point of the compound.

4.5.2 Insoluble matter The percent insolubles shall be calculated as follows:

$$I = \frac{A-B}{W} \times 100$$

Where:
A = Final filter paper weight
B = Initial filter paper weight
W = Weight of sample
I = % wt. insoluble matter

Insoluble matter = < 0.01 %

Result Conforms

4.5.7 Flash point: The flash point of the concentrated cleaning compound (Type I, II, III and IV) shall be determined in accordance with ASTM D 56 (Tag Closed Cup) and for materials that have a tendency to form a surface film under the test conditions, use ASTM D 93. The flash point of the 10% solution in distilled water (Type I only) shall be determined in accordance with ASTM D 92.

No flash point observed to initial boiling point (212°F).

Result Conforms

3.5.1 Chemical requirements (continued):

4.5.8 Emulsion characteristics: Twenty ml of a 25% by volume solution (Types I and II) of the cleaning compound (12.5% by volume solution for Types III and IV) shall be placed in a 50 ml glass stoppered graduated cylinder. Twenty ml of lubricating oil conforming to MIL-PRF-2104, grade 10W, shall be added. An emulsion shall be formed by 10 inversions of the graduated cylinder followed by a vigorous 15-second shake. After the emulsion has stood for 5 minutes, the 15-second shake shall be repeated. At 5 minutes and 8 hours for the Type I and at 5 minutes and 24 hours for the Types II, III and IV cleaners, the amount of free water and cleaner which separates from the lubricating oil shall conform to the requirements of Table I.

Amount of free water remaining:
After 5 minutes ***fewer than 5 mls*** ***After 24 hours:*** ***>11 mls***

Result Conforms*

4.5.21 Cleaning Efficiency: The cleaning efficiency of the cleaning compound shall be reported as the average of three test results and shall conform to the requirements of Table I.

Cleaning Efficiency: 98.5% Result Conforms*

4.5.23 Terpene hydrocarbons (Type I only): An approved test procedure shall be used. Result Not applicable

3.5.2 Residue Rinsibility: When a freshly prepared solution of the cleaning compound is tested in accordance with 4.5.4, it shall not leave any residue or stains. A freshly prepared solution is defined as one being prepared no longer than 30 minutes prior to testing. The weight change shall be not greater than that obtained with standard hard water tested under the same conditions.

No residue nor stains; no weight change Result Conforms*

3.6 Physical properties (All types unless otherwise noted).

3.6.1 Heat stability: The concentrated cleaning compound, when tested in accordance with 4.5.5, shall show no marked color change or precipitation and shall not corrode or stain the AMS 5046 (SAE 1020) steel strip (a slight darkening of the steel strip shall not be objectionable). Layering or separation shall constitute failure if it does not return to its original homogeneous state upon cooling.

Visible corrosion of steel strip; rust-colored precipitation; no layering
Result Does not conform

** Test performed using "as received" solution (ready to use) instead of dilution required by specification.*

3.6.2 Cold stability: The concentrated cleaning compound shall return to its original homogeneous condition when tested in accordance with 4.5.6.

Compound returned to original homogeneous condition after 5 cycles

Result Conforms

3.6.3 Rheology (Type III only).

3.6.3.1 Consistency: When tested as specified in 4.5.24, the concentrated cleaning compound shall flow between 10 and 20 centimeters in 10 seconds. The product shall also exhibit rheology, which enables it to meet the sprayability requirement.

Result Not applicable

3.6.3.2 Sprayability: The concentrated cleaning compound, when dispensed at 45 psig and tested in accordance with 4.5.25, shall give satisfactory spray characteristics and deposit a uniform layer on a vertical surface 3 feet away from the nozzle.

Result Not applicable

3.7 Effect on metals (All types unless otherwise noted).

3.7.1 Hydrogen embrittlement: When tested in accordance with 4.5.9, the concentrated cleaner (all types) and a 10% solution of the cleaner (Types I, II, and IV only) in distilled water shall not cause hydrogen embrittlement of cadmium plated or IVD aluminum coated AISI 4340 steel.

Specimens: Type 1c, ***cadmium*** *plated in accordance with Treatment B of ASTM F519*

As received: ***No failures within 150 hours.***
Dilute (10 %): ***Not performed***

Result Conforms*

Specimens: Type 1c, grit blasted, ***IVD Aluminum*** *plated per MIL-DTL-83488D, Cl 2, Ty I.*

As received: ***No failures within 150 hours.***
Dilute (10 %): ***Not performed***

Result Conforms*

** Test performed using "as received" solution (ready to use) instead of dilution required by specification.*

3.7.2 Total immersion corrosion: When tested in accordance with 4.5.10 (ASTM F 483), the concentrated cleaning compound (all types) and a 10% solution of the cleaning compound (Types I, II and IV only) in distilled water shall not show any indication of staining, etching, pitting, or localized attack on any of the panels, or cause a weight change of an average of three (3) test panels greater than that shown in Table II. A slight discoloration of the panels shall not be objectionable. The cleaning compound shall not layer or separate for the duration of the test.

Table II Total Immersion Corrosion Requirements

Alloy	Weight Loss ($mg/cm^2/168hrs$)		
	Maximum allowed	**As received**	**10 %**
Magnesium (AZ 31B-H24) AMS 4377 surface treatment per SAE AMS-M-3171, Ty III	0.50	**0.07**	**NOT PERFORMED**
Aluminum, SAE AMS-QQ-A-250/4, T3 surface treatment per MIL-A-8625, Type I, Class I	0.15	**0.02**	
Aluminum, SAE AMS-QQ-A-250/4, Bare T3 Alloy	0.15	**0.02**	
Aluminum, SAE AMS-QQ-A-250/12, Bare T6 Alloy	0.15	**0.01**	
Titanium, SAE AMS-T-9046, 6Al-4V Cl III, Comp. C	0.10	**0.01**	
Steel, AMS 5046, Grade 1020	0.25	**0.02***	
Steel, 410 SS, Silver Plated per SAE AMS 2410	0.10	**0.02**	

Slight discoloration

Result Conforms*

3.7.3 Low-embrittling cadmium plate corrosion: Steel panels coated with low-embrittling cadmium plate immersed in the concentrated cleaning compound (all types) and a 10% solution of the cleaning compound (Types I, II and IV only) in distilled water shall not show a weight change greater than 0.14 mg/cm^2 for 24 hours when tested in accordance with 4.5.11.

As received: ***0.07* $mg/cm^2/24hrs$***
Dilute (10 %): *Not performed*

Result Conforms*

** Test performed using "as received" solution (ready to use) instead of dilution required by specification.*

3.7.4 Effects on unpainted metal surfaces: The concentrated cleaning compound (Type III only) and a 10% solution (Types I, II and IV only) of the cleaning compound in distilled water shall not cause streaking, stains or other deposits that cannot be easily removed with water when tested in accordance with 4.5.12.

Result Conforms*

3.7.5 Sandwich corrosion: When tested in accordance with 4.5.16, the concentrated cleaner (all types) and a 10% solution (Types I, II and IV only) shall show no corrosion in excess of that shown by control test coupons in ASTM D 1193, Type IV, reagent water.

	2024-T3 Bare Anodized	2024-T3 Alclad	7075-T6 Bare Anodized	7075-T6 Alclad
As received	**1**	**1**	**1**	**1**
Dilute (10%)	**Not performed**			
Control	1	1	1	1

[1]staining/etching in excess of 25% of the surface area

Result Conforms*

3.7.6 Wet adhesion tape test (Types II and IV): A ten (10) percent solution of the cleaning compound, when used as directed, shall remove soil from a painted surface in preparation fro repainting such that paint applied after cleaning with the compound shall adhere to the surface when tested in accordance with 4.5.27.

COATING SYSTEM	OBSERVATIONS
SET 1: Primer: MIL-PRF-85582, Type I, Class 1B Waterborne Epoxy Topcoat: MIL-PRF-85285 Type I High Solids Polyurethane, Color # 34092	**Coating system showed no sign of damage.**
SET 2: Primer: MIL-PRF-23377, Type I, Class C High Solids Epoxy Topcoat: MIL-PRF-85285 Type I High Solids Polyurethane, Color # 34092	**Coating system showed no sign of damage.**
SET 3: Primer: TT-P-2760, Type I, Class C High Solids Elastomeric, Polyurethane Topcoat: MIL-C-85285 Type I High Solids Polyurethane, Color # 34092	**Coating system showed no sign of damage.**

Result Conforms*

** Test performed using "as received" solution (ready to use) instead of dilution required by specification.*

3.8 Effect on painted surfaces: The concentrated cleaning compound (Type III only) and a 25% solution (Types I, II and IV) of the cleaning compound in distilled water shall not cause streaking, blistering, discoloration or a permanent decrease in film hardness of more than one (1) pencil hardness level when tested in accordance with 4.5.13. The Type I material shall be tested using only the (H) Polyurethane paint systems.

PANEL SET	RESULT	
	Conc.	25 %
E (Epoxy topcoat) Primer: MIL-PRF-23377, Ty I, Class C High-Solids Epoxy Primer Topcoat: MIL-PRF-22750 Epoxy Topcoat, Color #: 17925	**Pass**	Not performed
H (Polyurethane) Primer: MIL-PRF-23377, Ty I, Class C High-Solids Epoxy Primer Topcoat: MIL-PRF-85285 Ty I, Polyurethane, High Solids, Color #: 17925	**Pass**	
F (Enamel) Primer: MIL-PRF-23377, Ty I, Class C High-Solids Epoxy Primer Topcoat: TT-E-529 Enamel, Semi-gloss, Color #: 27925	**Pass**	

Result Conforms*

3.9 Stress crazing of MIL-PRF-5425 and MIL-PRF-25690 (Type A and C) acrylic plastics: The concentrated product (Type III only) and a 10% solution (Types I, II and IV) in distilled water shall not cause stress crazing or staining of acrylic plastics when tested in accordance with 4.5.14.

Material	**As received**	Dilution (10%)
MIL-PRF-5425 (Type A)	***Crazing: Fails**	Not performed
MIL-PRF-25690 (Type C)	**No crazing: Pass**	Not performed

Result *Does not conform

** Test performed using "as received" solution (ready to use) instead of dilution required by specification.*

3.10 Stress crazing of polycarbonate plastic: The concentrated product (Type III only) and a 10% solution (Types I, II and IV) in distilled water shall not cause stress crazing or staining of polycarbonate plastic conforming to MIL-P-83310 when tested in accordance with 4.5.15.

Material	**As received**	Dilution (10%)
MIL-P-83310 (Polycarbonate)	**Crazing: Fails**	Not performed

Result *Does not conform

3.11 Long-term storage stability: After being stored for a period of 12 months, in accordance with 4.5.17, the cleaning compound shall not layer, separate, precipitate or corrode the shipping container. Plastic containers shall not show leakage nor any cracking, crazing, or softening. All cleaning compounds shall meet the requirements of paragraphs 3.5.1, 3.7.1, 3.7.2, 3.15, and 3.16 of this specification.

Result Not performed

3.12 Hot dip galvanizing corrosion: The concentrated product (Type III only) and a 10% solution of the cleaning compound (Types I, II and IV) in distilled water shall not show a weight change of an average of three (3) test panels greater than 0.14 mg/cm2 when tested in accordance with 4.5.18.

As received: ***0.04 mg/cm²***
Dilute (10%): *Not performed*

Result *Conforms

3.13 Workmanship: The cleaning compound shall be a liquid having a uniform and homogenous appearance. The cleaning compound shall be manufactured from materials that shall produce a product harmless to metal surfaces and humans when used as directed.

Result To be Cert. by Mfr.

** Test performed using "as received" solution (ready to use) instead of dilution required by specification.*

3.14 Effect on polysulfide sealants: The concentrated cleaning compound (Type III only) and a 25% solution (Types I, II and IV) of the cleaning compound in distilled water shall not change the durometer hardness of the polysulfide sealant by more than 5 units when tested in accordance with 4.5.19.

Sealants:	***MIL-S-81733 Type 1:***	***< 5 units hardness change***
	MIL-S-8802 Type 1:	***< 5 units hardness change***

Result Conforms*

3.15 Rubber compatibility: The concentrated cleaning compound (Type III only) and a 25% solution (Types I, II and IV only) of the cleaning compound in distilled water shall not change the durometer hardness more than 5 units when tested in accordance with 4.5.20.

Rubbers:	***AMS 3204:***	***< 5 units hardness change***
	AMS 3209:	***< 5 units hardness change***

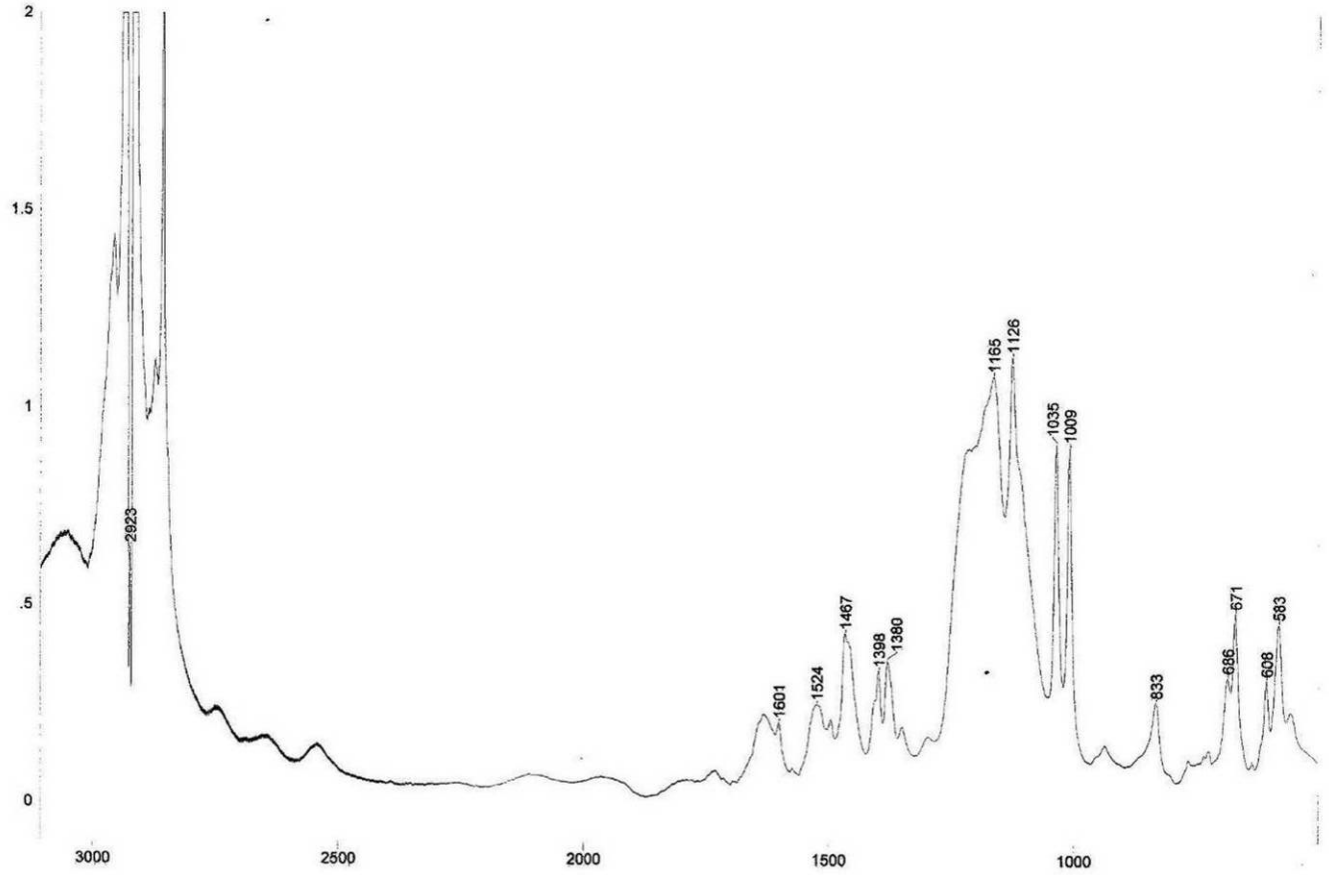
Result Conforms*

3.16 Effect on polyimide insulated wire: The cleaning compound, when tested according to 4.5.26, shall not cause dissolution, cracking, or dielectric breakdown (leakage) of the polyimide insulated wire in excess of that produced by distilled water.

Polyimide wire did not exhibit dissolution, cracking or dielectric breakdown.

Result Conforms

** Test performed using "as received" solution (ready to use) instead of dilution required by specification.*



Absorbance / Wavenumber (cm-1)

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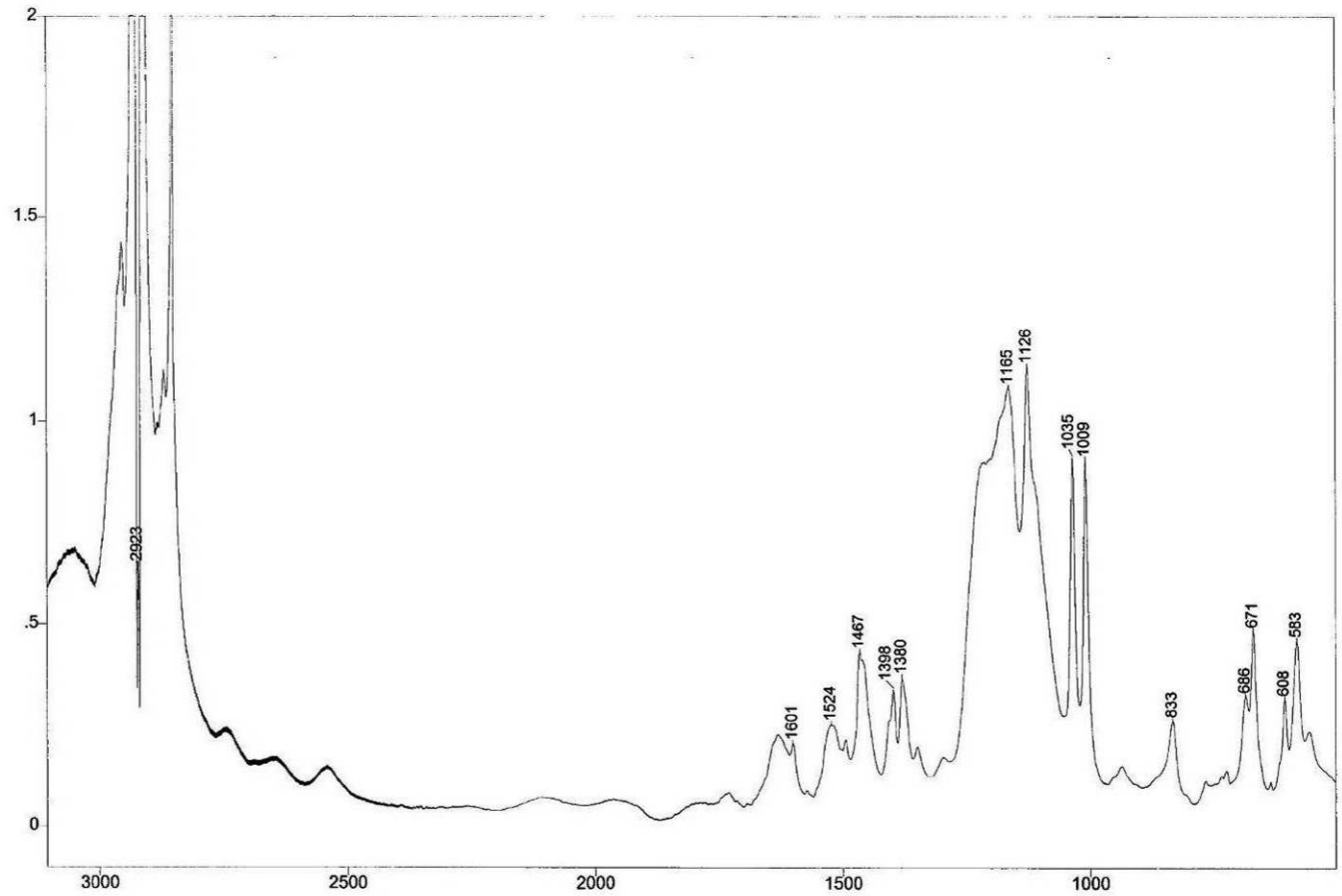

Absorbance / Wavenumber (cm-1)

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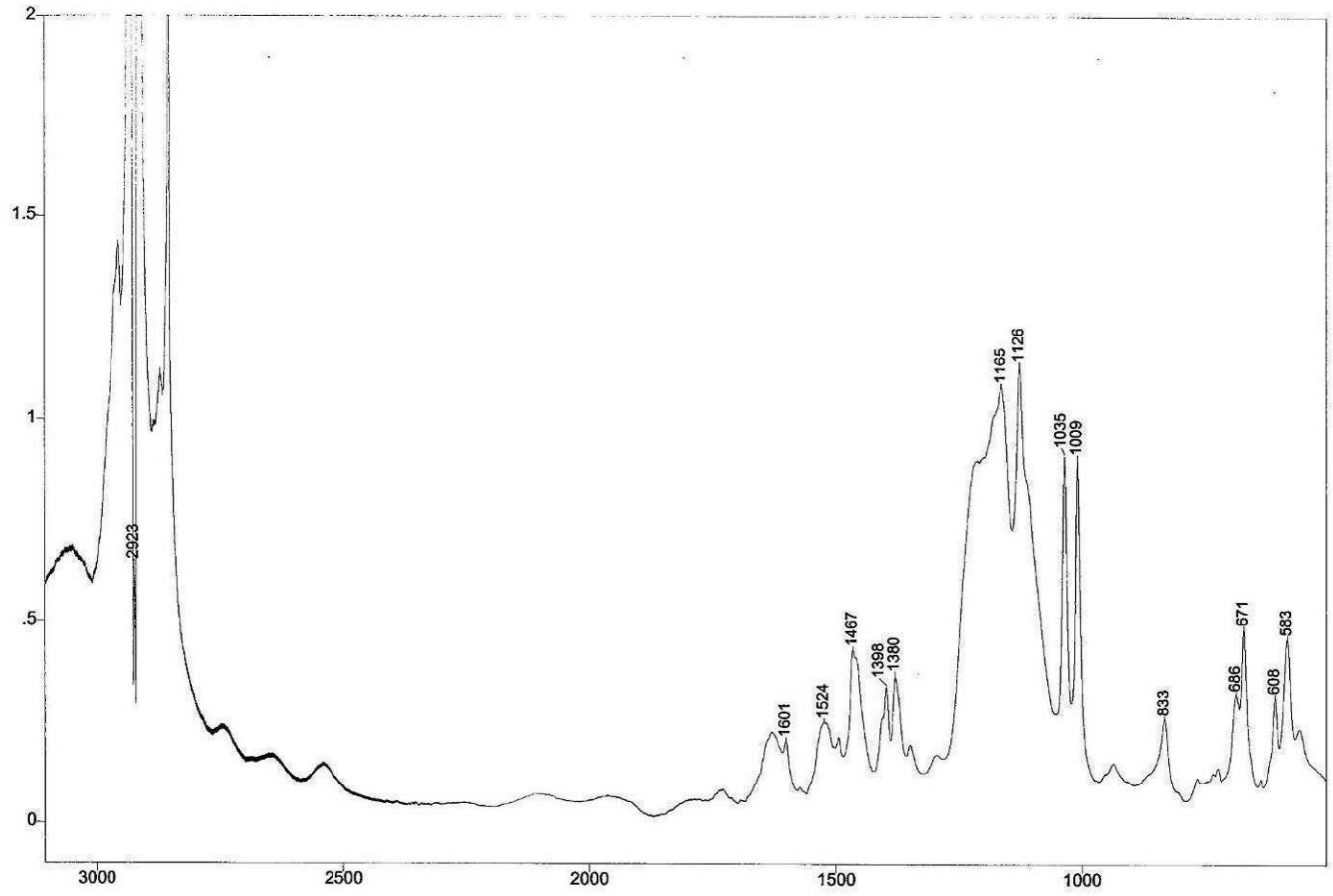

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SMI, Inc.

12219 SW 131 Avenue
Miami, Florida 33186-6401 USA

Phone: (305) 971-7047
Fax: (305) 971-7048

Attn: Sara Kuczek
Battelle Memorial Institute
505 King Avenue
Columbus, OH 43201-2693

Date: 04-Mar-2005

SMI/REF: 04DEC086

Product: **Eagle Kleen III Lot # C531-84-1** *(received 01-December-2004)*

Dilution: Ready to use

ASTM D 323
Test Method for Vapor Pressure of
Petroleum Products (Reid Method)

SCOPE

This test method provides procedures for the determination of vapor pressure of gasoline, volatile crude oil, and other volatile petroleum products.

SUMMARY OF TEST METHOD

The liquid chamber of the vapor pressure apparatus is filled with the chilled sample and connected to the vapor chamber that has been heated to 37.8°C (100°F) in a bath. The assembled apparatus is immersed in a bath at 37.8°C (100°F) until a constant pressure is observed. The reading, suitably corrected, is reported as the Reid vapor pressure.

Sample	Reid Vapor Pressure
Eagle Kleen III	<0.2 mm Hg @ 20°C

Respectfully submitted,

Patricia D. Viani, SMI Inc.

SMI, Inc.

12219 SW 131 Avenue
Miami, Florida 33186-6401 USA

Phone: (305) 971-7047
Fax: (305) 971-7048

Attn: BATTELLE MEMORIAL INSTITUTE
505 King Avenue
Columbus, OH 43201

Date: 07-Mar-2005
SMI REF: 04DEC086

PRODUCT: **EAGLE KLEEN III** (Lot # C531-84-1)
(received 01-Dec-2004)

40 CFR 796.3100: AEROBIC AQUATIC BIODEGRADATION
Code of Federal Regulations
Environmental Protection Agency
Title 40: Protection of Environment
Part 796: Chemical Fate Testing Guidelines
Shake Flask Method

Summary of Results:

Based on dissolved organic carbon analysis:

"EAGLE KLEEN III" = 87.1 % Biodegradable in 28 days

See Appendix A for graphical representation of Biodegradability vs. Time .

PROCEDURE

I. Introduction

This procedure provides a way to determine the rate and extent of aerobic biodegradation that might occur when chemical substances are released to aquatic environments. A high biodegradability result in this test provides evidence that the test substance will be biodegradable in natural aerobic freshwater environments. A low biodegradability result may not necessarily indicate poor biodegradation, as other factors may interfere, such as inhibition of the microbial inoculum by the test material.

EPA 796.3100: AEROBIC AQUATIC BIODEGRADATION

II. Principle of the Test Method

The method consists of a 2-week inoculum buildup period during which the microbes are allowed to adapt to the test compound. The acclimated media containing a defined amount of test compound is added to specially equipped Erlenmeyer flasks. The test media is sampled periodically and analyzed for dissolved organic carbon (DOC). A reservoir filled with barium hydroxide is utilized to measure the amount of carbon dioxide evolved. The degree of biodegradation is determined by comparison of the extent of DOC disappearance and the amount of carbon dioxide liberated. Control flasks containing no test compounds are run simultaneously and are used to estimate the degree of ultimate biodegradation. Reference substances which will exhibit ultimate biodegradation may be run simultaneously to check the activity of the inoculum. If the reference samples do not exhibit at least 60 percent of theoretical maximum carbon dioxide, and at least 70 percent DOC removal within 28 days, the test will be regarded as invalid and shall be repeated using different inoculum.

This method is believed to be appropriate for a screening test which has solely an acceptance but no rejective function.

III. Test Procedure

The total organic carbon (TOC) of the test compound is first determined by analysis or calculation if the formulation is known. Determination of the minimum inhibitory concentration is useful to insure that the test compound will not be inhibitory to the microbes at the required concentration. The shake flask apparatus is assembled utilizing a 2-liter Erlenmeyer flask and a 50 ml centrifuge tube. The tube containing 10 mls of barium hydroxide will be suspended over the contents of the flask in such a way that liberated carbon dioxide may diffuse into the barium hydroxide, while allowing the contents of the tube to be removed for analysis without spilling into the test media. Glass tubing may be utilized as access into the flask for sparging, venting, and sampling.

Stock solutions I, II, and III are prepared (see Appendix B), along with 0.2 N barium hydroxide and 0.1 N HCl. Acclimation medium is prepared by adding 1 ml each of stock solutions I, II and III to 1 liter of distilled, deionized water (DIW). The microbial inoculum is obtained from sewage and soil or from Polyseed and is added to the acclimation medium. Test compounds are added incrementally during the acclimation period at concentrations equivalent to 4, 8, and 8 mg/L carbon on days 0, 7, and 11, respectively. On day 14, the medium is ready for use in the test.

EPA 796.3100: AEROBIC AQUATIC BIODEGRADATION

Biodegradability test flasks are prepared by adding 100 mls of acclimation medium to 900 mls of DIW along with 1 ml each of solutions I, II, and III to the 2-liter Erlenmeyers. Additional test compound equivalent to 10 mg/L carbon is added to the flasks. Ten mls of barium hydroxide are added to the suspended reservoirs in each flask and 10 mls are also saved for use as a titration blank. Flasks are sparged with carbon dioxide-free air, sealed and placed on a shaking table (approx. 125 rpm) at 20 - 25 deg C in the dark. Test flasks should be run in triplicate and sampling should occur at time zero and at least four other times to allow for a smooth plot of biodegradation. Each sample for DOC analysis is first centrifuged or filtered through a 0.45 micrometer or smaller pore diameter. On the day prior to terminating the test, 3 mls of 20 percent sulfuric acid are added to release carbonate bound carbon dioxide.

IV. ANALYTICAL MEASUREMENTS

The quantity of carbon dioxide evolved is measured by titration of the entire barium hydroxide sample with 0.1 N HCl to the phenolphthalein end point, blank subtracted. Theoretically, 10 mg of carbon is converted to 0.833 mmol of carbon dioxide. Absorbed carbon dioxide precipitates as barium carbonate, causing a reduction in alkalinity by the equivalent of 16.67 ml of 0.1 N HCl for complete conversion of the test compound carbon to carbon dioxide. Therefore, the percent theoretical carbon dioxide evolved from the test compound is calculated at any sampling time from the formula:

$$\% \, CO_2 \text{ evolution} = [(TF - CF)/16.67] \cdot 100$$

where:

TF = mls of 0.1 N HCl used in titration of test flask

CF = mls of 0.1 N HCl used in titration of control flask

EPA 796.3100: AEROBIC AQUATIC BIODEGRADATION

The DOC analysis is performed using a suitable organic carbon method. The percent DOC disappearance from the test compound is calculated from the formula:

$$\% \text{ DOC removal} = [1 - (DTF_x - DCF_x)/(DTF_o - DCF_o)] \cdot 100$$

where:

DTF = Dissolved organic carbon from test flask
DCF = Dissolved organic carbon from control flask

o = Day zero measurements
x = Day x measurements

V. REPORT OF RESULTS

Inoculum: Polyseed and Mixed inoculum

Date Received: Jan, 2004

Source: Fisher Scientific and Metro-Dade County Water & Sewer Authority

Storage: Ambient temperature, used within 24 hours

Minimum Inhibitory Concentration: MIC < 3.125 % (non-inhibitory to microbes at concentrations lower than 3.125%)

Percent Biodegradation based on DOC analysis:

EAGLE KLEEN III:	**87.1 %** after 28 days (see Table 1)
Reference (Sodium citrate):	92.6 % after 28 days (see Table 1)

EPA 796.3100: AEROBIC AQUATIC BIODEGRADATION

Summary: Since the test compound was found to be over 70 % biodegradable based on the DOC analysis, it is reasonable to assume that the substance will undergo rapid and ultimate biodegradation in aerobic aquatic environments, also known as "ready biodegradability". The test is validated by the fact that the reference compound, sodium citrate, exhibited a biodegradability over 70%.

The percent biodegradability based on carbon dioxide evolution is typically lower than that of the DOC based numbers. In this case, the carbon dioxide evolution measured was significant, both on the test compound and on the reference, and the results generally agree.

Respectfully submitted,

Patricia D. Viani
SMI, Inc.

EPA 796.3100: AEROBIC AQUATIC BIODEGRADATION

TABLE I - DISSOLVED ORGANIC CARBON (DOC) VALUES

Sample: **EAGLE KLEEN III**

		DAY 0	DAY 7	DAY 14	DAY 21	DAY 28
	A	35.7	11.6	9.2	8.3	7.3
	B	39.1	9.3	8.8	7.2	7.4
	C	37.0	15.2	13.8	9.9	8.5
AVERAGE		37.3	12.0	10.6	8.5	7.7
CORRECTED AV		34.0	8.7	7.2	5.1	4.4
% BIODEGRADED		**N/A**	**74.3%**	**78.7%**	**85.1%**	**87.1%**

Reference: Sodium Citrate

	A	38.0	14.2	6.8	5.2	5.5
	B	39.9	9.1	6.9	6.2	6.0
	C	39.1	8.4	7.3	6.7	6.5
AVERAGE		39.0	10.6	7.0	6.0	6.0
CORRECTED AV		35.7	7.3	3.6	2.6	2.6
% BIODEGRADED		N/A	79.6%	89.8%	92.6%	92.6%
BLANK	A	3.5	3.1	3.3	3.7	3.0
	B	3.0	3.6	3.6	3.2	3.7
	C	3.4	3.2	3.2	3.3	3.4
AVERAGE		3.3	3.3	3.4	3.4	3.4

EPA 796.3100: AEROBIC AQUATIC BIODEGRADATION

Table II - Titration Data for CO2 Evolution

Sample: **EAGLE KLEEN III**

			DAY 7	DAY 14	DAY 21	DAY 28	
	A		6.8	8.8	15.8	18.0	
	B		5.6	12.4	16.0	18.0	
	C		6.0	9.2	16.6	17.6	
AVERAGE			6.1	10.1	16.1	17.9	
CORRECTED AVG			11.9	7.6	1.9	0.2	
% BIODEGRADED			20.9%	13.4%	3.3%	0.4%	
mls theoretical:		56.7				**TOTAL=**	**38.0%**

Reference: Sodium Citrate

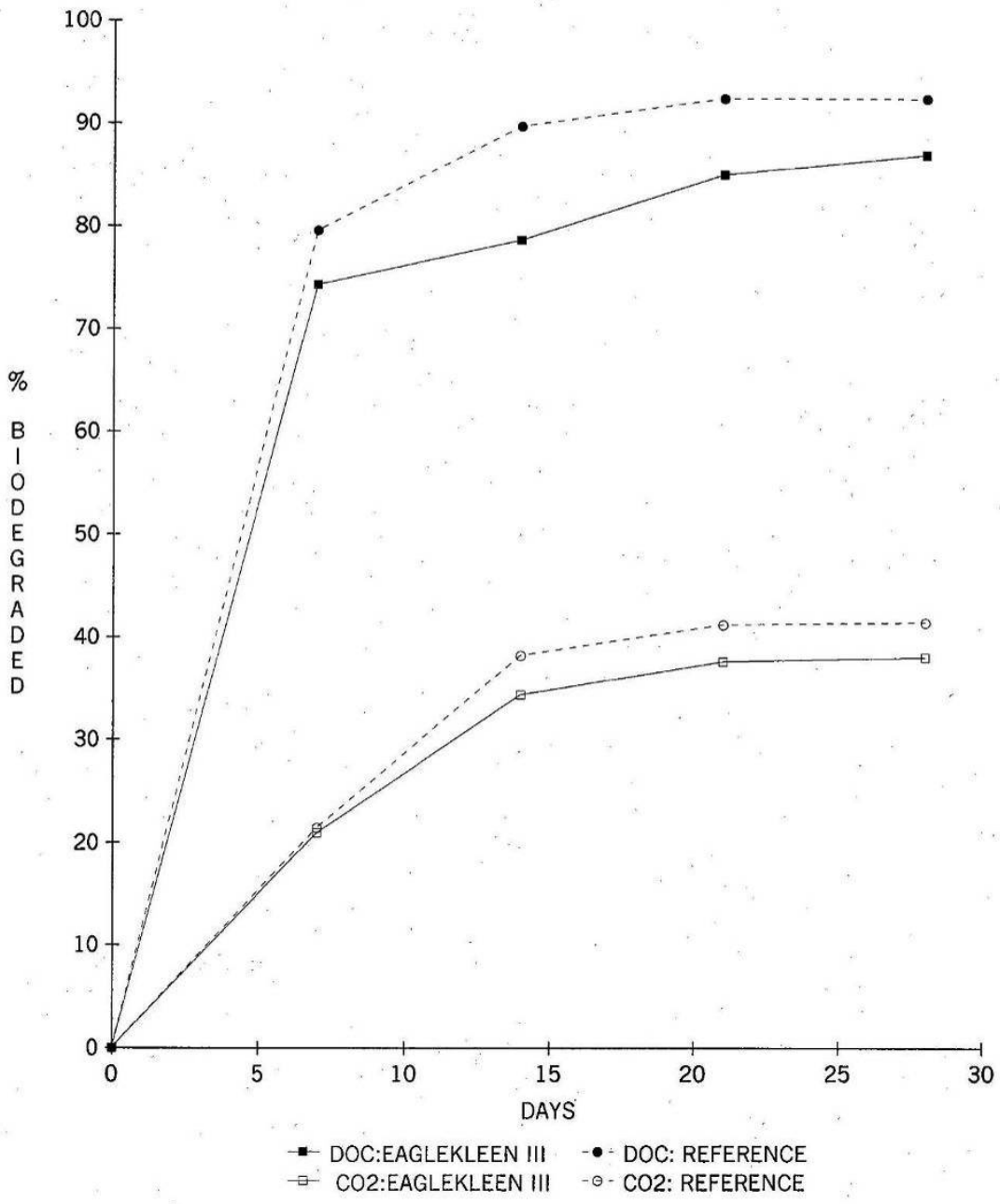
	A		4.6	7.8	17.0	18.0	
	B		6.0	9.2	14.8	17.8	
	C		5.2	6.2	16.8	18.0	
AVERAGE			5.3	7.7	16.2	17.9	
CORRECTED AVG			12.7	10.0	1.8	0.1	
% BIODEGRADED			21.4%	16.8%	3.0%	0.2%	
mls theoretical:		59.5				TOTAL =	41.4%
BLANK	A		18.2	18.0	18.2	17.8	
	B		18.2	17.4	18.0	17.8	
	C		17.6	17.8	17.8	18.6	
AVERAGE			18.0	17.7	18.0	18.1	

EPA 796.3100: AEROBIC AQUATIC BIODEGRADATION

Appendix A

BIODEGRADABILITY VS. TIME

EAGLE KLEEN III



Appendix B

STOCK SOLUTIONS I, II, AND III

SOLUTION I:	35	g/L	NH_4Cl
	15	g/L	KNO_3
	75	g/L	$K_2HPO_4 \cdot 3H_2O$
SOLUTION II:	25	g/L	$NaH_2PO_4 \cdot H_2O$
	10	g/L	KCl
	20	g/L	$MgSO_4$
	1	g/L	$FeSO_4 \cdot 7H_2O$
	adjust pH of Soln II to 3.0		
SOLUTION III:	5	g/L	$CaCl_2$
	0.05	g/L	$ZnCl_2$
	0.5	g/L	$MnCl_2 \cdot 4H_20$
	0.05	g/L	$CuCl_2$
	0.001	g/L	$CoCl_2$
	0.001	g/L	H_3BO_3
	0.0004	g/L	MoO_3

SMI, Inc.

12219 SW 131 Avenue
Miami, Florida 33186-6401 USA

Phone: (305) 971-7047
Fax: (305) 971-7048

Attn: Nick Conkle
Battelle Memorial Institute
505 King Ave
Columbus, OH 43201

Date: 24-Jun-2005

SMI/REF: 04JUN604

Product: **EAGLE KLEEN III**
(received 20-Jun-2005)

Dilution: Ready to use

Modified testing (product was tested neat - [undiluted])
MIL-PRF-87937D (24 Sep 2001)
CLEANING COMPOUND, AEROSPACE EQUIPMENT
Type IV - Heavy Duty, Water Dilutable Cleaning Compound

3.9 Stress crazing of MIL-PRF-5425 and MIL-PRF-25690 (Type A and C) acrylic plastics: The concentrated product (Type III only) and a 10% solution (Types I, II and IV) in distilled water shall not cause stress crazing or staining of acrylic plastics when tested in accordance with 4.5.14.

Material	**As received**	Dilution (10%)
MIL-PRF-5425 (Type A)	**Crazing: Fails**	Not performed
MIL-PRF-25690 (Type C)	**No crazing: Pass**	Not performed

Result Does not conform

3.10 Stress crazing of polycarbonate plastic: The concentrated product (Type III only) and a 10% solution (Types I, II and IV) in distilled water shall not cause stress crazing or staining of polycarbonate plastic conforming to MIL-P-83310 when tested in accordance with 4.5.15.

Material	**As received**	Dilution (10%)
MIL-P-83310 (Polycarbonate)	**Crazing: Fails**	Not performed

Result Does not conform

** Tests performed using "as received" solution (ready to use) instead of dilution required by specification.*

Respectfully submitted,

Patricia D. Viani, SMI Inc.

Appendix E

SMI Results from Eagle Kleen III Vapor Pressure Testing

SMI, Inc.

12219 SW 131 Avenue
Miami, Florida 33186-6401 USA

Phone: (305) 971-7047
Fax: (305) 971-7048

Attn: Sara Kuczek
Battelle Memorial Institute
505 King Avenue
Columbus, OH 43201-2693

Date: 04-Mar-2005

SMI/REF: 04DEC086

Product: **Eagle Kleen III Lot # C531-84-1** *(received 01-December-2004)*

Dilution: Ready to use

ASTM D 323
Test Method for Vapor Pressure of
Petroleum Products (Reid Method)

SCOPE

This test method provides procedures for the determination of vapor pressure of gasoline, volatile crude oil, and other volatile petroleum products.

SUMMARY OF TEST METHOD

The liquid chamber of the vapor pressure apparatus is filled with the chilled sample and connected to the vapor chamber that has been heated to 37.8°C (100°F) in a bath. The assembled apparatus is immersed in a bath at 37.8°C (100°F) until a constant pressure is observed. The reading, suitably corrected, is reported as the Reid vapor pressure.

Sample	Reid Vapor Pressure
Eagle Kleen III	<0.2 mm Hg @ 20°C

Respectfully submitted,

Patricia D. Viani, SMI Inc.

Appendix F

SMI Results from Eagle Kleen III Toxicity Testing

SMI, Inc.

12219 SW 131 Avenue
Miami, Florida 33186-6401 USA

Phone: (305) 971-7047
Fax: (305) 971-7048

Attn: Nick Conkle
Battelle Memorial Institute
505 King Avenue
Columbus, Oh 43201-2693

Date: 09-Dec-2005

SMI/REF: 05XCT045

Product: **EAGLE KLEEN III (50540-98-5)** *(received 27-Oct-2005)*

Dilution: Ready to Use

Partial testing in accordance with:
AMS 1424F
Deicing/Anti-icing, Fluid, Aircraft SAE Type I
(Fluid is ready to use)

3.1.4.4 Aquatic Toxicity: Formulated fluid shall be tested in accordance with EPA (40CFR 797.1300 and 797.1400, revised July 1, 1989 and 40 CFR 136.3) or OECD (Organization for Economic Cooperation and Development Guidelines for Testing of Chemicals, Methods 202 and 203) procedures using test species required by regulatory agencies for permitted discharges. Examples include: fathead minnows (96-hour LC_{50}), *Ceriodaphnia dubia* (48-hour EC_{50}), *Daphnia magna* (48-hour EC_{50}) and rainbow trout (96-hour LC_{50}). The LC_{50} (for fish) or EC_{50} (for invertebrates) concentration (the highest concentration at which 50% of the organisms do not survive the test period) shall be given in milligrams per liter.

EPA 40 CFR 797.1300 DAPHNID ACUTE TOXICITY TEST
Daphnia magna, static system
48 hour LC_{50}: 25 mg/L

EPA 40 CFR 797.1400 FISH ACUTE TOXICITY TEST
Pimephales promelas, static system
96 hour LC_{50}: 30 mg/L

Result Informational

Respectfully submitted,

Patricia D. Viani, SMI, Inc.

SMI, Inc.

12219 SW 131 Avenue
Miami, Florida 33186-6401 USA

Phone: (305) 971-7047
Fax: (305) 971-7048

Attn: Nick Conkle
Battelle Memorial Institute
505 King Avenue
Columbus, Oh 43201-2693

Date: 09-Dec-2005

SMI/REF: 05XCT046

Product: **SAFETY KLEEN (50540-98-24)** *(received 27-Oct-2005)*

Dilution: Ready to Use

Partial testing in accordance with:
AMS 1424F
Deicing/Anti-icing, Fluid, Aircraft SAE Type I
(Fluid is ready to use)

3.1.4.4 Aquatic Toxicity: Formulated fluid shall be tested in accordance with EPA (40CFR 797.1300 and 797.1400, revised July 1, 1989 and 40 CFR 136.3) or OECD (Organization for Economic Cooperation and Development Guidelines for Testing of Chemicals, Methods 202 and 203) procedures using test species required by regulatory agencies for permitted discharges. Examples include: fathead minnows (96-hour LC_{50}), *Ceriodaphnia dubia* (48-hour EC_{50}), *Daphnia magna* (48-hour EC_{50}) and rainbow trout (96-hour LC_{50}). The LC_{50} (for fish) or EC_{50} (for invertebrates) concentration (the highest concentration at which 50% of the organisms do not survive the test period) shall be given in milligrams per liter.

EPA 40 CFR 797.1300 DAPHNID ACUTE TOXICITY TEST
Daphnia magna, static system
48 hour LC_{50}: 125 mg/L

EPA 40 CFR 797.1400 FISH ACUTE TOXICITY TEST
Pimephales promelas, static system
96 hour LC_{50}: >70,000 mg/L*

Note: Product left a distinct layer on top of water layer. Since product was not water soluble, results are suspect.

Result Informational

Respectfully submitted,

Patricia D. Viani, SMI, Inc.

SMI, Inc.

12219 SW 131 Avenue
Miami, Florida 33186-6401 USA

Phone: (305) 971-7047
Fax: (305) 971-7048

Attn: Nick Conkle
Battelle Memorial Institute
505 King Avenue
Columbus, Oh 43201-2693

Date: 09-Dec-2005

SMI/REF: 05XCT047

Product: **TURCO 6849 SOLN 20% by volume (50540-98-13)** *(received 27-Oct-2005)*

Dilution: Ready to Use

Partial testing in accordance with:
AMS 1424F
Deicing/Anti-icing, Fluid, Aircraft SAE Type I
(Fluid is ready to use)

3.1.4.4 Aquatic Toxicity: Formulated fluid shall be tested in accordance with EPA (40CFR 797.1300 and 797.1400, revised July 1, 1989 and 40 CFR 136.3) or OECD (Organization for Economic Cooperation and Development Guidelines for Testing of Chemicals, Methods 202 and 203) procedures using test species required by regulatory agencies for permitted discharges. Examples include: fathead minnows (96-hour LC_{50}), *Ceriodaphnia dubia* (48-hour EC_{50}), *Daphnia magna* (48-hour EC_{50}) and rainbow trout (96-hour LC_{50}). The LC_{50} (for fish) or EC_{50} (for invertebrates) concentration (the highest concentration at which 50% of the organisms do not survive the test period) shall be given in milligrams per liter.

EPA 40 CFR 797.1300 DAPHNID ACUTE TOXICITY TEST
Daphnia magna, static system
48 hour LC_{50}: 150 mg/L

EPA 40 CFR 797.1400 FISH ACUTE TOXICITY TEST
Pimephales promelas, static system
96 hour LC_{50}: 225 mg/L

Result Informational

Respectfully submitted,

Patricia D. Viani, SMI, Inc.

SMI, Inc.

12219 SW 131 Avenue
Miami, Florida 33186-6401 USA

Phone: (305) 971-7047
Fax: (305) 971-7048

Attn: Nick Conkle
Battelle Memorial Institute
505 King Avenue
Columbus, Oh 43201-2693

Date: 09-Dec-2005

SMI/REF: 05XCT048

Product: **RUST BLOC SOLN 4% by weight (50540-98-20)** *(received 27-Oct-2005)*

Dilution: Ready to Use

Partial testing in accordance with:
AMS 1424F
Deicing/Anti-icing, Fluid, Aircraft SAE Type I
(Fluid is ready to use)

3.1.4.4 Aquatic Toxicity: Formulated fluid shall be tested in accordance with EPA (40CFR 797.1300 and 797.1400, revised July 1, 1989 and 40 CFR 136.3) or OECD (Organization for Economic Cooperation and Development Guidelines for Testing of Chemicals, Methods 202 and 203) procedures using test species required by regulatory agencies for permitted discharges. Examples include: fathead minnows (96-hour LC_{50}), *Ceriodaphnia dubia* (48-hour EC_{50}), *Daphnia magna* (48-hour EC_{50}) and rainbow trout (96-hour LC_{50}). The LC_{50} (for fish) or EC_{50} (for invertebrates) concentration (the highest concentration at which 50% of the organisms do not survive the test period) shall be given in milligrams per liter.

EPA 40 CFR 797.1300 DAPHNID ACUTE TOXICITY TEST
Daphnia magna, static system
48 hour LC_{50}: 79,200 mg/L

EPA 40 CFR 797.1400 FISH ACUTE TOXICITY TEST
Pimephales promelas, static system
96 hour LC_{50}: 33,500 mg/L

Result Informational

Respectfully submitted,

Patricia D. Viani, SMI, Inc.