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OZONE DEPLETING CHEMICAL (ODC) REPLACEMENT - ALTERNATIVE CLEANING SOLVENTS AND LUBRICANTS

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The purpose of this study was to identify suitable alternative cleaning solvents and lubricants to replace 1,1,2-trichloro-1,2,2-trifluoroethane (CFC-113) and 1,1,1-trichloroethane (TCA) in mechanical degreasing operations, in an anti-seize material used to lubricate high-torque fittings, and in electrical cleaning operations. The study was conducted at the Engineering Propulsion Laboratory (EPL) Air Force Plant PJKS. Over sixty candidate materials were evaluated. This study was demonstrative not only in eliminating ODC usage but also in selecting materials which could improve cleaning and operational requirements. Because many of the analysis and test techniques can be easily integrated into other industry applications, investigation of these techniques in other aerospace industry programs and processes is recommended.

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TABLE OF CONTENTS

TABLE OF CONTENTS (CONCLUDED)

APPENDIX

LIST OF TABLES

LIST OF FIGURES

LIST OF FIGURES (CONCLUDED)

EXECUTIVE SUMMARY

Martin Marietta was contracted by the government to complete a study to replace ozone depleting chemicals (ODCs) in use at Air Force Plant PJKS. This facility is an Air Force owned, contractor operated facility located at Martin Marietta Technologies, Inc. (MMTI) in Denver Colorado. Applications involving ODCs at this facility have included 1,1,1-trichloroethane (TCA) and 1,1,2-trichloro-1,2,2-trifluoroethane, also known as Freon-113® or Chlorofluorocarbon 113 (CFC-113). These two chemicals have been used extensively at PJKS for over twenty years. The three primary areas of use investigated in this study are in mechanical degreasing operations, in an anti-seize material used to lubricate high-torque fittings, and in electrical cleaning operations.

As part of this study, the chemistry and physics of cleaning operations were evaluated. Performance characteristics of good cleaners include the ability to dislodge the contaminant, which is favored by a low surface tension cleaner. The dissolution of the contaminant was also discussed along with several empirical and theoretical descriptions of solubility. Dispersion of the contaminant in the cleaner is also important because this process prevents recontamination of the cleaned surface after removal from the cleaner. Finally, a family of solvents was generated using a three-component solubility parameter, and this description was useful in identifying solvents required for dissolving a particular chemical residue.

Over sixty replacement chemicals were identified as candidates for mechanical degreasing operations. From this initial set, ten chemicals showed exceptional promise, and these were evaluated in depth for cleaning power, materials compatibility, propellant compatibility, and soils loading. The latter criteria was a measure of the reusability of the cleaner after processing contaminated parts. Four of these ten cleaners, Bioact 280, Biogenic Regent, EP921, and Parts Prep were taken through validation and demonstration tests in which conformance to applicable process specifications and cleaning performance of actual facility hardware were evaluated. Of these four cleaners, only Biogenic Regent displayed unacceptable performance (high non-volatile residues after cleaning Drilube contaminants). All of the remaining three candidates are acceptable replacements for TCA and CFC-113, although special precautions must be taken when using EP921 for propellant service. The preferred alternative cleaning solvent is Bioact 280; EP921 and Parts Prep will be retained as back-up cleaners.

For replacement of an anti-seize compound, three requirements were identified. These were the ability of the compound to lubricate threads and prevent metal galling, the ability of the compound to protect against environmental corrosion, and the ability of the compound to protect against metal seizure after exposure to high temperatures. Candidate replacements included Fel-Pro C5A (vendor modified to be ODC-free), Anti-Seize Special, Loctite A-S 767, and Dow Molykote 1000. All of these candidates performed well in the lubrication and corrosive atmosphere test, but only the Dow Molykote 1000 was able to protect fittings from galling after exposure to 1200°F.

In electrical cleaning operations, three uses of ODC solvents were identified: contact cleaning, tape head cleaning, and strain gage bonding. In contact cleaning, the objective was to replace the existing ODC cleaner, Kontact Restorer, with a suitable ODC-free substitute. The importance of combining contact lubrication with cleaning was demonstrated in a contact life cycle test in which proper lubrication reduced wear and maintained electrical performance. Of the five replacement contact cleaners evaluated, three performed better than the existing cleaner, and are easily implemented in the Instrumentation Shop at the PJKS facility. They are Miller Stephenson MS938/CO2, Zip Chem CS Cleaner, and CRC Contact Cleaner HF. The Miller Stephenson formulation is a hydrochlorofluorocarbon (HCFC) and will be subject to future environmental regulations. In tape head cleaning, important criteria for selecting alternatives was the lack of residue upon evaporation, and compatibility with tape drive materials. For this reason, both high evaporation rates and high chemical purity were required. Heptane and pure grain ethyl alcohol were selected as alternative tape head cleaners. In strain gage bonding, both the adhesive formulation, and the substrate cleaner contained TCA. For the adhesive, Micromeasurements Co. reformulated its product using isopropyl alcohol (IPA) as a carrier solvent. Tests performed at Martin Marietta showed improved adhesive performance with this substitution. For the cleaner, both Bio-T-Max (a water soluble terpene blend) and EP921 (which contains propylene carbonate and d-limonene) cleaned better than the existing TCA.

This project was demonstrative in not only eliminating chemicals known to deplete the ozone layer, but also in selecting compounds which could improve cleaning and operational requirements. Because many of the analysis and test techniques used to identify and select alternatives can be easily integrated into other defense industry applications, application and/or modification of the these technologies for other aerospace industry pollution prevention programs is recommended.

Preface

This final report was prepared by Martin Marietta Technologies, Inc. PO Box 179, Denver, Colorado 80201 in accordance with Contract F04701-85-C-0019 Modification Number T00827, Martin Marietta Corporation Non-UPI Support Task Order (NUSTO) Task Proposal No. 93-018, " Ozone Layer Depleting Substance (OLDS) Elimination Projects", for Wright Patterson Air Force Base, Dayton, OH 45433. Efforts documented in this report were performed between May 1993 and May 1994. Captain Norman LeClair, Captain William Shelton, and Mr. Jason Lewis were the Air Force Project Officers.

1.0 INTRODUCTION

1.1 Objective

The objective of this effort was to find suitable alternatives to ODCs used in specific projects at Air Force property PJKS. PJKS is Air Force owned, and is operated by Martin Marietta in Denver, Colorado. This effort specifically addressed the use of CFC-113 and TCA used for parts degreasing, the use of Fel-Pro C5A anti-seize compound which contains TCA, and the use of CFC-113 and TCA in electrical and electronic parts cleaning. The specific objectives are listed below.

- Identify suitable alternatives to ODCs at PJKS by April 1994.
- Investigate alternatives to CFC-113 and TCA for mechanical parts cleaning.
- Investigate alternatives to Fel-Pro C5A
- Investigate alternatives to CFC-113 and TCA for electrical parts cleaning.
- Demonstrate that the performance of alternatives is equivalent to or better than existing ODCs in the performance categories listed below
    - Removes contamination.
    - Compatible with materials.
    - Compatible with propellants.
    - Compliant with safety and environmental requirements.
    - Meets minimum cleanliness requirements of governing specifications.
    - Lubricating properties (anti-seize)
    - Sealing properties (anti-seize)
    - Service temperature range (anti-seize)
    - Exhibits low contact resistance after cleaning (electrical)
    - Protects contacts against mechanical wear (electrical)
- Provide sound technical information to facilitate the use of test results at other government sites.
- Identify the specifications and operations which require ODC use.
- Determine the costs of the alternatives including process and life-cycle costs.

1.2 Background

The Montreal Protocol and the 1990 Clean Air Act Amendments mandate Freon-113® (CFC-113), other chlorinated fluorocarbons (CFCs) and 1,1,1-Trichloroethane (TCA) be banned from production after December 31, 1995. These chemicals will become significantly more expensive each year, and will not be readily available after 1995. In response to these concerns, the Air Force has formulated policy that prohibits purchase of these solvents for Air Force use after April 1, 1994. The Air Force has also made their policies applicable to Government Owned Contractor Operated (GOCO) properties. Martin Marietta Technologies, Inc. (MMTI) operates the Engineering Propulsion Lab (EPL), a laboratory located on Air Force plant called PJKS. In order to fulfill current and future contracts with the Air Force and others, Martin Marietta must institute processes compatible with the new environmental guidelines, and these processes must be fully evaluated and qualified to ensure technical performance and mission success requirements are met.

Through the Titan IV contract, the Air Force has funded a study to investigate alternatives to ozone depleting chemicals (ODC) in use at PJKS. The study will not only investigate eliminating ODC usage at PJKS, but will also provide other GOCOs and Air Force facilities with the technical information needed to eliminate ODCs from other operations. The resultant technology benefits both the Air Force and MMTI. This study deals specifically with the following processes and operations that currently use ODCs at the Air Force PJKS facility.

- CFC-113 for parts cleaning and degreasing

- TCA as a parts cleaner and degreaser and as surface cleaner prior to bonding strain gages

- CFC-113 used for cleaning electrical contacts, computers, and tape heads

- Fel-Pro C5A anti seize compound

1.3 Individual Task Descriptions

The original task descriptions for this NUSTO activity as specified in the Statement of Work are described in the following paragraphs:

1.3.1 Task 1 Substitute for CFC-113 Instrument Cleaner. The purpose of this task was to study alternatives to CFC-113 used for cleaning electrical contacts, computer, and other instrumentation tape heads. The alternative must leave no residue on the cleaned surface. To qualify the alternative, a survey of suppliers was conducted and appropriate testing was conducted. Selected solutions from this study shall be implemented into the operational areas and appropriate specifications and procedures will be updated as an effort subsequent to this study.

1.3.2 Task 2 CFC-113 Parts Cleaning and Degreasing Substitutes. The purpose of this task was to study alternatives to CFC-113 products used for parts cleaning and degreasing. The study encompassed operations including the Valve Shop, Fabrication Shop, and Instrumentation Shop. Supplier surveys were also conducted to identify alternatives that do not contain ozone depleting chemicals. Product evaluations and validation testing were performed as appropriate prior to implementing solutions into the operational area. Manufacturing Specifications and procedures shall be revised for the use of substitute commodities as an effort subsequent to this study.

1.3.3 Task 3 TCA Parts Cleaning and Degreasing Substitutes The purpose of this task was to study alternatives to TCA used for general parts and components cleaning, surface degreasing prior to painting, and surface preparation prior to attaching (bonding) strain gages. Supplier surveys were conducted to identify alternatives that do not contain ozone depleting chemicals. Product evaluations and validation testing as appropriate were performed prior to implementing solutions. Manufacturing Specifications and procedures shall be revised for the use of substitute commodities as an effort subsequent to this study.

1.3.4 Task 4 Fel-Pro C5A Anti Seize Compound Substitute. The purpose of this task was to identify substitutes for Fel-Pro C5A Anti Seize compound. Supplier surveys were conducted to identify alternatives that are do not contain ozone depleting chemicals. Product evaluations and validation testing were performed as appropriate prior to implementing solutions. Manufacturing Specifications and procedures shall be revised for the use of substitute commodities as an effort subsequent to this study.

1.3.5 Task 5 Strain Gage Preparation Prior to Bonding The purpose of this task was to identify substitutes for the cleaning and bonding of strain gages. Supplier surveys were conducted to identify alternatives that do not contain ozone depleting chemicals. Product evaluations and validation testing were performed as appropriate prior to implementing solutions. Manufacturing specifications and procedures shall be revised for the use of substitute commodities as an effort subsequent to this study.

Martin Marietta, with the Air Force Project Officer approval, combined like tasks together to derive benefits from technical synergy and to realize economies of common work packages. For this reason, the performance of this contract was divided into three major work packages: 1) mechanical cleaning; 2) anti-seize replacement; and 3)electrical cleaning. A description of these three work packages follows in subsequent sections, and is used as common format throughout the remainder of this report.

1.4 Scope

1.4.1 Mechanical Cleaning

Gross cleaning and degreasing operations on mechanical hardware are performed prior to a number of final processing steps including painting and precision cleaning.

Gross cleaning occurs at several locations on PJKS. The valve shop is a work area which refurbishes valves and components for pneumatic and propellant systems. Personnel at the valve shop clean and degrease parts by immersion in an ultrasonic bath of TCA and by spraying CFC-113 with a wand supplied by a small 10 gallon pressurized tank. Other areas occasionally perform gross cleaning by using spray bottles and rags to wipe the surfaces.

1.4.2 Anti-seize Compound

An anti-seize lubricant, Fel-Pro C5A, is used at the PJKS facility on fittings and threaded fasteners exposed to high temperature and/or corrosive environments. Fel-Pro C5A contains TCA used as a carrier solvent.

1.4.3 Electrical Cleaners

Electronic tape heads and electrical contacts presently require the use of CFC-113 or TCA as cleaners. Strain gages are bonded to test hardware using an adhesive which contains TCA as a carrier solvent. Surfaces to be bonded are also presently wiped with TCA prior to the application of this adhesive. ODC-free alternatives to these materials must be identified.

This report contains the program objectives, a description of existing cleaning processes, the identification of potential alternatives, the evaluation of alternatives, and the screening tests performed to select ODC-free cleaning solvents and lubricants. This report also presents results of the demonstration and validation tests and the final product selections based on these results.

2.0 DESCRIPTION OF EXISTING PROCESSES

2.1 Mechanical Processes

Gross cleaning occurs in the Valve Shop (Building T-5) located on the PJKS facility. Cleaning processes are performed to maintain the facility systems and to preclean parts prior to precision cleaning in the clean room, also located in the Valve Shop. Gross cleaning is performed to achieve visibly clean surfaces and remove gross contaminants such as oils, greases, and hydrocarbon fluids. Cleanliness is verified by visual inspection and may be aided by wipe tests, water break tests, ultraviolet inspection and special lights and mirrors.

Precision cleaning is used to attain a high degree of cleanliness, typically required for critical applications. Precision cleaning is performed in a controlled environment (a clean room) and for this degree of cleanliness, contaminants are not visible to the human eye. Cleaning fluids are filtered, controlled, and routinely checked for contamination. Cleanliness is verified by particle count, non volatile residue (NVR) analysis, and total filterable solids (TFS) quantification.

The basic gross cleaning process is illustrated in Figure 1 and includes: degreasing using TCA in an ultrasonic, deionized water spray rinse, nitric-hydrofluoric acid passivation (stainless steel parts only), mild detergent cleaning in a heated (150 °F) ultrasonic bath, a final deionized water rinse, and drying in an oven. A small ten gallon tank of CFC-113 is used to spray some parts instead of immersion in the TCA bath. Spray cleaning is performed when the part material is not compatible with TCA, or to rinse off lubricant residues, dust or thread tape particles during reassembly of components.

A variety of parts are cleaned at the Valve Shop ranging from disassembled parts of valves to tubing, flanges, and fittings. The size of the parts cleaned is limited to the size of the baths, which are approximately 3 ft by 2 ft by 2 ft. Both metallic and nonmetallic parts are cleaned. The most commonly processed metal is stainless steel. However, aluminum, carbon steel, brass, bronze and copper are also cleaned. The non-metals are mainly soft goods for valves and components and include Teflon ®, Kel - F ®, nylon, rubber, and other plastics. The contaminants are also of many different varieties. The typical ones include: propellant compatible lubricants, grease, hydraulic fluid, vacuum pump oil, machining oil, gear oil, propellant residues, and dust.

After gross cleaning the parts are visually examined for any indication of contaminants or residues. If contaminants remain, the part is then recleaned. Tenacious contaminants are brushed or wiped off. Following precision cleaning, the final rinse fluid is collected and analyzed for nonvolatile residue (NVR) and particulates in accordance with the general requirements outlined in MIL-STD-1246B and other specifications listed in Table 1. Cleaning operations are performed eight hours of every work day for a total of 248 days per year.

The local area procedure (EPL SOP 8.1) describes the Valve Shop cleaning operation most accurately. Military standards and industry standards are the basis for the Martin Marietta implementing processes and these processes were used to develop the local area procedures. The military specifications do not dictate the use of specific solvents, but do provide recommendations which include the use of halogenated solvents. The specific solvents are identified in industry specifications or in Martin Marietta procedures.

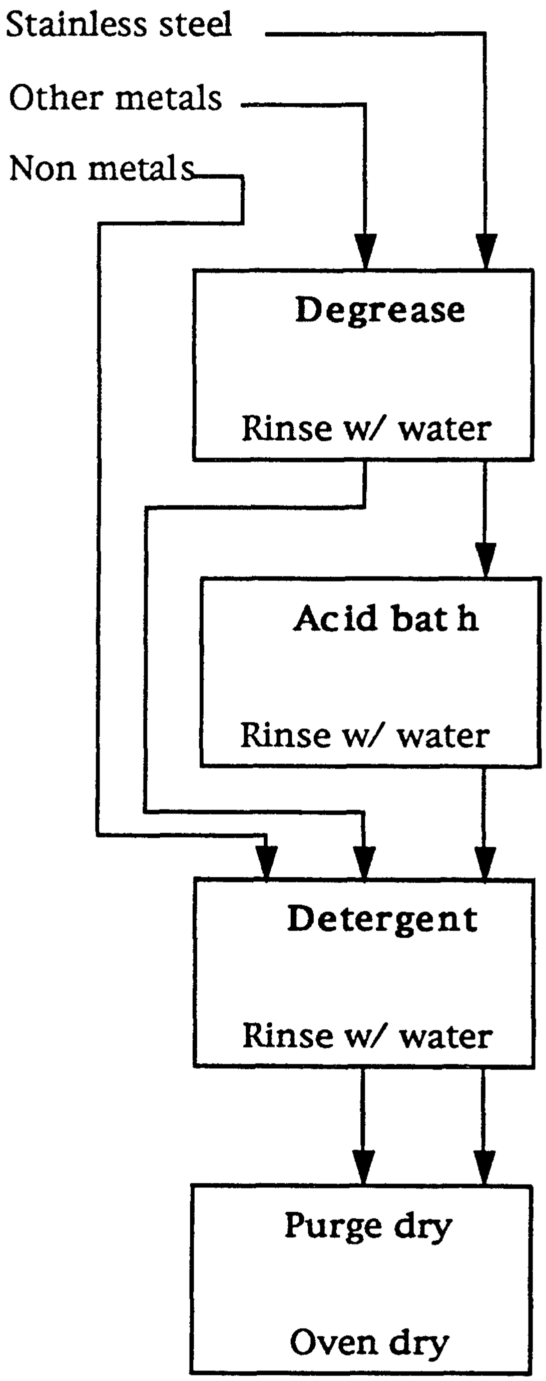

FIGURE 1 GROSS CLEANING OPERATIONS FLOWCHART

TABLE 1 CLEANING SPECIFICATIONS AND STANDARDS USED AT AIR FORCE PJKS FACILITY

Number	Revision	Title
MP 50018	6	Paint Removal
MP 50036	10	Cleaning, Alkaline, Non-Etch
MP 50042	23	Cleaning, Descaling, and Passivation of Corrosion and Heat Resistant Steel and Nickel Alloys
MP 50042 Appendix 5	3	Cleaning, Descaling, and Passivation of Corrosion and Heat Resistant Steel and Nickel Alloys. Effectivity: Titan Launch Vehicles
MP 50045	3	Degreasing, Aqueous
MP 50046	12	Degreasing, Vapor
MP 50307	3	Decontamination of Components Exposed to Propellants
MP 50309	NEW	General Cleaning of Equipment
MP 50405 Supplement 2	11	Contamination Control Specification Requirements for Operational Fluids and Gases
MP 50405 Supplement 3	13	Contamination Control Specification - Fluids and Equipment (Cleanliness Levels)
MP 50405-1	9	Contamination Control Cleaning - Detail Cleaning Methods
MP 72115	21	Cleanliness Controls for Spacecraft/Systems
MP 72115-5	10	Cleaning Procedures for Spacecraft/Systems Propulsion Equipment
KSC-C-123	F	Surface Cleanliness of Fluid Systems, Specification for
MIL-STD-1246	B	Military Standard, Product Cleanliness Levels and Contamination Control Program
EPL SOP 8.1	B	Cleaning of Parts to EPS 50405 Requirements

2.2 Anti-Seize Compound

The anti-seize compound is used at the PJKS facility as an aid for the separation of threaded fasteners and fittings that are subjected to high temperature or to corrosive environments. The compound is applied to fittings or fasteners prior to assembly, provides a lubricating coating that seals out moisture and prevents corrosion of the threads, and allows fittings that are exposed to high temperature to be later separated.

The following documents list the requirements for anti-seize compounds used to prevent threaded fasteners from seizing; however, the PJKS facility does not use anti-seize compounds for deliverable and/or flight hardware. The primary use for anti-seize compounds is for facility equipment. Presently there is no governing specification for the use of anti-seize compounds at the PJKS facility.

-- MIL-A-907 "High Temperature Anti-seize Thread Compound"
--SAE AMS-2518 "Graphite-Petroleum Anti-seize Compound"

2.3 Electrical Processes

2.3.1 Tape Heads (Large)

CFC-113 and TCA are used to clean video tape heads and other magnetic tape heads used for test data acquisition at the PJKS facility. The cleaner is used to remove contaminants (dust, oil, residues from magnetic tape) as well as to prolong the life of the heads by reducing abrasion. In addition to the commercial video cassette recorders (VCRs) used to record audio and video test events, a digital Honeywell recording device, and a high speed video recorder are used to record digital test data and high speed video test events, respectively. Cleaning of the magnetic tape heads with CFC-113 and TCA typically occur at regularly scheduled maintenance intervals, or when the video picture quality or signal-to-noise ratio for the digital recorder is poor. Upon cleaning, the units are returned to service.

The electronic area of PJKS performs tests and records data on large tape systems. The standard tape recording unit is the Honeywell Model 101 tape drive, although some other Honeywell units (Models 96 and 97) are also used. The standard maintenance procedure for the equipment is to clean all tape contacting surfaces before every test and more often as needed for longer duration tests. According to the manufacturer "Maximum cleanliness, especially within the tape path areas, is essential for optimum performance". The manufacturer (now Metrum) had previously recommended cleaning with CFC-113. Cleaning is performed using either a lint free cloth or a cotton swab. The manufacturer now recommends cleaning the magnetic heads with pure ethyl alcohol and the Capstan rollers with heptane. The capstan has a polyurethane coating which could be detrimentally affected by the alcohol.

Contaminants to be removed from tape heads include residues from the machine (tape particles both polyester and binders, head particles of ferric oxide, and glass particles from glass spacers), residues from the operation (dirt, dust, and oxides) and air borne contaminants (spores, organic particulates, skin cells, hydrocarbons, and smoke residues). Contaminants can also deposit from other processes at the facility or from the general environment since the electronics test area is open to the air.

Process requirements for large tape heads include removal of the contaminants, especially particulate; materials compatibility with the tape heads and Capstan rollers; and the purity of the cleaner to prevent micro contamination upon evaporation. The objective of the cleaning process is to produce a correctly functioning tape drive with no deterioration in recording ability.

2.3.2 Tape Heads (Small-VCRs)

The PJKS facility also has several smaller video cassette recorders (VCRs) which are used for both playback and recording of information and images. These units are manufactured by Panasonic and Sony and include both 1/2 inch and 3/4 inch tape sizes. The standard recommended cleaner for these machines is CFC-113. Since this type of equipment is required for present and future test applications, it was included in the current evaluation. The facility is also in the process of ordering a high speed video recording and playback unit from NAC Corporation. Many of the same materials are used in the production of the small VCRs, and considerations such as contamination and material compatibility are valid for these units as well.

2.3.3 Contacts

ODCs are currently used to clean electrical contacts used in amplifiers at the PJKS facility. These amplifiers are typically installed in electronic racks and used for data acquisition during the performance of engineering tests of aerospace hardware. These amplifiers are also used to calibrate electrical test devices during qualification and acceptance testing of flight hardware. The contacts are part of a rotary switch installed in the amplifier module. Channels can be selected by rotating the switch through eleven contact points placed circumferentially around one wafer disk. Several wafers are placed along the axis of rotation to comprise the complete switch. A photograph of a typical electrical contact used in an electrical amplifier is shown in Figure 2. Amplifiers are removed from storage and placed into service based on the frequency of testing and required usages. During extended periods of storage, contacts installed in the amplifiers are inspected and tested for cleanliness and contact resistance, respectively. Contacts displaying excessive contamination or high electrical resistivity are cleaned with a commercial cleaner (Kontact Restorer®), reinspected, and retested prior to installation into an amplifier to be used for data acquisition or instrument calibration purposes. Kontact Restorer is formulated with trichlorofluoromethane (CFC-11), and also has a hydrocarbon lubricant which aids in reducing metal wear upon cycling the contact position.

The PJKS facility uses hundreds of signal conditioning amplifiers in its test areas and these units contain n hermetically sealed wafer switches which are presently cleaned with Kontact Restorer. These contacts fail by s of function or by noise generation, until restored by cleaning. The typical procedure for cleaning is to spray with the cleaner and exercise the contact until smooth performance returns. The contamination can be a build-up of particulates such as dirt or dust, residues left from material deterioration, or the deposition of a slick non-conductive coating.

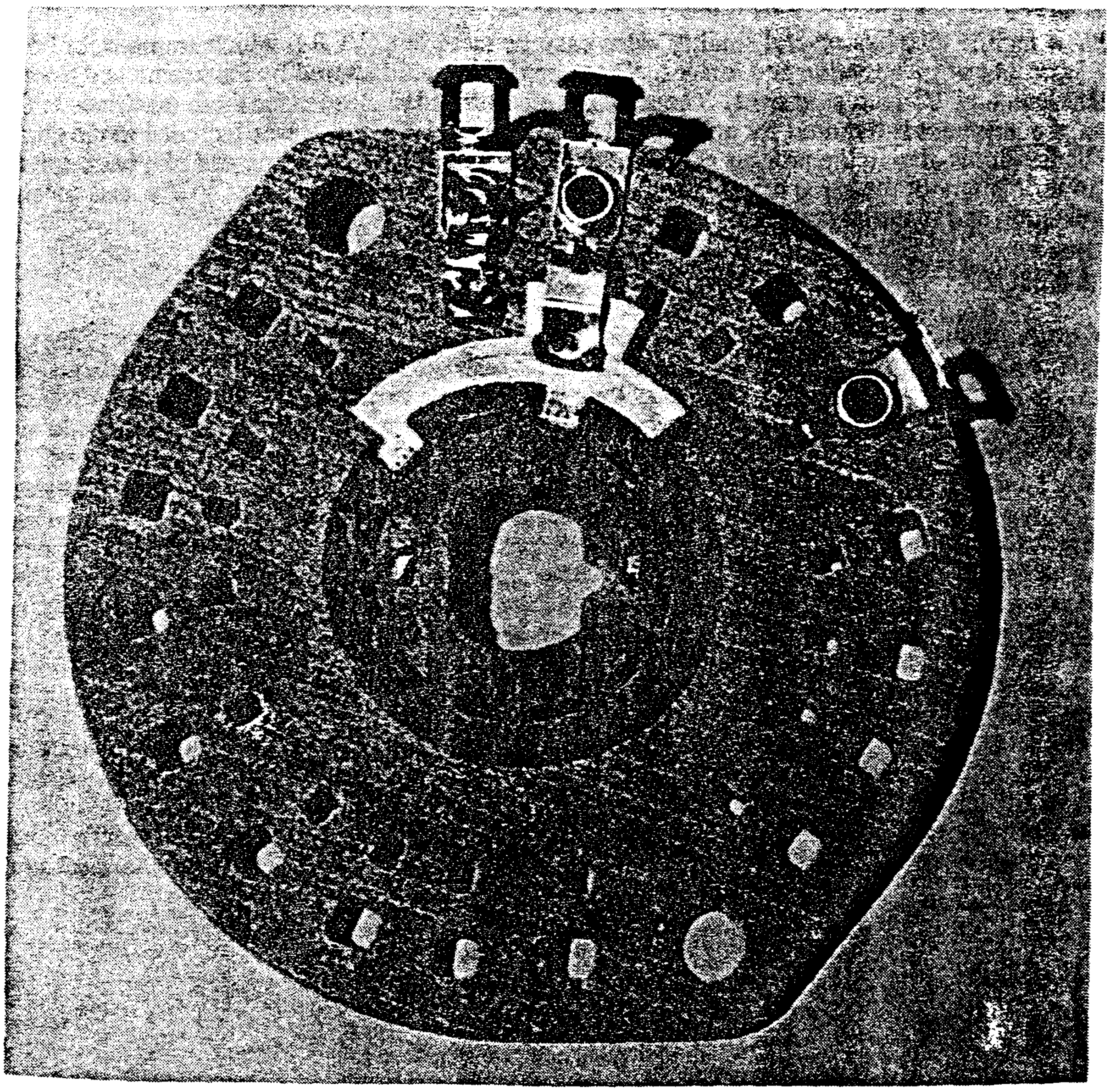

FIGURE 2 SINGLE WAFER REMOVED FROM AMPLIFIER ROTARY CONTACT SWITCH (MAGNIFIED)

2.3.4 Installing Instrumentation (Strain Gauges)

Strain gages are small calibrated electrical wires used to measure dimensional changes in test fixtures, fabricated hardware components (such as propellant tanks), and material samples. These gages are adhesively bonded to the surface of interest and are used to measure the degree a material or surface will elongate under applied stress. As the material surface elongates, the strain gage wire attached to that surface also elongates proportionally. As the wire elongates, the diameter of that wire is reduced, resulting in increased electrical resistivity. A measurement of the wire resistance therefore is indicative of the degree of elongation, or strain, of the strain gage wire and also of the substrate to which it is attached. This measurement is very accurate, and higher reliability can be obtained by this method than by optical or dimensional methods. The present application of strain gages to material surfaces require surface degreasing prior to bonding using TCA, and adhesive bonding using M-Bond 200® (Micromeasurements Group) which uses TCA as a carrier solvent. The company has since changed its adhesive formulation, and isopropyl alcohol is now used as the carrier solvent for this adhesive. Adhesive bonding is accomplished in accordance with Martin Marietta Non-Standard Process (NSP) 00309 "Installation of Instrumentation". This procedure requires the following surface preparation steps:

1. Wipe the surface to be bonded with TCA solvent.
2. Abrade with 220 grit sandpaper.
3. Remove any residual particulates with cotton swabs moistened with TCA or MEK
4. Apply chemical conditioner
5. Wipe the surface until visually clean.
6. Apply chemical neutralizer
7. Wipe the surface until visually clean.

Figure 3 shows an example of strain gages bonded to two types of tensile specimens. Lap shear tensile specimens for 6061 aluminum and G-10 fiberglass are also included in this photograph and were used to evaluate the adhesive shear strength of the replacement ODC-free adhesive. Lap shear tensile specimens also were used to evaluate the ability of the alternative cleaners to produce a bondable surface.

The PJKS facility performs the installation of instrumentation for all programs in its electronic area. This instrumentation may include strain gauges, miniature temperature sensors, thermocouples, accelerometers, heater tape, thermistors, thermostats and accelerometers. The controlling document used is Non Standard Process NSP00309 "Installing of Instrumentation". Solvent cleaning with TCA prior to bonding is specified in the process. The cleaning step is very important since without removing all the contaminants the adhesive would not bond the instrumentation properly, or the attached instrumentation would break loose inappropriately. The contaminants involved include the normal oils from previous manufacturing processes (drilling, tapping, cutting and finger oils). The surfaces to which the instrumentation would be bonded include all metals (aluminum, titanium, stainless steel, iron, etc.) as well as composites such as carbon-carbon and carbon graphite. The composite surface presents a unique concern because of its tendency to absorb fluids (including contaminates) much like a blotter and then releasing those fluids upon exposure to a vacuum environment (space environment).

The manufacturer of strain gauges (Micromeasurements Group) provides a complete system including the adhesive for their bonding. The catalyst for the adhesive (M-Bond 200) contains TCA as a carrier but the manufacturer offers another version using isopropyl alcohol (IPA). Since this adhesive is an integral part of the instrumentation process the alternative adhesive needs to be qualified for use.

This activity contains many of the same concerns and issues for the general metal cleaning, and advances made in this area will be applied to electronics cleaning.

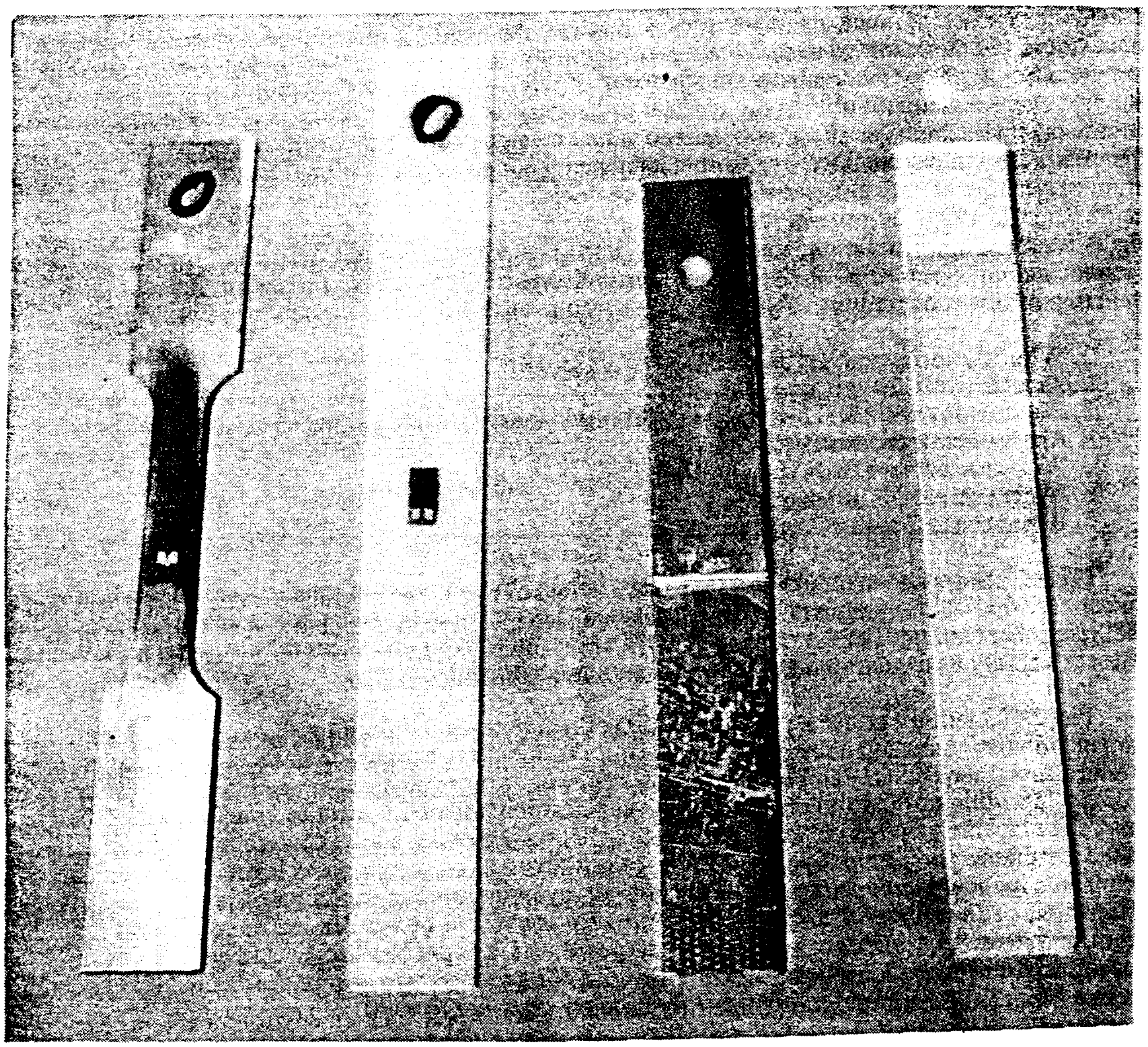

FIGURE 3 STRAIN GAGES BONDED TO TENSILE SPECIMENS (LEFT TWO MATERIALS); LAP SHEAR TEST SPECIMENS (RIGHT TWO MATERIALS)

2.4 Desired Output of Cleaning Process

The desired feature of a replacement cleaner for TCA and CFC-113 is to effectively remove contaminants from surfaces being cleaned without any degradation in end-item performance. For this reason, the alternative cleaners must demonstrate an ability to clean a wide variety of greases, oils, and other residues typically prevalent on aerospace and facility hardware, while maintaining program critical requirements such as materials compatibility, propellant compatibility, and low residue upon cleaner removal. In addition, the use of the cleaner should not impose any safety or environmental hazards, or incompatibilities with other chemicals used in the life cycle of the hardware. Flammability and Volatile Organic Compound (VOC) emissions are a current concern for many of the hydrocarbon solvents prevalently used in industry today. For gross cleaning applications for non-critical hardware, the hardware should be visibly clean after completing the cleaning cycle, and should show no signs of residual contaminates when visual aids such as blacklight inspection, magnification, water break tests, or cloth wipe tests are employed. In precision cleaning applications for program critical hardware (such as propellant tanks and subsystems), the hardware must also conform to required measurements for Non Volatile Residue (NVR), particulate count, and Total Filterable Solids (TFS) as required by the governing standards and applicable cleaning specifications. The final criteria is the suitability of the hardware for the intended mission. Frequently microcontamination which may be acceptable in commercial applications will be disastrous in aerospace applications. Contamination may clog fine mesh capillary screens used in propellant systems, resulting in shunted fuel flows, or during the course of a long duration flight may corrode or degrade critical hardware in interplanetary satellites. For these reasons, both the direct and indirect effects of alternative cleaners on aerospace hardware should be properly evaluated.

2.5 Special Concerns and Considerations

2.5.1 Entrapment Areas Because CFC-113 and TCA are volatile organic cleaners, removal of the cleaner from the processed part is relatively easy. The part may be baked in an oven, purged with hot dry gas, or simply dried in air to evaporate residual solvent. Many of the alternative cleaners commercially available do not have the high evaporation rate that provides this advantage. For solvents having a surface tension and/or boiling point higher than these ODC solvents, a unique problem arises. The cleaners may not adequately reach entrapment areas in complex systems, or may be very difficult to remove from entrapment areas once present. In these cases, additional steps must be taken to ensure parts are effectively cleaned and residual cleaner is adequately removed. Mechanical agitation or pressurized flushing may be required during the cleaning operation. An additional solvent rinse with a highly evaporating solvent (such as IPA) may be required at the completion of the cleaning process. Residual cleaner should be soluble in the solvent selected for the final rinse, such that all traces of the cleaner are removed during this step.

2.5.2 Propellant Compatibility Because propulsion hardware is processed at the Air Force Plant PJKS, selected cleaners must be chemically compatible with the rocket propellants subsequently used in that hardware. Rocket propellants may include hypergolic storable propellants (Aerozine-50 fuel and nitrogen tetroxide oxidizer), cryogenic propellants (liquid hydrogen fuel and liquid oxygen oxidizer), or other special propellant blends and test fluids (such as liquid ammonia). Of particular concern are the rocket oxidizers, nitrogen tetroxide and liquid oxygen, which react with a number of hydrocarbons and substituted hydrocarbons explosively. Selected cleaners used in propellant systems must therefore completely remove all traces of hydrocarbon contaminants during the cleaning cycle, and must either be non-reactive with the propellants or be completely removed prior to charging the hardware with propellants. A number of tests to demonstrate propellant compatibility, such as pressure rise, drop-impact sensitivity, and reaction monitoring using Fourier Transform Infrared (FTIR) Spectrometry may be used to evaluate this concern.

2.5.3 Corrosion and Material Compatibility Chemical cleaners selected as replacements for ODC cleaners must not degrade the materials of construction in contact with the cleaners. Metallic hardware comprised of stainless steel, aluminum, and titanium should show no evidence of corrosion or oxidation after exposure to the cleaners. Other metallic items typically used in valve components and other such parts, such as brass, bronze, copper, cadmium plated fasteners, galvanized materials, as well as electrical solder should similarly demonstrate acceptable corrosion resistance in contact with candidate replacement cleaners. Non-metals, such as Buna, EPR, and Viton rubbers, fiberglass-epoxy materials, and graphite epoxy materials should not show loss in mechanical properties or functional use after exposure to the cleaner. Plastics are prone to craze, fog, or become embrittled in the presence of some solvents, and rubber materials may swell and deform in the presence of incompatible solvents. Materials intended for use in critical or customer deliverable hardware should be evaluated for corrosion and materials compatibility using candidate replacement cleaners.

2.5.4 Electrical Conductivity In addition to the cleaning requirements specified above for mechanical hardware, electrical performance requirements must be maintained after cleaning. These requirements include the proper functioning of switches, relays, tape heads, and other electrical and electronic devices. Positive electrical continuity must be demonstrated on parts which have been cleaned using alternative cleaners. In most cases, the verification of proper electrical conductivity (or resistivity) of affected parts and components is sufficient to demonstrate acceptable performance. In some unique electrical applications (such as bonding strain gages to equipment), demonstration of mechanical (bond strength) performance may also be used to verify the proper operation of alternative cleaners.

3.0 CHEMISTRY AND PHYSICS OF ACTION

3.1 Introduction

As part of a program to eliminate the use of ozone depleting chemicals at a Government Owned Contractor Operated facility located at Martin Marietta Technologies, an evaluation of the chemical and physical properties of effective cleaning solvents was initiated. In the past, both 1,1,1-trichloroethane (TCA) and 1,1,2-trichloro-1,2,2-trifluoroethane (CFC-113) have been used in degreasing mechanical hardware (such as tubing, valves, and propellant tanks), degreasing and cleaning electrical hardware (such as video tape heads, electrical contacts, and for strain gage preparation), and as a carrier solvent in formulating anti-seize compounds which are applied to fasteners to facilitate assembly and disassembly. These particular solvents are also used in other unique applications, such as decontaminating rocket oxidizer flowmeter assemblies, cleaning of operational tanks for liquid oxygen (LOX) use, cleaning of large and/or complex systems, as a referee test fluid for vibration and acoustic testing, and in soldering and lubrication applications. Both TCA and CFC-113 have been used in cleaning applications in the aerospace industry for over thirty years. TCA and CFC-113 were the cleaners of choice in the Aerospace industry because they were readily available, inexpensive, efficient at removing a wide variety of organic contaminants, quickly evaporating, safe to use, and nonflammable. Unfortunately, these two chemicals as well as a variety of other chlorinated hydrocarbons have been identified as contributing to depletion of the ozone layer which in turn promotes skin cancer. As a result of the Montreal Protocol and the 1990 Clean Air Act Amendments, ozone depleting chemicals will be banned for use in the United States after 1995. Because of the heavy reliance on TCA and CFC-113 for these aerospace usages, an understanding of the scientific basis for the uses of these chemicals as effective cleaners was initiated.

3.2 Objective

The purpose of this Section is to describe the principles of cleaning processes, their application to existing ODC solvents (TCA and CFC-113) and to aerospace hardware, and to provide a more systematic approach to the selection and evaluation of alternative cleaners.

3.3 Cleaning Methods

Cleaning is defined simply as the removal of an unwanted dirt or contamination from the material, part, or system being cleaned. Effective cleaning may be accomplished by a variety of techniques. Mechanical cleaning, such as brushing, abrading, grit blasting, shot peening, or high pressure impingement is often used to mechanically remove contamination from the surface of interest. This process is usually the first of several steps in a cleaning cycle, and is designed to aggressively remove intractable contaminants such as scale, rust, and paint residues from untreated surfaces prior to performing more exacting cleaning processes. Chemical cleaning is subsequently used to remove gross contamination such as greases, oils, machining dye residues, and fingerprints from the part. Parts may be cleaned by suspending them in a vapor degreaser, by immersion in liquid solvent, by spraying with the cleaner, or by wiping them with a cloth saturated with the cleaning solvent.

In vapor degreasing, the part being cleaned is typically suspended above a heated solvent, and the solvent vapors are allowed to condense on the part. During the condensation process, contaminants are removed from the part, and the condensed solvents containing the dissolved contaminants drip from the part back into the heated solvent reservoir. Condenser coils are placed above the processed part to recycle the solvent into the heated bath. Vapor degreasing allows for the replenishment of fresh solvent onto the part during condensation, so the part becomes cleaner as it remains in the degreaser. When the part temperature reaches the temperature of the solvent vapor, no more liquid will condense on the part, and the part is removed from the vapor degreaser.

Immersion methods typically rely on the chemical nature of the cleaner to remove contamination from the processed parts. Many types of chemical cleaners are used to effectively clean contaminated hardware. Organic cleaners rely on the chemical similarity between the cleaner and the residue to effectively dissolve and disperse the residue into the cleaner. Organic solvents may be aliphatic (naptha, stoddard solvent, kerosene, mineral spirits), aromatic (toluene, xylene) chlorinated (perchloroethylene, trichloroethane, methylene chloride), glycol ethers (butyl cellosolve, butyl carbitol), or oxygenated (methanol, ethanol, isopropyl alcohol). A considerable amount of effort has been performed on developing water-based cleaners as possible replacements for organic solvents. Aqueous cleaners are being considered because the chlorinated hydrocarbons (such as TCA) and the chlorofluorocarbons (such CFC-113) are restricted for use because of their ozone depleting characteristics, and because many of the other common organic solvents (such as acetone and methyl ethyl ketone, or MEK) are volatile organic compounds (VOC) and contribute to atmospheric air pollution. Aqueous cleaners usually have an active cleaning agent (such as d-limonene) which dissolves the contaminants, ionic detergents to solubilize the cleaned residue, and surfactants to reduce the surface tension and allow penetration of the cleaner into the soil. Because aqueous cleaners do not typically have the high affinity for hydrocarbon contaminants that organic solvents do, aqueous cleaning is typically augmented using some form of mechanical action (Ref 1). Alkaline cleaners are used to react the free alkalies in the cleaners with reactive fatty acids in the organic oil to form water soluble soaps, which are flushed from the part. During immersion cleaning, parts are suspended into liquid cleaner, and after a period of time, are removed from the bath. Solvent baths usually contain dissolved contaminants from parts processed previously, and upon removal from the bath, some of this contamination will remain on the part. Parts processed by immersion cleaning are seldom cleaned adequately for critical or exacting applications. Therefore, immersion cleaning in solvent is more likely to be used as a preliminary, rather than a final cleaning method. In many cases, immersion cleaning is also augmented by some form of mechanical action. An ultrasonic bath, for example, imparts high frequency energy to the cleaning process and also induces cavitation in the bath. Cleaning solutions may also be stirred, flushed, or recirculated to add convective cleaning actions.

Solvent spraying and high pressure impingement are being used more frequently in hardware processing. Direction of a high pressure liquid onto the part aids in mechanically cleaning the part, and allows the cleaner to contact recesses and blind surfaces in the part typically not assessable in immersion techniques. Impingement processes typically introduce a hydrodynamic shear force to the contaminant, and both immiscible fluids or compressed gases have been used in this application (Ref 2). Some solvents which are marginal cleaners from a chemical standpoint, such as isopropyl alcohol, can be very effective at cleaning when coupled with an impingement process.

For large or intricate surfaces, in which vapor degreasing, immersion, or impingement of the cleaner is impractical, hand wiping of the surface with the cleaner remains a viable cleaning method. In this method, a lint-free clean cloth is saturated with cleaning solvent, and the surface of the part is wiped successively until a wiping using a fresh cloth leaves no visible residue on the cloth. Highly evaporating solvents, such as acetone or TCA create a problem for hand wiping applications, in that the contamination removed from the part is typically redeposited as the solvent is evaporated. In this case the contamination is not removed, but merely smeared around the part surface. Recontamination of Titan IV payload fairings after solvent wiping with MEK has been discovered during qualification testing (Ref 3).

Hardware cleaning at Air Force Plant PJKS is typically performed by solvent immersion in quiescent solvent baths, by immersion in ultrasonic baths, or by low-pressure spray applications (such as large tank cleaning), The replacement of cleaning chemicals therefore is highly reliant on the chemical nature of the cleaning solvent and its chemical and physical properties. These properties will be discussed in the following sections.

3.4 Contaminant Removal

During the cleaning process, the contaminant must first be removed from the material surface, at which point dissolution and dispersion of the contaminant in the cleaning solution takes place. Contaminant removal is therefore regarded as a critical step in the cleaning process, and is governed by the phenomenon of surface tension.

The effect of surface tension on an oily contaminant may be described in terms of the work of adhesion, as depicted in Figure 4 (Ref 4). On the left side of the figure, an oil residue adheres to a solid surface by surface tension (γ_{so}). The surface free energy for detachment of the soil from the solid surface therefore is :

$$\Delta G = \gamma_{final} - \gamma_{initial} = \gamma_{wo} + \gamma_{sw} - \gamma_{so}$$

This change is spontaneous for conditions in which $\Delta G< 0$. This occurs when the surface tension of the solid-oil interface (γ_{SO}) is greater than the surface tension at the cleaner-oil interface (γ_{WO}) plus the surface tension at the cleaner-solid interface (γ_{SW}). Detachment of the soil from the solid occurs when the liquid surface tension of the cleaner is lower than that of the contaminant. A similar result of the surface free energy may be envisioned if the soil is considered to be a fluid having a contact angle (θ) between the liquid and the wetted surface, as represented in Figure 5. In Figure 5, the complement of the contact angle, $1-\theta$ is shown for clarity. A highly wetting liquid will have a contact angle of zero. A non-wetting liquid will have a contact angle of 180°. The low surface tension cleaner displaces the contaminant having the higher surface tension, at the solid interface. Poor wetting and adhesion of the contaminant to the solid surface results in the coalescence of the contaminant into droplets due to the dominating internal bulk attractive forces in the contaminant. These contaminant droplets are characterized as having a large contact angle, and are easily removed from the solid surface.

A cleaner, therefore, must have a lower surface tension than that of the contaminant to spread over the surface of the contaminant and lift it from the solid surface. This phenomenon helps to explain why some aqueous based cleaners (such as Daraclean®) can effectively remove fluorinated greases (such as Krytox®) from hardware without dissolving them. If the surface tension of the cleaner or solvent is higher than the contaminant, it will not readily wet the surface of the contaminant, and inadequate removal of the contaminant occurs. This principle is demonstrated by the use of the "water break test", which is a measure of the cleanliness of metallic surfaces prior to painting or bonding. In this test, water is applied to the cleaned metal surface. If the water forms a uniform water layer spread over the entire surface area, the surface is considered clean and free from any surface contamination. Parts that have not been adequately cleaned will result in a broken water layer. Contaminated areas having a surface tension lower than water will cause the water to bead at those locations, indicating surface contamination. The part must then be recleaned prior to painting or bonding. Water is selected for this test because its surface tension is relatively high, being approximately 72 dynes/cm. Many common contaminants have surface tensions dramatically lower than water. For example, silicone oil, petroleum lubricating oil, and glycerol have surface tensions of 21, 29, and 63 dynes/cm, respectively (Ref 5).

The low surface tension of the cleaner or solvent has another beneficial effect. It allows the solvent to penetrate pores and abrasions in the substrate so that these internal surfaces may also be adequately cleaned. These pores and abrasions are present in all rough surfaces created by chemical etching, by abrading with sand paper, by the creation of an oxide layer (anodizing), or by chemical conversion treatments of the metal. Proper cleaning of these porous areas substantially adds to the surface area to which the coating or adhesive may adhere.

The characteristic of a cleaned metallic surface is one of high surface energy. It is this high surface energy that provides the greater force of attraction to a liquid such as an adhesive or coating which is applied to it. Clean metallic surfaces such as aluminum are very polar and have high surface energies in the vicinity of 500 dynes/cm (Ref 6). Most liquid adhesives or coatings have surface tensions in the range of 30 to 70 dynes/cm. This difference between the surface tension of the coating and the surface energy of the metal substrate results in spontaneous wetting of the metal with the coating, providing a uniform coating of maximum adhesive strength. One negative aspect of high surface energy substrates is their tendency to absorb low energy contaminants and thereby lower their own surface energy. Metal surfaces that are carefully and rigorously cleaned are very susceptible to contamination and corrosion (by water vapor and acid gases) due to removal of protective organic films and must be processed immediately or protected during the production cycle (Reference 7).

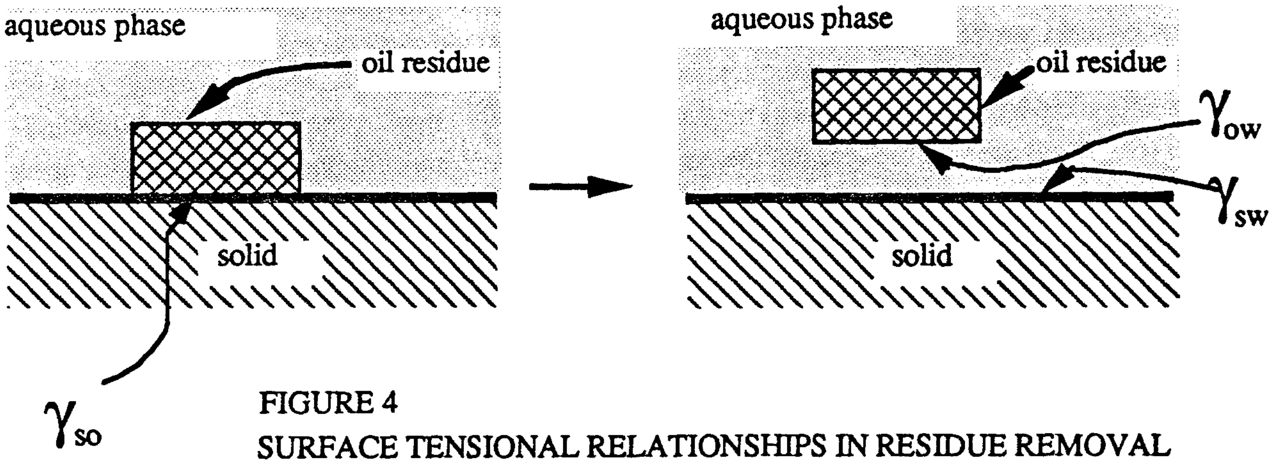

FIGURE 4
SURFACE TENSIONAL RELATIONSHIPS IN RESIDUE REMOVAL

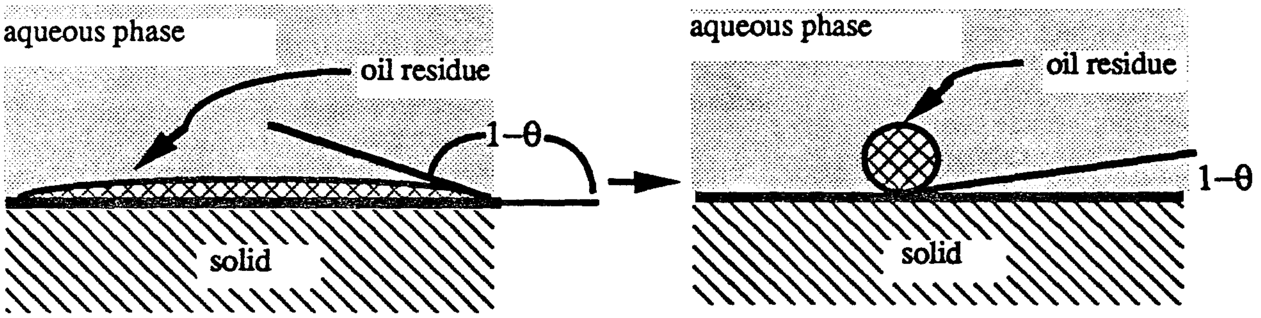

FIGURE 5
CONTACT ANGLE RELATIONSHIPS IN RESIDUE REMOVAL

3.5 Solvency

For many years, the use of chemical solvents to clean and degrease surfaces was established by empirical, rather than theoretical means (Reference 8). For example, it is common in the chemical solvent industry to characterize the comparative strength of industrial solvents by measuring its relative ability to dissolve common substances. One such measurement is the Kauri-Butanol or K_B value (Ref 9). Kauri gum, a hard copal resin derived from the kauri pine of New Zealand, is readily soluble in butanol, but insoluble in hydrocarbons. The K_B value, performed in accordance with ASTM D1133 (Ref 10), is the measure of the volume of solvent required to produce turbidity in a standard solution containing kauri gum dissolved in butanol. The higher the K_B value, the stronger the solvent and the more effective it is at dissolving organic compounds. K_B values for solvents such as methylene chloride and TCA are high, being 136 and 124, respectively. Other solvents such as mineral spirits and Stoddard solvent have K_B values of 37 and 33 (Reference 11). Fluorocarbon solvents such as Freons® range between 31 and 60. As a comparison K_B values for naturally occurring cleaning materials such as d-limonene or citrus oil are 62 (Reference 12), much lower than TCA, but higher than most Freon® compounds.[1]

Even rudimentary treatments of cleaning action have led to very useful descriptions of the fundamental physical processes involved. From these early investigations, the ability of a solvent to dissolve a contaminant was seen as a problem of the solubility of substances, which was closely related to the chemical principles of cohesion and constitution. In the dissolution of a substance, the cohesive forces holding the molecules of the substance together are displaced by opposing forces, acting between the solvent and the substance. Two solubility rules were generally accepted as valid. In the case of isomeric or otherwise similar compounds, the higher melting substance is frequently the less soluble one. For example, phenanthrene (melting point 100°C) is very soluble in ether, but anthracene (melting point 217°C) is only slightly soluble in ether. The second solubility rule states that the more volatile substance is also more soluble. Thus, the compound that has a lower boiling point dissolves more readily in the solvent. These rules are often valid because the melting point and boiling point of a pure substance are measurements of the intermolecular forces used to cohesively bind the substance together. The stronger the intermolecular forces, the more work that is required to remove the molecules from the lattice structure.

Although these general rules were seen to be valid for many applications, there were frequent cases of the opposite trend, in which substances having the higher melting or boiling points were more soluble. In these cases, the nature of the cohesive forces, in addition to the strength, was an underlying factor in the solubilities of these substances. One solubility rule that brings into the foreground the nature of the cohesive forces may be expressed briefly in the words "Like Dissolves in Like". "Like" in this context means the presence of like or structurally similar groups in the molecule. For example, the lower paraffin hydrocarbons such as pentane, hexane, and heptane are mutually miscible in all proportions, as are the lower aliphatic alcohols. TCA is an effective defatting solvent because it is structurally similar to hydrocarbon oils, waxes, and greases it removes. In some fluorinated greases such as Krytox®, only solvents identically similar to the contaminant (such as Tribolube®, a fluorinated solvent) effectively dissolves them. Hexane and methanol, being members of different chemical families, are not miscible in all proportions. Polar solvents, such as water, methanol, and ammonia do not effectively dissolve non-polar contaminants, such as hydrocarbon oils, because the energy produced by solvation cannot overcome the strong bonds within the solvent.

The dissolving character of the solvent must therefore overcome the nature as well as the strength of the forces holding the substance together. Since these early descriptions of solubility of substances, which include an understanding of the strength of the intermolecular forces (melting point, boiling point, vapor pressure) in the substance as well as the nature of these forces, advances in surface physics and solution chemistry have greatly improved our understanding of the physical processes involved in cleaning processes.

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[1] Experimental methods for determining KB numbers are not capable of measuring solvents that contain water. The double bond to oxygen in methyl ethyl ketone (MEK) and acetone also apparently affects this measurement, so values for these solvents have not been determined.

3.6 The Solubility Parameter

Early studies of the solubility of substances were useful in characterizing solvents based on empirical data and on simple "rule-of-thumb" principles, but a more rigorous and especially useful approach was developed theoretically by Hildebrand (Ref 13). Solvation of the contaminant is governed by solution chemistry, and thus the application of the principles of thermodynamics of liquid mixtures apply. Again, for spontaneous solution of one compound into another, the Gibbs Free Energy of mixing, expressed by the following equation, must be negative.

$$\Delta G_{mix} = \Delta H_{mix} - T\Delta S_{mix}$$

Where: ΔG_{mix} is the Gibbs Free Energy upon mixing (calories/mole)
ΔH_{mix} is the enthalpy of mixing (calories/mole)
ΔS_{mix} is the entropy of mixing (calories/mole-K)
T is the absolute temperature (K)

Because ΔS, a measurement of the randomness or chaotic state of the system, is positive for the dissolution process, the controlling term for the prediction of the dissolution process is the enthalpy (heat content) term. If ΔH is negative, or a small positive number, than the process could be considered spontaneous. An expression was developed for the heat of mixing and is presented in Reference 14.

$$\Delta H_{mix} = V_t [(\frac{\Delta E_1^v}{V_1})^{0.5} - (\frac{\Delta E_2^v}{V_2})^{0.5}]^2 \phi_1 \phi_2$$

Where: ΔH_{mix} = Overall heat of mixing (calories)
V_t = Total mixture volume (cm^3)
ΔE^v = Energy of vaporization (calories/mole)
V = Molar Volume (cm^3/mole)
ϕ = Volume fraction

The term $\Delta E^v/V$, the energy of vaporization per unit volume can be taken as a measure of the internal pressure. It is a measurement of the energy required to totally separate (vaporize) the molecules of 1 cm^3 of material to infinity by overcoming the intermolecular forces holding the material together. It is often called the cohesive energy density. The square root of the cohesive energy density is of primary importance in the heat of mixing term and Hildebrand defined it as the material's solubility parameter (δ). In other words:

$$\delta = (\frac{\Delta E^v}{V})^{0.5}$$

is the expression most commonly used for the solubility parameter and δ has the units of $(\text{Energy/Vol})^{0.5}$. This terms also converts to $(\text{Pressure})^{0.5}$, with three of the most common units being $(J/m^3)^{1/2} = 4.889 \times 10^{-4}\ (cal/cm^3)^{1/2} = 1 \times 10^{-3}\ MPa^{1/2}$. As the definition implies, δ changes gradually with the temperature, hence it must be calculated at the temperature for which solubility is required.

The heat of mixing (and thus the Gibbs Free Energy of Mixing) is negative when $[\delta_1-\delta_2]^2$ is minimized. This minimization occurs when $\delta_1=\delta_2$, or when the solubility parameter of the first material (solvent) is identical to the solubility parameter of the second material (contaminant). According to this theory, two substances should completely mix and dissolve when the solubility parameters of the two materials are equal. The result of this theoretical treatment has very significant implications. First, it demonstrates the molecular basis of the rule-of-thumb often used in the solvent industry "Like dissolves in Like". Secondly, if the solubility parameter of a contaminant can be determined or estimated, solvents with similar solubility parameters can be chosen to effectively dissolve that contaminant. Although the determination of the solubility parameter of a specified contaminant is complex and not regularly determined, estimation techniques as well as identification of chemical families with similar solubility parameters can be effective tools in the selection of alternative solvents.

Upon further observation, one recognizes the influence of cohesive forces in the development of the heat of mixing equation. The solubility parameter may be calculated from a number of solution properties (Reference 15), for example:

from the heat of vaporization (as calculated from reliable vapor pressure measurements)

$$\delta = \left[\frac{D(\Delta H_v - RT)}{M}\right]^{1/2}$$

Where: D = Liquid density, g/cm^3
ΔHv = Heat of vaporization, calories/mole
R = Ideal gas constant, 1.9872 calories/mole-K
T = Absolute temperature, K
M = Molecular weight, grams/mole

From surface tension (γ)

$$\delta = 4.1\left(\frac{\gamma}{V^{1/3}}\right)^{0.43}$$

From the van der Waal constant for the gas (a)

$$\delta = \frac{a^{1/2}}{V}$$

From critical pressure (p_c)

$$\delta = 1.25 p_c^{1/2}$$

The influence of several important factors have been oversimplified by this theory, nevertheless, it is widely used as a formulating tool. Its development and application has been for non-polar molecules, such as normal chain hydrocarbons. For non-polar contaminants and solvents (which may be expected upon removal of a hydrocarbon oil from a metal surface using an organic solvent) a reliable method for the prediction of solubility is to compare the solubility parameters of the solvent and the contaminant. Solution occurs when the proposed components of the solution have about the same solubility parameter. In polar molecules (such as water, acetone, and aliphatic alcohols), the energy of the system is decreased because of the orientation of dipolar molecules, and the estimation of the heat of solutions derived from the solubility parameter differences may not be accurate. The presentation of a three component solubility parameter, incorporating contributions due to dipole-dipole interactions and hydrogen bonding, in addition to the dispersion forces commonly found in non-polar molecules is discussed in the following sections.

3.7 Hydrogen Bonding

The main weakness of the solubility parameter is that it was only applicable with non-associating materials (such as the non-polar hydrocarbons) so using the numbers by themselves could be misleading. For example, when a solvent for the softening of Nordel 1040 EPDM rubber was sought, both xylene and ethyl acetate were identified as having the proper solubility parameters (18.2 $MPa^{0.5}$ and 18.6 $MPa^{0.5}$, respectively). In practice, the xylene greatly swelled the rubber (indicating good solvation properties), while the ethyl acetate left it untouched (Ref 16). The apparent difference in the similar solubility parameters and the observed solution properties was attributed to the highly associative bonds present in ethyl acetate. The intermolecular forces in this solvent are similar to the forces in many such solvents in which a hydrogen atom is covalently bound to a highly electronegative atom such as fluorine, oxygen, chlorine, and nitrogen. This special bond is called a hydrogen bond, and is prevalent in such liquids as water, ammonia, and hydrofluoric acid. The apparent solubility parameter of water (calculated by the three component solubility parameter to be discussed in subsequent sections) is quite large $\delta = 48.7$ $MPa^{0.5}$, owing to the strong hydrogen bonding of water and to its small molar volume. Such liquids exhibit abnormally high boiling points and heats of vaporization because of the increased cohesive forces in the liquid. A proposed classification of chemical solvents into three major groups has been proposed:

Strong hydrogen bonds - alcohols, amines, amides, aldehydes, acids
Moderate hydrogen bonds - esters, ketones, ethers, glycol monoethers
Poor hydrogen bonds- aliphatics, aromatics, chlorinated hydrocarbons, nitrohydrocarbons

3.8 Solvent Polarity

In addition to the solubility parameter, other useful properties of the solvent include its polarity and polarizability. The polarity of the solvent is a measurement of the charge distribution in the molecule and is expressed as the dipole moment μ which is equal to the magnitude of the charge, e, multiplied by the distance d, between the centers of charge (Reference 17).

$$\mu = e \ X \ d$$

where M = dipole moment measured in Debye units ($1D = 10^{-18}$ esu X cm)
e = charge in electrostatic units (esu), and
d = distance between center of charge in Angstroms (Å).

A non-polar molecule (such as carbon tetrachloride, benzene, or methane) has a symmetric distribution of charge resulting in a zero net dipole moment, while a polar molecule (such as chloroform or acetone) has a positive dipole moment. Non-polar contaminants (such as oils and hydrocarbon residues) are best dissolved by non-polar solvents (such as hexane and carbon tetrachloride). Polar residues, such as inorganic salts, are best dissolved by polar solvents (such as acetone, isopropyl alcohol, or water). When both the solvent and the contaminant molecules possess permanent dipole moments, small interactions result by alignment of the two dipoles in a linear configuration. Dipole interactions also can usually occur between individual functional groups of two molecules, so that solvent and contaminant molecules having polar functional groups will selectively interact, the interaction being stronger for groups with larger dipole moments. A few examples of group dipole moments are presented in Table 2 below (Ref 18).

TABLE 2. FUNCTIONAL GROUP DIPOLE MOMENTS (in debyes)

amine, -N=	0.8-1.4	halogen, -F, -Cl -Br, -I	1.6-1.8
ether, -O-	1.2	ester, -COO-	1.8
sulfide, -S-	1.4	aldehyde, -CHO	2.5
thiol, -SH	1.4	ketone, -CO-	2.7
carboxylic acid, -COOH	1.7	nitro, $-NO_2$	3.2
hydroxy, -OH	1.7	nitrile, -C≡N	3.5
		sulfoxide, -SO-	3.5

A critical property of an effective cleaner is its ability to dissolve many different types of contaminants, i.e., both polar and non-polar residues. Polarization of a liquid molecule occurs when their electronic distribution is altered by the electric fields present on the surfaces to which they are applied. A liquid with high polarizability, therefore, will be effective at dissolving both polar and non-polar contaminants (Ref 19). Dispersion forces are those attractive forces arising between highly polarizable molecules. In a polarizable substance, the symbol p is used to represent the dipole moment induced in one cubic centimeter of a non-conducting, or dielectric, material.

One additional intermolecular interaction is used to characterize a solvent's polarity. The dielectric interaction refers primarily to the interaction of sample ions with liquids having a high dielectric constant, such as water or lower aliphatic alcohols. In this type of interaction, a charged ion (such as a salt residue) can polarize the molecules in the surrounding solvent, resulting in increased attractive forces between the solvent and the charged residue, and aiding in the dissolution of the contaminant. These interactions are quite strong and drive the dissolution of ionic or ionizable materials in polar phases such as water and methanol (Ref 20).

Each of the four interactions discussed, hydrogen bonding, dipole, dispersion, and dielectric interactions in combination contribute to the polarity of the solvent, and determine its unique ability to dissolve substances of similar polarity.

3.9 Chemical Families

Hansen (Ref 21) expanded on the Hildebrand solubility parameter and included the major cohesive strength factors which include dispersive forces (δ_d), non-dispersive polarity (δ_p), and hydrogen bonding (δ_h) and combined them into an effective (or total) solubility parameter (δ_t).

$$\delta_t^2 = \delta_d^2 + \delta_p^2 + \delta_h^2$$

Values of solubility parameters and their individual components for various solvents can be found in Refs 22 and 23.

Methylene chloride has a published solubility parameter of about 9.7 $(\text{cal/cm}^3)^{0.5}$ which equates to 19.8 $(\text{MPa})^{0.5}$. This value can also be estimated from observed measurements of the dispersive, polar, and hydrogen bonding contributions as follows:

$$\delta_t^2 = \delta_d^2 + \delta_p^2 + \delta_h^2$$

$$\delta_t^2 = 13.4^2+9.5^2+12^2$$
$$=144 \text{ MPa}$$

$$\delta_t = 20.3\ (\text{MPa})^{0.5}$$

This method of estimating the total solubility parameter of a solvent or material is extremely powerful because it provides for hydrogen bonding and dipole interactions in addition to the dispersive forces originally formulated by Hildebrand. While the inception of the solubility parameter is useful in defining solvation of non-polar molecules displaying only weak dispersive forces, the more recent work using the three component solubility parameter expands the application to include polar and hydrogen bonded solvents. If the solubility parameter of a contaminant is known, or can be estimated, a solvent or group of solvents can be selected to provide maximum dissolution of that contaminant. A corollary to this observation is that if one solvent has the demonstrated capability to dissolve a particular type of contaminant, another solvent having a similar total solubility parameter can be found which will also be effective at dissolving that contaminant. Thus one may identify chemical families, having similar total solubility parameters, which may be effective at dissolving a particular type of contaminant.

A chart relating δ_p vs δ_h for various solvents has been prepared by Moran (Ref. 24). These two contributions have been selected because they vary the most over the solvents evaluated. The δ_d of the solvents studied only spanned from 10.3 (Acetonitrile) to 20.1 $MPa^{0.5}$ (Propylene Carbonate). Figure 6 presents solubility charts for a variety of solvents and solvent families, and the key alongside the charts give the dispersive parameter (δ_d) in parenthesis.

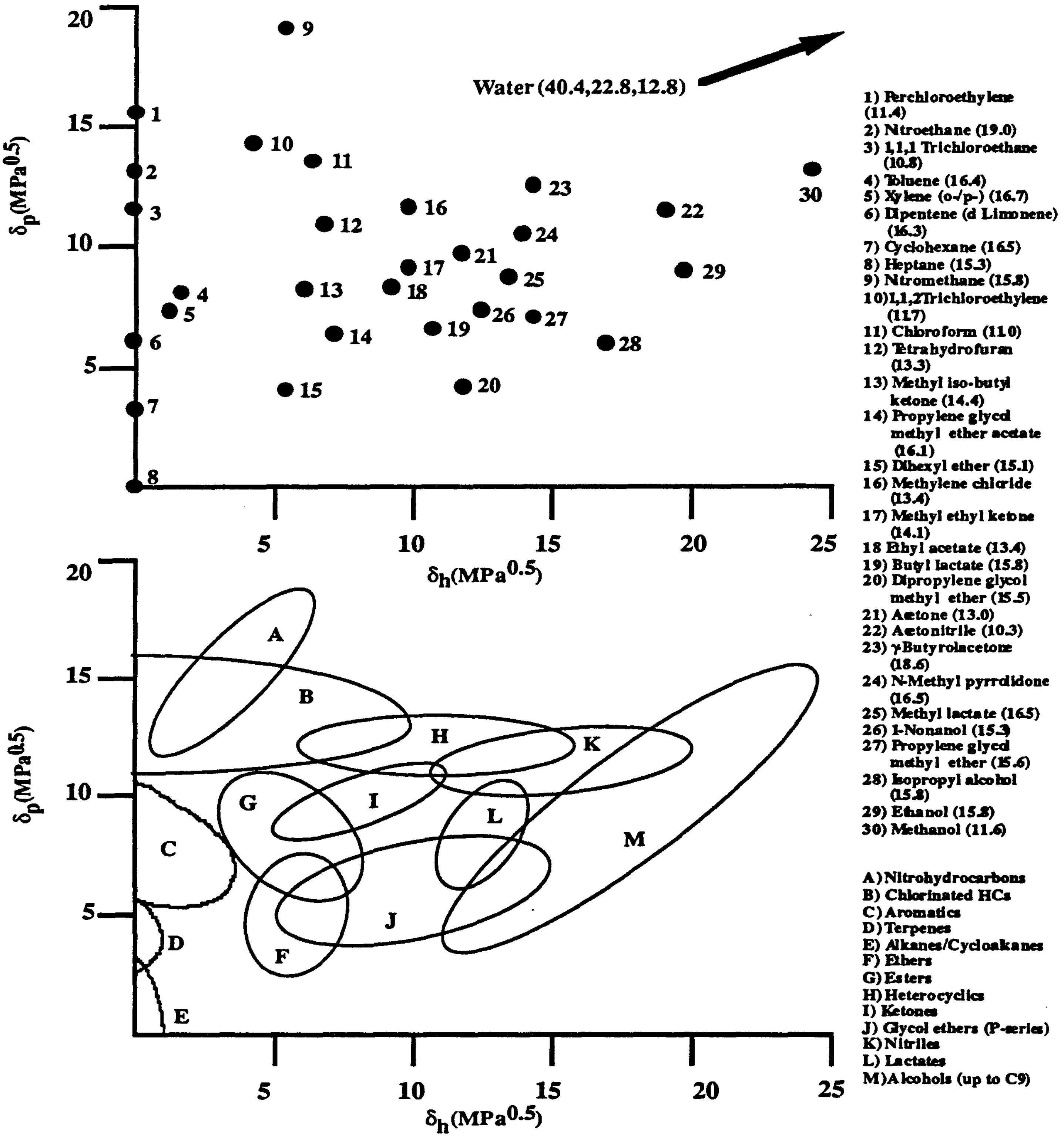


FIGURE 6 SOLUBILITY CHARTS FOR VARIOUS SOLVENTS AND CHEMICAL FAMILIES
(reproduced from reference 16)

3.10 Other Useful Solvent Properties

The choice of a good solvent, in addition to its high solvency for oil , grease, and other contaminants, depends upon the application for which the solvent is intended. For example, in vapor degreasing, a desirable feature of the solvent is to have a low heat of vaporization and low specific heat to maximize the amount of solvent which will condense on the part, and minimize the heat input requirements to the system (Ref 25). In addition the boiling point should be high enough to ensure adequate condensation, but low enough to allow good separation of the contaminant from the solvent by simple distillation. The cleaner used for vapor degreasing should also display a high vapor density (in comparison with air) to minimize loss of solvent to the atmosphere.

Cleaning by immersion frequently requires mechanical agitation or applied ultrasonic energy to allow the solvent to penetrate recesses, and to reduce the cleaning time of complex shapes (Ref 13). In addition, the solvents may be heated to improve the cleansing action, although the cleaning temperature should be maintained well below the flash point. Chlorinated solvents can be heated, but the evolved fumes are highly toxic, and require properly designed equipment and adequate ventilation for personnel health.

Selection requirements for industrial solvents must encompass other parameters other than performance requirements (solvency of the contaminant). The solvent must display no chemical attack on the part to be cleaned, either by metal corrosion or non-metallic embrittlement or swelling. Chlorinated solvents, such as TCA, usually contain stabilizers to retard acid generation in the solvent and subsequent metallic corrosion. The solvent should leave no residue upon removal which could affect the integrity of the part in its intended use. The cleaner should be safe to use (or engineered to be safe) by personnel working with the solvent, and should not contribute to environmental damage upon its disposal. Fire hazards associated with using the solvent should be minimized. The extremely low flammability of the halogenated hydrocarbons (TCA and CFC-113) explain their prevalent industrial use today (Reference 26). The solvent in addition should be commercially available at minimal cost.

3.11 Summary

Several physical and chemical parameters of a commercial solvent have been discussed. These include a low surface tension for removing adherent contaminants from surfaces, the selection of a solubility parameter (δ) which matches as closely as possible the solubility parameter of the contaminant being dissolved, a discussion of the polarity of solvents, and the desire to obtain a solvent with a high polarizability such that its ability to dissolve and disperse many different types of contaminants is achieved. The use of the Kauri-Butanol value as an empirical indicator of solvent strength has also been addressed. Finally, a classification of chemical families has been discussed, using a three dimensional solubility parameter. A chart of chemical families has been presented, which can be very useful to the materials engineer who must select from a variety of different solvents for a particular cleaning application.

Values of selected physical constants for a variety of industrial solvents are included in Table 3.

TABLE 3
PHYSICAL CONSTANTS FOR COMMON INDUSTRIAL CLEANING SOLVENTS

Solvent	Surface Tension (dynes/cm)$_{^{\circ}C}$	Solubility Parameter (MPa)$^{0.5}$	Polarity, μ (Debye)	Polarizability (10^{-24} cm^3)	K_B
Benzene	28.88_{20}	18.8	0	10.32	112
Acetone	23.32_{20}	20.2	2.88	6.33	N/A
Methyl Ethyl Ketone (MEK)	23.97_{25}	19.0		8.13	N/A
Isopropyl Alcohol (IPA)	20.9_{30}	23.5	1.66	7.61	
1,1,1-Trichloroethane (TCA)	25.56_{20}	17.4	1.78	10.7	124
1,1,2-Trichloro-1,2,2-Trifluoro Ethane (Freon-113®)	17.[illegible]$_{20}$	14.9			31
Carbon Tetrachloride	26.15_{25}	17.6	0	11.2	104
Chloroform	27.16_{20}	19.0	1.01	9.5	
Methylene Chloride	28.12_{20}	19.8	1.60	6.48	136

Notes: Benzene, carbon tetrachloride, and chloroform are no longer used as cleaning solvents because of their toxic nature. Their inclusion here is designed to illustrate the chemical properties which have been used to predict good solvent behavior. Physical property data used for this table have been taken from References 4, 11, and 27.

4.0 METRICS AND SELECTION CRITERIA

4.1 Mechanical Cleaners

Because of the large number of ozone safe cleaners available, cleaner properties and characteristics were used to eliminate some of the candidates prior to demonstration and validation testing. Some of these characteristics are universal, such as surface tension and vapor pressure. Other characteristics are specific for the application of cleaning parts in the Valve Shop at PJKS, such as propellant compatibility. Safety, environmental compliance, and cost were all general concerns which were part of the selection criteria. The characteristics were divided into four broad categories: chemical & physical properties, safety & health, environmental, and cost. These characteristics along with the desired ranges are incorporated in Table 4. The right side of Table 4 indicates how the characteristic was evaluated: literature, analysis or test. Literature indicates that the information is already available from vendors, reference material or published reports. Analysis is defined as predictions, judgments, and calculations based on sound technical practice and knowledge. Test is defined as collecting information through trial and experimentation.

The chemical and physical properties that were evaluated are surface tension, density, vapor pressure, contaminant solubility, residue, material compatibility, propellant compatibility, and solvent life. Surface tension is one factor which determines how well a cleaner can remove contaminants from a surface. Most effective cleaners have surface tensions of less than 30 dynes/cm at 20 °C. The density of a fluid determines the ability of the cleaner to pick up and carry away particulate contamination. Therefore, a higher density is more desirable. The vapor pressure is a measure of volatility. Quick evaporation simplifies the drying process following cleaning and alleviates leaving cleaner residues in small cavities. However, quick evaporation may cause contaminants to be redeposited on evaporation or may leave residues. High vapor pressures also result in higher vapor concentrations in the work area which may affect worker health, and an increase in chemical emissions which can be an environmental concern. Surface tension, density and vapor pressure information can be obtained from the vendor literature, and testing is not required. The primary measure of a cleaning fluid is its ability to dissolve contaminants.

TABLE 4 MECHANICAL CLEANING REQUIREMENTS

Category	Specific property	Desired range	Deter mine by *		
			Literature	Analysis	Test
Chemical & Physical	surface tension	< 30 dynes/cm at 20 °C	X		
	density	N/A	X		
	vapor pressure & volatility	< 45 & > 18 mm Hg at 20 °C	X		
	Kauri- Butanol value	> 60	X		X
	contaminant removal	unique contaminants solubility			X
	low residue & NVR	< 0.1 mg/l < 1.0 mg/ft ^{2}			X
	material compatibility	w/ metal & soft goods	X	X	X
	propellant compatibility	w/ hydrazine, N_2O_4, ...	X	X	X
	solvent life	N/A		X	X
Safety & Health	flammability	< 3, rating in CISH book	X	X	
	flash point	> 150°F	X	X	X
	toxicity	non toxic	X		
Environmental	regulation limitations	VOC < 200 g/l	X	X	
	waste disposal	Treated on site & low cost.		X	
	friendly	less is better		X	
Cost	solvent	Low cost is desirable.		X	
	disposal/process	< $700/drum.		X	
	capital equipment	< $200K		X	

* Literature is information already available from vendors, reference material or published reports. Predictions, judgments and calculations, based on sound technical practice and knowledge are analysis. Test is collecting information through trial and experimentation.

A standard measure of solvent strength is the Kauri-Butanol (K_B) value which can be determined in accordance with ASTM D1133. However, this application has specific and unique contaminants: the solubility of those contaminants are typically determined in a laboratory. Residues left from the cleaners may affect the parts for propellant service and for the application of coatings and surface finishes. Therefore, the amount and composition of residues was determined by reviewing vendor literature and by conducting appropriate tests. Materials being cleaned must not be degraded by the cleaner; and since much of the hardware will contact liquid rocket propellants, the compatibility of the cleaner with propellants must also be evaluated. Finally, solvent life should be determined by appropriate testing. Solvent life is defined as the number of contaminated parts that may be processed before the solvent must be discarded and replaced with fresh solvent. Solvent life is important in determining the process costs and the environmental impact of waste disposal.

Personnel safety and health is a primary concern in the aerospace industry. Flammability, toxicity, and volatility of the alternate cleaners require evaluation . The flash point is the main determinant of the flammability. However, proper equipment design and common precautions can be taken in order to safely use flammable substances. Therefore, alternatives may not have been eliminated solely on flammability, but may have been more expensive due to the required equipment and controls needed to eliminate the hazard. The Industrial Hygiene Dept. researched toxicity in reference books such as Properties of Dangerous Materials by Sax and Registry of Toxic Effects of Chemical Substances (RTECS). In addition to chemical toxicity, sensitivities to the skin and respiratory systems were evaluated to determine the personnel protective equipment and ventilation requirements. The use of special equipment to mitigate hazards was identified because they would affect the capital equipment and/or process costs.

Environmental considerations were also important. This study was initiated because of the concern that CFCs are depleting atmospheric ozone. Alternatives had to be evaluated to prevent trading one harmful chemical for another. Regulatory limitations, waste disposal, and basic environmental friendliness were considered during the evaluation process. Current and anticipated future regulations may limit the usage or impact the cost of the alternative cleaners. The desired environmental objective of the selected cleaner is to eliminate ozone depleting chemicals with minimal impact to waste disposal and while maintaining the lowest cost. Finally, the nature of the cleaner may make it more friendly to the environment than others. The consumption of less resources and energy during the cleaning process results in better environmental conservation.

Finally, the cost of the different cleaners had to be evaluated. The costs can be divided into three categories. Solvent cost is the procured cost of the cleaner. Process and operating costs are maintenance labor and material costs. Capital equipment costs are the new equipment that is required, including any safety items such as vent hoods and vapor monitors. These costs were estimated from vendor information and by evaluating test results. The costs were compared with each other and with the existing process.

4.2 Anti-Seize Compounds

The three metrics that were used to evaluate the anti-seize compounds are sealing properties, lubricating properties and ability to withstand exposure to temperatures of 1200 °F. A good alternative should have the ability to seal the threaded surfaces against moisture penetration. It also should provide good lubricating properties to allow the proper clamping force for a given applied torque. Finally, the anti-seize compound should retain its release properties after repeated exposure to high temperatures. The torque required to separate fasteners after exposure should not exceed the torsional stress allowable for the fastener.

4.3 Electrical Cleaners

4.3.1 Contact Cleaners

For electrical contacts, technical requirements of substitute cleaners include the ability to remove the contaminant, materials compatibility (no corrosion for metals), fast evaporating, no unwanted residues, the ability to reduce arcing and RFI, and the ability of the cleaned contacts to perform their function over a wide temperature range. Contact life and performance is degraded by a number of environmental and operational parameters, among them contact wear, arc erosion, contamination, frettage corrosion, and contact bounce. Contact wear occurs when the contacts slide across each other during normal contact switching. This sliding causes metal erosion and wear which decreases the surface area in contact by the adjoining metals and results in increased electrical resistivity. Contact wear may be minimized by properly designing the electrical contact, by specifying a smooth surface finish, by selecting proper construction materials, by controlling the operating temperature, and by maintaining the proper contact pressure. Arc erosion occurs when electrical energy is transferred by sparking from one contact to the other. In this case, ionizing air is the transfer medium instead of the adjoining electrically conductive metals. Contamination of contacts include atmospheric oxidation and silicone contamination, both of which can increase the resistance of the contacts beyond required operating values. When contacts degraded by excessive mechanical wear are exposed to a corrosive environment (such as humid air or acidic gases), metallic corrosion of the worn contacts is accelerated. Finally, contact bounce may occur as the switch position is cycled repetitively. Contact bounce is a low amplitude vibration that may also increase contact wear and contribute to frettage corrosion. All of these parameters contribute to electrical and mechanical degradation of the contacts, and cleaning and lubrication of the contacts at prescribed maintenance intervals helps to minimize these problems.

In addition to cleaning processes, good contact cleaners will also have a lubricant to minimize contact wear and erosion. Specific advantages of including a lubricant in a contact cleaner are listed in Table 5.

TABLE 5 ADVANTAGES OF LUBRICATED CONTACTS

Clean and Dry Contacts	Lubricated Contacts
Surface Finish is Not Perfect	Small Imperfections are "Filled" with Lubricant, Effective Contact Area is Increased
Wear is Continuous	Wear is Minimized
Arc Erosion and Contact Bounce Cannot be Reduced or Eliminated	Arc Erosion and Contact Bounce will be Minimized or Eliminated
Frettage Corrosion is Continuous	Frettage Corrosion will be Minimized or Eliminated
Oxidation Readily Occurs	Oxidation will be Eliminated on Some Metals
Contamination is Present	Contamination Effects are Minimized
Silicone Contamination is Difficult to Remove	Silicone Contamination may not Occur, or will be Easily Removed upon Occurrence

Metrics for selecting an ODC-free cleaner for the existing contact cleaner currently in use at AF Plant PJKS include the following:

1. Cleaning Ability. The cleaner must demonstrate adequate removal of contaminants and other residues which degrade mechanical and electrical performance. In addition, proper lubrication of the contacts should be provided such that restoration of electrical performance occurs after repetitive switching cycles. The purpose of this metric is to demonstrate removal of common contaminants (oils, dust, and particulates) from common contact materials (copper, brass, and tin and silver plated copper). This performance requirement was verified by test.

2. Compatibility. The cleaner must be chemically compatible with materials of construction used in the design and production of the electrical contacts. In particular, no adverse chemical reaction between the contacts and the cleaner should occur which could potentially degrade the performance or function of the electrical contacts in their intended applications. This performance requirement was verified by analysis.

3. Fast Evaporating. The cleaner must evaporate quickly from the contacts, such that residual cleaner residues are adequately removed prior to using the contacts in intended applications. This performance requirement was verified by test.

4. No Residues. The cleaner shall leave no residues upon evaporation, other than required lubricants. This performance requirement was verified by test.

5. Wide Temperature Range. The candidate cleaner shall display a wide temperature use range as required by the operating temperature requirements of the applicable electrical contacts, and/or electrical components. This performance requirement was verified by review and analysis of pertinent data.

4.3.2 Tape Head Cleaners

For tape head cleaning, the primary issues for selection of the replacement chemical were the cleaning ability of the cleaner, the purity of the cleaner (no residues upon evaporation), and the compatibility of the cleaner with contacted materials. The cleaner should have a low vapor pressure and flash off quickly. It should be readily available at a modest cost. In addition, any alternative cleaner must be non hazardous to the environment and operationally safe for the user. Areas requiring cleaning in a magnetic tape head include the tape path (all areas in contact with the magnetic tape), the magnetic heads themselves, and all other system components. Dust, metal oxide deposits, and particulates can cause abrasion of the magnetic heads or the Capstan rollers which guide the tape. In addition, the cleaner must leave no residue upon evaporation, must be chemically compatible with the materials in contact with the cleaner, and should be safe to use by the individual servicing the unit. Acceptable performance of the tape drive system should be demonstrated after cleaning the tape heads. For critical applications (such as recording and playback of digital data for test measurements), the signal-to-noise ratio of the dirty and cleaned tape heads can be compared. For routine VCR applications, the picture quality or visual inspection of the tape head after cleaning is adequate to verify contaminant removal and lack of residue after evaporation.

4.3.3 Strain Gage Bonding

For cleaning prior to installing strain gauges, the cleaner must remove the contaminant and permit successful bonding of the instrumentation to the desired surfaces. The cleaner must be compatible with the material substrate, the adhesive, and the instrumentation and must not leave any unwanted residues. In addition, the alternative carrier (IPA) for the strain gauge adhesive requires qualification for use in the process.

Bonding strain gages properly to test hardware and materials require that three conditions be achieved:

1. Adhesive strength of materials bonded with "new" ODC-free adhesive must meet or exceed adhesive strength of existing ODC adhesive, under expected operating temperatures.

2. Cleaners selected to replace the existing TCA degreasing solvent must provide a chemically clean surface to which optimum bond strengths can be achieved. In this application bond strengths achieved with replacement ODC-free cleaners must meet or exceed bond strengths achieved using TCA as a cleaner.

3. Strain gages applied by cleaning with ODC-free cleaners and bonded using ODC-free adhesives must remain intact on material surfaces during tension, such that premature separation of the gages from the hardware does not occur.

5.0 ALTERNATIVE CLEANERS

5.1 Approach

5.1.1 Literature Search. Cleaning alternatives for mechanical degreasing, electrical degreasing and replacement of an anti-seize compound were investigated using computer assisted library searches. Three major data bases were accessed: National Technical Information Service (NTIS) 1985-1993; Applied Science and Technology (1983-1993) and Aerospace (1989-1993). These searches identified over 150 articles related to the elimination of ozone depleting chemicals in industry and government applications. These articles were carefully screened, and the pertinent articles were obtained and reviewed for application to the present study. Articles used in this effort are listed in the Reference Section.

5.1.2 Government/Industry Users. Telephone calls were placed to users of the present ozone depleting chemicals to inquire about substitution progress. In particular, aerospace engineering operations similar to Air Force Plant PJKS were contacted. These included the Jet Propulsion Laboratory (Pasadena, CA), Marshall Space Flight Center (AL), Kennedy Space Center (FL), Vandenberg AFB (CA), the Aerospace Corporation (Los Angeles, CA), and others. Communication with these using groups has been very beneficial in designing and enacting a plan for elimination of these chemicals at PJKS.

5.1.3 Chemical Manufacturers. Both the current manufacturers of TCA (Dow Chemical Company) and CFC-113 (Du Pont de Nemours) were contacted with respect to alternative cleaners. Dow Chemical indicated that no drop in replacement for TCA was currently being marketed, however, they would work with Martin Marietta to design and test suitable alternatives to meet specific applications. Du Pont has formulated alternative cleaners, based on glycol ether-hydrocarbon blends. The Axarel® product line is a result of their research into alternative cleaners. Martin Marietta does currently use Axarel products in electrical manufacturing operations, and Du Ponts's formulations were some of the 60 candidates considered for electrical and mechanical cleaning operations. As will be described in subsequent sections, the Du Pont products were eliminated during the selection process.

5.1.4 Environmental Conferences, Symposiums, and Working Groups. As part of an integrated ODC replacement team, several team members attended or presented results at a variety of conferences related to ozone layer depletion elimination or pollution prevention. For example, two Martin Marietta employees (S. Prince and E. King) presented papers on alternative cleaning and cleaning verification methods, respectively, at the 1994 HazMat/Pollution Prevention Symposium held at Wright Patterson AFB 21-22 April 1994. Attendance at these and similar conferences has been instrumental in providing two-way communication needed to share lessons learned in solvent replacement and ODC elimination.

5.2 Identification Of Candidates

5.2.1 Mechanical Cleaning

The purpose of the selection process was to conduct screening tests, demonstration tests, and validation tests on the most promising cleaning candidates and to eliminate alternatives which don't meet the selection criteria. Preselection of the original list of candidates was performed by reviewing available literature, and by communicating with chemical vendors. The cleaners evaluated during this program are shown in Table 6, and Table 7 shows values of the selection criteria, where available. These criteria are: surface tension, density, vapor pressure, contaminant solubility, material compatibility, flammability, flash point, toxicity, and regulation limitations. Several support groups at Martin Marietta were asked to assist in the preselection process. Industrial Hygiene, Occupational Safety & Health, and Environmental Management performed evaluations of the flammability, toxicity, regulatory limitations, and waste disposal. The remaining evaluations were made by the assembled project teams. Decisions made on these cleaning candidates (either retention for subsequent evaluations, or elimination), as well as the evaluation conducted by the Martin Marietta Industrial Hygiene Department are included in Appendix A.

TABLE 6 LIST OF CANDIDATE ODC-FREE CLEANERS, MECHANICAL CLEANING

Product Name	Manufacturer	Description	Cleaner Type	Application
Alcohol	J.T. Baker Inc.	ethyl, methyl, IPA	alcohol	final cleaning
Aquinox SSA				
Armaclean E-2001	Church & Dwight Inc.	Inorganic salts, buffered	Alkaline	Heated 160 F.
AVD	Petroferm	Aliphatic ester & Perfluorocarbon		Complex vapor degreasing equipment
Axarel 52	DuPont	Aliphatic Hydrocarbons & Diisobutly DBE	Semi-aqueous	Immersion (room temp or elevated)
Axarel 2200	DuPont	1-Propoxy-2-Propanol & hydrocarbons	Hydrocarbon	Spray bottle & wipe.
Axarel 6100	DuPont	Aliphatic Hydrocarbons & Diisobutly DBE	Semi-aqueous	immersion room temperature
Bio Clear	Bio-Chem Systems	Water (80%), d-Limonene & surfactants.	Aqueous	Ultrasonic bath - immersion- no rinse
Bio-T-Max	Bio-Chem Systems	d-Limonene	Terpene	Ultrasonic bath - immersion
Bioact 120	Petroferm	Terpene (65-95%) & surfactants	Terpene	Cold tank immersion
Bioact 280	Petroferm	Aliphatic esters	Hydrocarbon	Cold tank immersion or heated
Biogenic Regent	Rochester Midland	Aliphatic hydrocarbons & d-Limonene (25-30%)	Hydrocarbon	Immersion bath, pure.
Breakthrough	Inland Technology	C12 -C13	Hydrocarbon	
Brulin 815 GD	Brulin	Water	Alkaline	Immersion, heated (160 F)
Citra Safe	Inland Technology	d-Limonene, >95%	Terpene	
Citrasolv		d-Limonene	Terpene	
Citrex	Inland Technology	NMP & d-Limonene	Organic	
Daraclean 220	W.R. Grace	No glycol ethers.	Alkaline	Immersion.
Daraclean 235XL	W.R. Grace	No silicates or glycol ethers	Aqueous	Heated, ultrasonic bath-immersion
Daraclean 282	W.R. Grace	Glycol ether.	Aqueous	Heated, ultrasonic bath - immersion
Desoclean 20	DeSoto		Organic	
Electrolube	Phoenix Systems			
Electron	Sentry	Refined aliphatic hydrocarbons & terpenes	Hydrocarbon	Apply & wipe off or dip tank.
EP-921	Inland Technology	Propylene carbonate & d-Limonene	Organic	
EZE 431	EZE Products Inc.	Petroleum distillate	Hydrocarbon	
EZE 662P	EZE Products Inc.			Vanishing oil
Formula 624 GD	Quaker	Silicic acid, sodium hydroxide	Alkaline	Immersion, heated.
HFC Vapor				
Iso-Prep	Inland Technology	C12-C13, distilled	Hydrocarbon	
JPX Degreaser	Jayne Products	Water (50%), terpene, glycol ether	Semi-aqueous	Immersion, room temp.
Kerosene	various	Hydrocarbon fraction	Hydrocarbon	
MOK	Boeing	Water soluble	Proprietary	
n-methyl pyrolidone	J.T. Baker Inc.	Heterocyclic compounds	Water soluble	
Partsprep	Sentry	NMP	Semi-aqueous	Heated (150 F) bath.
Perfluorocarbons	generic			
PF Degreaser	P-T Technologies	Aliphatic hydrocarbons & citrus	Organic	
PF-145-HF	P-T Technologies	Aromatic, aliphatic hydrocarbons	Hydrocarbon	
PM Acetate	generic	glycol ether		
Prep Rite	Sentry	Enamel, varnish, acrylic, latex	Paint remover	

TABLE 6 LIST OF CANDIDATE ODC-FREE CLEANERS, MECHANICAL CLEANING (CONCLUDED)

Product Name	Manufacturer	Description	Cleaner Type	Application
Purasolv ELS	Purac			
Safe-strip	Sentry	Even epoxy paints	Paint remover	
Safer Bio-Degreaser	Glinton Industries Inc.	Water, glycol ether.	Aqueous	Spray, wipe or dip. Dilute as necessary.
Safety-Kleen	Safety-Kleen Corp	Naptha solvent	Organic	Cleaning & degreasing
ShopMaster	Buckeye International Inc.	Water & glycol ether.	Aqueous	Dip tank or spray. Can be heated.
ShopMaster A/C	Buckeye International Inc.	Water & glycol ether. No silicates.	Aqueous	Dip tank or spray. Can be heated.
ShopMaster LpH	Buckeye International Inc.	Water & glycol ether. Low pH.	Aqueous	Dip tank or spray. Can be heated.
Skysol	Inland Technology	d-Limonene	Organic	
Sonacor 103	Sonicor	Glycol ether?	Alkaline	
Teksol EP	Inland Technology	C-10, C-9 & d-Limonene	Hydrocarbon	
Turco 3878 LF-NC	Elf AtoChem	Some glycol ethers	Aqueous	Immersion. Cold or heated (140 F).
Turco 4215-NC-LT	Elf AtoChem	Glycol ether. No silicates or chromates.	Aqueous	Immersion w/ ultrasonic. Heated (110 F).
Turco 6759	Elf AtoChem	Ketone, toluene, acetate, IPA.	Organic	Handwipe cleaning.
Turco 6776	Elf AtoChem	Epoxies, polyeurethanes, Formic acid.	Paint remover	Apply & scrap off.
Turco 6778	Elf AtoChem	Silicates & no glycol ethers.	Aqueous	Immersion. Cold or heated (140 F).
Turco 6780	Elf AtoChem		Aqueous	
Turco 6813	Elf AtoChem	Epoxies, enamels. Ammonium hydrate.	Paint remover	Apply & scrap off.
Vortex	Sentry	d-Limonene (>85%) & surfactants	Terpene	Must rinse
X-Caliber	Inland Technology	NMP & d-limonene	Organic	electrical components

TABLE 7 COMPARATIVE PHYSICAL AND CHEMICAL PROPERTIES

Alternative	1	2	3	4	5	6	7	8	9	10	11	Keep ?
Alcohol (IPA)		.79						53				N
Aquinox SSA												I
Armaclean E-2001												I
AVD (advanced vapor degreasing)												Y
Axarel 52	27	0.83	<0.1		yes		1	210	1.5 ppm TLV			Y
Axarel 2200		0.82	2		yes		2	111	1.5 ppm TLV			N
Axarel 6100	26	0.81	<1		yes		2	145	1.5 ppm TLV			Y
Bio Clear												Y
Bio-T-Max		.863			most	no ox	2	146	1	no	no	Y
Bioact 120		0.85	1.6		yes		2	117				N
Bioact 280		0.87	<0.01				1	140				Y
Biogenic Regent		.78	0.2	28			2	147	1	no	no	Y
Break through			<2					150				N
Brulin 815 GD												Y
Citra Safe	29.8	0.84	<2					132				N
Citrasolv												N
Citrex		0.86	<2		yes			144	1	no	no	Y
Daraclean 220	35	1.08										N
Daraclean 235XL		1.02				no ox		200	1	no	no	Y
Daraclean 282	30 @2%	1.02				no ox		212	1	no	no	Y
Desoclean 20								46				N
Electrolube												N
Electron		0.78	10				2	147				I
EP-921								146	no	no	no	Y
EZE 431		0.80	12			no ox	2	122		no	no	I
EZE 662P		0.77	12			no ox	2	105	no	no	no	N
Formula 624 GD		1.08				no ox				no	no	I

TABLE 7 COMPARATIVE PHYSICAL AND CHEMICAL PROPERTIES (CONTINUED)

Alternative	1	2	3	4	5	6	7	8	9	10	11	Keep ?
HFC Vapor												N
Iso-Prep		0.75	<10	28			2	104		no	no	N
JPX Degreaser		0.95	<.054					145				Y
Kerosene		0.80	<0.1		most	no ox	yes	125	1	no	no	N
MOK		1.2	<0.1		most	ox?	2	230	1	no	no	N
n-methyl pyrrolidone		.81				no	very	37.4	3	no	no	N
Partsprep		1.01	<0.3				2	193				I
Perfluorocarbon												N
PF Degreaser		0.76	>1		yes	no ox	comb	144	1	no	no	I
PF-145-HP		0.85	>1		yes	no ox	yes	145	1	no	no	N
PM Acetate		.97	3.7		yes	no ox	comb	45.5	1	no	no	N
Prep Rite		1.08					1	197	3			N
Purasolv ELS												N
Safe-strip							2	197	2			N
Safer Bio-Degreaser		1.01	18		most	no ox	no	0	1	no	no	I
Safety-Kleen		0.80	2				1	148	1	no	no	Y
Shopmaster	29	1.03					0		0	no	no	Y
ShopMaster A/C	29	1.03					0			no	no	Y
ShopMaster LpH	29	1.03					0		1	no	no	Y
Skysol		0.77	2			no ox		152	1	no	no	N

TABLE 7 COMPARATIVE PHYSICAL AND CHEMICAL PROPERTIES (CONCLUDED)

Alternative	1	2	3	4	5	6	7	8	9	10	11	Keep ?
Sonacor 103		1.01										Y
Teksol EP		0.77	<10					112				N
Turco 3878 LF-NC												I
Turco 4215-NC-LT												Y
Turco 6759		0.83	19					51				N
Turco 6776		1.05										N
Turco 6778												I
Turco 6780			25					111				N
Turco 6813												N
Vortex		0.88	<1				2	119	1			N
X-Caliber		0.92	<2					155				N

1- Surface tension in dynes/cm at 20°C
2- Density in lbs/ft^3
3- Vapor pressure in mm Hg at 20 °C
4- Kauri-Butanol value
5- Is it compatible with materials being cleaned?
6- Is it compatible with propellants?
7- Flammability class.
8- Flash point in °F.
9- Is it considered toxic?
10- Are any regulatory limitations limiting usage? Are any anticipated?
11- Can the waste be treated on site?

Y = YES
N = NO
? = Questionable
I = More Information Needed

5.2.2 Anti-seize Compounds

Initial selection of candidate replacement compounds was performed upon review of vendor literature. While many manufacturers produce anti-seize compounds, our search was limited to high temperature compounds. To further define boundaries for the selection process, candidates were selected from nationally recognized corporations such as Dow Corning and Loctite. This was done to ensure availability and consistency of the product.

The prescreening process yielded five potential candidates. One from the Dow Corning Corp., one from the Loctite Corp., two from Anti-Seize Technology, and the last from the Fel-Pro Chemical Products Co. The Fel-Pro product was selected because we currently utilize an anti-seize product manufactured by the Fel-Pro Company that contains an ozone depleting chemical (ODC).

The material safety data (MSD) sheets for these five candidates were sent to the industrial hygiene department for evaluation. Of the five, one was rejected. The Anti-Seize Technology Moly-lit product was removed from the list because it contained lead oxide and was undesirable from a health standpoint. The remaining four candidates selected for evaluation are listed below.

1- Loctite Anti-seize 767 (1600° F)
2- Anti-Seize Technology Anti-seize Special (2000° F)
3- Dow Corning Molykote 1000 (2100° F)
4- Fel-Pro C5A High Temp Anti-seize Compound (1800° F)

Table 8 lists the candidate anti-seize materials and their chemical composition. All of the candidates are similar in that they all use some sort of a petroleum base material that contains powdered metals and graphite.

TABLE 8 ALTERNATIVE ANTI-SEIZE COMPOUNDS

CANDIDATE	COMPOSITION	% (wt.)
Loctite 767	Mineral Oil	65-70 %
	Copper	10-15 %
	Aluminum	5-10 %
	Graphite	1-3 %
	Silica	1-3 %
Dow Molykote 1000	Mineral Oil	48 %
	Calcium Fluoride	20 %
	Graphite	10 %
	Copper	7 %
	Disodium Sebacate	4 %
	Silica	2 %
Anti-Seize Technology	Petroleum Grease/Oil	not available
	Copper	21.9 %
	Aluminum	3 %
	Graphite	not available
	Corrosion Inhibitors	trade secret
Fel-Pro C5A	Petroleum Base	not available
	Copper and Graphite	40 %

5.2.3 Electrical Cleaners.

Candidate replacement cleaners have been identified for electrical contacts, video tape heads, and cleaning prior to bonding strain gages. A brief discussion of the candidates identified for these three applications follows.

5.2.3.1 Contact Cleaners

Several commercial contact cleaners free of ozone depleting chemicals are currently available from the respective manufacturers. Contact cleaners evaluated by Martin Marietta for this ODC elimination program are listed in Table 9.

TABLE 9 ALTERNATIVE CONTACT CLEANERS

Cleaner	Manufacturer	Remarks
Kontact Restorer	Chemtronics	Present Cleaner (contains CFC-11)
MS-939 Contact Re-Nu	Miller Stephenson	1,1-Dichloro-1fluoroethane/methanol
ASP#1M	Poly Chem	Diacetone blend
C.S. Cleaner	Zip Chemical	Hydrocarbon, terpene blend
Deoxit 100	Caig Labs	Mineral oil
MS-938/CO2 Safezone Contact Re-Nu	Miller Stephenson	1,1-Dichloro-1-fluoroethane/methanol
HF Contact Cleaner	CRC Industries	Petroleum Distillate
Contact Cleaner 2000	CRC Industries	1,1-Dichloro-1-fluoroethane

In addition to the cleaners presented above, several lubricants were also tested to evaluate anti-wear properties. These lubricants are listed in Table 10.

TABLE 10 ALTERNATIVE CONTACT LUBRICANTS

Lubricant	Manufacturer	Remarks
Contact Treatment Grease CG52B	Electrolube	synthetic oil
ReNu lube	Miller Stephenson	
MS938,MS939	Miller Stephenson	1.1-Dichloro-1-fluoroethane/methanol
WD40	WD40	petroleum product
Deoxit 100	Caig Laboratories	mineral oil
Kontact Restorer	Chemtronics	CFC-11 & hydrocarbon lubricant

5.2.3.2 Tape Head Cleaners

Chemical solvents evaluated for cleaning video tape heads are included in Table 11

TABLE 11 ALTERNATIVE TAPE HEAD CLEANERS

Solvent	Manufacturer	Remarks
CFC-113	Omnisolve	existing cleaner, ODC
TCA	EM Science	existing cleaner, ODC
Isopropyl Alcohol	Unknown	technical grade
Ethyl Alcohol	Mallinkrodt	reagent grade, denatured (5% IPA)
Heptane	Aldrich Gold Label	reagent grade
Ethyl Alcohol	McCormick Distilling Co.	absolute, dehydrated, 200 proof

Additional cleaners originally considered as replacement candidates, but not evaluated further include:

Cleaner	Manufacturer	Remarks
Head Cleaner II	Chemtronics	alcohol blend
Isoclene	Automated Facilities	IPA
Video Head Cleaner	Electrolube	water based
hydrochlorofluorocarbons	DuPont Chemical	impending environmental restrictions

5.2.3.3 Strain Gage Bonding

The following adhesive systems were evaluated as part of the strain gage bonding replacement effort:

TABLE 12 ALTERNATIVE STRAIN GAGE ADHESIVES

Adhesive	Manufacturer	Remarks
M-Bond 200 (old)	Micromeasurements	Contains TCA
M-Bond 200 (new)	Micromeasurements	Contains IPA

The following alternative cleaners were evaluated for surface preparation prior to bonding:

TABLE 13 ALTERNATIVE SURFACE CLEANERS FOR STRAIN GAGE BONDING

Cleaner	Manufacturer	Remarks
TCA	Dow Chemical Corp	Existing ODC
EP921	Inland Technology	Propylene Carbonate, d-limonene
Bio-T-Max	Golden Technologies	d-limonene
CFC-113	DuPont Chemical	Existing ODC
Isopropyl Alcohol	J.T. Baker Chemical	
Ethyl Alcohol	J.T. Baker Chemical	
Biogenic Regent	Rochester Midland	Aliphatic Hydrocarbons, d-limonene

6.0 TEST METHODS AND RESULTS

6.1 Mechanical Cleaning

Initially, over 60 cleaners were identified as possible replacements for TCA and CFC-113 in mechanical degreasing operations. These initial candidates were identified from current literature sources and upon communication with various chemical manufacturers. The list included representatives from different chemical families: aqueous cleaners, semi-aqueous cleaners, organic solvents, alkaline cleaners, and terpene cleaners. Figure 7 shows the decision tree used to provide technical screening of these 60 candidates, and to provide final selections for implementation at the PJKS facility.

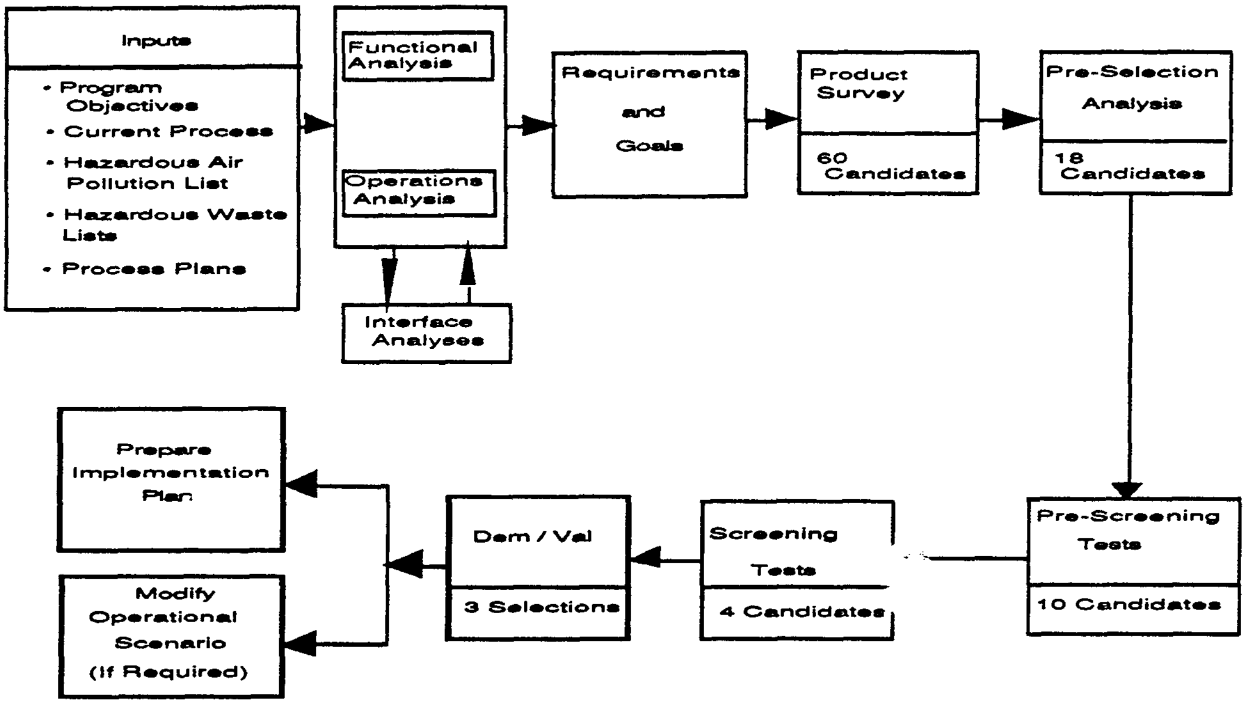


FIGURE 7 FLOWCHART MECHANICAL CLEANER SELECTION

6.1.1 Prescreening

Criteria used to eliminate cleaners from the original list of approximately 60 candidates considered include: 1) lack of support from vendor, manufacturer, and/or distributor. Lack of support was defined as inadequate product information, no material safety data sheets, etc. 2) environmental concerns or disposal issues; 3) chemical hazards such as flammability, noxious odor, toxicity, etc.; 4) incorrect product for the intended ap[illegible]ion; and 4) chemical similarity to another product scheduled for test. As a result of the prescreening pr[illegible] 18 cleaners were selected for further evaluation.

The following is a list of the 18 candidates:

Bioact 280
Citrex
EP 921
n-methylpyrrolidone
PF-145-HP
Turco 3878LF-NC
Biogenic Regent
Daraclean 235XL
Electron
Partsprep
Shopmaster
Turco 3878
Bio-T-Max
Daraclean 282
Brulin 815 GD
PF Degreaser
ShopmasterA/C
Shopmaster LpH

A prescreening test was devised to measure the cleaning power of the eighteen candidates. The purpose of the prescreening test was to eliminate several of the candidates such that eight to ten cleaners could be evaluated more rigorously under more exacting test conditions (screening tests).

6.1.1.1 Cleaning Performance Test. 2" X 6" X .125" test coupons were prepared from 6061 aluminum, carbon steel, and 304 stainless steel sheet stock. The coupons were precleaned using TCA in an ultrasonic bath and then cleaned in an ultrasonic bath of hot soapy water. Cleaning was followed by a deionized water rinse and oven drying. The coupons were then contaminated with Drilube 822™ (MMS N306), hydraulic oil (Mil-H-6083) and lithium based grease (Mobil EP2™). These contaminants were applied to the metal surfaces using cotton swabs. Parallel, equally spaced, applications of each of the three contaminants were made to each coupon. The applications were approximately 1 inch long and 1/4 inch wide and were made at one end of the coupon (Figure 8). The coupons were maintained at ambient temperature until degreasing using the alternative cleaners was performed. Cleaning was accomplished by immersing the coupons in a glass beaker containing one of the alternative cleaners. The beaker was suspended in the ultrasonic bath for 5 minutes, at a frequency of 47kHz. Figure 9 shows the test fixture for the cleaning performance test.

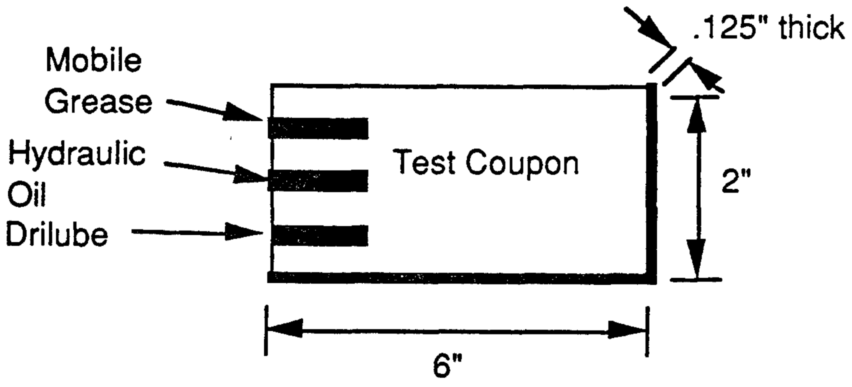


FIGURE 8 PRE-SCREENING TEST COUPON

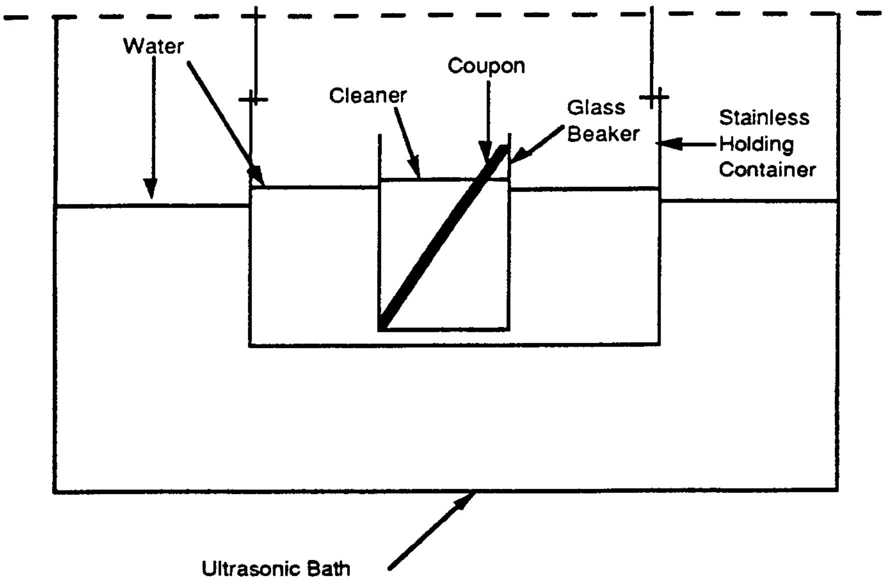


FIGURE 9 CLEANING PERFORMANCE TEST FIXTURE

At the completion of the five minute cleaning period, test coupons were removed from the cleaners, inspected visually for residual contamination, and evaluated using a water break test. A maximum of five points was awarded the cleaner if no residues remained, and if water applied to the surface uniformly wet the cleaned metal surface. Points were subtracted from this value based on breaks in the water layer and on visible remaining contamination as follows:

Cleaning Defect	Points Subtracted
water break	.2 to 1.5
slight water break	.1 to .5
Drilube not removed	1 to 2
grease not removed	1 to 2

TCA and CFC-113 were included in this evaluation as baseline cleaners. Results of the cleaning performance test for the eighteen candidate cleaners are included in Table 14. Detailed test conditions and results for these tests are included in Appendix B. Using this Prescreening test data, ten cleaners (including TCA and CFC-113) were selected for additional testing. Turco 3878, although displaying a relatively high rating, was not considered further because of environmental disposal concerns.

TABLE 14 PRESCREENING TEST RESULTS

cleaner	rating	cleaner	rating	cleaner	rating
Bioact 280*	4.7	Biogenic Regent*	4.0	Citrex*	4.9
Daraclean 282*	4.0	EP-921*	4.8	Brulin 815GD*	4.0
JPX Degreaser*	4.9	Partsprep*	5.0	TCA*	4.9
CFC-113*	4.9	Bio-T-Max	3.0	Daraclean 235XL	3.0
Electron	2.5	Formula 624 GD	3.8	PF Degreaser	2.5
PF-145-HP	2.0	Shopmaster FF	3.0	Shopmaster A/C	4.0
Shopmaster LpH	1.5	Turco 3878	4.5	Turco 3878 LF-NC	3.[illegible]

* Selected for Additional Testing

6.1.2 Screening Tests

The purpose of the screening tests was to evaluate the cleaning power, soils loading, materials compatibility, and propellant compatibility of the selected candidate cleaners. Bioact 280, Biogenic Regent, Citrex, Daraclean 282, EP-921, Formula 815, JPX Degreaser and Partsprep were used in the as received condition, un-diluted. Two cleaners, Daraclean 282 and Formula 815, were diluted with deionized water to a 25% cleaner to water solution strength, per manufacturers instructions. CFC-113 and Trichloroethane were included in the test for comparison and baseline purposes. Raw data for all screening tests are included in Appendix B.

6.1.2.1. Cleaning Power Tests. In the cleaning power evaluation, test coupons (2x4x0.125 inches) were fabricated from 304 stainless steel sheet stock. The coupons were initially prepared for testing by grit blasting using 220 grit, followed by cleaning using TCA and hot soapy water. The coupons were then rinsed in deionized water and oven dried. Drilube 822™, Mil-H-6083 hydraulic oil, Mobil EP2™ grease and Dykem™ machining dye were applied to the test coupons (Figure 10). Some of the contaminated coupons were cleaned immediately, and some of the contaminated coupons were aged by baking at 100°F for seven days prior to cleaning. Aged contaminants were more difficult to remove by the cleaning process. The aging process was representative of the status of actual hardware processed at EPL, and proved to be a useful discriminator in the cleaning power tests. Cleaning was performed in an ultrasonic bath (fifteen minutes cleaning period), and also by mechanical stirring (thirty minutes cleaning period). At the end of the selected cleaning periods, coupons were removed from the cleaning baths and examined for remaining residue. Because the Dykem machining dye was not considered a prevalent contaminant source at EPL, it was applied to coupons, but not included in the evaluation. Visual examination was augmented by blacklight inspection and a waterbreak test. The degree of contamination remaining was estimated numerically. In this assessment, indications of "slight remaining contaminant" was assessed one point. Indications of "significant remaining contaminant" was assessed three points. A summary of results for each of the ten cleaners with aged contaminants and cleaned ultrasonically is shown in Figure 11. For this figure, individual points for all inspection types (visual inspection, blacklight inspection, waterbreak) and contaminants (grease, hydraulic oil, and drilube) were totaled. A similar summary of results for the cleaners with aged contaminants and cleaned by mechanical stirring is shown in Figure 12. A photograph of test coupons removed after completing the cleaning power test by mechanical stirring is shown in Figure 13. One interesting result of these tests is that the existing ODC cleaners TCA and CFC-113 performed poorly using mechanical stirring, but were very good cleaners in an ultrasonic bath. This result is attributed to the inducement of cavitation in these high vapor pressure liquids which greatly improves the cleaning process.

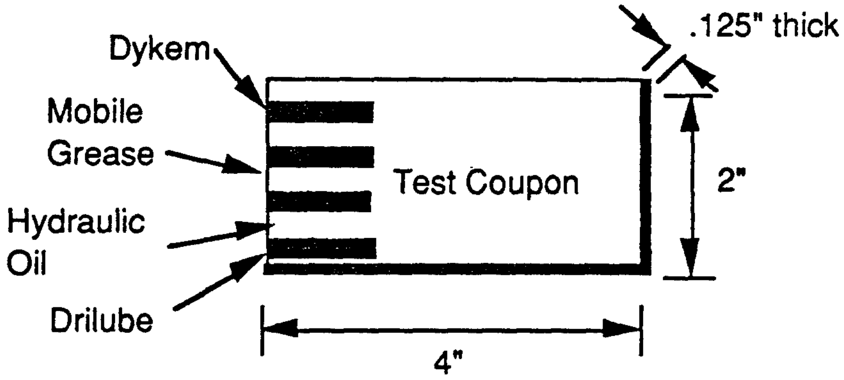


FIGURE 10 SCREENING TEST COUPON

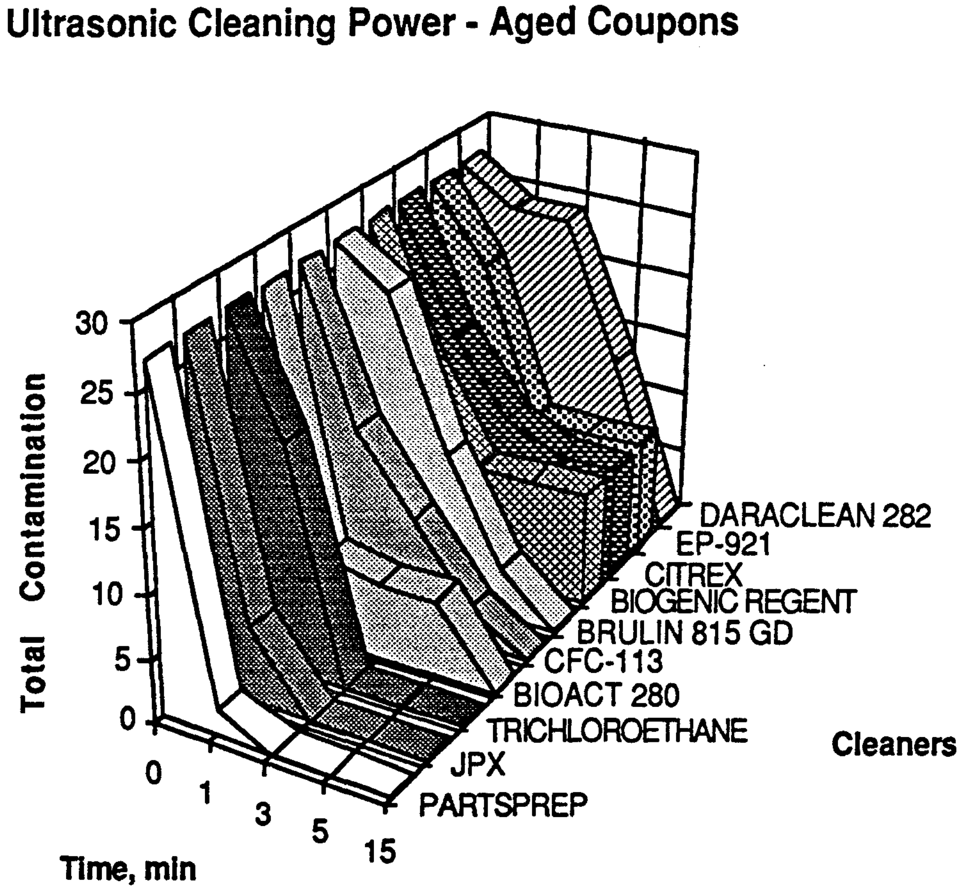


FIGURE 11 RESULTS, ULTRASONIC CLEANING POWER TEST

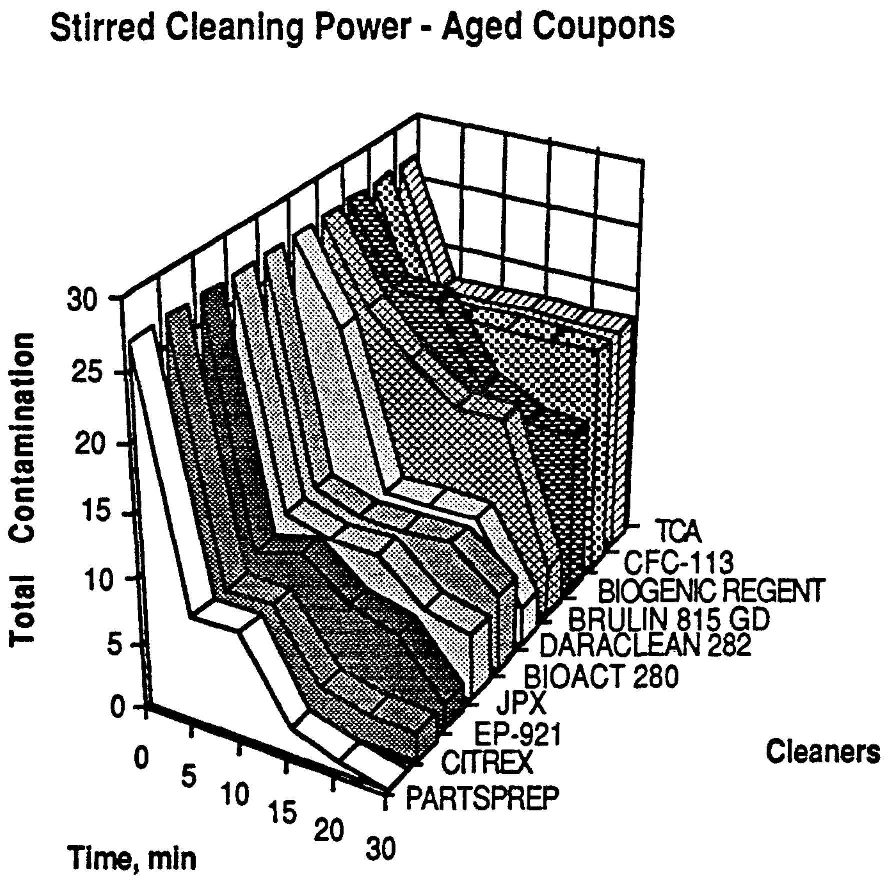


FIGURE 12 RESULTS, STIRRED CLEANING POWER TEST

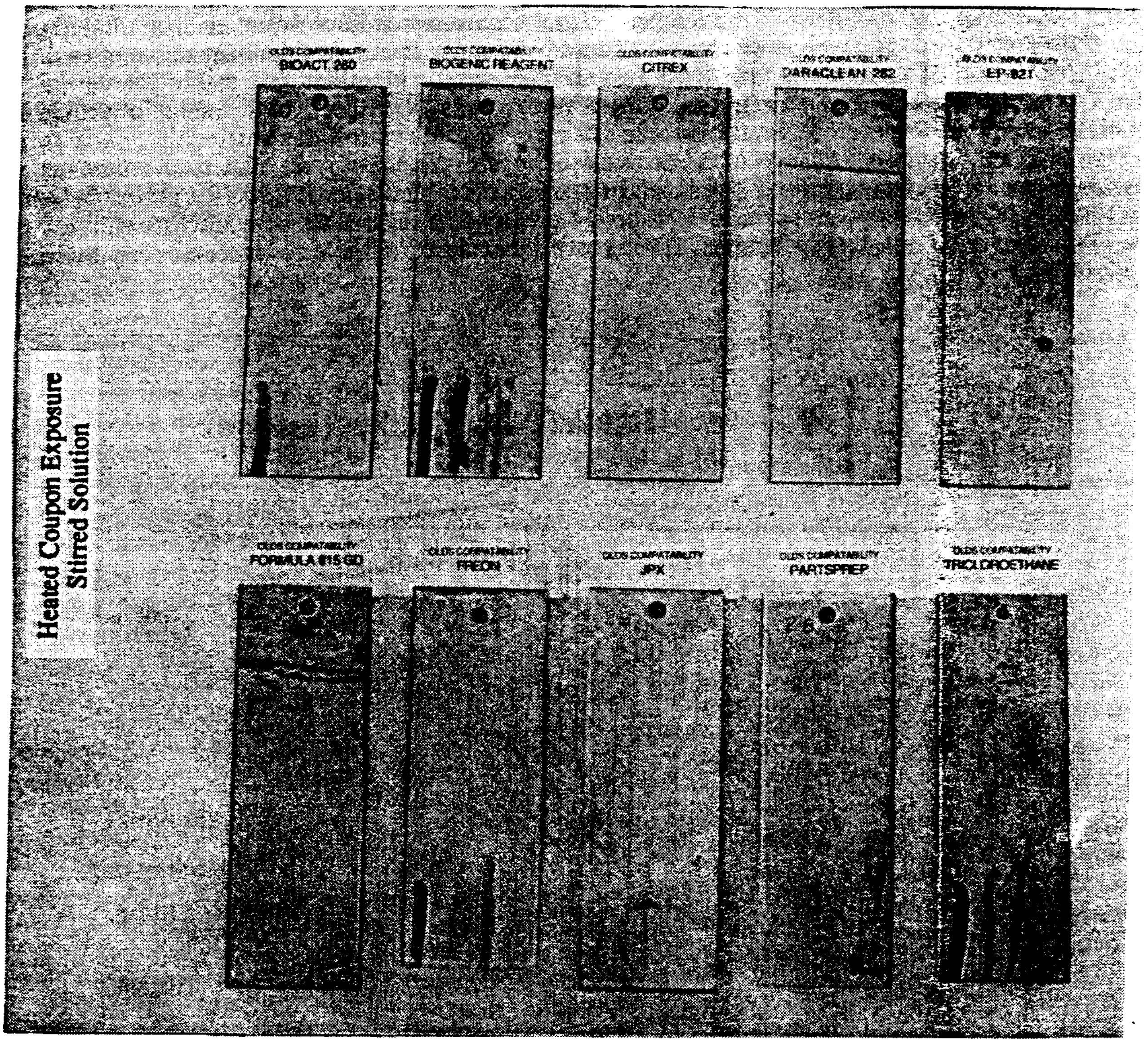


FIGURE 13 CLEANING RESULTS, 30 MINUTE STIRRED SOLUTIONS

6.1.2.2 Materials compatibility. The eight candidate cleaners as well as TCA and CFC-113 were evaluated for compatibility with materials typically processed at EPL. These included aluminum, 304 stainless steel, carbon steel, brass, Plexiglas™, titanium, copper, galvanized iron, Kel-F™, graphite/epoxy, and G-10 fiberglass. Other hardware tested included cadmium plated nuts and bolts, bronze fittings, solder samples, as well as Viton™, ethylene propylene rubber (EPR) and Buna-N rubber gaskets and O-rings. Materials were placed into glass bottles containing 250 milliliters of the cleaners. Three exposure conditions were attained: liquid phase exposure, vapor phase exposure, and exposure at the liquid-vapor interface. A visual inspection of each sample was made at the end of 24 hours, at the end of 7 days, and then weekly after that for a total of six weeks exposure. At each of the sampling periods, the coupons were rated with respect to the amount of visible corrosion or material degradation. For each observation of significant or obvious corrosion or material degradation, three points were assessed. At the completion of the six week exposure, the points were totaled and compared. A compilation of results from this test for all combined materials, combined materials less the rubber O-rings, and for metal samples only is found in Appendix B. Figure 14 presents the results for compatibility with metallic samples. Photographs of metal test specimens after six weeks exposure to the various cleaners are included in Figures 15 through 23.

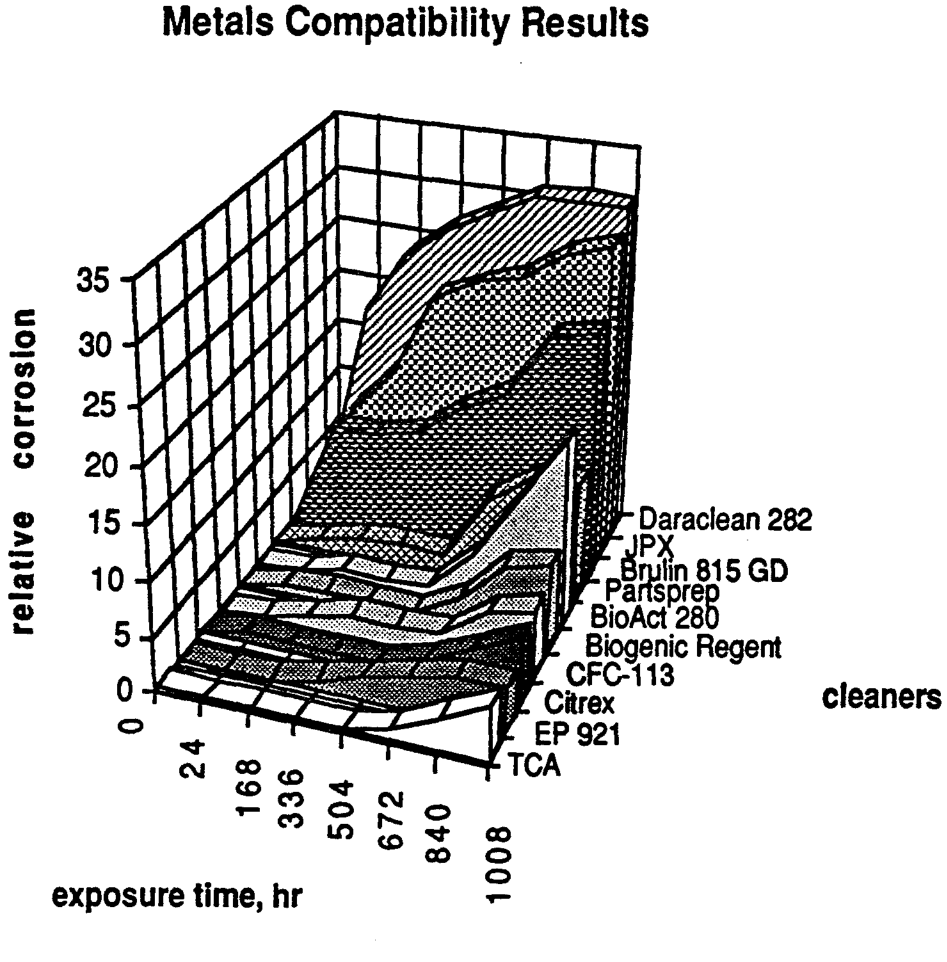


FIGURE 14 RESULTS, METALS COMPATIBILITY

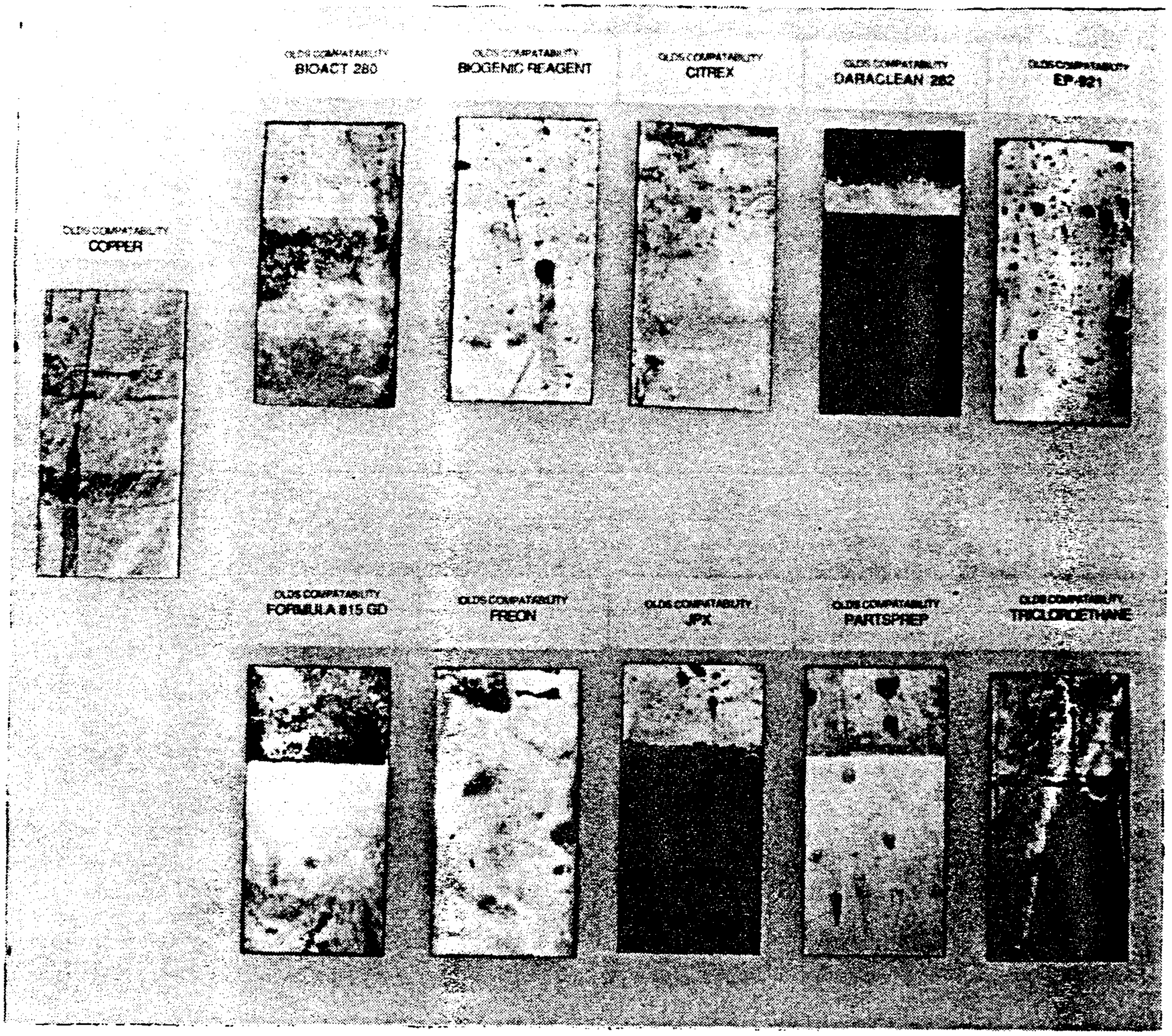


FIGURE 15 COPPER COUPONS AFTER SIX WEEKS EXPOSURE TO CLEANERS

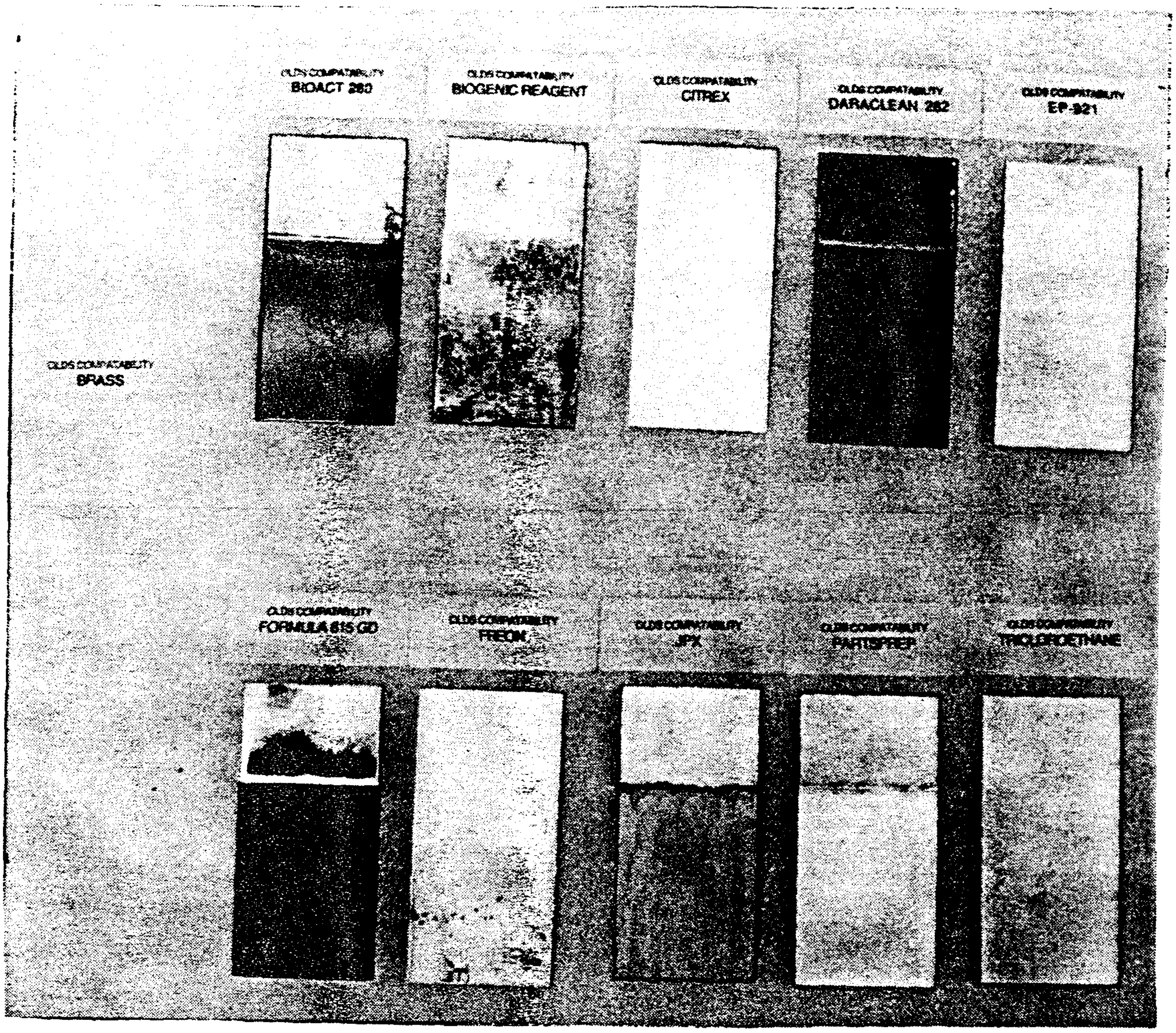


FIGURE 16 BRASS COUPONS AFTER SIX WEEKS EXPOSURE TO CLEANERS

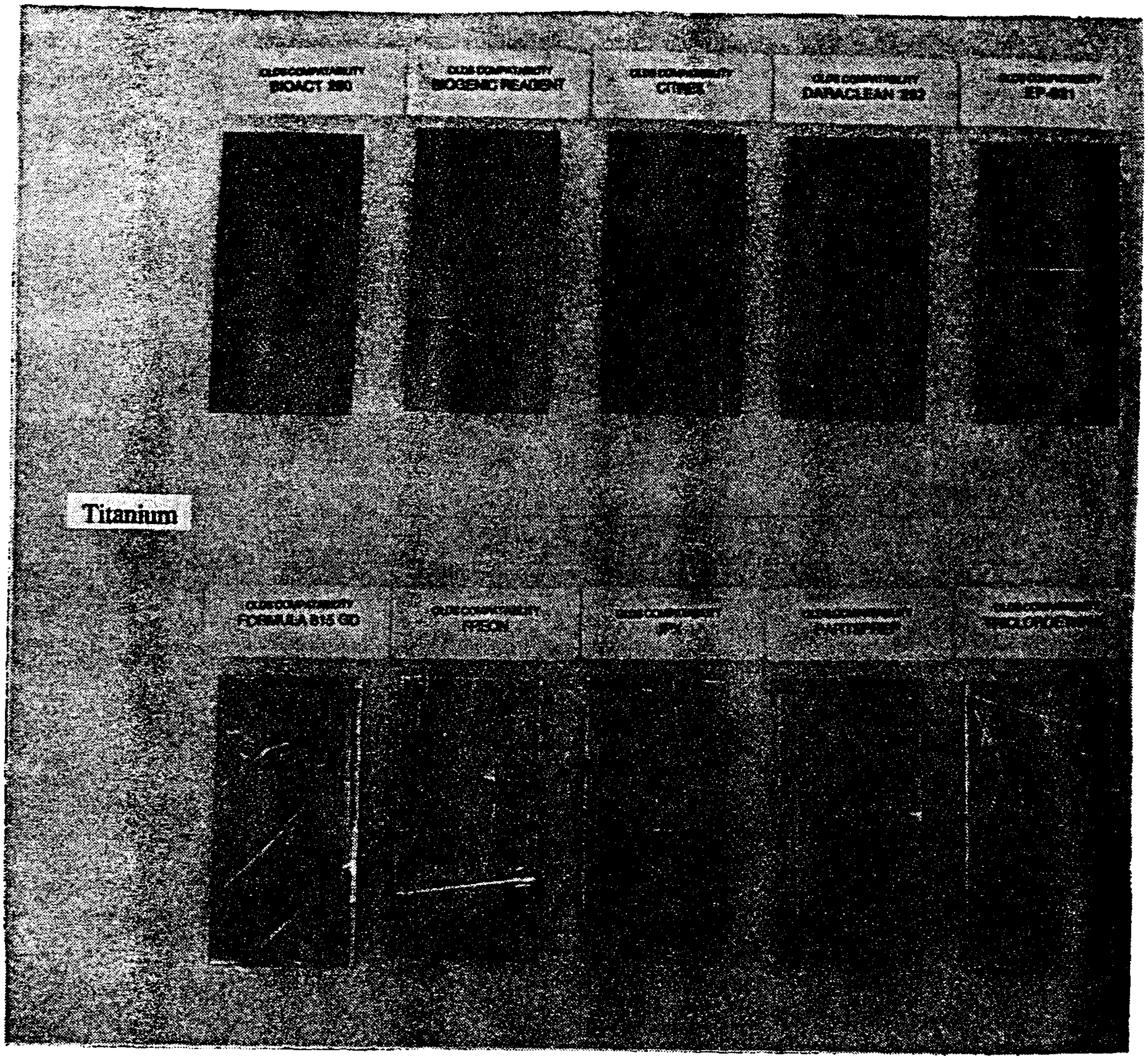


FIGURE 17 TITANIUM COUPONS AFTER SIX WEEKS EXPOSURE TO CLEANERS

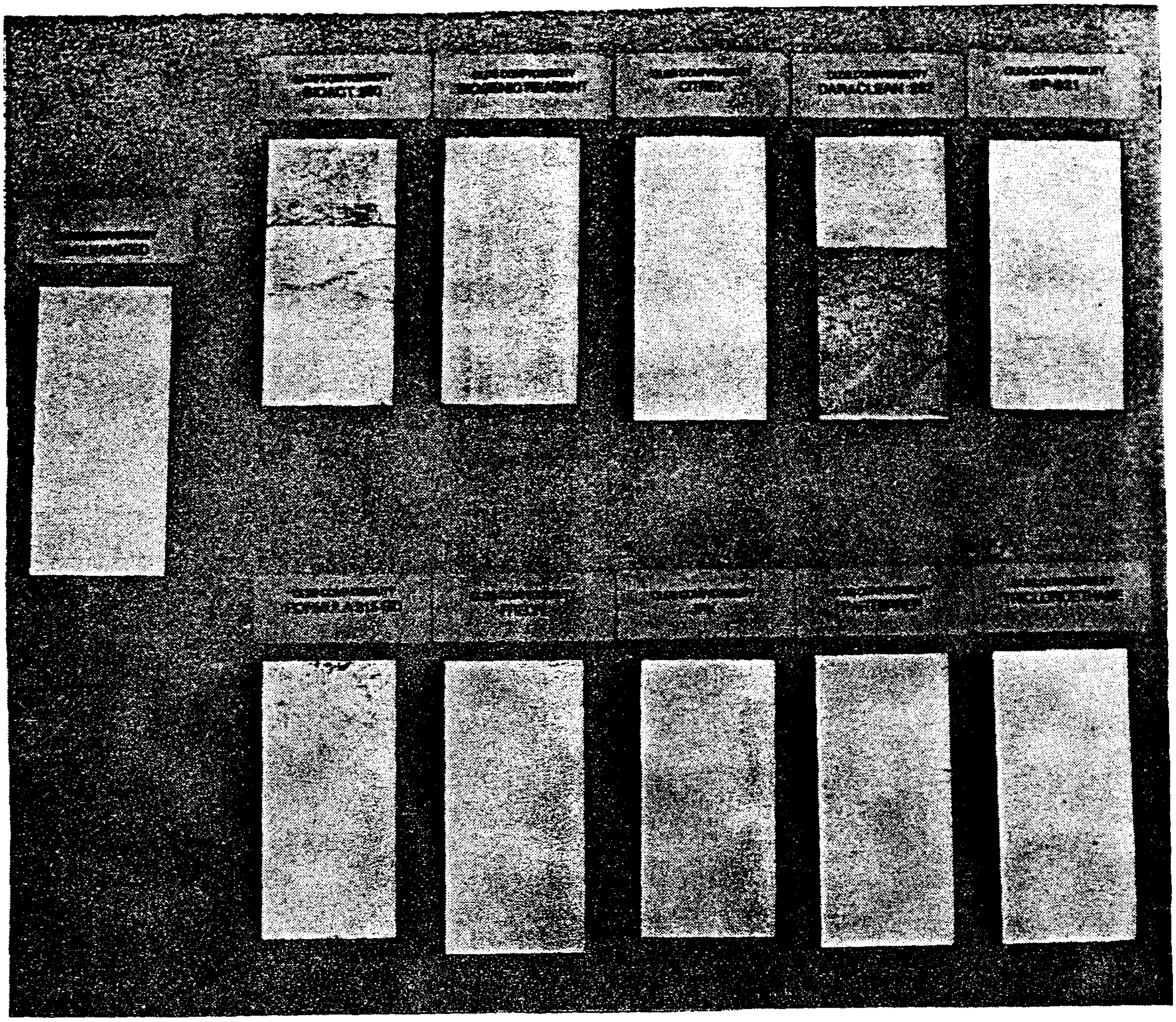


FIGURE 18 GALVANIZED IRON COUPONS AFTER SIX WEEKS EXPOSURE TO CLEANERS

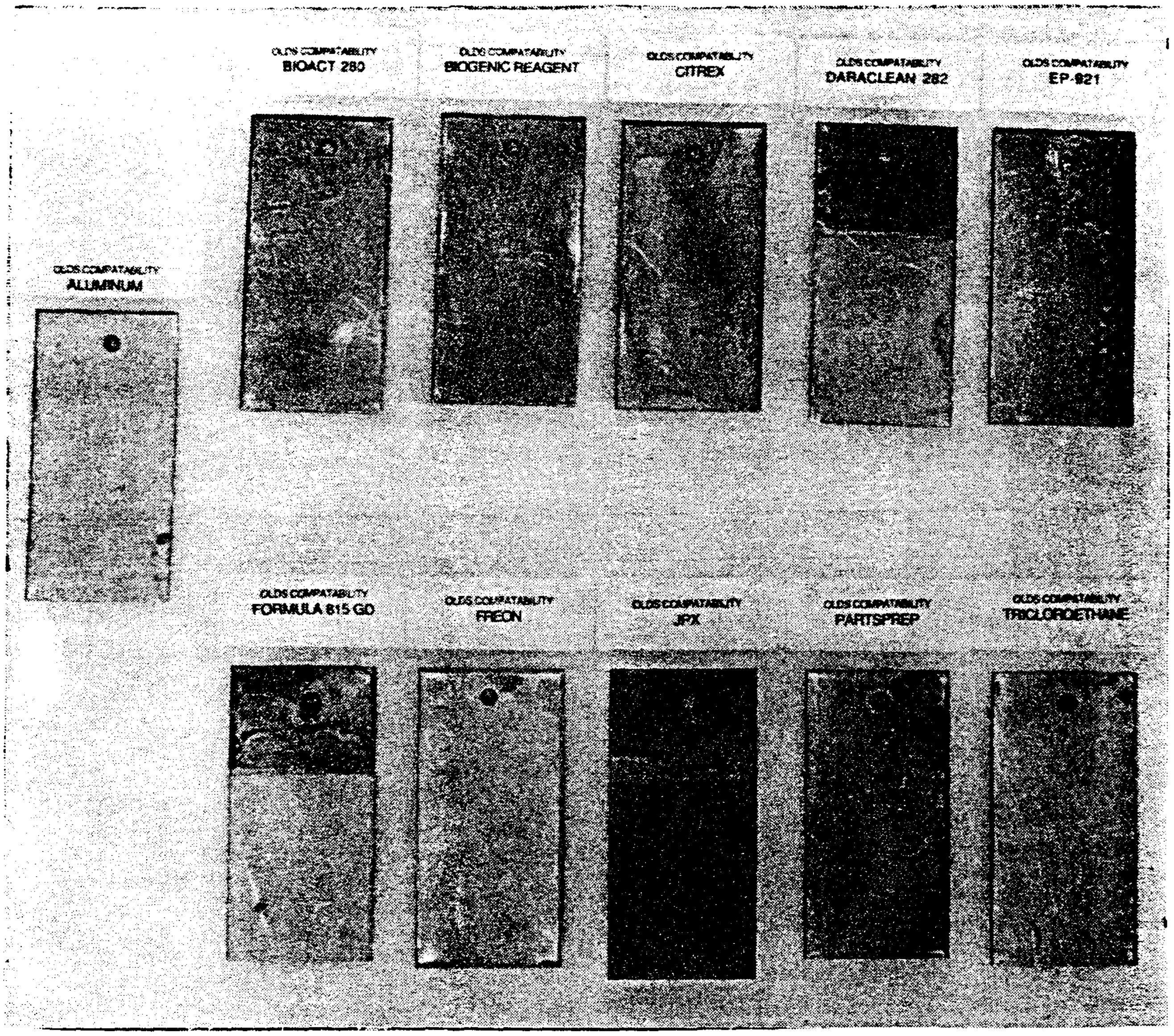


FIGURE 19 ALUMINUM COUPONS AFTER SIX WEEKS EXPOSURE TO CLEANERS

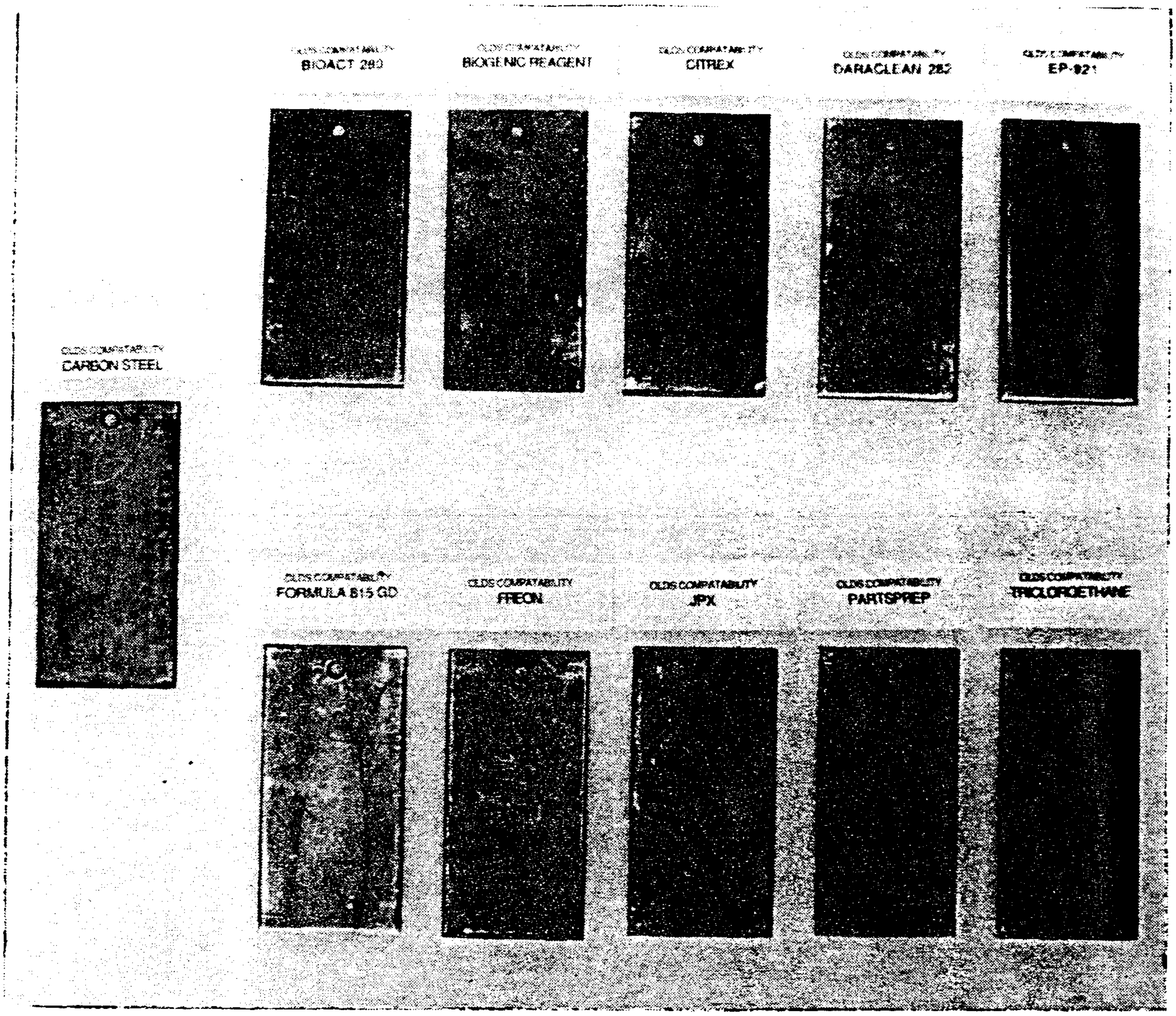


FIGURE 20 CARBON STEEL COUPONS AFTER SIX WEEKS EXPOSURE TO CLEANERS

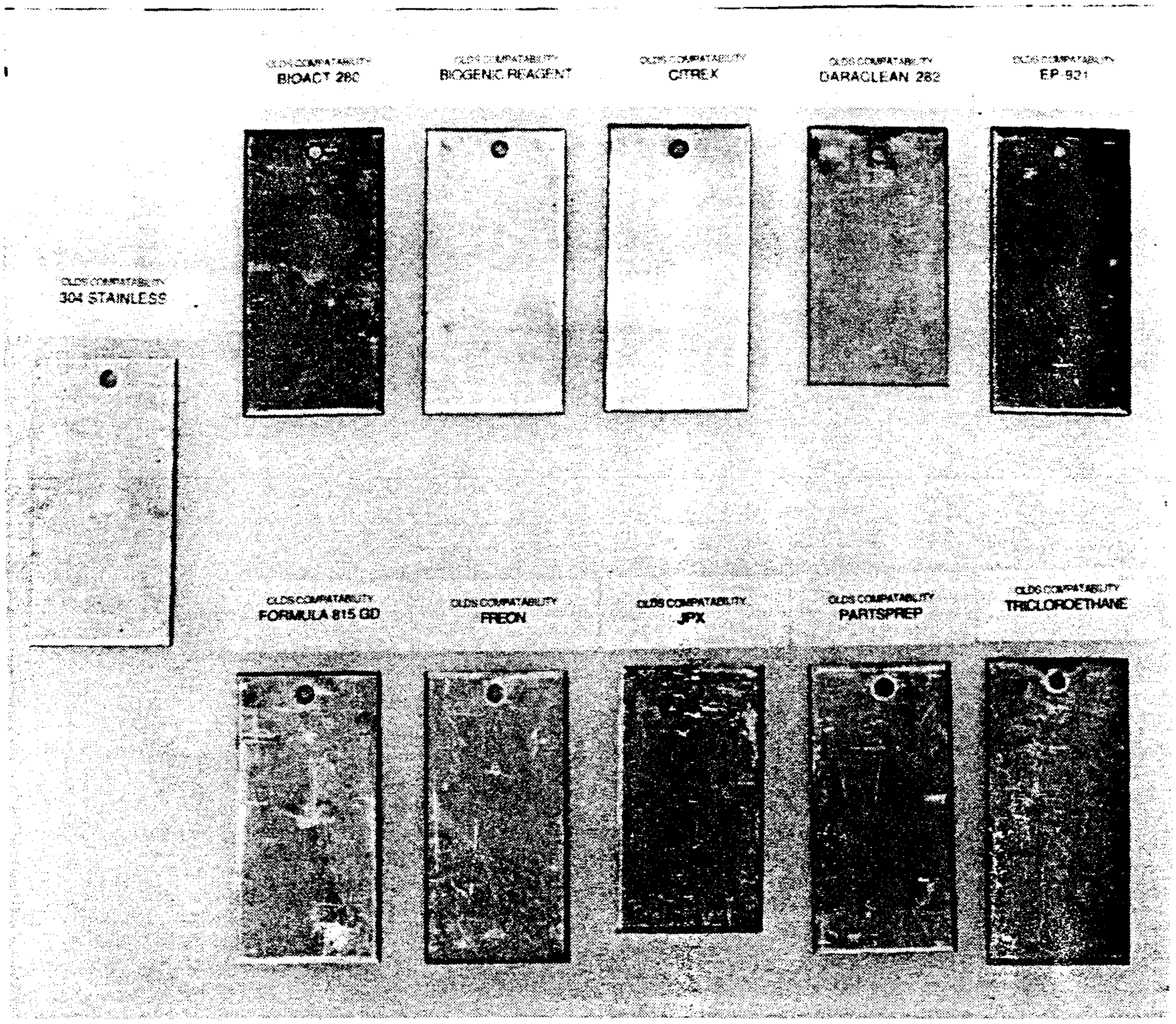


FIGURE 21 STAINLESS STEEL COUPONS AFTER SIX WEEKS EXPOSURE TO CLEANERS

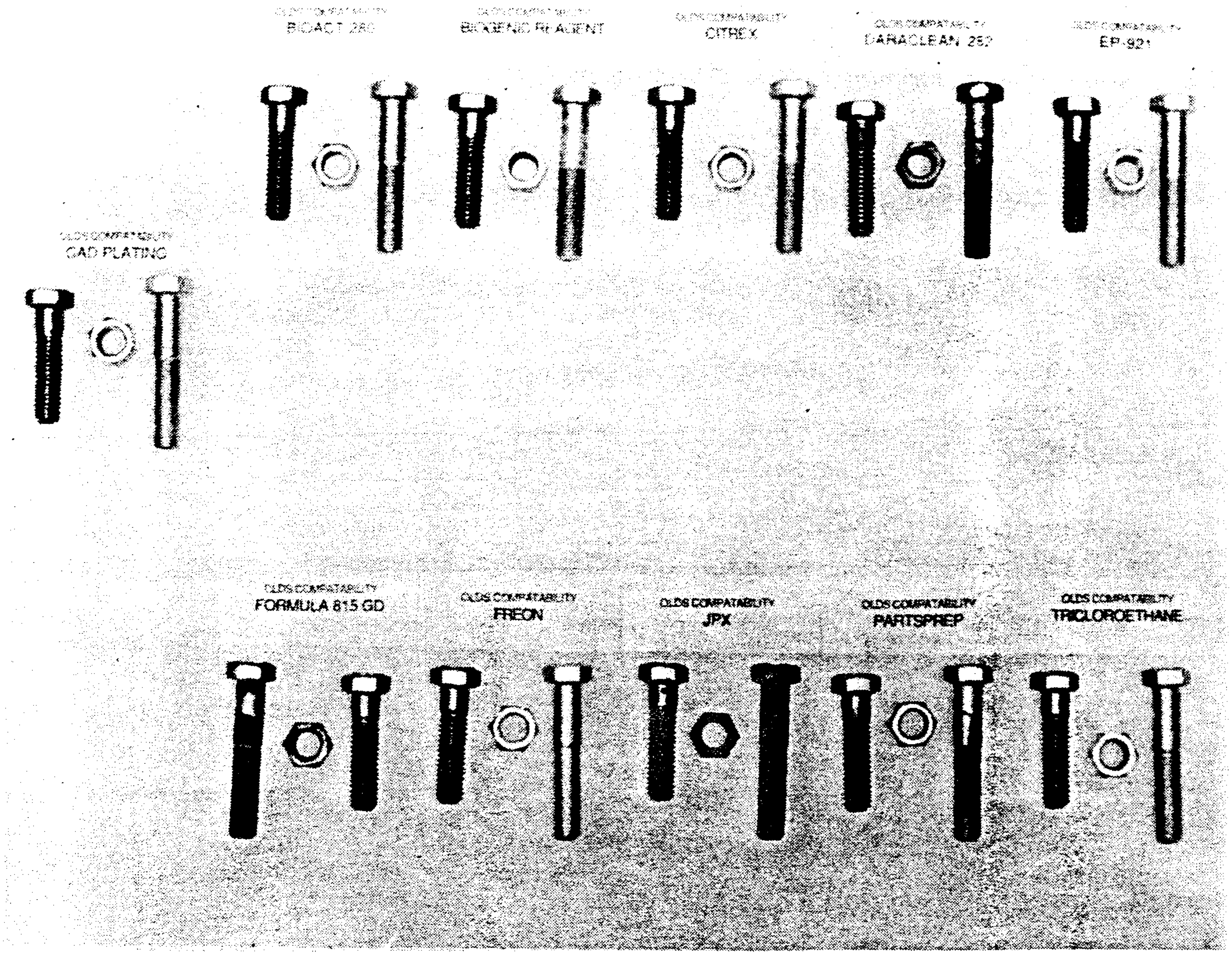


FIGURE 22 Cd-PLATED BOLTS AFTER SIX WEEKS EXPOSURE TO CLEANERS

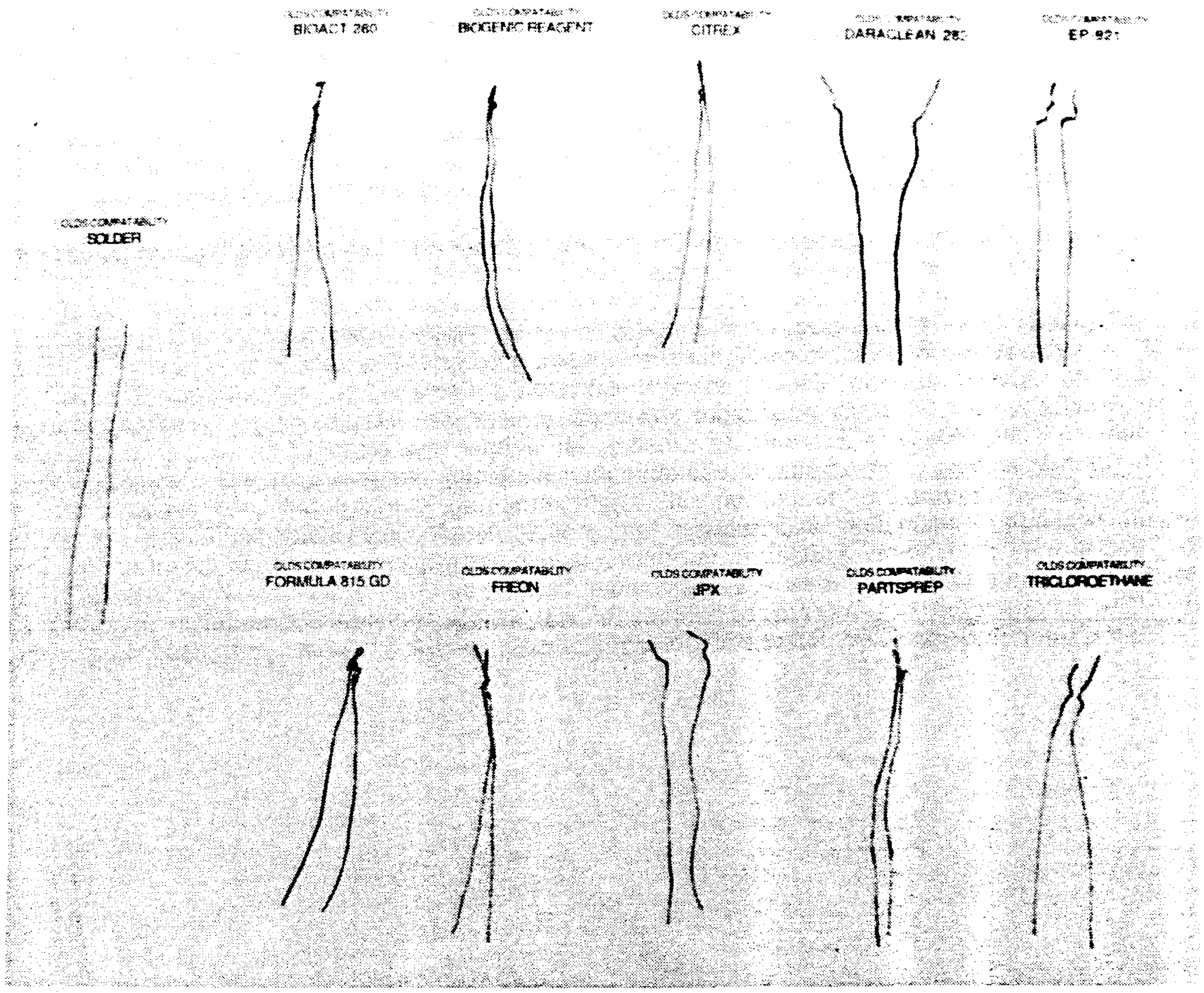


FIGURE 23 SOLDER AFTER SIX WEEKS EXPOSURE TO CLEANERS

6.1.2.3 Soils Loading. The soils loading test consisted of pre-loading the cleaners with 30 weight moto oil, and measuring the ability of the cleaner to clean metal coupons contaminated with Drilube 822 or hydraulic oi. The results of this test were used to determine the frequency of cleaner changeout after cleaning contaminated hardware.

Two sets of test coupons were prepared from 2x4 inch 304 stainless steel. One side of each coupon was grit blasted with 220 grit media in a grit blast cabinet. Grit blasting was followed by cleaning with TCA and hot soapy water in separate ultrasonic baths. Cleaning was followed by a deionized water rinse and oven drying.

One set of cleaned coupons was contaminated with 30 weight motor oil. The other set was contaminated with Drilube 822. Contamination was applied to the grit blasted side of the coupon. The contaminated coupon was introduced into a stirred bath containing 300 milliliters of the candidate cleaner. The coupon was allowed to remain in the cleaner until the contamination was visibly removed. This step demonstrated that the cleaner would remove the oil contamination. After cleaning the first coupon, a measured sample of oil (initially 15 milliliters) was added to the cleaning solution. This oil addition simulated the removal of contamination from the coupon during the cleaning process. After the oil addition was made, another contaminated coupon was immersed into the cleaning bath. At intervals of 1, 3, 5 and 15 minutes, the coupon was removed for visual, waterbreak and ultraviolet light inspections. The amount of contamination remaining on the coupon was assessed by the point system as described previously. Indications of "slight remaining contaminant" was assessed one point. "Significant remaining contaminant" was assessed three points. Points were totaled and cleaners were ranked, with the lowest number of points indicating optimum cleaner performance. For each cleaning solution, the cleaning/inspection/ oil addition sequence was repeated until the coupon was cleaned or the cleaner was no longer effective. This test procedure was observed for both the oil and Drilube contaminated coupons.. Figure 24 shows the loading test result for Bioact 280. Results of all loading tests in tabular format are presented in Appendix B.

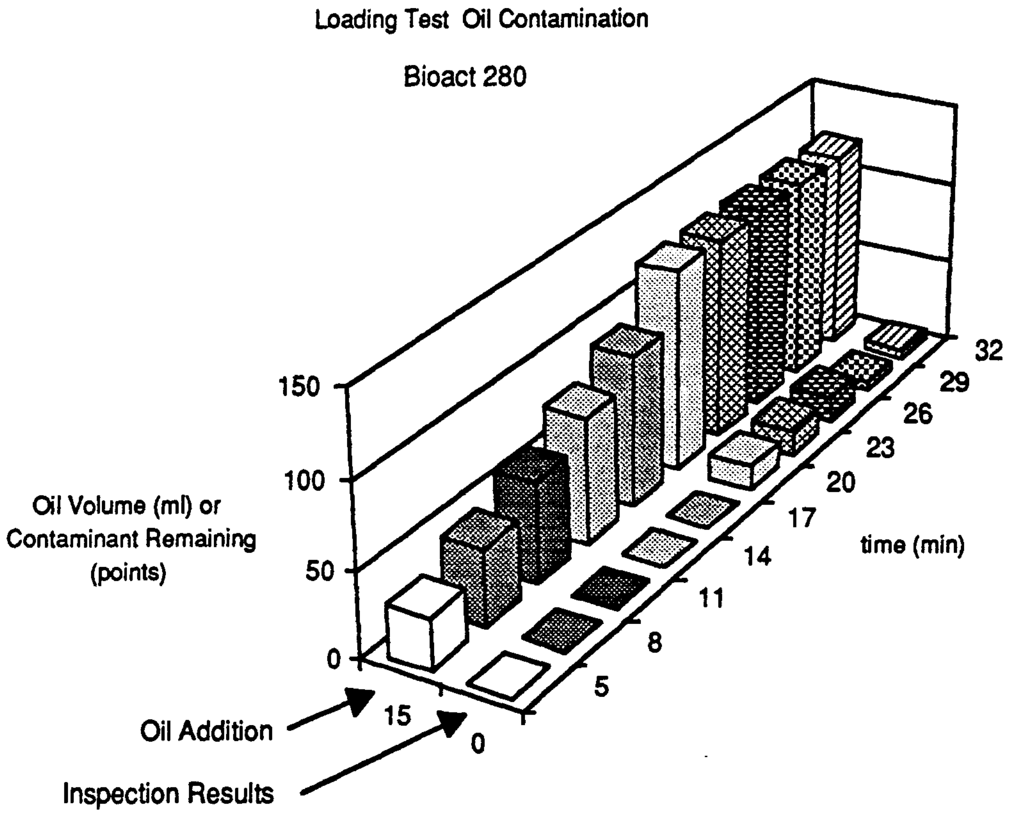


FIGURE 24 OIL LOADING TEST RESULTS, BIOACT 280

The columns in the background of the graph indicate the cumulative oil additions made to the cleaning solution. As indicated by the graph, 15 mls. of oil was added at the start of the test and an additional 15 mls. was added after five minutes. The columns in the foreground depict inspection results for contamination remaining on the coupon. The first data point depicts inspection after five minutes, and no evidence of coupon contamination was observed. After 20 minutes, over 100 mls of oil was added to the cleaner, and some residual contamination was observed on the coupon surface. This particular cleaner demonstrated an excellent cleaning ability after loading with 30 weight motor oil, and would likely require infrequent changeouts during processing of contaminated hardware.

6.1.2.4 Propellant Compatibility Test. In the propellant compatibility test, small amounts of cleaner were added to liquid rocket propellants (Aerozine-50 and nitrogen tetroxide) in a sealed glass pressure vessel. The reaction pressure and temperature were monitored using an electronic data logger. Rapid rises in temperature and/or pressure indicated a chemical incompatibility between the cleaner and the propellant. This test is discussed in more detail in Section VII. No adverse reactions were observed between the cleaners and the liquid rocket fuel, Aerozine-50. Reactions with liquid rocket oxidizer, nitrogen tetroxide, were predictable based on the chemical composition of the cleaners and are summarized in Table 15.

TABLE 15 PROPELLANT COMPATIBILITY TEST RESULTS WITH NITROGEN TETROXIDE

No Reaction	Moderate Reaction	Violent Reaction
TCA	Daraclean 282	JPX Degreaser
CFC-113	Biogenic Regent	Citrex
Bioact 280	Brulin 815 GD	EP-921
	Partsprep	

6.1.2.5 Screening Test Summary and Selection of Final Test Candidates.

A summary of screening test results for the eight candidate cleaners tested, as well as TCA and CFC-113, are presented in Table 16. Four of the eight cleaners were eliminated from further consideration based on the results of the screening test: Citrex experienced a violent reaction when mixed with nitrogen tetroxide. Daraclean 282 did not clean as efficiently as some of the other cleaners and corroded brass hardware as well as the cadmium plated bolts. JPX degreaser was incompatible with nitrogen tetroxide and non-metallic materials were degraded by this cleaner. Brulin 815 GD showed some indications of metals incompatibility after the six week exposure period. Although the EP 921 reacted with nitrogen tetroxide, this cleaner was retained because of its good compatibility with metallic and non-metallic components. Applications involving this cleaner would be limited to hardware unexposed to liquid rocket propellants, or to hardware in which all traces of residual propellant have been removed prior to cleaning. In addition to the EP-921, demonstration and validation tests were conducted on the remaining three cleaners (Biogenic Regent, Bioact 280, and Partsprep).

TABLE 16 SCREENING TEST RESULTS

Cleaner	Industrial Hygiene †	Odor	Ultrasonic Cleaning Power *	Stirred Clean. Power *	Soils Loading/Drillube *	Metals Compat. *	Oxidizer Compat.	Fuel Compat.	Environmental †	Soils Loading/Oil *	Surface Tension	Non Metals Compat. *	Cost in $/gal
Bioact 280		Slight	4	5	Fail	6	Yes	Yes		1	28	3	16
Biogenic Regent		Good	7	8	1	5	Marginal	Yes		1	22	6	16
Citrex	D	Slight	8	2	2	3	No	Yes	A	4	30	8	34
Daraclean 282	E	Mild	10	6	Fail	10	Marginal	Yes	B	4	33	2	15
EP 921		Slight	9	3	Fail	2	No	Yes		5	27	4	28
Formula 815 GD		Mild	6	7	Fail	8	Marginal	Yes	B	6	32	1	13
Freon			5	9	Fail	4	Yes	Yes		7	17	5	52
JPX Degreaser	E	Strong	2	4	Fail	9	No	Yes	A	3	28	10	11
Partsprep	D	Mod.	1	1	Fail	7	Marginal	Yes		8	40	9	36
Trichloroethane			3	10	Fail	1	Yes	Yes		2	27	7	7

† A-Flash Point makes Haz Waste.
B-Level HAP
C-Chromium Content makes Haz Waste.
D-Contains NMP.
E-Contains Gycol Ethers.

* scored values:
lowest number = best performance
highest number = worst performance

6.1.3 Demonstration and Validation

The purpose of the demonstration and validation tests was to demonstrate cleaning performance on facility hardware and to validate applicable process specifications. Two inch pneumatic valves and Barksdale™ control valves were used to demonstrate cleaning of facility hardware. A photograph of such a valve scheduled for cleaning is shown in Figure 25. These valves are assembled from a variety of metallic and non-metallic components and were disassembled prior to cleaning (Figure 26). Preliminary inspection of the valves typically indicated oil residue and corrosion on stainless steel castings, minor rust and oil on the spring mechanism, Drilube lubricant on cartridge end plates and cylinders, grease and corrosion on brass threads, and silver paint, orange paint, and residual tape adhesive on the cartridge body. The parts were placed in a basket and suspended in a pump agitated bath containing the cleaner at room temperature for 45 - 60 minutes. The pump agitated bath was only used for the Bioact 280, Partsprep, and EP-921 cleaners. The materials were inspected, removed from the bath, and transferred to an ultrasonic bath for an additional 20-60 minutes until cleaning was considered complete. The parts were rinsed with deioinized water, rinsed with hot soapy water, rinsed again with deionized water, and oven dried. Figure 27 shows a valve after cleaning with Bioact 280 and after final painting. Parts processed with Bioact 280 or EP-921 were visually clean after removal from the bath, and exceeded results expected from TCA cleaning. Parts processed using Biogenic Regent and Partsprep were still contaminated with Drilube after cleaning, and results were generally poorer than those expected from TCA cleaning. The following paragraphs discuss the results of the demonstration tests.

BIOACT 280

Test Valve #1 was disassembled and a pre-cleaning inspection revealed: stainless steel castings had oily grime and corrosion. Spring had some rust and oil. Drilube was observed on cartridge end plates, actuation rod and cylinder. Rust was present on some bolts. Grease was present on beveled seats. Grease and rust were observed on snap rings. Large brass fitting had grease on threads and corrosion internally. Barksdale casting were fairly clean. Large seat showed corrosion and grease. Paint, silver and orange, and residual tape adhesive were present on cartridge body.

The small parts were placed in a basket and the basket was placed in the pump agitated bath. Initial solution temperature was 68 °F. The basket was removed at 5 minutes intervals for visual inspection of the parts. At the end of 60 minutes, Drilube was still visually apparent, though at approximately 10 % of the initial amount. The cleaning solution temperature had increased to 108 °F. Heating of the bath occurred due to gear pump friction. At that point, the cleaner was drained from the agitated bath and paced in a stainless steel bucket, which was placed in an ultrasonic bath. The basket, containing the small parts, was then immersed into the cleaning solution and the ultrasonic bath was turned on. After 10 minutes significant debris was observed floating on the surface of the solution. After 10 additional minutes, the basket was removed from the cleaning solution and visually inspected. All evidence of Drilube contamination on the parts had been removed with a single exception of an extremely heavy deposit of Drilube. Such a deposit would normally have been wiped off prior to starting the cleaning process. Old tape adhesive, magic marker and orange paint was not removed, but approximately 50% of the silver paint used for cosmetic purposes, was removed. The remainder of the valve parts were subsequently cleaned in the same cleaning solution in the ultrasonic bath with similar results, except no other heavy Drilube deposits were noted. All parts were then processed through a normal cleaning sequence of water rinse, acid bath and water rinse(stainless steel parts only), ultrasonic soap and hot water and final oven drying. All parts were visually clean and generally exceeded results expected from trichloroethane cleaning.

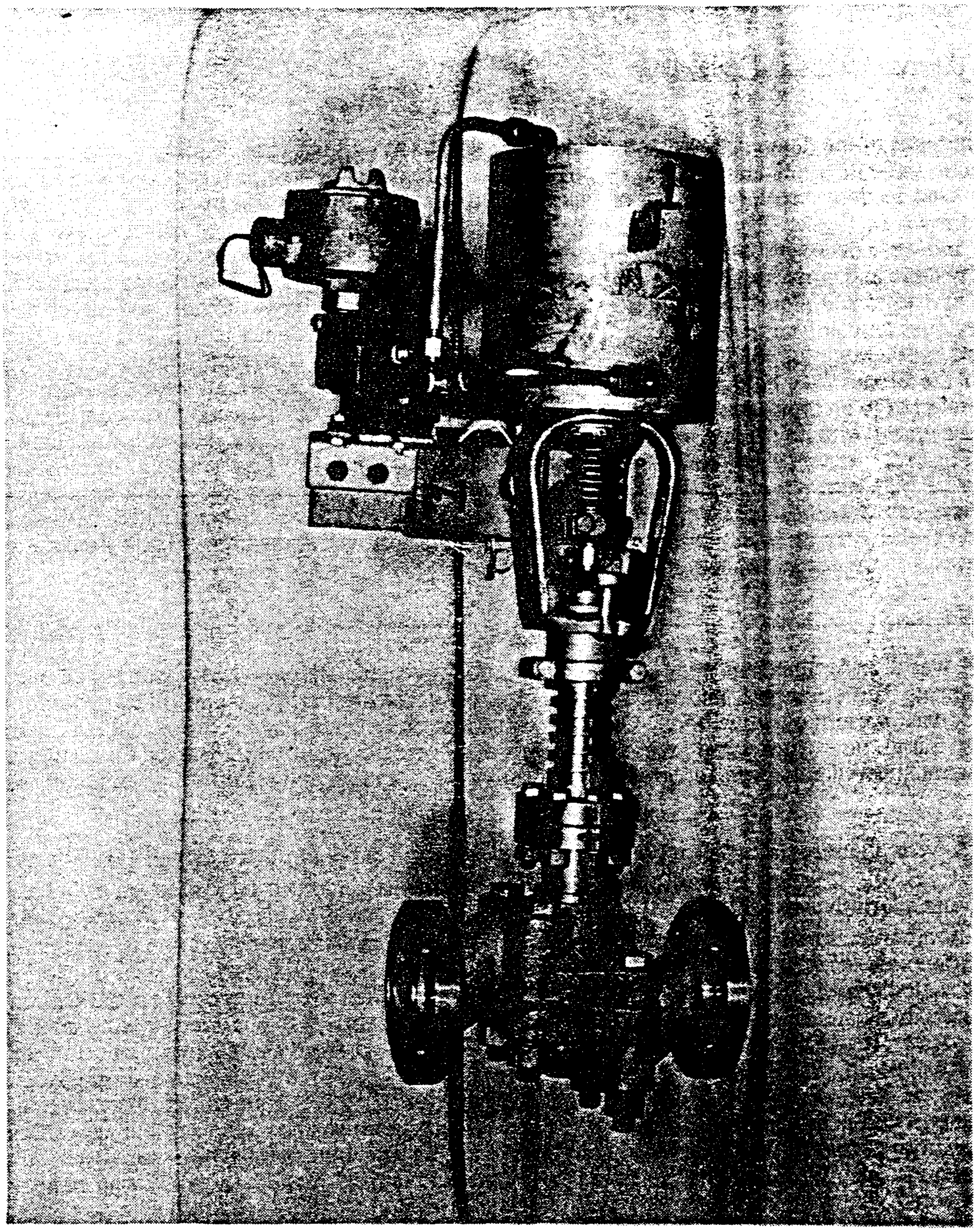


FIGURE 25 VALVE ASSEMBLY PRIOR TO CLEANING

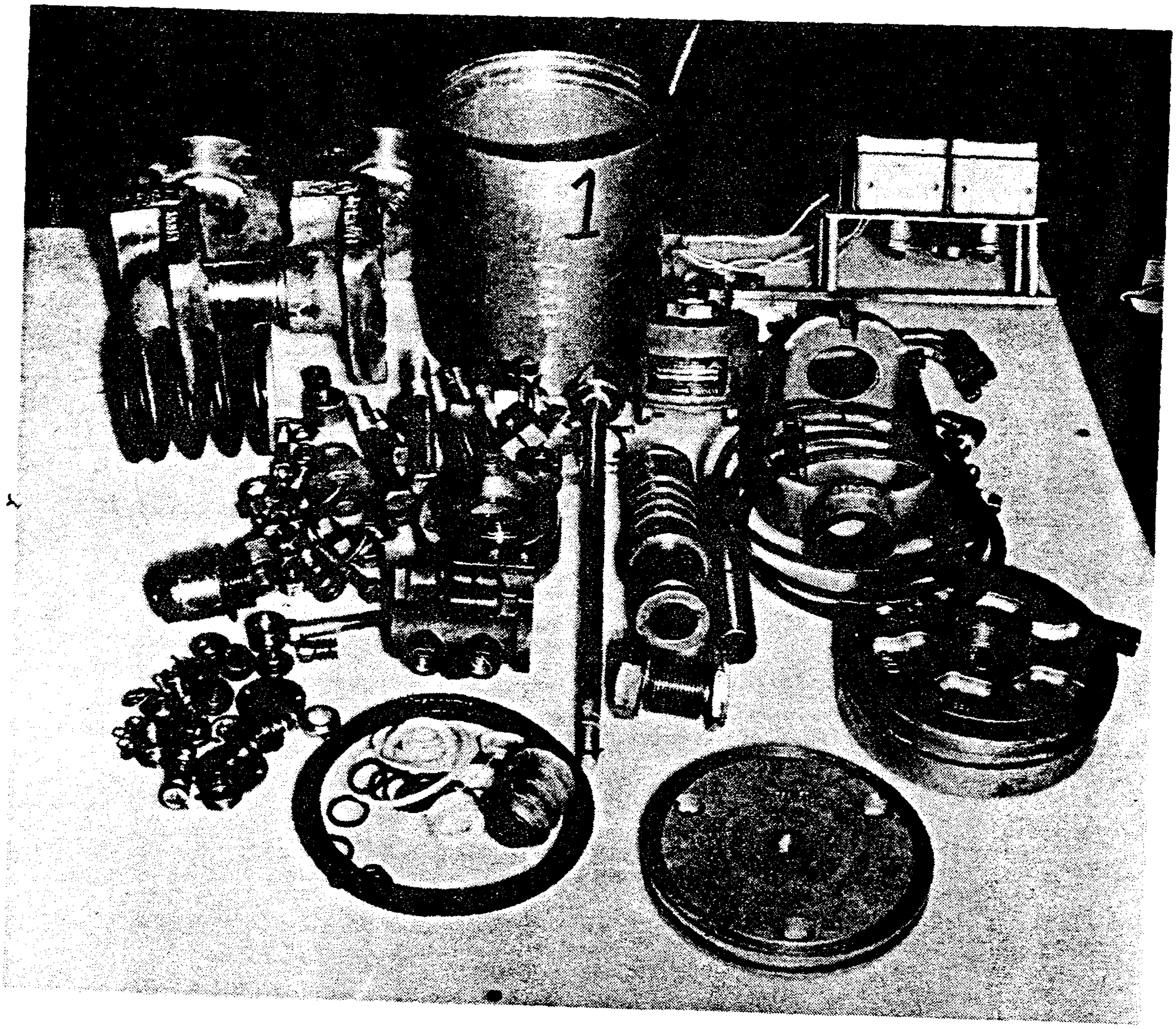


FIGURE 26 COMPONENT PARTS UPON DISASSEMBLY OF VALVE

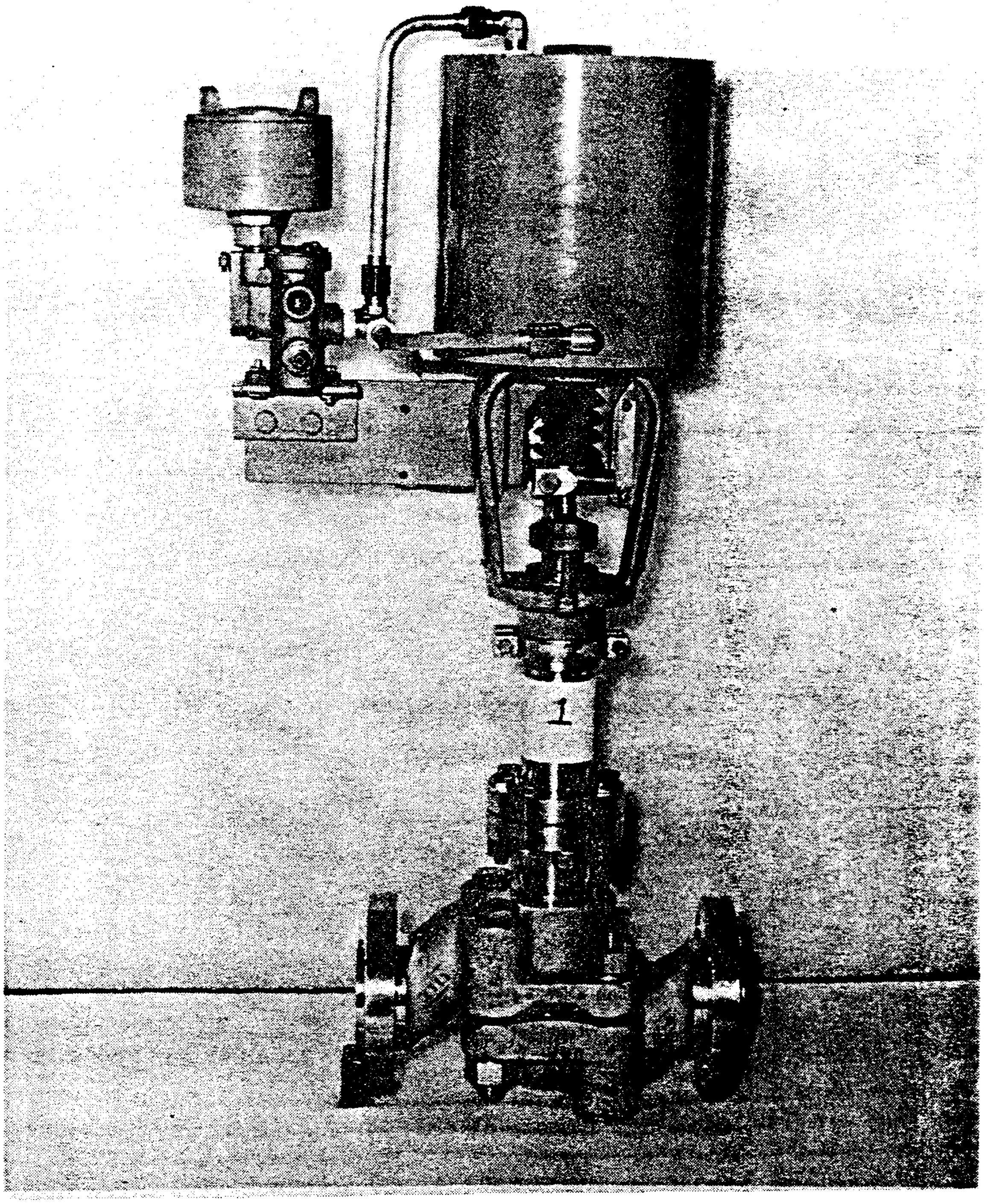


FIGURE 27 VALVE AFTER CLEANING AND PAINTING

BIOGENIC REAGENT

Test Valve #4 was disassembled and a pre-cleaning inspection revealed: stainless steel castings had oily grime and corrosion. Spring had minor rust and oil. Drilube was observed on cartridge end plates and cylinder. Grease was present on actuation rod. Bolts were relatively clean. Grease was observed on beveled seats. Heavy grease and rust were present on snap rings. Large brass fitting had slight grease on threads and corrosion. Barksdale casting had old grease residue and some corrosion. Large seat showed corrosion and grease. Paint, silver and orange, and residual tape adhesive were present on cartridge body with metal chips and internal grease.

The pump agitated bath was not used for this test. Initial solution temperature was 72 °F. A basket containing the small parts was paced in a stainless steel bucket. The bucket contained the cleaning solution and was placed in the ultrasonic bath. After 20 minutes of ultrasonic cleaning, the basket was removed from the cleaning solution and visually inspected. Those parts with Drilube contamination remained contaminated with clumps of Drilube. Some of the silver paint was softened but not removed without mechanical cleaning. At intervals of 10 minutes to a total of 50 minutes, inspections indicated the cleaner does not remove the Drilube from the parts surfaces. The remainder of the valve parts were subsequently cleaned in the same cleaning solution, in the ultrasonic bath, with similar results. All parts were then processed through a normal cleaning sequence of water rinse, acid bath and water rinse(stainless steel parts only), ultrasonic soap and hot water and final oven drying. All parts with original Drilube contamination were still contaminated. These results were not satisfactory and were considerably poorer than those expected from trichloroethane cleaning or any of the other three cleaners used for the demonstration and validation tests.

EP-921

Test Valve #2 was disassembled and a pre-cleaning inspection revealed: stainless steel castings had oily grime and corrosion. Spring had some rust and oil. Drilube was observed on cartridge end plates, actuation rod and cylinder. Rust was present on some bolts and activation rod. Grease was observed on beveled seats. Grease and st were present on snap rings. Large brass fitting had grease on threads and internal corrosion. Barksdale asting exhibited corrosion and some grease. Large seat showed corrosion and grease. Paint (silver, brown and orange), residual tape adhesive, and magic marker were present on the cartridge body.

The small parts were placed in a basket and placed in the pump agitated bath. Initial solution temperature was 68 °F. The basket containing the small parts was removed at 5 minutes intervals for visual inspections. At the end of 60 minutes, only caked dried Drilube was visually apparent. The cleaning solution temperature had increased to 104 °F. Heating was attributed to gear pump friction. The cleaner was drained from the agitated bath and placed in a stainless steel bucket, which was placed in an ultrasonic bath. The basket containing the small parts was immersed into the cleaning solution and the ultrasonic bath was turned on. After 10 additional minutes, the basket was removed from the cleaning solution and the parts were visually inspected. All evidence of Drilube contamination on the parts had been removed. Old tape adhesive, magic marker and orange paint was removed, and approximately 90% of the silver paint used for cosmetic purposes was removed. The remainder of the valve parts were subsequently cleaned in the same cleaning solution in the ultrasonic bath. No Drilube deposits remained. All parts were then processed through a normal cleaning sequence of water rinse, acid bath and water rinse(stainless steel parts only), ultrasonic soap and hot water and final oven drying. All parts were visually clean and exceeded results expected from trichloroethane cleaning. More of the incidental markings nominally found on this type of valve (paint, tape adhesive residue etc.) was removed than is typical of trichloroethane cleaning.

PARTSPREP

Test Valve #3 was disassembled and a pre-cleaning inspection revealed: stainless steel castings had oily grime and corrosion. Spring had rust and oil. Drilube was present on cartridge end plates and cylinder. A heavy brown residue was observed on the actuation rod. Rust was present on some bolts. Grease was observed on beveled seats. Grease and rust were present on snap rings. Large brass fitting had grease on threads and internal corrosion. Barksdale casting had rust, grease and slight corrosion. Large seat showed corrosion and grease. Paint (silver and orange) and residual tape adhesive present on cartridge body.

The small parts were placed in a basket and the basket placed in the pump agitated bath. Initial solution temperature was 72 ^{O}F. The basket containing the small parts was at 5 minutes intervals for visual inspections. At the end of 45 minutes heavy Drilube contamination was still apparent on the parts. The cleaning solution temperature had increased to 137 ^{O}F due to gear pump friction. The cleaner was drained from the agitated bath and placed in a stainless steel bucket, which was placed in an ultrasonic bath. The basket containing the small parts, was immersed into the cleaning solution and the ultrasonic bath was turned on. After 20 minutes, the basket was removed from the cleaning solution and the parts were visually inspected. Parts with Drilube contamination were coated with a thin layer of Drilube over their entire surface. A gray deposit of an unidentifiable material was noted on the 1/2 inch bolts. This deposit may have been a baked-on thread lubricant. It was not evident on the valves cleaned by the other cleaners. Almost all of the old tape adhesive, magic marker and paint was removed. This cleaner cleaned these residues better than the other cleaners tested. The remainder of the valve parts was subsequently cleaned in the same cleaning solution, in the ultrasonic bath, with similar results. All parts were then processed through a normal cleaning sequence of water rinse, acid bath and water rinse(stainless steel parts only), ultrasonic soap and hot water and final oven drying. All parts with original Drilube contamination were still contaminated. These results were not satisfactory and were generally poorer than those expected from trichloroethane cleaning.

Validation Test

To validate process specifications, parts were contaminated with vacuum oil, grease, Drilube and Krytox™. These parts consisted of metal blocks with blind holes, stainless steel tubes with 90° bends and attached "B" nuts, and brass valve shuttles and spacers (Figure 28). The contaminated parts were aged at 100°F for seven days, and subsequently cleaned with the four candidate cleaners in an ultrasonic cleaning bath. The cleaning process consisted of ultrasonic cleaning, hot soapy water rinsing, rinsing with deionized water, and oven drying. Some mechanical brushing was used as necessary to aid in dislodging the contaminants. After cleaning, the parts were rinsed using methylene chloride, and the solvent was evaporated to determine the non volatile residue (NVR). The goal of this process was to attain parts conforming to Level 100 A of the Martin Marietta Manufacturing Process Specification MP50405 "Contamination Control Specification". This specification requires an NVR equal to or less than one milligram residue per 100 milliliters solvent. Results from this test are included in Figure 29. Biogenic Regent did not meet this standard with respect to cleaning Drilube and Krytox contaminants, and EP-921 was considered marginal.

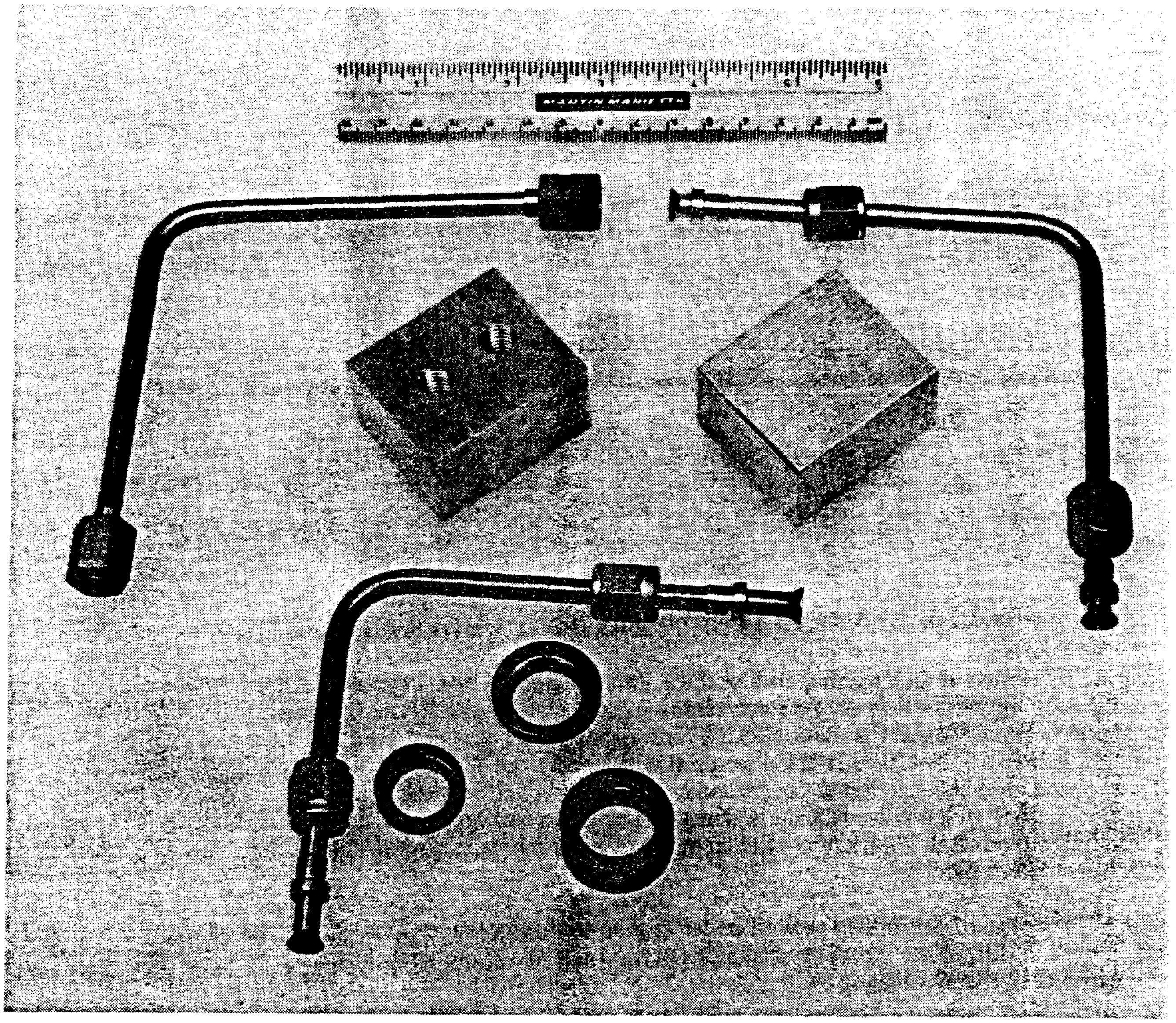


FIGURE 28 VALIDATION TEST HARDWARE

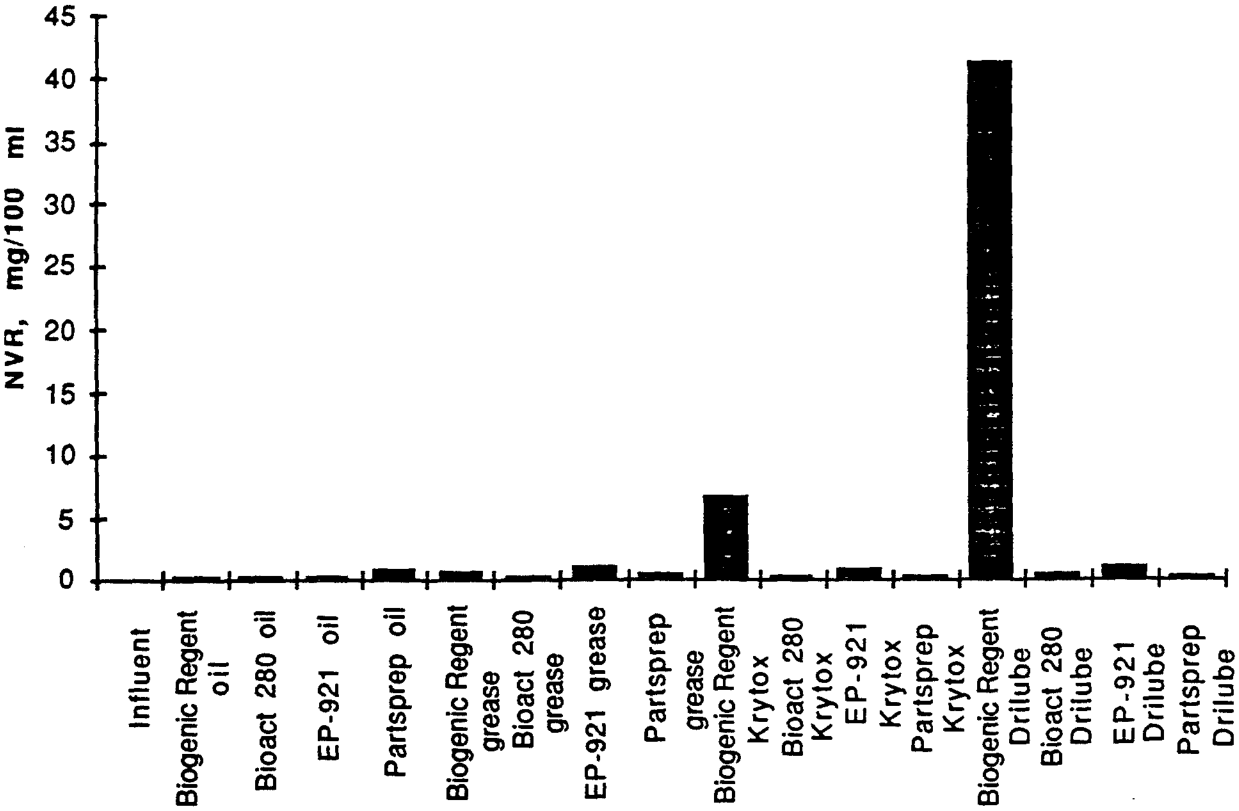


FIGURE 29 VALIDATION TEST NON VOLATILE RESIDUE

The apparent difference in the cleaning ability of the Partsprep and EP-921 cleaners is attributed to cleaning actual facility hardware (demonstration tests) versus cleaning hardware in which contamination was artificially introduced and thermally aged (validation tests). The Bioact 280 is the preferred substitute cleaner for implementation at PJKS, but EP-921 and Partsprep will be retained for specific cleaning applications. Biogenic Regent was eliminated because it had not passed either of the requisite demonstration or validation tests. A summary and ranking of these four cleaners with respect to the demonstration and validation tests is presented below. Excluded from this summary are the cleaning effectiveness on Krytox contamination. NVR results using this contaminant were inconsistent.

<u>Bioact 280:</u> This cleaner performed as well or better than trichloroethane. Heavy deposits of Drilube required mechanical wiping during the cleaning process. This additional requirement, however, was also typical of similar contaminated parts processed using TCA.

<u>Biogenic Regent:</u> This cleaner did not adequately remove Drilube contamination at levels comparable to TCA. Upon cleaning, the heavy Drilube contamination was thinned but redepositied on surfaces previously free from contamination. Biogenic regent was not effective in removing Drilube from contaminated parts. This cleaner failed the validation test.

<u>EP-921:</u> EP-921 removed all contaminants at levels consistently better than those expected from TCA. In addition, contamination not typically removed by TCA (such as paint, tape adhesive residue, etc.) were partially removed using this cleaner.

<u>Partsprep:</u> Partsprep cleaned contamination not typically removed using TCA (paint, tape adhesive residue, etc.). Moderate amounts of Drilube contamination were not removed.

6.2 Anti-seize Compounds

6.2.1 Test Description

In order to evaluate the performance of the alternative anti-seize compounds, three different tests were devised.

Bolt Force vs Torque tests were performed to evaluate the lubrication properties of the alternative anti-seize compounds. In this test, the desired result for the alternative compounds tested was a constant coefficient of friction, which would indicate the absence of metal galling. The clamping force that a bolt exerts on a pipe flange is proportional to the torque applied, and the slope of the line yields the coefficient of friction for a given diameter bolt. The coefficient of friction is a factor used to determine the torque required to achieve the desired clamping force.

Salt Fog testing was performed in accordance with ASTM B117 to evaluate the ability of the alternative materials tested to exclude moisture and prevent corrosion.

The last test performed was thermal cycling. Vendor literature for each of the candidates selected indicated a maximum service temperature range of at least 1600°F. At the PJKS Facility, service use never exceeds 1200° F, so this temperature was selected as the test temperature.

A photograph of the alternative anti-seize compounds tested and the hardware used in the evaluation is shown on Figure 30.

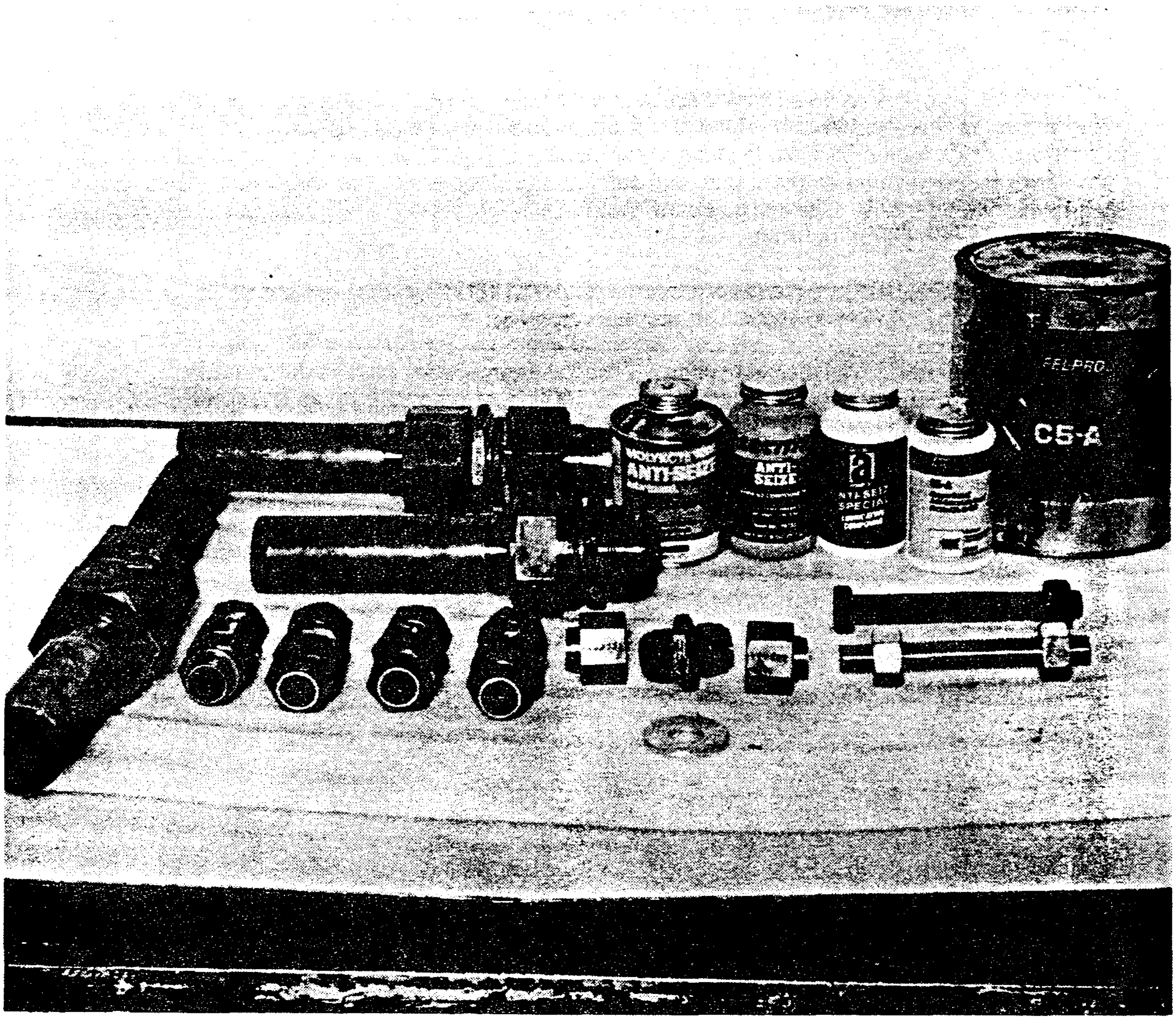


FIGURE 30 ANTI-SEIZE COMPOUNDS AND HARDWARE

Materials used to perform evaluation testing were selected based on typical fasteners and fittings used at the PJKS Facility. Two common materials used to bolt flanges together are ASTM A193 and SAE Grade 8. The ASTM A193 bolts used were a high strength stainless steel and the SAE Grade 8 studs used were an alloyed carbon steel. Most test fixtures used at PJKS include 304 stainless steel tubing with AN 37° flair fittings. The AN 37° flair fittings used in salt fog and thermal cycling were 304 stainless steel.

Bolt Force vs. Torque testing was performed using 3/4 inch SAE Grade 8 bolts and ASTM A193 studs. Salt fog testing was performed using these bolts and studs as well as 1" and 2" 37° flair AN unions, sleeves and B-nuts. Thermal cycling tests were performed with the AN fittings only. All of the test hardware used for testing was new, and had been degreased and cleaned to a visually clean (VC) level using TCA prior to starting the tests.

6.2.1 Bolt Force vs. Torque

In order to determine the load as a function of torque applied, a bolt force tester was used (Figure 31). This device is essentially a load cell that measures the tensional load as the bolt or stud is tightened. Both of the bolt and stud materials were tested using each of the candidate anti-seize materials as well as the existing Fel-Pro-C5A formulation which contains TCA. Tests were also performed for comparison purposes using mineral oil and a perfluorinated grease commonly used at PJKS.

A typical torque vs. load test was performed as follows: 1) For each test performed a new clean bolt or stud was selected. 2) The threaded area of the fastener was liberally coated with the anti-seize compound. 3) The fastener was installed into the bolt load tester and a flat washer and nut were then threaded onto the end. 4) Additional anti-seize compound was applied to the load bearing surface of the nut. 5) Torque was applied to the nut using a calibrated torque wrench until the load was 5000 lb. The torque required to achieve the 5000 lb. load was recorded on a data sheet. 6) The load was increased in 5000 lb increments, and data was recorded up to a load of 30,000 lb. 7) The nut was then loosened and without any further application of anti-seize compound the load was then reapplied in 5000 lb increments up to 30,000 lb. Again, data was recorded for each 5000 lb increment. 8) This process was repeated one more time for a total of three cycles. 9) After 3 cycles were completed, additional anti-seize compound was applied to the threaded surface and one more cycle to 30,000 lb was made in 5000 lb increments. These repeated cycles were performed to evaluate whether or not the results were repeatable without reapplication of the compound.

Results of the bolt force vs torque tests are presented in Figure 32. These results demonstrate that all anti-seize lubricants performed adequately and exhibited a constant coefficient of friction. No evidence of metal seizure was noted for any of the compounds tested.

No one anti-seize compound was superior in this test. The Loctite 767 and the Dow Molykote 1000 yielded consistent results on the SAE Grade 8 material, however results with the ASTM A193 were more scattered. The Anti-seize Tech A-S Special yielded fairly consistent results when applied to the ASTM A193 material, yet more data scatter was observed with the SAE Grade 8 material. Both the original and new Fel-Pro C5A products performed adequately, but showed some scatter on both of the bolt and stud materials. Tabulated data for these test are presented in Appendix C. Friction factors were calculated using Equation 1.

$$T = kdF \qquad \text{(Eq 1)}$$

where:

T= torque applied (ft-lb)
k= friction factor (dimensionless)
d= bolt diameter (ft)
F= bolt/stud load (lb)

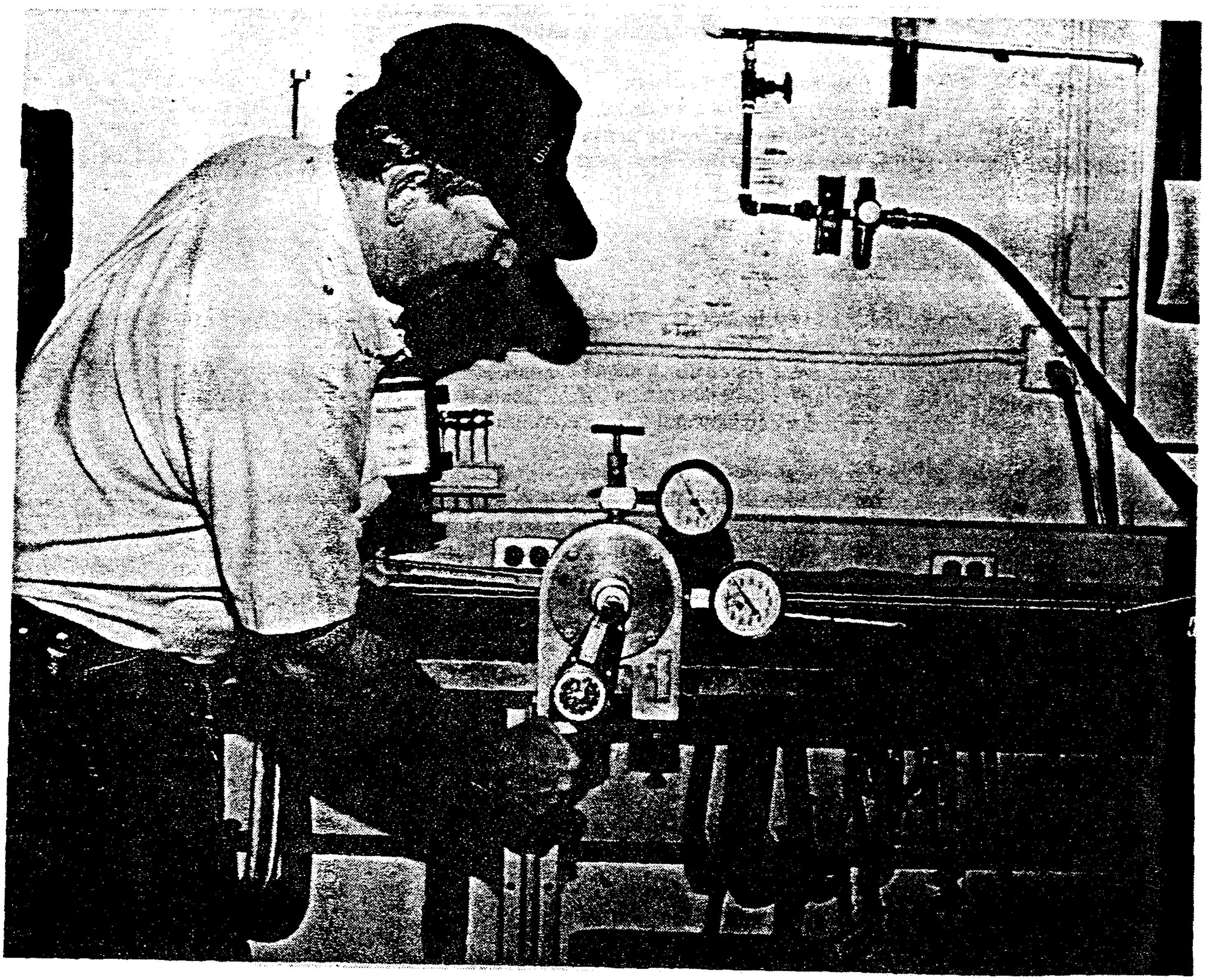
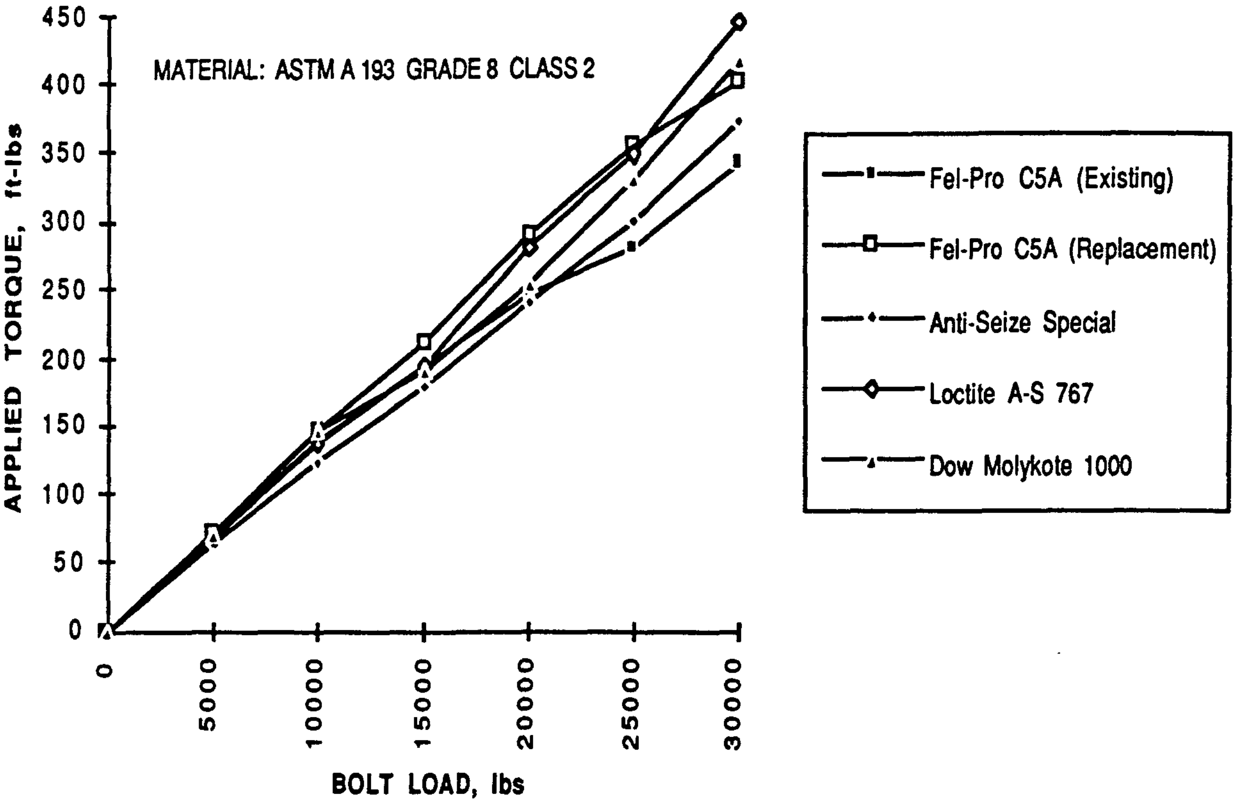


FIGURE 31 BOLT FORCE TESTER

FIGURE 32 TORQUE VS CLAMPING FORCE TEST, ANTI-SEIZE LUBRICANT



6.2.2 Salt Fog Exposure

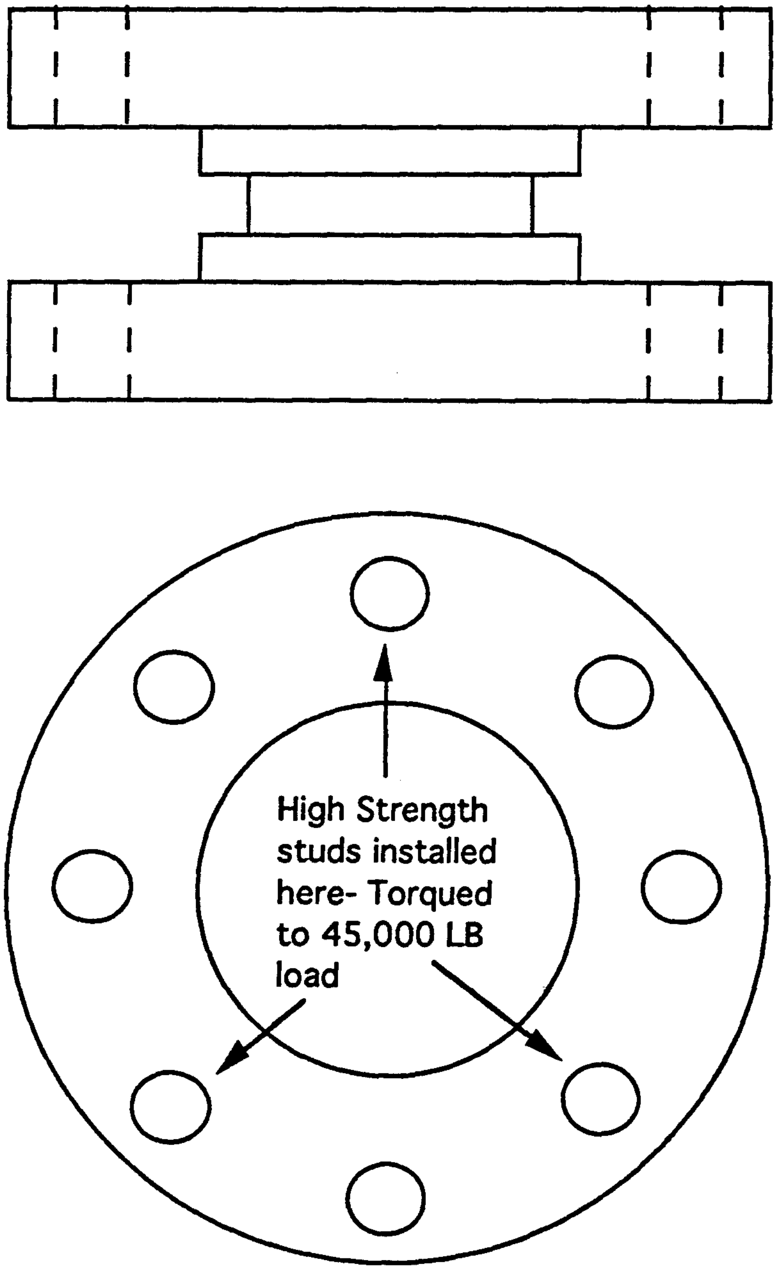
Test specimens for salt fog testing were prepared using new hardware precleaned to VC levels. Two halves of an eight hole ring seal flange were assembled (Figure 33) with three high strength studs, triangulated in three of the eight holes. These three studs were then torqued to apply a 45,00 lb load on each stud. This allowed the five remaining holes to be used for the SAE Grade 8 bolts loaded to 30,000 lb. Preloading the three high strength studs to 45,00 lb. allowed each of the SAE Grade 8 bolts to be loosened later without altering the preload on the other test bolts installed into the ring flange assembly.

Five test items (SAE Grade 8) were prepared by applying the anti-seize compound to the threads and assembling them into the ring seal flange using a flat washer and a nut. Again, additional compound was applied under the load bearing surface of the nut. Once the five bolts with the five different anti-seize compounds were installed into the flange assembly, each bolt was tightened to achieve a load of 30,000 lb.

Torque values obtained from the first run of the bolt force vs load tests were used to determine the torque required to achieve a 30,000 lb load.

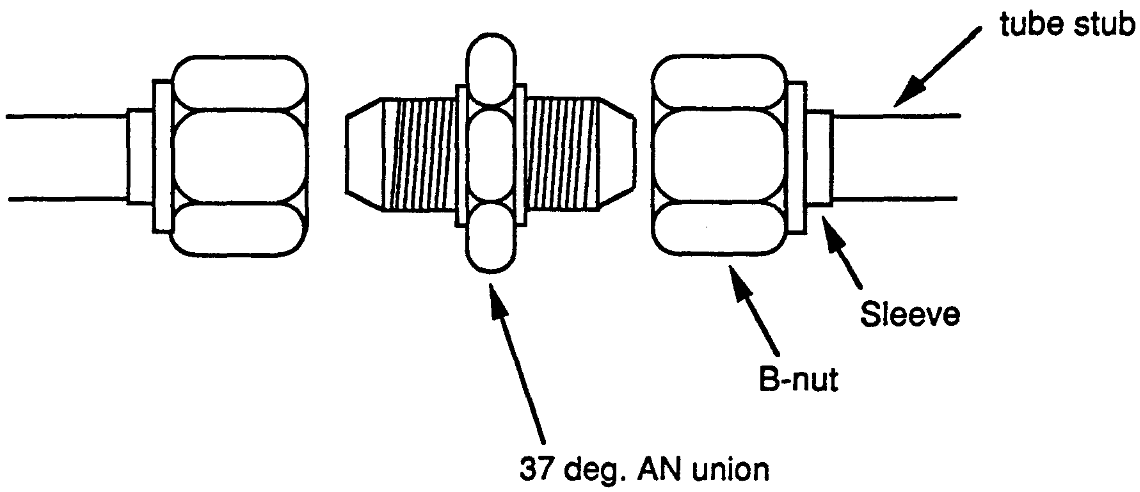
The five ASTM A193 studs were installed into a single 300 lb. flange after the anti-seize compounds were applied. Each of these studs was torqued to produce a 30,000 lb load using data from the torque vs. load tests.

FIGURE 33 RING SEAL FLANGE ASSEMBLY USED FOR SALT FOG TESTING



Additional specimens for salt fog testing were prepared using 304 stainless steel 37° flair AN fittings. Both 1" and 2" unions were prepared with short tubing stubs and sleeves with B-nuts (Figure 34). The threads were liberally coated with one of the anti-seize compounds and assembled. The B-nuts were then torqued to 100 and 400 ft-lb, respectively, for the 1" and 2" fittings.

FIGURE 34 A-N FITTING USED IN SALT FOG AND THERMAL CYCLE TESTS



Testing was performed in a salt fog chamber operated in accordance with ASTM B117. Exposure conditions were 95°F with a 5% by weight solution of sodium chloride (NaCl) in deionized water. Exposure time was 67 hours of continuous salt spray. At the conclusion of the salt fog exposure, the test hardware was removed from the chamber and allowed to dry. Each bolt/stud was evaluated on the basis of separation (breakaway) torque, free running torque, and visual inspection of thread condition.

Each of the anti-seize compounds performed well under the salt fog conditions. Inspection of the hardware after exposure revealed that most of the excess anti-seize compound had been washed away from the exposed threads. The 304 stainless steel fittings and the ASTM A193 materials survived the exposure well with very little corrosion. There were some small areas of surface rust, probably due to free iron on the surface of these materials. The SAE Grade 8 material did not fare as well. Corrosion was present on all of the surfaces, with an increased amount on surfaces that were not covered with the anti-seize compound. In all cases, irregardless of the material or the anti-seize compound used, all of the hardware disassembled easily. Separation torque for all hardware tested was less than or equal to the initial applied torque and the free running torques were negligible. Examination of the threads revealed no damage. A summary of the applied and separation torques for each material and anti-seize compound tested are included in Appendix C.

6.2.3 Thermal Cycle Tests

Test specimens for thermal cycle tests were prepared using 304 stainless steel 37° flair AN fittings. Both 1" and 2" unions were prepared with short tubing stubs and sleeves with B-nuts . Configuration of test hardware was identical to that prepared for the Salt Fog Test (Figure 34). The threads were liberally coated with one of the anti-seize compounds and assembled. The B-nuts were then torqued to 100 and 400 ft-lb, respectively, for the 1" and 2" fittings. The criteria for thermal cycling were as follows:

1) Three cycles to 1200° F minimum.
2) 30 minute hold time at 1200° F for the first and third cycles and 7.5 hours for the second cycle
3) Test hardware must be allowed to cool to ambient (70° F) prior to initiating the next heating cycle.
4) No temperature ramp rates were specified, however the cooling was not to be performed by immersion of the test hardware into some quenching media i.e. oil or water, but rather by allowing the test hardware to cool in the air.

Thermal cycling was performed in an electric furnace in the Quality Control Laboratory at Martin Marietta. After the conclusion of the thermal cycling, the torque required to separate the fittings was measured along with the free running torque. Visual observations of the fittings were made.

Examination of the hardware after thermal cycling revealed evidence of thermal degradation. All of the hardware had been blued by the heat, and the exposed anti-seize compound was dried out and cracked. The carrier grease had evaporated, leaving behind the various metal compounds that act as thread lubricants. Touching the exposed material caused it to flake off from the fittings.

Results from the thermal cycling tests were conclusive. Of the five anti-seize compounds tested, only the Dow Molykote 1000 protected the 1" and 2" hardware from metal seizure and allowed complete disassembly of the fittings.

When attempting to loosen the B-nuts from the 1" unions, the breakaway or separation torque could be measured in all cases; however only the Dow Molykote 1000 and the original Fel-Pro C5A prevented the fittings from galling within one turn of the B-nut. The remaining three compounds did not provide enough lubrication, and the free running torques increased rapidly until the B-nut had seized on the union. In all five cases, the breakaway torque measured was just below two times to over three times the initially applied torque.

A "crows foot" adapter was broken during an attempt to measure the separation torque of B-nuts on the 2" unions. A 24" crescent wrench and a 4 foot extension bar were subsequently used to evaluate which fittings could be separated. The fittings treated with the original Fel-Pro C5A were inseparable, even with two people pulling on the extension bar. The fittings treated with Anti-Seize Special, Loctite 767 and the new Fel-Pro C5A formulation broke free but seized within a half of a turn. The fittings treated with Dow Molykote 1000 broke free and the B-nut could easily removed from the union by hand. There was no visible damage to the threads, and no visible evidence of smeared metal which would indicate potential galling.

6.3 Electrical Cleaning

6.3.1 Contact Cleaning Two methods were used to evaluate the effectiveness of electrical contact cleaners. The first method evaluated the ability of the alternative cleaners to remove accumulations of contaminants and to restore the electrical resistance of the contacts to original specifications. The second method evaluated the ability of the cleaners and lubricants to protect the contacts from wear and abrasion during normal switching operations.

For the contact cleaner test, Neff rotary gain switches were obtained directly from the manufacturer in an "as received" condition, and also were removed from field service amplifiers scheduled for maintenance. The latter switches had accumulated at least ten years of service. Each set of switches was initially degreased with CFC-113, and baseline resistance readings were taken using a Kiethley digital volt meter model 2001. The field amplifier contacts were subsequently cleaned with the alternative cleaners, using manufacturer's recommended practices. After cleaning, the contact switches were rotated by hand for ten cycles. Contacts were degreased using CFC-113 (to remove residual cleaner and lubricants), and the resistance was remeasured and recorded. The cleaning cycle was repeated a minimum of three times until further cleaning indicated no noticeable improvement in the contact resistance.

Figure 35 shows the initial contact resistance of an as received switch. The average contact resistance for these contacts was 0.0049 Ohms, and the manufacturer's specification was 0.010 Ohms. The contact resistance of the as received switch was well within the manufacturer's specified value.

Figure 36 shows the results of the contact resistance measurements of a field service switch, before and after cleaning with the existing ODC cleaner (Kontact Restorer). The average initial resistance measurement for all switch positions was 0.2043 Ohm, about twenty times the manufacturer's specified value. After one cleaning cycle, the average resistance was improved to 0.0219 Ohms, but still exceeded the manufacturer's specification. After three cleaning cycles, the average measured electrical resistance fell to 0.0107 Ohms, slightly in excess of specification. No further improvement of this resistance was made in subsequent cleaning cycles.

Figure 37 shows the results of one of the ODC-free cleaners evaluated (HF Contact Cleaner™). The average resistance of these contacts was 0.3308 Ohms, over thirty times the manufacturer's specification. After one cleaning cycle, this average measurement dropped to 0.0101 Ohms. Further cleaning cycles dropped this value to 0.0061 Ohms. This cleaner performed significantly better than the existing Kontact Restorer. One drawback of this particular cleaner is that it did not readily evaporate, and further clean up was required.

Results for all other ODC-free contact cleaners tested are included in Figures 39 through 43 .

FIGURE 35 CONTACT RESISTANCE MEASUREMENTS, NEW SWITCH

NOTES:

1) RESISTANCE READINGS OF THE NEW WAFERS BY MANUFACTURES SPECIFICATION IS 0.010 OHMS
2) CLEANED 5 NEW WAFERS IN FREON AND TOOK RESISTANCE READINGS.
3) RESISTANCE READINGS ARE 4 WIRE MEASUREMENTS USING A KEITHLY MODEL 2001.
4) CONTACTS ARE MADE OF COPPER ALLOY AND SLIVER PLATED.

NEW WAFER RESISTANCE READINGS IN OHMS:

SWITCH POSITION	WAFER 1	WAFER 2	WAFER 3	WAFER 4	WAFER 5
1	0.0049	0.0052	0.0045	0.0052	0.0048
2	0.0052	0.0051	0.0039	0.0048	0.0037
3	0.0065	0.0049	0.0047	0.0051	0.0051
4	0.0072	0.0049	0.0053	0.0054	0.0047
5	0.0052	0.0041	0.0057	0.0062	0.0052
6	0.0053	0.0043	0.0052	0.0049	0.0057
7	0.0044	0.0059	0.0048	0.0059	0.0039
8	0.0046	0.0051	0.0045	0.0042	0.0037
9	0.0051	0.0049	0.0037	0.0061	0.0048
10	0.0039	0.0049	0.0029	0.0055	0.0045
11	0.0045	0.0048	0.0022	0.0051	0.0041
Average	0.0052	0.0049	0.0043	0.0053	0.0046

NOTE: THE ABOVE RESISTANCE READINGS WERE STABLE AND REPEATABILITY WAS +/- .006
THE AVERAGE RESISTANCE READING OF ALL CONTACTS IS .0049 OHMS.
THIS FIGURE WILL BE BASELINE.

FIGURE 36 RESULTS, CONTACT CLEANING, OLD SWITCH, KONTACT RESTORER

SWITCH POSITION	*KONTACT RESTORER WAFER NO. 1*				
	INITIAL	NO. 1	NO. 2	NO. 3	NO.4
1	0.0961	0.0149	0.0142	0.0101	0.0085
2	0.0751	0.0181	0.0107	0.0107	0.0078
3	0.0462	0.0206	0.0188	0.0134	0.0087
4	1.329	0.0151	0.0172	0.0141	0.0151
5	0.0621	0.0124	0.0112	0.0042	0.0089
6	0.0372	0.1101	0.0075	0.0061	0.0097
7	0.1135	0.0079	0.0207	0.0083	0.0104
8	0.0367	0.0087	0.0103	0.0077	0.0092
9	0.137	0.0101	0.0086	0.0081	0.0102
10	0.0981	0.0068	0.0126	0.0053	0.0106
11	0.216	0.0163	0.0133	0.0294	0.0173
AVERAGE	0.2043	0.0219	0.0132	0.0107	0.0106

FIGURE 37 RESULTS, CONTACT CLEANING, OLD SWITCH, HF CONTACT CLEANER

SWITCH POSITION	*CRC CONTACT CLEANER HF WAFER NO. 1*				
	INITIAL	NO. 1	NO. 2	NO. 3	NO. 4
1	0.0103	0.0086	0.0069	0.0068	0.0068
2	0.7251	0.0125	0.0088	0.0072	0.0064
3	0.3042	0.0193	0.0079	0.0056	0.0057
4	0.1031	0.008	0.0075	0.0067	0.0053
5	0.1961	0.0098	0.0082	0.0062	0.0056
6	0.2959	0.0098	0.0041	0.0063	0.0056
7	N/A	N/A	N/A	N/A	N/A
8	0.0212	0.0122	0.0092	0.0065	0.0069
9	0.0162	0.0089	0.0441	0.0064	0.0062
10	1.592	0.0043	0.0056	0.0079	0.0069
11	0.0435	0.0076	0.0042	0.0071	0.0058
AVERAGE	0.3308	0.0101	0.0107	0.0067	0.0061

FIGURE 38 CONTACT CLEANING, OLD SWITCH, MS-938/CO2

MILLER STEPHENSON CONTACT RENU MS 938/CO2

WAFER NO. 1

SWITCH POSITION	INITIAL	NO. 1	NO. 2	NO. 3	NO. 4
1	0.0395	0.0031	0.0043	0.0051	0.0051
2	0.0475	0.0072	0.0043	0.0039	0.0039
3	0.0695	0.0041	0.0047	0.0047	0.0046
4	0.1559	0.0069	0.0048	0.0037	0.0039
5	0.0311	0.0109	0.0043	0.0044	0.0045
6	N/A	N/A	N/A	N/A	N/A
7	0.0855	0.0132	0.007	0.003	0.0036
8	0.0655	0.0074	0.0037	0.0027	0.0031
9	0.0816	0.0046	0.0052	0.0053	0.0055
10	0.1096	0.0052	0.0037	0.0045	0.0042
11	0.3651	0.0181	0.0079	0.0041	0.0045
AVERAGE	0.1051	0.0081	0.0051	0.0042	0.0043

FIGURE 39 CONTACT CLEANING, OLD SWITCH, C.S. CLEANER

ZIP CHEM. CS CLEANER

WAFER TEST

SWITCH POSITION	*WAFER NO. 1*				
	INITIAL	NO. 1	NO. 2	NO. 3	NO. 4
1	0.0042	0.0037	0.0036	0.0036	0.0037
2	0.0052	0.0041	0.0041	0.0041	0.0041
3	0.3256	0.0035	0.0038	0.0037	0.0041
4	0.0131	0.0043	0.0044	0.0044	0.0043
5	0.0071	0.0034	0.0045	0.0041	0.0044
6	0.112	0.0037	0.0039	0.0049	0.0041
7	0.0162	0.0044	0.0043	0.0044	0.0045
8	0.0093	0.0082	0.0045	0.0046	0.0048
9	0.0122	0.0052	0.0053	0.0055	0.0053
10	0.0127	0.0055	0.0052	0.0052	0.0056
11	0.0193	0.0054	0.0051	0.0054	0.0052
AVERAGE	0.0488	0.0047	0.0044	0.0045	0.0046

FIGURE 40 CONTACT CLEANING, OLD SWITCH MS-939

MILLER STEPHENSON RENU /LUBE MS 939/SAFE ZONE

WAFER TEST

SWITCH POSITION	*WAFER NO. 1*				
	INITIAL	NO. 1	NO. 2	NO. 3	NO. 4
1	0.2563	0.0143	0.0151	0.0068	0.0052
2	0.0265	0.0185	0.0139	0.0062	0.0063
3	0.0141	0.0079	0.0124	0.0087	0.0051
4	0.0847	0.0061	0.0116	0.0071	0.0061
5	0.0174	0.0065	0.0099	0.0075	0.0059
6	0.0247	0.0152	0.0082	0.0057	0.0055
7	0.0751	0.0088	0.0067	0.0069	0.0061
8	0.2814	0.0095	0.0286	0.0051	0.0051
9	0.0391	0.0911	0.0096	0.0057	0.0053
10	0.0135	0.0086	0.0075	0.0072	0.0071
11	N/A	N/A	N/A	N/A	N/A
AVERAGE	0.0833	0.0187	0.0124	0.0067	0.0058

FIGURE 41 CONTACT CLEANING, OLD SWITCH, ASP#1M

POLY CHEM ASP # 1M (ULTRASONIC BATH 3 MIN. AT TIME

WAFER TEST

SWITCH POSITION	*WAFER NO. 1*			
	INITIAL	NO. 1	NO. 2	NO. 3
1	0.0098	0.0069	0.0064	0.0054
2	0.0133	0.0078	0.0055	0.0057
3	0.0099	0.0062	0.0063	0.0066
4	0.0115	0.0059	0.0061	0.0059
5	0.0258	0.0113	0.0069	0.0072
6	1.25	0.0109	0.0059	0.0057
7	0.0375	0.0083	0.0064	0.0062
8	0.0306	0.0055	0.0056	0.0058
9	0.0107	0.0073	0.0069	0.0068
10	0.0295	0.0074	0.0071	0.0071
11	N/A	N/A	N/A	N/A
AVERAGE	0.1429	0.0078	0.0063	0.0062

FIGURE 42 CONTACT CLEANING, OLD SWITCH, CONTACT CLEANER 2000

SWITCH POSITION	*CRC CONTACT CLEANER 2000*				
	INITIAL	NO. 1	NO. 2	NO. 3	NO. 4
1	0.3154	0.0069	0.0042	0.0044	0.0045
2	0.1901	0.0081	0.0062	0.0055	0.0057
3	0.1584	0.1453	0.0098	0.0063	0.0059
4	0.0181	0.0104	0.0151	0.0054	0.0055
5	0.0619	0.0403	0.0206	0.0097	0.0064
6	0.6421	0.0659	0.0223	0.0101	0.0075
7	0.0124	0.0119	0.0085	0.0061	0.0055
8	0.0127	0.0099	0.0084	0.0065	0.0053
9	0.0159	0.0109	0.0092	0.0062	0.0058
10	0.0155	0.0093	0.0098	0.0081	0.0063
AVERAGE	0.14425	0.03189	0.01141	0.00683	0.00584

These cleaners were evaluated by instrumentation shop personnel conducting the tests. The following ratings resulted from this evaluation.

RATING: CLEANING TEST

1. Excellent Miller Stephenson MS938/CO_2 (1,1 - Dichloro -1-Fluoroethane & Methanol)

Their product evaporates quickly after application. Resistance readings became stable after three applications. The average resistance readings were comparable to the new wafer readings, though not as stable. This material comes in a spray can containing a directed application tube. Would use in instrumentation shop. This material is a hydrochlorofluorocarbon (HCFC), and has impending environmental restrictions. For this reason, it is not a viable alternative at the present time.

1. Odor - Ethereal
2. Flash point - None
3. Requires good ventilation
4. Requires gloves
5. Health (2)
6. Flammability (1)
7. Reactivity (1)

2. Excellent Zip Chem CS Cleaner (Hydrocarbon, Terpenes)

When applied to the switches, this product does not evaporate and a large amount of an oily substance remained behind. This product had stable resistance readings after two applications. These measurements were comparable to the new wafers, although not as stable. In spite of the heavy oil loading in this product, it was rated highly. This material comes in a spray can containing a directed application tube. Would use in instrumentation shop.

1. Mild citrus odor
2. Flash point 112°F
3. Good ventilation
4. Requires gloves

3. Good CRC Contact Cleaner HF (Petroleum Distillate)

This product evaporates quickly. Reading became stable in four applications. This material comes in a spray can containing a directed application tube. Would use in instrumentation shop.

1. Negligible odor
2. Good ventilation
3. Requires gloves
4. Flashpoint 142°F

4. Good Miller Stephenson MS939 Safezone Contact Cleaner ReNu and Lube

This product comes in a bottle, and is identical to the MS-938/CO2 aerosol product except for its application (liquid application vs spray application). This product leaves a slight oil film. Readings became stable in four applications. Because this product contains a HCFC, it was not considered a viable alternative at this time.

1. Negligible odor
2. Good ventilation
3. Requires gloves

5. Good CRC Contact Cleaner 2000 (1,1 - Dichloro -1- Fluoroethane)

This product leaves too much oil after application. Readings became stable in four applications. Drawbacks of this material include oil cleanup, and the need for four applications for the resistance measurements to become stable. This material comes in a spray can containing a directed application tube. Would use in instrumentation shop. Because this product contains a HCFC, it was not considered a viable alternative at the present time.

1. Odor - Ethereal
2. Good ventilation
3. Requires gloves
4. Health (2)
5. Flammability (2)
6. Reactivity (0)

6. Fair Poly Chem ASP #1 (Di-acetone alcohol blend)

This product was used in an ultrasonic bath at three minutes per application. Application of product needs to be performed in a vent hood. The resistance reading became stable after two applications.

1. Odor - extremely strong
2. Requires gloves
3. Flashpoint 143°F

7. Fair Chemtronics Kontact Restorer (contains Freon-11®). Presently used in shop.

This product evaporates very quickly. The readings became stable after four applications. Final resistance measurements after cleaning were higher than desired.

8. Fair Deox-It 100 (Mineral Oil)

This product is 100% oil. The reading became stable after four applications. This product was too messy and the results were not good enough to warrant use.

1. No special requirements
2. Flashpoint 240°C

For the contact wear test, contacts were removed from field service amplifiers and initially degreased with CFC-113. Initial resistance measurements were made using a Keithley digital volt meter model 2001. The alternative cleaners and lubricants were applied to the contacts on various wafers in the rotary switches. One wafer remained unlubricated, which served as a test control. Switches were cycled using a D.C. motor at approximately 30 RPM. At the completion of 5,000 revolutions, the multi-layer contacts were again degreased with CFC-113 to remove any residual cleaner and lubricants. Resistance measurements of the contacts were retaken. Contacts which had lubricants applied previously were then relubricated at this point. The switches were again cycled for another 5,000 cycles using the D.C. motor, lubricants removed, and resistance measurements taken. This process was repeated for a total exposure of 45,000 cycles. The electrical resistance of the contacts lubricated with the alternative cleaners/lubricants were then compared with the resistance of the unlubricated contacts. Resistance measurements of the contacts lubricated with the existing ODC cleaner-lubricant (Kontact Restorer™) were also taken for comparison. Figure 43 shows a photograph of the test device for measuring contact wear.

Figure 44 shows the results of the contact wear test. After 45,000 cycles, the unlubricated contact demonstrated excessive metallic wear, and the contact resistance increased from 0.0161 ohm to 0.0565 ohm. The use of lubricants or cleaners with lubricants improved the wear performance of the contact, and also maintained or improved the measured contact resistance after 45,000 cycles.

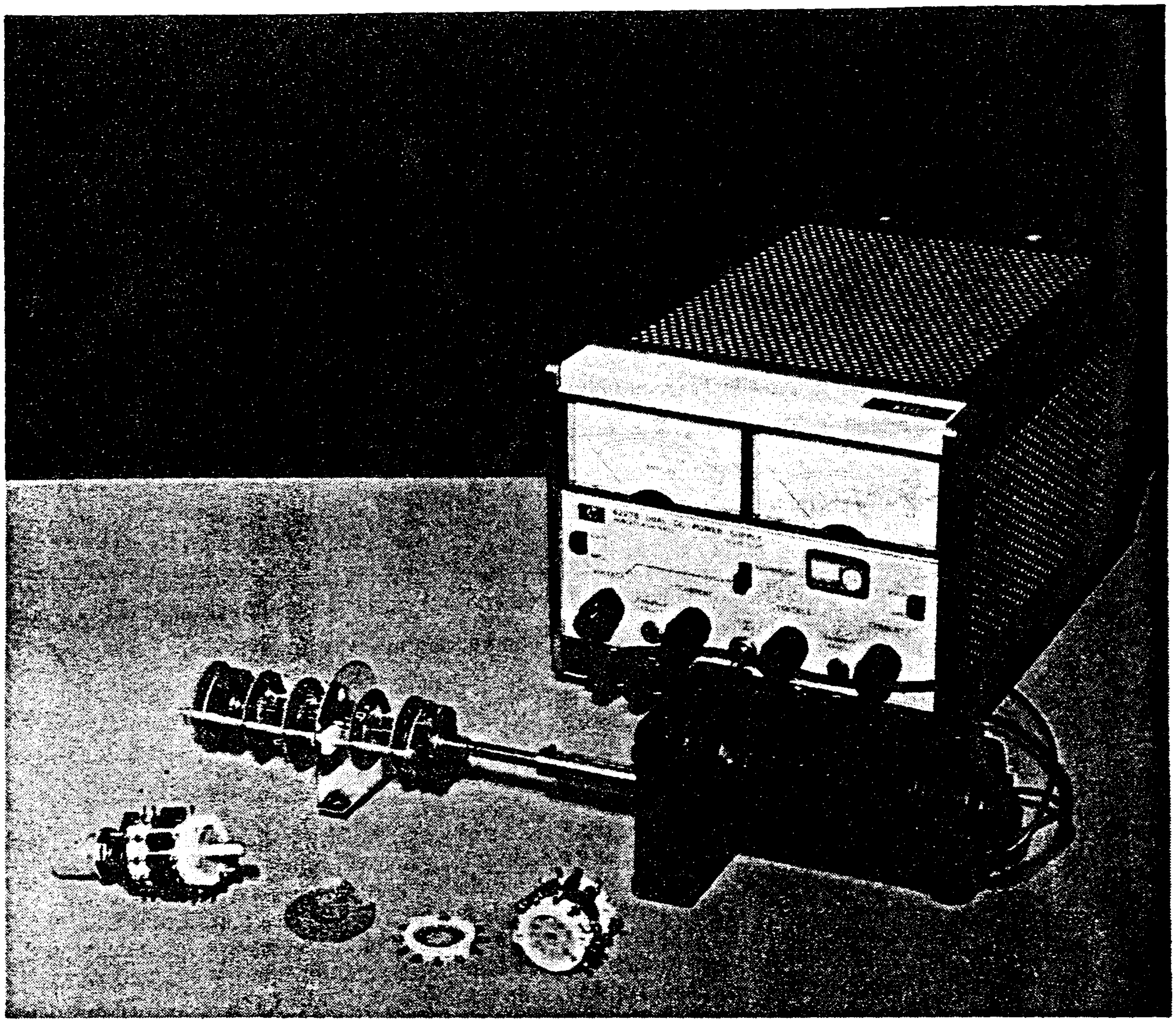


FIGURE 43 CONTACT WEAR TEST APPARATUS

FIGURE 44 RESULTS, CONTACT WEAR TEST

SWITCH LIFE CYCLE TEST

1) WAFERS WERE INITIALLY CLEANED WITH FREON
2) INITIAL RESISTANCE READINGS WERE TAKEN WITH A KEITHLEY D.V.M. MODEL 2001.
3) THE LUBRICANTS WERE THEN APPLIED TO THE VARIOUS WAFERS.
4) SWITCHES WERE THEN CYCLED USING A D.C. MOTOR AT APPROX. 30 RPM.
5) WAFERS WERE THEN CLEANED WITH FREON.
6) RESISTANCE MEASUREMENTS WERE TAKEN
7) WAFERS THEN HAD LUBES REAPPLIED.
8) STARTED THE NEXT CYCLE TEST

WAFER	CLEANER	INITIAL	5K CYCLES	10K CYCLES	15K CYCLES	20K CYCLES	25K CYCLES	30K CYCLES	35K CYCLES	40K CYCLES	45K CYCLES
1	No lube	0.0161	0.0234	0.0207	0.019	0.0099	0.0105	0.0181	0.0261	0.0391	0.0565
2	Electrolube	0.0155	0.0134	0.0202	0.0189	0.0132	0.0089	0.0097	0.0097	0.0095	0.0081
3	ReNu lube	0.018	0.0276	0.0287	0.0136	0.0211	0.0103	0.0121	0.0131	0.0119	0.0103
4	MS939	0.0175	0.0257	0.021	0.0181	0.0119	0.0089	0.0102	0.0111	0.0105	0.0112
5	WD 40	0.0146	0.0101	0.0192	0.0189	0.017	0.0102	0.0072	0.0081	0.0089	0.0101

WAFER	CLEANER	INITIAL	5K CYCLES	10K CYCLES	15K CYCLES	20K CYCLES	25K CYCLES	30K CYCLES	35K CYCLES	40K CYCLES	45K CYCLES
1	No lube	0.0191	0.0152	0.0147	0.0162	0.0183	0.0161	0.0169	0.0289	0.0506	0.0681
2	Deoxit	0.0229	0.0184	0.0155	0.0148	0.0157	0.0175	0.0155	0.0165	0.0149	0.0131
3	KT.Restorer	0.0162	0.0221	0.0165	0.0155	0.0205	0.0169	0.0179	0.0201	0.0199	0.0197

These lubricants were evaluated by instrumentation shop personnel conducting the tests. The following ratings resulted from this evaluation.

RATING: WEAR TEST

NOTE: All lubes were applied only at the start of each cycle test.

1. Excellent — Miller Stephenson 938/CO_2

   This product evaporates quickly but leaves a slight oily film which is preferable for longevity of wafer switches. This product did an excellent job of preventing wear of the contacts. Because this product contains a HCFC, however, it is not considered a viable alternative at the present time.

2. Good — Deox-It 100

   This product leaves too much oil. This oil may attract contaminants that could cause erosive contact wear. However, in a controlled situation such as those experienced during the testing regimen, the product did a good job of lubricating the contacts and protecting against excessive wear.

3. Good — Miller Stephenson 939 Contact Safezone RENU and Lube

   This product leaves a slight oily residue on the switch contacts. This product did a good job in preventing contact wear. Because it contains a HCFC, this material is not considered a viable alternative at the present time.

4. Good — WD-40

   This product would not normally be used on electrical switches but was used as a baseline lubricant for comparison purposes. This product leaves too much of an oily residue behind, which in the long term would attract contaminants. However, the product did a good job in preventing contact wear.

. Fair Electrolube

This product actually does a better job of preventing contact wear. However, it is a lubricating grease similar to Vaseline® that is applied by a tube. It would be cumbersome to apply on wafer switches in close quarters and is too thick for use at the PJKS facility.

6. Fair Chemtronics Kontact Restorer (Contains Freon-11®)

This product evaporates very quickly and leaves a slight oil residue. However, in comparison to the products evaluated above, this product did not adequately protect the contacts from excessive wear.

6.3.2 Tape Head Cleaning. An initial attempt was made to measure the signal-to-noise ratio in tapes played from a dirty tape head versus those played from a clean tape head. This difference in the signal-to-noise ratio would establish a cleanliness criterion for tape head cleaning, especially for magnetic tape heads used in data acquisition. The dirty tape head was prepared by continuously playing a blank magnetic tape for approximately 5 days until the tape head was visibly contaminated with magnetic particles. A 10KHz signal was recorded on a clean magnetic tape, and played on both the contaminated (dirty) tape head, as well as a tape head recently cleaned per manufacturer's instructions. The signal read from both the dirty and clean tape heads, however, did not display any differentiation in signal profile or noise level. For this reason, this approach was abandoned as a measurement criterion of the cleanliness level of the tape head.

A more viable approach for the selection of alternative tape head cleaners was to evaluate the amount of ,sidue remaining after evaporation. Effective cleaning of magnetic tape heads is accomplished by the use of a chemically pure solvent, and the presence of a film or residue after evaporation must be precluded. Residue remaining after evaporation of the cleaning solvent was evaluated by two methods. In the first method, magnetic tape heads were removed from various recording devices and inspected for cleanliness prior to testing. Candidate testing solvents (including CFC-113, heptane, ethyl alcohol, and isopropyl alcohol) were placed on the tape head, and allowed to air dry. The drying process was observed under a microscope at 30X magnification. After solvent drying was complete, the tape heads were visually inspected for remaining residue. Significant residue after evaporation would result in rejection of the cleaner. In the second method, the Non Volatile Residue (NVR) of the cleaning solvents was measured. In this technique, the solvent was evaporated to dryness, and the remaining material was weighed on an analytical balance. The non volatile residue is reported as milligrams of residue per 100 milliliters of solvent.

The final evaluation of the alternative cleaning solvents was performed by servicing video tape heads removed from the field using the manufacturers recommended cleaning procedures. In this evaluation, a visual inspection of the cleanliness level of the tape head, amount of residue remaining, and any adverse effect on any of the other materials contacted by the cleaner was the criterion for selection of an alternate cleaning compound.

Non volatile residues and visual observations for the various solvents evaluated as tape head cleaners are incorporated in Table 17. Reagent isopropyl alcohol, ethyl alcohol, and heptane were tested in VCR heads. These solvents all performed adequately. Ethyl alcohol and heptane are the recommended replacement tape head cleaners. For cleaning Capstan® rollers, heptane must be used because of the incompatibility of Capstan with ethyl alcohol.

TABLE 17 TAPE HEAD CLEANING RESULTS

Solvent	NVR (mg/100ml)	Visual Observations (30X magnification)
CFC-113	<0.1	particles remained after cotton swab wipe
Isopropyl Alcohol	2.0*	obvious film and residue remained after evaporation
1,1,1- TCA	0.7	
Reagent IPA	0.8	
n-Heptane	0.1	good cleaning ability, evaporates relatively slowly
Ethyl Alcohol	0.4**	good cleaning ability, evaporates quickly

Notes: * Film Remained After Evaporation
** Swells Capstan Rollers

6.3.3 Cleaning Prior to Installation of Strain Gages

Four tests were performed to evaluate the bonding of strain gages to test articles: an adhesive evaluation test, a cleaning prior to bonding test, a strain gage creep test, and a strain gage bonding test.

In the adhesive evaluation test, the existing ODC adhesive system (M-Bond 200 formulation containing TCA) and the replacement ODC-free adhesive system (M-Bond 200 formulation containing IPA) were applied to lap shear test panels, allowed to cure, and tested for lap shear strength. Lap shear panels were prepare from 304 stainless steel and aluminum sheet stock and conformed to requirements specified in ASTM D1002-72 "Standard Test Method for Strength Properties of Adhesives in Shear by Tension Loading (Metal to Metal)". Test panels were used in the "as-received" condition, had only nominal contamination from handling and skin oils, and were precleaned with CFC-113 prior to processing in accordance with NSP 00309 "Installation of Strain Gages". NSP 00309 specifies the use of TCA as the degreasing solvent, followed by abrasion and conditioning steps. After the adhesive was adequately cured, test panels were equilibrated at either room temperature (70°F nominal), 150°F, -40°F, or -250°F and separated using a tensile test machine (MTS Systems Corporation). These test temperatures were selected as representative of the temperature extremes experienced by actual test hardware. Mean stress required to separate the panels in 10^3 psi (or ksi) were recorded for each of the test panels, and the resultant stress for the new adhesive was compared to the stress obtained from the old adhesive, the higher stress being indicative of superior adhesive strength.

Table 18 shows the results of the adhesive performance test. These test results indicate superior adhesive strength using the replacement M-Bond 200 formulation which incorporates IPA as the carrier solvent instead of TCA.

TABLE 18 LAP SHEAR TEST RESULTS OF OLD AND NEW ADHESIVE

LAP SHEAR SCREENING OF OLD/NEW CATALYST, ROOM TEMP	12/3/93	MEAN STRESS, psi
S/S OLD		500
S/S NEW		855
ALUM OLD		532
ALUM NEW		680
LAP SHEAR SCREENING OF OLD/NEW CATALYST, 150 DEGREES	12/3/93	MEAN STRESS, psi
S/S OLD		575
S/S NEW		875
ALUM OLD		408
ALUM NEW		993
LAP SHEAR SCREENING OF OLD/NEW CATALYST, -40 DEGREES	12/3/93	MEAN STRESS, psi
S/S OLD		342
S/S NEW		435
ALUM OLD		246
ALUM NEW		339
LAP SHEAR SCREENING OF OLD/NEW CATALYST, -250 DEGREES	12/3/93	MEAN STRESS, psi
S/S OLD		152
S/S NEW		255
ALUM OLD		250
ALUM NEW		317

In the cleaning prior to bonding test, stainless steel, aluminum, and G-10 fiberglass epoxy lap shear specimens were contaminated with a variety of contaminants, the contaminated parts aged for two hours at room temperature, and the parts cleaned in accordance with NSP 00309. Contaminants included Mystic Metal Mover, Cooltool, Moly-D, Trimsol, and WD-40. Samples were also handled without gloves, so skin oils were also present. In this test, degreasing was performed with one of five candidate replacement cleaners: EP921, Bio-T-Max, Isopropyl Alcohol, Ethyl Alcohol, or Biogenic Regent. The use of EP921, Bio-T-Max and Biogenic reagent required a final water rinse to remove residual cleaner. TCA and CFC-113 were also included to evaluate their cleaning effectiveness relative to the alternative cleaners. An uncontaminated lap shear specimen which had been solvent wiped with TCA was used as the baseline test article. After cleaning, lap shear specimens were bonded with the new ODC-free adhesive and measured for separation stress. The objective of this test was to obtain an alternative cleaner with equivalent, or superior, properties as the existing TCA solvent.

Figure 45 shows the results of the cleaner evaluation test, using aluminum as the test surface. Figure 46 shows the results of the cleaner evaluation test, using G10 fiberglass epoxy as the test surface. In these figures the baseline sample was an uncontaminated specimen which was wiped with TCA prior to bonding. This baseline was used as a measurement of the ideal or maximum lap shear bond strength achieved under the conditions tested. All other samples were contaminated with the oil mixture, cleaned with the candidate replacement cleaners, and measured for lap shear strength in a tensile test machine.

FIGURE 45 ALUMINUM LAP SHEAR RESULTS, ALTERNATIVE CLEANERS

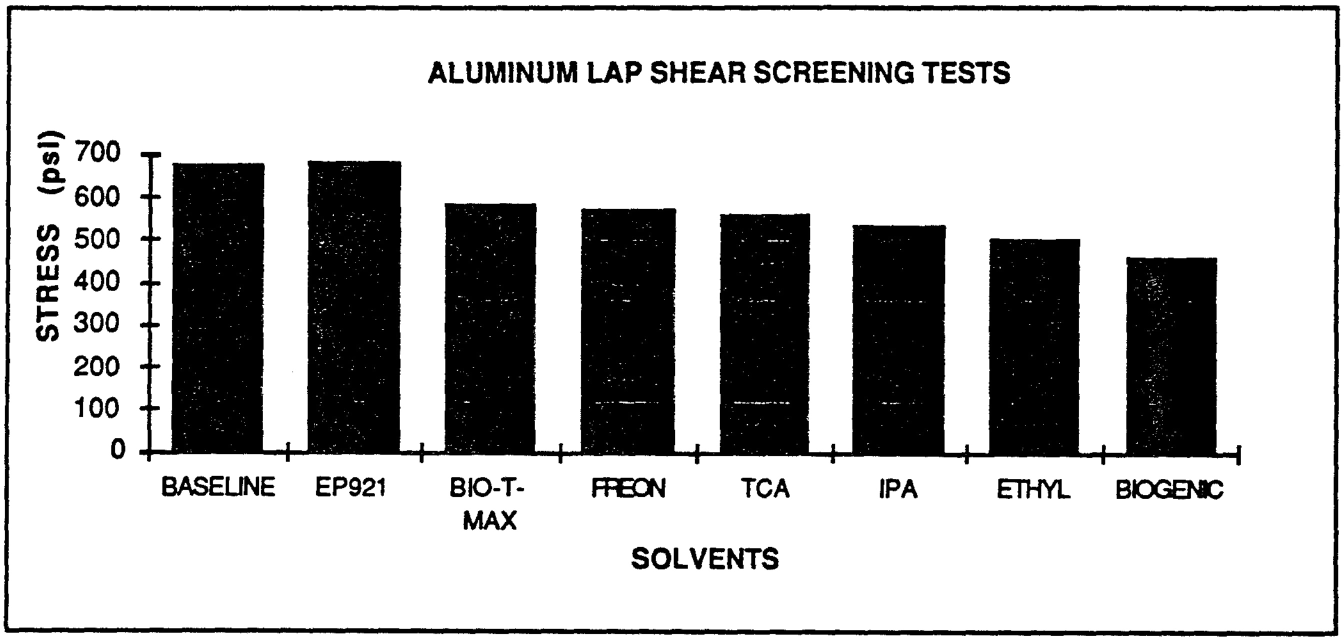
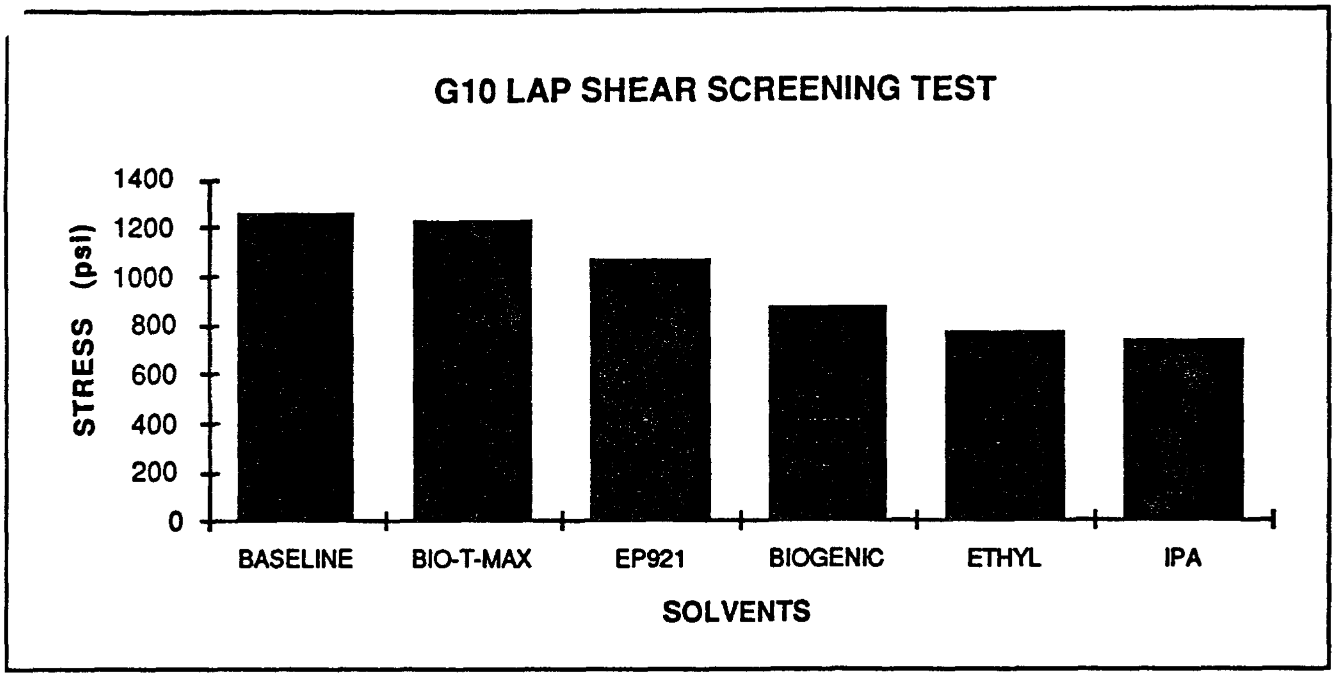


FIGURE 46 G-10 FIBERGLASS LAP SHEAR RESULTS, ALTERNATIVE CLEANERS



Creep is used to determine the extent of deformity under an applied load. In the strain gage creep test, test specimens were loaded to a predetermined value (within the elastic limits of the material), and the load held onstant for a given period of time. The load was then reduced and the relaxation of the material was monitored. aterials tested in this evaluation included 2024 aluminum, 304 stainless steel, and G10 fiberglass laminates. For the stainless steel and aluminum samples, stress was applied to 90% of the material's yield strength and the stress held constant for twenty minutes. The fiberglass epoxy tensile samples were loaded in a uniaxial tensile machine to a load equivalent to 75% of the material's ultimate strength and this stress was also held constant for twenty minutes. The strain gage output versus time under constant load was recorded. These tests were performed at ambient temperature (70°F). Three test specimens for each test condition were included. The objective of this test was to compare the creep rate of strain gages bonded with the existing ODC adhesive versus the creep rate of strain gages bonded with the replacement ODC-free adhesive.

No significant differences were observed in creep rates between strain gages bonded with the previous ODC adhesive and gages bonded with the new ODC-free adhesive for stainless steel, aluminum, and G-10 materials.

In the strain gage bonding test, strain gages were bonded to aluminum substrates using the new ODC-free adhesive formulation. One side of the sample remained uncontaminated and was wiped with TCA to remove residual oils. The other side was contaminated and cleaned using the alternative cleaners. Contaminants and alternative cleaners were identical to those used in the cleaning prior to bonding test. An additional sample, having both sides uncontaminated and precleaned with TCA to remove residual oils was tested to determine the baseline variability of this test method. Tensile stress was applied to the substrate, causing a strain (elongation) on the strain gage. Tensile specimens were designed and tested in accordance with ASTM E-8 "Standard Test Method for Tension Testing of Metallic Materials". Strain (Percent Elongation) was measured using the output of the calibrated strain gage. An applied stress vs strain curve of the strain gage was recorded for each of the alternative cleaners and compared to the curve obtained for cleaning with TCA. The objective of this test was to record no premature separation of the strain gage from the test article using the candidate replacement cleaners, and to obtain similar stress vs strain profiles for these cleaners as the existing TCA cleaner.

Results of the strain gage bonding test are incorporated in Table 18. No significant difference was seen between the TCA cleaned gage and the ODC-free cleaned gage, for all samples tested. Strain data obtained from uncontaminated surfaces wiped with TCA were essentially identical to data obtained from intentionally contaminated surfaces cleaned with ODC-free cleaners. Yield and modulus data had standard deviations in the 1% range for both groups tested.

TABLE 19 STRAIN GAGE BONDING RESULTS

"AS-IS" CONDITION- TCA CLEANED

S/N	COUPON NUMBER	YIELD LOAD (kips)	YIELD STRENGTH (kips)	ULTIMATE LOAD (ksi)	ULTIMATE STRENGTH (ksi)	YOUNG'S MODULUS (Msi)
1	BL/BL TRIC	1.505	47.48	2.146	67.72	10.24
2	BL/BT MAX	1.500	47.42	2.152	68.05	10.58
3	BL/IPA	1.514	47.77	2.145	67.69	10.07
4	BL/BIOG	1.500	47.32	2.171	68.51	10.65
5	BL/EP921	1.514	48.65	2.173	69.82	10.76
6	BL/TRIC	1.493	47.96	2.150	69.08	10.54
	MEAN	1.504	47.77	2.156	68.48	10.47
	STANDARD DEVIATION	0.008	0.490	0.013	0.842	0.26
	COEF oF VAR	0.570	1.040	0.603	1.230	2.50

"CONTAMINATED"- SOLVENT'S CLEANED

S/N	COUPON NUMBER	YIELD LOAD (kips)	YIELD STRENGTH (kips)	ULTIMATE LOAD (ksi)	ULTIMATE STRENGTH (ksi)	YOUNG'S MODULUS (Msi)
1	BL/BL TRIC	1.504	47.46	2.146	67.72	10.28
2	BT MAX	1.500	47.42	2.152	68.05	10.70
3	IPA	1.514	47.77	2.145	67.69	10.20
4	BIOG	1.500	47.32	2.171	68.51	10.67
5	EP921	1.514	48.65	2.173	69.82	10.60
6	TRIC	1.493	47.96	2.150	69.08	10.71
	MEAN	1.504	47.81	2.156	68.48	10.53
	STANDARD DEVIATION	0.008	0.470	0.013	0.842	0.23
	COEF oF VAR	0.570	0.990	0.603	1.230	2.14

7.0 PROPELLANT COMPATIBILITY AND RESIDUE ANALYSIS

7.1 Introduction

The objective of the mechanical cleaning part of the PJKS Phase I program was to select cleaners for use in the Engineering Propulsion Laboratory (EPL) Valve Shop. The current cleaning solvents CFC-113 and TCA have been identified as Class 1 Ozone-Depleting Chemicals (ODC's) which must be removed from Air Force facilities. Tasks performed at the Chemical Technology Laboratory (CTL) in support of this program included determination of:

1. Non-volatile residues (NVR's) from test specimens which had been cleaned in candidate cleaners,
2. Surface contamination on the test specimens described above using Fourier Transform Infrared Spectroscopy (FTIR), and
3. Compatibility of candidate cleaners with propellants used at EPL, Aerozine-50 and nitrogen tetroxide.

Approximately sixty cleaners were identified as potential ODC replacements. Screening was performed through data reviews, discussions, and preliminary testing. Cleaners tested after screening was complete are listed in Table 19.

TABLE 20 CLEANERS TESTED

No.	Cleaning agent	Composition
1.	TCA	CCl_3CH_3
2.	CFC-113	CCl_2FCF_2Cl
3.	Citrex	N-methyl pyrollidone and d-limonene (25-30%)
4.	JPX Degreaser	Water (50%), terpene, glycol ether
5.	EP - 921	Propylene carbonate and d-limonene
6.	Bioact 280	Aliphatic esters
7.	Partsprep	Water and N-methyl pyrollidone
8.	Biogenic Reagent	Aliphatic hydrocarbons and d-limonene
9.	Daraclean 282	Water, glycol ether, additives
0.	Formula 815 GD	Water and alkaline additives

7.2 Non-Volatile Residue Determination

7.2.1 Approach Stainless steel and aluminum test specimens which had been contaminated and cleaned were provided by the EPL. They were transferred to 150 ml beakers, and 100 ml methylene chloride, Omnisolv™ Grade, was added. The beakers were then exposed to ultrasound for ten minutes. During testing, two beakers spilled because of the weight of the specimens. The specimens were removed, and the solvent dried with mild heat in tared aluminum weighing pans. After the methylene chloride had evaporated, the pans were transferred to a 45°C oven for 30 minutes. The pans were then cooled and reweighed. Because methylene chloride did not remove the most of the Dykem marking fluid, a visual assessment of the amount of remaining blue Dykem was made.

7.2.2 Results and Discussion Results summarized in Table 20 include the weight of the non-volatile residues (NVR) and a visual assessment of the amount of remaining Dykem. Assessment values for Dykem ranged from 0 (no Dykem remaining on the specimen) to 3 (no evidence of removal by cleaner). The reporting limit was 0.2 mg based on a blank value of 0.1 mg/100 ml. Important results include:

1. Several alternative cleaners were more effective than CFC-113,
2. Of the candidate cleaners, Citrex, EP-921, and Partsprep were the most effective in removing contaminants,
3. Biogenic Reagent and Bioact 280 produced a low NVR, but were less effective in removing Dykem,
4. Daraclean 282 produced a high NVR, but effectively removed Dykem, and
5. Generally, more polar cleaners are effective in removing Dykem.

TABLE 21 NON-VOLATILE RESIDUES FROM CLEANING POWER DEMONSTRATION

Test Num.	Cleaner	Material	Treatment	Weights Tare, g	Final, g	NVR, mg	Report, mg	Blue Appearance (a)
1	TCA	Aluminum	As-is	1.3956	1.3958	0.2	(b)	3
2		CRES	As-is	1.4107	1.4110	0.3	0.3	3
3	CFC-113	Aluminum	As-is	1.3886	1.3890	0.4	0.4	3
4		CRES	As-is	1.3994	1.3998	0.4	0.4	3
5	Citrex	Aluminum	As-is	1.4051	1.4053	0.2	(b)	0
6		CRES	As-is	1.4063	1.4066	0.3	0.3	0
7	JPX Degreaser	Aluminum	As-is	1.3941	1.3944	0.3	0.3	2
8		CRES	As-is	1.4112	1.4115	0.3	0.3	2
9	EP-921	Aluminum	As-is	1.4129	1.4130	0.1	(b)	0
10		CRES	As-is	1.3973	1.3975	0.2	(b)	1
11	Bioact 280	Aluminum	As-is	1.4070	1.4071	0.1	(b)	1
12		CRES	Baked	1.3949	1.3950	0.1	(b)	2
13	Partsprep	Aluminum	As-is	1.3866	1.3868	0.2	(b)	0
14		CRES	Baked	1.3889	(c)	(c)	(c)	0
15	Biogenic Regent.	Aluminum	As-is	1.3968	1.3970	0.2	(b)	2
16		CRES	Baked	1.4070	(c)	(c)	(c)	3
17	Daraclean 282	Aluminum	As-is	1.3848	1.3853	0.5	0.5	0
18		CRES	Baked	1.4022	1.4026	0.4	0.4	0
19	Formula 815 GD	Aluminum	As-is	1.3835	1.3846	1.1	1.1	0
20		CRES	Baked	1.4019	1.4029	1.0	1.0	1
21	None	Aluminum	Baked	1.4133	1.4177	4.4	4.4	3
22		CRES	Baked	1.4112	1.4163	5.1	5.1	2
23	None	Aluminum	Baked	1.3920	1.3963	4.3	4.3	3
24	Blank	(d)	(d)	1.4111	1.4112	0.1	(d)	(d)

(a) Appearance of blue stripe
- 0 = not visible; evidence that all removed by cleaner
- 1 = faint; evidence that most removed by cleaner
- 2 = moderate blue; evidence of some removal by cleaner
- 3 = Strong blue; no evidence of removal by cleaner

(b) Not detected at reporting limit (2 x blank value)
(c) No data; solvent lost during ultrasound
(d) Not applicable

7.3 Surface Evaluation By Fourier Transfer Infrared Spectroscopy

7.3.1 Approach The objectives were to provide additional assessment of the effectiveness of cleaners in removing contaminants and to determine whether the cleaner left residue on the surface. Stainless steel and aluminum test specimens which had been contaminated and cleaned were provided by EPL. A listing of the specimens analyzed is given in Table 21. They were analyzed by Fourier Transform Infrared Spectroscopy (FTIR) in the reflectance mode. Two scans were performed for each specimen. The first scan was from an area where no contaminants were present to provide a baseline. The second scan was from an area which had been contaminated and cleaned.

7.3.2 Results and Discussion Results are summarized in Table 21. Surface residues consistent with organic contamination were detected on the control specimens and on the stainless steel specimen which had been cleaned with Bioact 280. This result is surprising because no NVR's were detected on this specimen and the aluminum specimen contained no residue. There were gross differences in the spectra of the blank surfaces which may have resulted from the presence of oxide coatings, but are unrelated to the cleaning process.

TABLE 22 RESULTS OF SURFACE EVALUATION FROM CLEANING POWER DETERMINATION

Test Num.	Cleaner	Material	Treatment	Results
1	TCA	Aluminum	Baked	No difference from blank
2		CRES	Baked	No difference from blank
3	CFC-113	Aluminum	Baked	No difference from blank
4		CRES	Baked	No difference from blank
5	Citrex	Aluminum	Baked	No difference from blank
6		CRES	Baked	No difference from blank
7	JPX Degreaser	Aluminum	Baked	No difference from blank
8		CRES	Baked	No difference from blank
9	EP-921	Aluminum	Baked	No difference from blank
10		CRES	Baked	No difference from blank
11	Bioact 280	Aluminum	Baked	No difference from blank
12		CRES	As-is	Organic contaminants present
13	Partsprep	Aluminum	Baked	No difference from blank
14		CRES	As-is	No difference from blank
15	Biogenic Regent	Aluminum	Baked	No difference from blank
16		CRES	As-is	No difference from blank
17	Daraclean 282	Aluminum	Baked	No difference from blank
18		CRES	As-is	No difference from blank
19	Formula 815 GD	Aluminum	Baked	No difference from blank
20		CRES	As-is	No difference from blank
21	None	Aluminum	As-is	Organic contaminants present
22		CRES	As-is	Organic contaminants present
23	None	CRES	Baked	Organic contaminants present

7.4 Propellant Compatibility

7.4.1 Approach

Compatibility with propellants was determined in two phases:

a. Screening tests in which small amounts of cleaner and propellant were mixed in order to eliminate candidates which reacted violently with propellants, and

b. Detailed testing in which cleaners were added to propellants in closed containers where pressure and temperature changes could be modified.

Screening tests were performed to eliminate combinations which could cause explosive reactions in sealed chambers during detailed testing.

7.4.2 Screening Tests

Cleaners, nominal 1ml, were placed in small vials and transferred to the appropriate glove box. A small volume of propellant, either nitrogen tetroxide or Aerozine-50, was added and the resulting reaction was noted.

7.4.3 Detailed Testing

Separate apparatus were used for nitrogen tetroxide and Aerozine-50. Each apparatus (Figure 96) was a heavy-walled glass tube to which an aluminum top was attached with an aluminum flange and tetrafluoroethylene (TFE) seal. Each top was equipped with a pressure transducer, septum port, and relief valve set at 75 psig. The transducer and thermocouple were attached to a data logger which monitored test time, system temperature, and pressure. Propellant, 65 ml, was placed in the tube along with a magnetic stirring bar. After the temperature and pressure had equilibrated, 2 ml of cleaner was added through the septum port. The ullage volume was approximately 40 ml. The temperature and pressure were monitored continuously for the first fifteen minutes and every five minutes thereafter until the end of the test.

The initial plan was to test all of the cleaners listed in Table 20. However, the plan was modified as follows:

1. Two cleaners which reacted violently with nitrogen tetroxide during the screening test, Citrex and JPX Degreaser, were not tested further. Another cleaner, EP-921, which reacted likewise reacted violently was tested because it was one of the final candidate cleaners. However, the volume was reduced to 0.5 ml.
2. Water was tested with nitrogen tetroxide as a check on system performance, and
3. The four final candidate cleaners, and CFC-113 were tested with Aerozine-50.

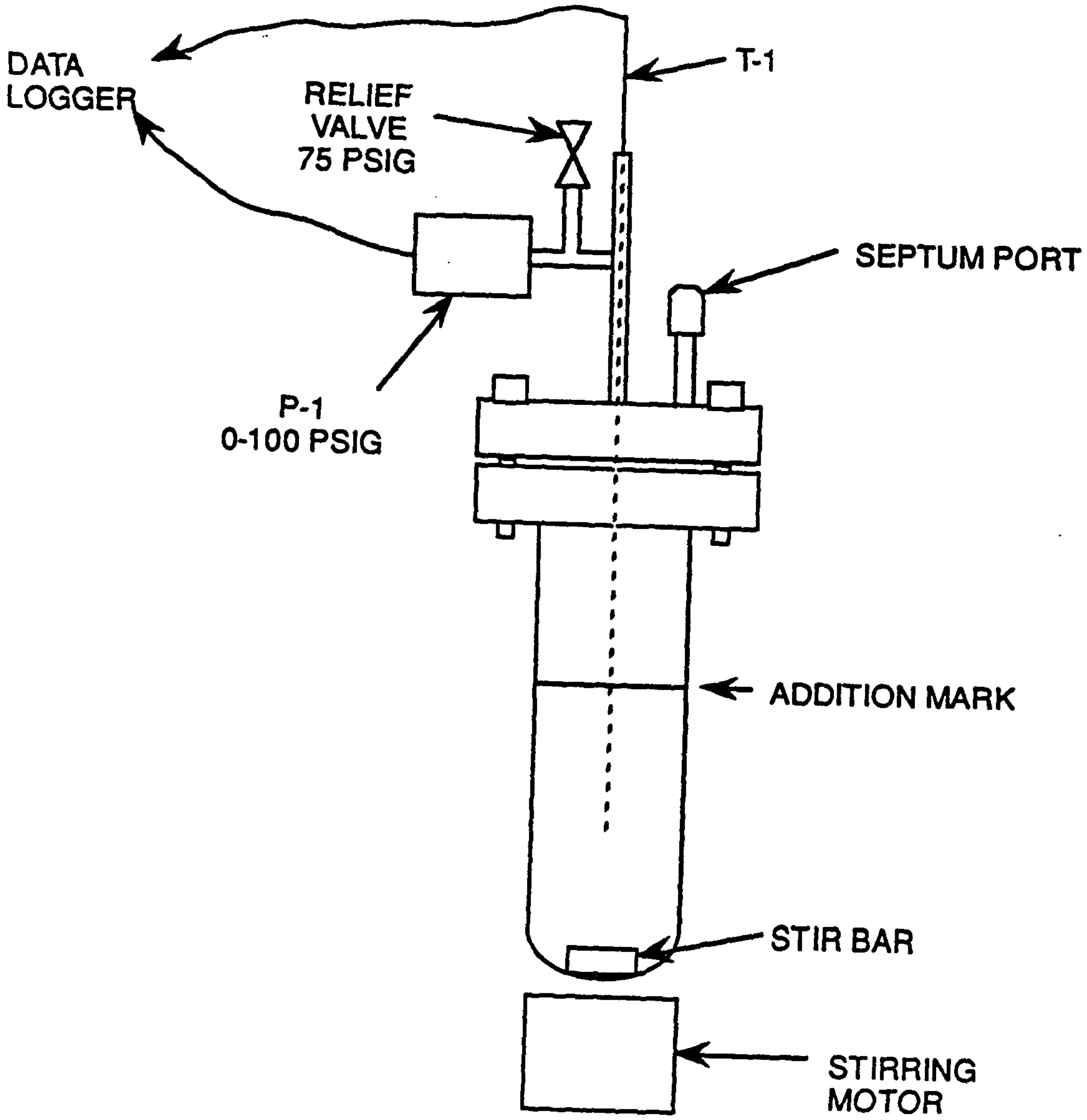


FIGURE 47 PROPELLANT COMPATIBILITY TEST SCHEMATIC

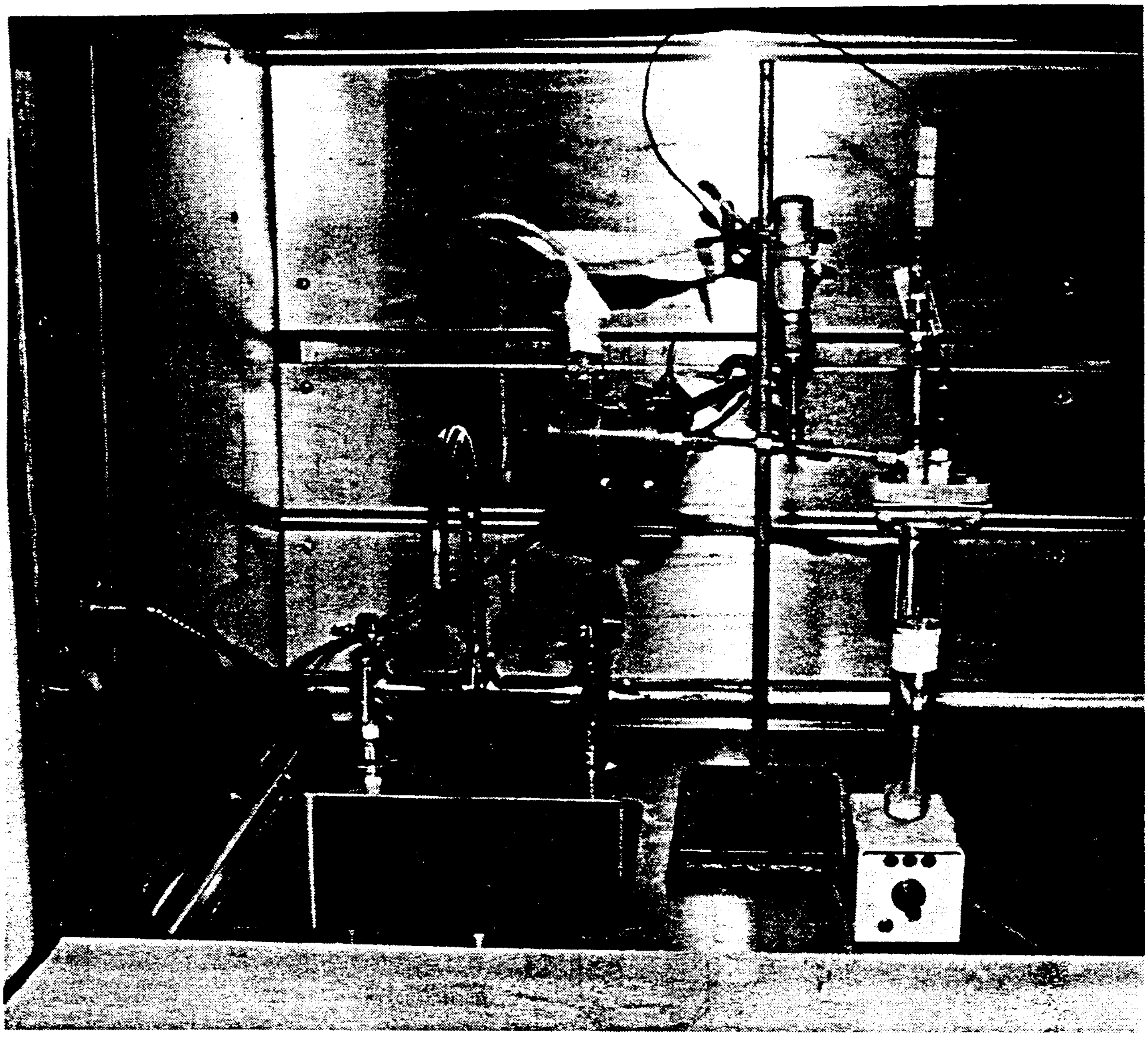


FIGURE 48 PROPELLANT COMPATIBILITY TEST PHOTOGRAPH

7.4.4. Results and Discussion Results of the screening tests are presented in Table 23. The data logger output for each of the detailed tests was reviewed and representative pressures and temperatures were plotted as functions of time after addition of cleaners. These graphs are presented in Appendix D. Data summaries from the graphs are given in Table 24.

7.4.4.1 Nitrogen Tetroxide

As shown in Table 24, reactions of nitrogen tetroxide with cleaners generally results in an increase in pressure and either increase or decrease in temperature. Some changes occurred immediately, others more slowly over the test interval. This section describes some chemical reactions of nitrogen tetroxide which might account for the observed temperature and pressure changes.

1. Some pressure change results from the changes in temperatures, either because of changes in ambient temperatures during the test interval or chemical reactions. At 70°F, a five degree rise in temperature will produce a pressure increase of *ca.* 2 psi.
2. In the absence of base, nitrogen tetroxide reacts endothermically with water with the production of nitric acid (HNO_3) and nitric oxide (NO) gas. If base is present, nitrogen tetroxide is exothermically converted to nitrate (NO_3^-) and nitrite (NO_2^-).
3. Nitrogen tetroxide oxidizes organic compounds, with resulting formation of nitric oxide or nitrogen gas. These reactions are likely to be exothermic, with some producing considerable heat. Nitrogen tetroxide can also react with organic compounds to form nitro compounds ($R\text{-}NO_2$) or nitrates ($R\text{-}ONO_2$). These reactions have been observed when nitrogen tetroxide is mixed with hydrocarbons and are expected with the organic constituents of the cleaners.

Species whose reactions with nitrogen tetroxide are of particular concern include:

1. *Terpenes, such as d-Limonene.* In the screening tests all of the cleaners containing terpenes showed instantaneous gas evolution. Similar results were obtained in the detailed testing. For example, immediately after nitrogen tetroxide was added to Biogenic reagent, the temperature rose 3 degrees and the pressure rose 34 psi.
2. *N-methyl Pyrrolidone.* Although the reaction was more gradual than with the terpenes, temperature and pressure rises were noted with Partsprep. During detailed testing, the temperature rose 7 degrees and the pressure rose 62 psi within the first thirty minutes. In the screening test, Citrex, a mixture of NMP and d-limonene reacted the most violently with nitrogen tetroxide, with immediate heat and evolution of copious yellow fumes.
3. *Propylene Carbonate.* The reaction with EP-921 was highly exothermic with a temperature rise of 10 degrees within 60 minutes even though the volume of cleaner was 0.5 ml instead of the two ml used for the other cleaners.

7.4.4.2 Aerozine-50

Reactions of the cleaners with Aerozine-50 were less severe than with nitrogen tetroxide. The absence of significant pressure increases indicates that the cleaners did not significantly oxidize either hydrazine or UDMH. Such oxidation would have resulted in formation of nitrogen gas. During the screening tests, two phases formed when non-polar solvents, such as TCA or CFC-113, mixed with hydrazine. Precipitates formed when additives to aqueous cleaners, such as Daraclean 282, mixed with the less polar Aerozine-50. Formation of two phases or precipitates can be a problem if they result in clogging or localized areas of contamination.

The absence of severe reactions does not necessarily mean that the cleaner is compatible with the propellant. For example, although CFC-113 did not react appreciably with Aerozine-50 during the test interval, it does react over time with hydrazine with formation of hydrogen chloride and hydrogen fluoride, both of which can corrode metals.
When Partsprep was mixed with Aerozine-50 there was a gradual temperature rise of almost nine degrees. This might be a result of the expected reaction of N-methyl pyrrolidone, which possesses a carbonyl (R-C=O) group, with hydrazine and UDMH. Carbonyl groups react with hydrazines to form hydrazones, compounds with an imino (R-C=N) group. This may have operational significance if unless the hydrazone is less volatile than the propellants.

TABLE 23 RESULTS OF PROPELLANT COMPATIBILITY SCREENING

Num.	Cleaner	Prop.	Results
1	JPX	N_2O_4	Violent gas evolution
1	JPX Degreaser	A-50	No apparent reaction - 2 phases
2	Daraclean 282	N_2O_4	Some gas evolution, similar to water
2	Daraclean 282	A-50	No apparent reaction - White precipitate
3	TCA	N_2O_4	No apparent reaction
3	TCA	A-50	No apparent reaction - 2 phases
4	CFC-113	N_2O_4	No apparent reaction
4	CFC-113	A-50	No apparent reaction - 2 phases
5	Citrex	N_2O_4	Violent reaction > 1a, gas evolution
5	Citrex	A-50	No apparent reaction - Mixed well
6	EP - 921	N_2O_4	Violent reaction, < 5a. Some gas evolution. Bubbled for several min.
6	EP - 921	A-50	No apparent reaction - Mixed well
7	Biogenic Regent	N_2O_4	Bubbling gas evolution
7	Biogenic Regent	A-50	No apparent reaction - 2 phases
8	Bioact 280	N_2O_4	No apparent reaction
8	Bioact 280	A-50	No apparent reaction - 2 phases
9	Formula 815 GD	N_2O_4	Some bubbling, foaming, & bumping
9	Formula 815 GD	A-50	No apparent reaction - white precipitate
10	Partsprep	N2O4	Gas evolution, < 6a, followed by delayed bubbling & burping
10	Partsprep	A-50	No apparent reaction - mixed well

TABLE 24 PROPELLANT COMPATIBILITY DATA SUMMARY

Nitrogen Tetroxide

Cleaner(a)	Duration, Min	Temperature/Pressure Events			Temperature/Pressure Events			Temperature/Pressure Events			Temperature/Pressure Events		
		Time(b)	*Temp.(c)*	*Pres.(d)*	*Time(b)*	*Temp.(c)*	*Pres.(d)*	*Time(b)*	*Temp.(c)*	*Pres.(d)*	*Time(b)*	*Temp.(c)*	*Pres.(d)*
Water	91	<1	-3.2	1.6	2	-2.6	-0.4	56	3.6	2.6			
TCA	102	<1	-0.5	-1.0	12	3.6	1.6	76	8.7	4.3			
CFC-113	36	<1	-1.1	-1.5	2	-0.5	0.0	21	1.6	1.1			
Bioact 280	61	<1	0.0	0.0	4	1.7	1.0	31	0.7	0.8	41	1.8	1.4
Biogenic Regent.	76	<1	0.0	33.5	<1	3.0	26.0	1	2.0	21.5	60	8.5	10.5
Daraclean 282	88	<1	0.0	0.0	3	-0.6	1.6	20	4.8	2.2	68	8.8	4.0
EP-921 (e)	275	<1	0.0	5.5	<1	0.0	3.0	50	10.0	6.0	160	11.8	7.7
Formula 815	115	<1	0.0	4.2	1-2	2.2	1.8	20	1.4	1.7	35	4.0	3.0
		65	1.8	2.6	80	3.7	3.7						
Partsprep	91	<1	0.0	10.0	<1	0.0	8.0	26	7.0	61.5	90	9.0	25.0

Aerozine-50

Cleaner(a)	Duration, Min	Temperature/Pressure Events			Temperature/Pressure Events			Temperature/Pressure Events			Temperature/Pressure Events		
		Time(b)	*Temp.(c)*	*Pres.(d)*	*Time(b)*	*Temp.(c)*	*Pres.(d)*	*Time(b)*	*Temp.(c)*	*Pres.(d)*	*Time(b)*	*Temp.(c)*	*Pres.(d)*
CFC-113	110	<1	-0.4	3.9	10	0.2	2.0	75	1.1	0.6	110	0.6	0.4
Bioact 280	160	<1	0.0	1.0	4	-1.3	-0.6	24	-4.0	0.1	140	-5.0	0.0
Biogenic Regent.	96	<1	0.2	0.2	10	0.3	0.2	96	6.7	0.3			
EP-921	73	<1	0.3	0.0	1-2	0.1	-0.7	18	-3.4	-0.1	68	-4.1	-0.1
Partsprep	280	<1	-1.5	1.0	1-2	-1.5	0.0	100	4.6	0.8	250	8.6	0.1

(a) Volume 2 ml unless otherwise noted
(b) Minutes after addition of cleaner
(c) Change in degrees F from start of test
(d) Change in psi from start of test
(e) Cleaner volume equals 0.5 ml

8.0 DISCUSSION

8.1 Technical Performance of Selected Alternatives

8.1.1 Mechanical Cleaning Four cleaners were selected for cleaning of valve hardware and conformance to manufacturing plan (MP) requirements of non volatile residue. These cleaners were Biogenic Regent, Bioact 280, EP-921, and Parts Prep. Hardware cleaned with Biogenic Regent resulted in elevated levels of non volatile residue (NVR) when Drilube contaminant was present. Of the remaining three candidates EP-921 had a violent reaction with N_2O_4, and Partsprep and Bioact 280 had some materials compatibility concerns. Any of these remaining candidates, however, would be acceptable as replacement solvents for TCA and CFC-113 used at the PJKS facility if allowances are made for these nonconformities. EP-921, for example, can be used to process hardware not intended for propellant usage if the proper controls can be implemented. Partsprep and Bioact 280 may be used for servicing propellant hardware, and appropriate steps should be taken to minimize exposure time to these cleaners because of materials compatibility concerns. The selection of replacement cleaners resulted in a compromise of desired features. The selection of Bioact 280 was based on delivering the features and requirements necessary to clean various types of hardware in the most optimum manner.

8.1.2 Anti-Seize Compound Each of the anti-seize compounds evaluated performed well in the Salt Fog and the Bolt Force vs. Torque tests. No corrosion damage was observed on materials coated with these candidates. Each compound evaluated exhibited a constant friction factor. For ambient temperature usage, any of the compounds evaluated would be an effective substitute for the existing Fel-Pro C5A. However, for fittings that will be exposed to high temperatures (1200°F) then only the Dow Molykote 1000 should be used.

8.1.3 Electrical Cleaning All of the ODC-free contact cleaners performed as well as or better than the existing Kontac Restorer. Results of the contact life cycle test reinforced the importance of selecting a cleaner that also has a contact lubricant in its formulation. For the tape head cleaners, selection of a high purity grade chemical solvent is important. Use of a high purity solvent eliminates problems with deposition of residues on the tape head surface after the solvent evaporates. Heptane and absolute ethyl alcohol were selected as the replacement cleaners for tape heads. Because ethyl alcohol is incompatible with the Capstan rollers contained in the Honeywell unit, heptane should be used to clean these materials. In strain gage bonding, the replacement ODC-free adhesive formulation had superior bonding results than the existing formulation. Replacement cleaners, such as Bio-T-Max and EP-921 also demonstrated acceptable surface preparation prior to bonding. Creep testing and strain gage bonding tests also validated the use of the replacement adhesive formulation and alternative cleaners for strain gage bonding applications.

8.2 Required Changes in Governing Specifications and Standards

Several internal (Martin Marietta) and external (Military Specifications) require revision to include the use of these alternative cleaners. The list of specifications and standards requiring revision is incorporated below:

Number	Title	Revision Required	Responsibility
MP50018	Paint Removal	Include alternatives to TCA as solvent wipe	Martin Marietta
MP50042	Cleaning,Descaling, Passivation	Include alternate degreasing solvents	Martin Marietta
MP50046	Degreasing, Vapor	Include alternatives to vapor degreasing	Martin Marietta
MP50309	General Cleaning of Equipment	Add alternative cleaners to existing list	Martin Marietta
MP50405	Contamination Control Cleaning	Include alternatives to TCA and CFC-113	Martin Marietta
MP72115	Cleaning Procedures for Spacecraft	Include alternatives to halocarbon fluid	Martin Marietta
KSC-C-123	Surface Cleanliness of Fluid Systems	Include replacements to CFC-113, TCE	NASA
MIL-STD-1246	Product Cleanliness Levels	Include allowances for alternative solvents	USAF
SOP 8.1	Cleaning of Parts to EPS 50405	Include alternatives to TCA and CFC-113	MM/EPL
NSP0039	Installation of Instrumentation	Include alternatives to TCA	Martin Marietta

8.3 Implementation Plan

The implementation plan for mechanical cleaning identifies required changes to pertinent standards and specifications (as detailed above), the selection of one of the three cleaning solvents for use, and the elimination of TCA and CFC-113 in all future operations at AF Plant PJKS. As part of this process, Martin Marietta also intends to characterize chemical waste streams resulting from the replacement cleaning chemicals, to ensure the cleaning of metallic hardware results does not generate hazardous waste, especially from dissolved metals such as cadmium or chromium. Incorporation of capital equipment into the PJKS Valve Shop is needed in order to accommodate these new cleaners. Equipment has been identified and includes an ultrasonic cleaning bath and hot water rinse tank. A request has been submitted internally for the acquisition of this equipment (Request For Job #193910, PE770945), but has not yet been approved or funded. Approval and purchase of this equipment will be followed closely to ensure timely installation to support ODC elimination at PJKS.

Because the anti-seize compound currently in use (Fel-Pro C5A) is used as a shop aid, and is not governed by any government, industry, or Martin Marietta specifications or standards, replacement of this material with the identified alternatives is easily implemented. Material substitution has already occurred. Martin Marietta is now using the ODC-free replacement to Fel-Pro C5A and has discarded all lots of the older ODC material. In addition, Martin Marietta will initiate the purchase of one or more of the replacement anti-seize compounds, especially the Dow Molykote 1000, for future applications at the PJKS facility. No change in personnel training or equipment requirements are necessary for implementation of replacement anti-seize compounds.

In electrical cleaning, several of the alternative cleaners are considered a "drop-in" replacement for the ODCs currently in use. Implementation of the alternative contact cleaners requires only elimination of the existing cleaner (Kontact Restorer) and purchase of the desired alternative. Because contact cleaning is not controlled by governing standards or specifications, no action other than material substitution is required. In like manner, the selection of pure grain ethyl alcohol and heptane as tape head cleaners requires only elimination of existing stock of CFC-113 and TCA and replacement with the newly identified chemicals. The use of reagent grade ethyl alcohol does require a tax stamp which Martin Marietta is currently pursuing. In some cases, such as the M-Bond 200 used to bond strain gages, the manufacturer has already issued an ODC-free replacement, and this replacement is now fully implemented by Martin Marietta. In this case, required changes to Non-Standard Process NSP00309 "Installation of Instrumentation" have been identified , which include both the formulation change of the M-Bond 200 adhesive and the specification of alternative cleaners.

8.4 Cost Benefit Analysis

8.4.1 Mechanical Cleaning Payback for implementation of alternative cleaning solvents for mechanical systems is calculated as follows:

Present materials cost of TCA:	150 gal/yr x $7 /gal = $1050/yr
Present materials cost of CFC-113	100 gal/yr x $52/gal = $5200/yr
Total materials cost (existing)	$6250/yr
Approximate labor cost (burdened)	2000hr/yr x $65/hr = $130,000/yr
Total labor and non-labor cost (existing)	$136,250/yr

Future materials cost of Bioact 280	200 gal/yr* x $16/gal = $3200/yr
Future approximate labor cost (burdened)	1600 hr/yr* x $65/hr = $104,000/yr
Total recurring cost	$107,200
Training (non-recurring)	120 hr x $65/hr = $7800
Capital Equipment Costs** (non-recurring)	$30,000
Specification Revision Labor (non-recurring)	160 hr x $65/hr = $10,400
Total non-recurring cost	$48,200

Payback Period = Cost (non-recurring)/(recurring present-recurring cost future)
= \$48,200/(\$136,250/yr-\$107,250/yr)
= 1.7 years

Notes: * improved materials cost and labor efficiency due to fewer solvent changeouts
** capital equipment to include upgrade to ultrasonic cavitation bath

8.4.2 Anti-Seize Replacement Because each of the candidate replacement anti-seize compounds costs about \$10/lb to \$11/lb and are approximately equivalent to the current pricing of the original Fel-Pro C5A, no additional materials cost are incurred by this material substitution. In addition, there are no additional training requirements, required specification changes, or changes in labor costs. Therefore no additional cost burdens or cost savings are incurred by replacing this ODC lubricant with one of the identified ODC-free alternatives. The real cost savings associated with the use of any of these products is due to the protection that they provide for the threaded fasteners. Currently the cost of a two inch 37 degree flair union made of stainless steel is about \$200.00.

8.4.3 Electrical Cleaners Because each of the identified replacement chemicals for contact cleaners, tape head cleaners, and strain gage cleaners and adhesives are "drop-in" replacements, no additional materials costs or labor costs are planned for the implementation of these cleaners. The only non-recurring cost associated with these materials substitutions is the revision to NSP00309 "Installation of Instrumentation" which is expected to consume about 32 labor hours. At an hourly labor rate of \$65/hr, this cost is \$2080.00

9.0 CONCLUSIONS AND RECOMMENDATIONS

In all applications studied (mechanical cleaning processes, anti-seize compounds, and electrical cleaning processes), alternatives to TCA and CFC-113 have been identified that exhibit identical or superior technical performance than the existing ozone depleting chemicals. Many of the identified alternatives are safe to use by operating personnel and are also environmentally friendly. Several possible replacements have been identified for each of the applications so that these chemicals may be similarly qualified for use. The use of multiple replacements allows for minimization of risk of reliance on one chemical system or manufacturer. Implementation of these alternative cleaners and lubricants is now in progress at Air Force Plant PJKS.

Several successful test technologies have been incorporated for the evaluation of alternative contact cleaners and lubricants at Air Force Plant PJKS. This facility has unique applications, such as the cleaning of propulsion hardware for propellant usage, cleaning of electronic test equipment, and the use of high temperature anti-seize compounds. The technologies developed under this contract could be effectively integrated into other solvent reduction initiatives within the Air Force, at other Government Owned Contractor Operated (GOCO) facilities, and in defense and aerospace manufacturing and engineering environments. Refinements in the technologies presented in this report could also be made to accommodate unique technical requirements as specified by requesting agencies.

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APPENDIX A

PRE-SELECTION RESULTS AND INDUSTRIAL HYGIENE REVIEW OF ORIGINAL LIST OF ALTERNATIVE ODC-FREE CLEANERS

Original Mechanical List

CLEANER	REASON FOR ELIMINATION
Alcohols	flammability, poor cleaning
Aquinox SSA	unable to find manufacturer/vendor
Armaclean E-2001	incorrect application-used for flux removal
AVD	cost, process requirements
Axarel 2200	incorrect application-solder paste removal
Axarel 52	hazardous
Axarel 6100	incorrect application-solder paste removal
Bio Clear	no supplier support
Bio-T-Max	poor cleaning results
Bioact Act 280	finalist
BioAct EC7	similar product
Biogenic Regent	finalist
Breakthrough	not recommended by vendor for our application
Brulin 815 GD	semifinalist, materials compatability, cleaning/diethanolamine is an A-level air pollutant
Citra Safe	similar product/flash point problem
Citrasolv	similar product
Citrex	semifinalist-extreme oxidizer compatability problem
Daraclean 220	similar product
Daraclean 235 XL	oxidizer compatability problem
Daraclean 282	semifinalist; poor cleaning results, material compat/glycol ethers are A-level air pollutants
Desoclean 20	low flashpoint
Electrolube	volatility, cost
Electron	similar product
EP-921	finalist
EZE 431	flammability
EZE 662P	incorrect application
Formula 624 GD	incorrect application/respiratory hazard
HFC Vapor	not commercially available
Iso-Prep	flammable
JPX Degreaser	semi-finalist
Kerosene	flammable, residue
MOK	proprietary, lack of support

Original Mechanical List

CLEANER	REASON FOR ELIMINATION
n-methyl pyrrolidone	disposal problems
Partsprep	finalist, leaves residue after loaded
Perfluorocarbons	cost
PF degreaser	poor cleaning results
PF-145-HP	oxidizer compatability problem
PM Acetate	combustable, not oxidizer compatible
Prep Rite	incorrect application
Purasolv ELS	hazardous, disposal
Safe-strip	incorrect application
Safer Bio Degreaser	similar product
Safety-Kleen	disposal, future disposal problems
Shopmaster A/C	discontinued by manufacturer
Shopmaster FF	poor cleaning/glycol ethers are A-level air pollutants
Shopmaster LpH	poor cleaning results
Skysol	not recommended by vendor
Sonacor 103	glycol ether
Teksol EP	incorrect application/flashpoint problem
Turco 3878 LC-NC	high chromium/diethylene glycol monobutyl ether is an A-level air pollutant.
Turco 4215-NC-LT	similar product
Turco 6759	flammable/hazardous waste
Turco 6776	incorrect application
Turco 6778	incorrect application-paint remover
Turco 6780	similar product
Turco 6813	incorrect application
Vortex	no supplier support
X-Caliber	incorrect appplication

TABLE OF PROPOSED OZONE DEPLETING SOLVENTS ALTERNATIVES
NUSTO 018 AND 018A
INDUSTRIAL HYGIENE REVIEW

July 14, 1994

MATERIAL	MSDS #	MANUFACTURER	RATING[1]			INDUSTRIAL HYGIENE REVIEW	STATUS 2	RECOMMENDATIONS
			F	T	R			
ANTI-SEIZE SPECIAL	T17370	ANTI-SEIZE TECH	1	1	0		(RM)	OK
ANTI-SEIZE MOLY-LIT	T17369	ANTI-SEIZE TECH	1	(1)	0	CONTAINS LEAD	(SUR)	NOT RECOMMENDED
ARMACLEAN		ARM AND HAMMER		(DS)			(P)	
BIO CLEAR		BIO CHEM						
BIO-T-MAX	T09469	BIO CHEM	2	0	0	D-LIMONENE	(RM)	OK
M.O.K	T12076	BOEING	1	(1)	0	NO INGREDIENT DATA	(P)	
BRULIN 815 GD		BRULIN		(DS)			(P)	
BUCKEYE SHOPMASTER	T17416	BUCKEYE	0	1	0	GLYCOL ETHERS	(RM)	OK
SHOPMASTER FF	T17417	BUCKEYE	1	1	0		(RM)	OK
SHOPMASTER AC	T17418	BUCKEYE	0	1	0	GLYCOL ETHER	(RM)	OK
ANTI-SEIZE 1000	T17176	DOW CORNING	1	2	0		(RM)	OK
AXAREL 2200		DUPONT						
AXAREL 6100		DUPONT						
AXAREL 52	T14814	DUPONT	1	2	0	1.5 PPM TLV (9% DIISOBUTYL DBE)	(SUR)	NOT RECOMMENDED
DESOCLEAN 20		DUPONT		(DS)			(P)	
VORTEX	T17399	ECOLINK SENTRY CHEM	2	1	0	D-LIMONENE	(RM)	OK
EZE 662P		EZE PRODUCTS	2	1	0	FLAMMABILITY FP 105 F	(NR)	
1-METHYL-2-PYRROLIDONE	C00512	GENERIC	1	2	1	MANUFACTURER RECOMMENDS A TLV OF 100PPM	(RM)	OK
ALCOHOLS IPA ETHANOL		GENERIC		(DS)		NEED SPECIFIC ALCOHOLS	(P)	
HFC VAPOR		GENERIC		(DS)		NEED SPECIFIC HFC	(P)	
KEROSENE	C00416	GENERIC	2	2	0	FLAMMABILITY AND SKIN IRRITATION	(NR)	NOT RECOMMENDED
P.M. ACETATE	C03892	GENERIC	2	2	O	GLYCOL ETHER (TOXICITY IN QUESTION)	(NR)	LIMITED USE NOT FULL ALTERNATIVE
PERFLUOROCARBONS		GENERIC		(DS)		NEED SPECIFIC PRODUCT NAME	(P)	
SUPER BIO SCUB	T17375	GLINTON	1	(1)	0	NO INGREDIENT DATA	(P)	
BREAKTHROUGH	T17413	INLAND TECH				NEED TO REVIEW	(P)	
CITRA SAFE B056	T17412	INLAND TECH				NEED TO REVIEW	(P)	
CITREX	T17414	INLAND TECH	2	1	0	GLYCOL ETHERS (1-BUTYOXY-2-PROPANOL	(P)	NEED TO RESEARCH
EP-921	T16182	INLAND TECH	2	1	0		(RM)	OK
ISO PREP	T17410	INLAND TECH	2	1	0	(104°F) LOW FLASH POINT	(NR)	NOT RECOMMENDED

MATERIAL	MSDS #	MANUFACTURER	RATING[1]			INDUSTRIAL HYGIENE REVIEW	STATUS[2]	RECOMMENDATIONS
			F	T	R			
SKYSOL	T17420	INLAND TECH	2	1	0	D-LIMONENE	(RM)	OK
TEKSOL	T17411	INLAND TECH	2	1	0	(112°F) LOW FLASH POINT	(NR)	NOT RECOMMENDED
X-CALIBER		INLAND TECH						
JPX DEGREASER		JAYNE PRODUCTS						
ANTI-SEIZE 767	T08344	LOCTITE	1	1	1		(RM)	OK
CEE BEE 777		MCGREAN CHEMICAL		(DS)		NEED SPECIFIC PRODUCT NAME	(P)	
PF	T16024	P-T TECHNOLOGIES	2	0	0		(RM)	OK
PF-145HP	T15659	P-T TECHNOLOGIES	2	(I)	0	NEED TOXICITY DATA	(P)	
AVD (ADVANCED SYS.		PETROFERM (NO ADD.)		(DS)			(P)	
BIOACT 120		PETROFERM						
ELECTROLUBE		PHOENIX SYSTEMS		(DS)			(P)	
PURASOLV ELS		PURAC		(DS)			(P)	
FORMULA 624GD		QUAKER CHEMICAL	0	2	0	SODIUM HYDROXIDE	(SUR)	
BIOGENIC REAGENT		ROCHESTER MIDLAND	?	2	0	FLAMMABILITY	(P)	
EZE 431		ROCKESTER MIDLAND						
ELECTRON	T17400	SENTRY CHEMICAL	2	1	0	(110°F) LOW FLASH POINT	(NR)	NOT RECOMMENDED
ELECTRON		SENTRY	2	(I)	0	NEED MORE TOX DATA		
PARTSPREP DEGREASER		SENTRY	1	2	0	NEED MORE TOX DATA	(P)	
PREP RITE	T17421	SENTRY		(I)		NEED MORE TOX DATA	(P)	
SAFE-STRIP		SENTRY	2	(I)	0	NEED MORE TOX DATA		
TURCO 6759	T17404	TURCO	4	2	0	FLAMMABLE	(NR)	NOT RECOMMENDED
TURCO 6780	T17405	TURCO	2	2	0		(RL)	OK
DARACLEAN 220		W. R. GRACE		(DS)				
DARACLEAN 282	T12602	W. R. GRACE	1	(I)	0	GLYCOL ETHERS (TOXICITY IN QUESTION)	(P)	
DARACLEAN 235XL		W. R. GRACE		(DS)			(P)	
AQUINOX SSA		Y						
CITRISOLV		Y		(DS)			(P)	
SONACORE 102M		Y						
		z						

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[1] RATING (F=FLAMMABILITY, T=TOXICITY, R=REACTIVITY- RATINGS 0=LOW HAZARD 4=HIGH HAZARD), (DS)=NEED MATERIAL SAFETY DATA, (I)=INGREDIENT MISSING/INADEQUATE (TD)=TOXICITY DATA LACKING OR INADEQUATE.

[2] STATUS AND RECOMMENDATIONS; (NA)=NOT APPROVED, (NR)=NOT RECOMMENDED, (SUR) STRONG USE RESTRICTIONS, (RL)=RECOMMENDED WITH LIMITED RESTRICTIONS, (RM)=RECOMMENDED WITH LITTLE RESTRICTIONS, (P)=PENDING FURTHER REVIEW.

APPENDIX B
MECHANICAL CLEANING TEST RESULTS

CLEANER	temperature	concentration %	rating		NOTES
1,1,1, TCA	ambient	100	4.9	*	sl WB
Bio T Max	ambient	100	3.0		Drilube not removed
BioAct 280	ambient	100	4.7	*	sl WB
Biogenic Regent	ambient	100	4.0	*	sl Drilube residue,sl WB
Citrex	ambient	100	4.9	*	sl WB
Daraclean 235XL	ambient	30	3.0		Drilube residue, WB
Daraclean 282	ambient	25	4.0	*	sl Drilube residue,sl WB
Electron	ambient	100	2.5		Drilube residue, WB
EP-921	ambient	100	4.8	*	WB
Formula 624 GD	150° F	15	3.8		Drilube residue, WB
Brulin 815 GD	ambient	100	4.0	*	sl Drilube residue, sl WB
CFC-113	ambient	100	4.9	*	sl WB
JPX Degreaser	ambient	100	4.9	*	sl WB
n-methylpyrrolidone	ambient	100	4.3	XX	Drilube residue, WB
Partsprep	140° F	100	5.0	*	all contaminants removed
PF Degreaser	ambient	100	2.5		Drilube not removed, WB
PF-145-HP	ambient	100	2.0		Drilube and Grease residue
Shopmaster FF	ambient	50	3.0		Drilube residue, WB
Shopmaster A/C	ambient	50	4 .0	ZZ	sl Drilube residue,sl WB
Shopmaster LpH	ambient	50	1.5		significant Drilube and Grease residue
Turco 3878	ambient	20	4.5	YY	sl Drilube residue,sl WB
Turco 3878 LF-NC	130° F	20	3.8		Drilube residue, WB

*	selected for additional testing
XX	eliminated for chemical and disposal concerns
YY	eliminated for flammability concerns
ZZ	eliminated, manufacturer discontinued production
sl	slight
WB	water break

Figure B-1 Prescreening Data summary

Figure B-2

freshly contaminated coupons	visual	blacklight	water	total
PARTSPREP	0	2	0	2
BIOACT 280	0	1	6	7
JPX	3	1	3	7
TRICHLOROETHANE	11	7	6	24
FREON	9	12	5	26
BIOGENIC REGENT	3	4	36	43
FORMULA 815 GD	17	18	9	44
EP-921	3	6	36	45
CITREX	3	7	36	46
DARACLEAN 282	15	19	27	61

aged coupons	visual	blacklight	water	total
PARTSPREP	0	2	0	2
JPX	3	0	3	6
TRICHLOROETHANE	6	6	6	18
BIOACT 280	0	1	18	19
FREON	13	7	7	27
FORMULA 815 GD	13	18	9	40
BIOGENIC REGENT	3	4	36	43
CITREX	3	7	36	46
EP-921	6	6	36	48
DARACLEAN 282	15	19	27	61

Contaminants: Grease, Hydraulic Oil, Drilube, Dykem.

Ultrasonic Cleaning Power Test Data (summation of all inspections)

Aged Coupons			TIME (min)			
Cleaner	0	1	3	5	15	Total
PARTSPREP	27	2	0	0	0	2
JPX	27	6	0	0	0	6
TRICHLOROETHANE	27	18	0	0	0	18
BIOACT 280	27	7	6	6	0	19
FREON	27	15	9	3	0	27
FORMULA 815 GD	27	24	12	4	0	40
BIOGENIC REGENT	27	16	9	9	9	43
CITREX	27	17	11	9	9	46
EP-921	27	21	9	9	9	48
DARACLEAN 282	27	24	24	13	0	61

Contaminants: Grease, Hydraulic Oil, Drilube. Dykem was applied to coupons but not considered as a contaminant during test

Figure B-3

Stirred Cleaning Power Test Data (summation of all inspections)						
Aged Coupons						
				TIME (min)		
CLEANER	0	5	10	15	20	30	TOTAL
PARTSPREP	27	8	8	2	1	0	19
CITREX	27	8	8	3	2	2	23
EP-921	27	9	10	7	6	2	34
JPX	27	10	9	9	6	5	39
BIOACT 280	27	10	9	9	9	6	43
DARACLEAN 282	27	21	9	9	9	3	51
FORMULA 815 GD	27	21	18	15	15	4	73
BIOGENIC REGENT	27	21	21	15	13	13	83
FREON	27	18	18	18	18	18	90
TCA	27	18	18	18	18	18	90
Contaminants: Grease, Hydraulic Oil, Drilube. Dykem was applied to coupons							
but not considered as a contaminant during test.							
Maximum Number of Points = 3 points x 3 contaminants x 3 inspections = 27							

CLEANER	SOLUTION	METHOD	COUPON	EXPOSURE	COUPON	visual	visual	visual	visual
	%		ID #	DATE	TREATMENT				
BIOACT 280	100	ULTRASONIC	6	11/23/93	ambient 1	pass	pass	pass	pass
BIOACT 280	100	ULTRASONIC	6	1/4/94	heated 7 days	pass	pass	pass	pass
BIOGENIC REGENT	100	ULTRASONIC	8	11/23/93	ambient 2	DL	pass	pass	pass
BIOGENIC REGENT	100	ULTRASONIC	8	1/4/94	heated 7 days	DL	pass	pass	pass
CITREX	100	ULTRASONIC	3	11/23/93	ambient 3	sl DL, sl G x	sl DL x	pass	pass
CITREX	100	ULTRASONIC	3	1/4/94	heated 7 days	sl DL, sl G x	sl DL x	pass	pass
DARACLEAN 282	25	ULTRASONIC	9	11/23/93	ambient 4	DL,G	DL,G	DL	pass
DARACLEAN 282	25	ULTRASONIC	9	1/4/94	heated 7 days	DL,G	DL,G	DL	pass
EP-921	100	ULTRASONIC	5	11/23/93	ambient 5	DL	pass	pass	pass
EP-921	100	ULTRASONIC	5	1/4/94	heated 7 days	DL,G	pass	pass	pass
FORMULA 815GD	25	ULTRASONIC	10	11/23/93	ambient 6	DL,G	DL,G	DL	pass
FORMULA 815GD	25	ULTRASONIC	10	1/4/94	heated 7 days	DL,G	DL,G	sl DL	pass
FREON	100	ULTRASONIC	2	11/23/93	ambient 7	DL, G	DL	pass, y	pass, y
FREON	100	ULTRASONIC	2	1/4/94	heated 7 days	DL, G,H	DL	sl DL	pass, y
JPX	100	ULTRASONIC	4	11/23/93	ambient 8	hvy DL	pass	pass	pass
JPX	100	ULTRASONIC	4	1/4/94	heated 7 days	hvy DL	pass	pass	pass
PARTSPREP	100	ULTRASONIC	7	11/23/93	ambient 9	pass	pass	pass	pass
PARTSPREP	100	ULTRASONIC	7	1/4/94	heated 7 days	pass	pass	pass	pass
TRICLOROETHANE	100	ULTRASONIC	1	11/23/93	ambient	DL, G	sl DL, G	sl DL	pass
TRICLOROETHANE	100	ULTRASONIC	1	1/4/94	heated 7 days	DL, G	pass	pass	pass

Figure B-4 Ultrasonic Cleaning Results

CLEANER	black light	black light	black light	black light	water break	water break	water break	water break
BIOACT 280	sl G	pass	pass	pass	fail G DL	pass ****	pass *	pass *
BIOACT 280	sl G	pass	pass	pass	fail G DL	fail G DL	fail G DL	pass *
BIOGENIC REGENT	DL,sl G	pass	pass	pass	fail *	fail *	fail	fail *
BIOGENIC REGENT	DL,sl G	pass	pass	pass	fail *	fail *	fail *	fail *
CITREX	DL,G x	s DL, x	pass	pass	fail	fail	fail	fail
CITREX	DL,G x	s DL, x	pass	pass	fail	fail	fail	fail
DARACLEAN 282	fail all	fail all	sl DL	pass	fail	fail	fail	pass *
DARACLEAN 282	fail all	fail all	sl DL	pass	fail	fail	fail	pass *
EP-921	DL, G	pass	pass	pass	fail *	fail *	fail	fail *
EP-921	DL, G	pass	pass	pass	fail *	fail *	fail	fail *
FORMULA 815GD	fail all	DL, G	DL	pass *	fail	pass *	pass *	pass *
FORMULA 815GD	fail all	DL, G	DL	pass *	fail	pass *	pass *	pass *
FREON	DL, G	DL	DL	pass	sl DL, sl G	sl DL, sl G	sl DL	pass
FREON	DL	DL	sl DL	pass	DL	DL	sl DL	pass
JPX	sl DL	pass	pass	pass	hvy DL	pass ,*	pass ,*	pass ,*
JPX	pass	pass	pass	pass	hvy DL	pass ,*	pass ,*	pass ,*
PARTSPREP	sl DL, sl G	pass	pass	pass	pass,*	pass,*	pass,*	pass,*
PARTSPREP	sl G	pass	pass	pass	pass,*	pass,*	pass,*	pass,*
TRICLOROETHANE	DL, G	sl DL	pass	pass	DL,G	pass, *	pass, *	pass, *
TRICLOROETHANE	DL, G	pass	pass	pass	DL,G	pass, *	pass, *	pass, *

Figure B-4 Ultrasonic Cleaning Results

CLEANER	
BIOACT 280	50% of Dykem remains.
BIOACT 280	* coating requiring alcohol rinse and blow drying with air to remove most of it
BIOGENIC REGENT	Dykem not affected.
BIOGENIC REGENT	* coating requiring alcohol rinse and blow drying with air to remove most of it
CITREX	x heavy coating of DL and G not removed. .
CITREX	Residue requiring futher cleaning present. Dykem removed after 1 minute.
DARACLEAN 282	Soution heated to 130o F. Dykem removed 95% after 1 min. 100% 3 min,
DARACLEAN 282	Water break requires alcohol rinse and air dry.
EP-921	Surface coating after cleaning, caused water break to fail.
EP-921	Dykem removed at 5 mins. Residue requiring futher cleaning
FORMULA 815GD	$, heavy DL coating not removed
FORMULA 815GD	Solution heated to 130 o F. 75 % Dykem removed
FREON	y Heavy DL not removed, 25% Dykem Removed
FREON	
JPX	* Surface coating, after cleaning, requires alcohol rinse and blow dry.
JPX	Dykem still present
PARTSPREP	* Surface coating, after cleaning, requires alcohol rinse and blow dry.
PARTSPREP	Dykem not removed
TRICLOROETHANE	* Surface coating, after cleaning, requires alcohol rinse and blow dry.
TRICLOROETHANE	Dykem not removed

Figure B-4 Ultrasonic Cleaning Results

				Compatability All Samples Included					
time (hrs)	0	24	168	336	504	672	840	1008	total
EP 921	0	0	0	1	3	3	5	5	17
CFC-113	0	0	1	2	3	3	9	10	28
TCA	0	1	2	2	4	9	11	11	40
Biogenic Regent	0	2	2	2	4	6	12	15	43
BioAct 280	0	0	2	2	2	8	15	20	49
Citrex	0	7	7	7	13	14	20	23	91
Brulin 815 GD	0	10	10	11	12	16	20	24	103
Partsprep	0	13	14	14	14	20	23	24	122
JPX	0	13	15	27	29	30	33	34	181
Daraclean 282	0	18	24	27	29	31	31	31	191
				Compatability Data less O-Ring Data					
time (hrs)	0	24	168	336	504	672	840	1008	
EP 921	0	0	0	0	1	1	3	3	8
TCA	0	0	0	0	0	3	5	5	13
Freon	0	0	1	2	2	2	5	5	17
Biogenic Regent	0	1	1	1	1	3	9	12	28
BioAct 280	0	0	1	1	1	7	12	17	39
Citrex	0	1	1	1	7	8	11	14	43
Partsprep	0	7	8	8	8	14	17	18	80
Brulin 815 GD	0	10	10	11	12	16	20	24	103
JPX	0	10	12	24	26	26	29	30	157
Daraclean 282	0	18	24	27	29	31	31	31	191

Figure B-5 Compatibility Test Results

			Compatability data for metallic samples only						
time (hrs)	0	24	168	336	504	672	840	1008	total
TCA	0	0	0	0	0	1	3	5	9
EP 921	0	0	0	1	2	3	4	4	14
Citrex	0	1	1	1	1	2	4	6	16
CFC-113	0	0	1	2	2	2	5	6	18
Biogenic Regent	0	1	1	1	1	3	7	8	22
BioAct 280	0	0	1	1	1	5	11	17	36
Partsprep	0	1	2	2	2	8	11	11	37
Brulin 815 GD	0	10	10	11	14	16	22	23	106
JPX	0	10	14	22	24	25	28	29	152
Daraclean 282	0	18	24	27	29	31	31	31	191

Figure B-5 Compatibility Test Results

LOADING TEST

OIL CONTAMINATION						
	All tests started with 300 mls of cleaning solution.					
	oil added	coupon #	stirring	temp F	result	notes
BIO-ACT 280	15 mls	1	2 min	76.8	pass BL	
	15 mls	6	3 min	79.2	pass BL	ultra sonic 30 sec. hot soapy H2O
	15 mls	12	3 min	83.8	pass BL	ultra sonic 30 sec. hot soapy H2O
	15 mls	21	3 min	83.9	pass BL	ultra sonic 30 sec. hot soapy H2O
	15 mls	17	3 min	83.7	pass BL	ultra sonic 30 sec. hot soapy H2O
	15 mls	4	3 min	83.8	pass BL	ultra sonic 30 sec. hot soapy H2O
	15 mls	5	3 min		fail WB	ultra sonic 30 sec. hot soapy H2O
	15 mls	10	3 min	81.1	fail WB	ultra sonic 30 sec. hot soapy H2O
	15 mls	10	3 min		fail WB	ultra sonic 30 sec. hot soapy H2O
		10	3 min		sl fail WB	ultra sonic 30 sec. hot soapy H2O
		10	3 min		sl fail WB	ultra sonic 30 sec. hot soapy H2O
BIOGENIC REGENT	15 mls	2	2 min	75.5	pass BL	
	15 mls	7	3min	75.6	pass BL	ultra sonic 30 sec. hot soapy H2O
	15 mls	19	3min	77.5	pass BL	ultra sonic 30 sec. hot soapy H2O
	15 mls	11	3min	77.2	pass BL	ultra sonic 30 sec. hot soapy H2O
	15 mls	20	3min		pass BL	ultra sonic 30 sec. hot soapy H2O
	15 mls	16	3min	76.2	pass BL	ultra sonic 30 sec. hot soapy H2O
	15 mls	18	3min	75.8	pass BL	ultra sonic 30 sec. hot soapy H2O
	15 mls	4	3min		fail WB	ultra sonic 30 sec. hot soapy H2O
	15 mls	9	3min	75.3	fail WB	ultra sonic 30 sec. hot soapy H2O
		9	3min		fail WB	ultra sonic 30 sec. hot soapy H2O
		9	3min		fail WB	ultra sonic 30 sec. hot soapy H2O
		9	3min		fail WB	ultra sonic 30 sec. hot soapy H2O
OIL CONTAMINATION	oil added	coupon #	stirring	temp F	result	notes
CITREX	15 mls	14	2 min	74	fail BL	

Figure B-6 Loading Test Data
page1

LOADING TEST

		14	3 min		pass	
	15 mls	17	3 min	77	fail	oil migrated on bottom edge
		17	4 min		pass	ultra sonic 30 sec. hot soapy H2O
	30 mls	16	3 min	80	pass	ultra sonic 30 sec. hot soapy H2O
	30 mls	6	3 min		fail	ultra sonic 30 sec. hot soapy H2O
		8	3 min		fail	ultra sonic 30 sec. hot soapy H2O
		8	3 min		fail	ultra sonic 30 sec. hot soapy H2O
		8	3 min		sl fail	ultra sonic 30 sec. hot soapy H2O
	30 mls	3	3 min		pass ?	water rinse
	30 mls	15	3 min		fail	ultra sonic 1 min. hot soapy H2O
Daraclean 282	15 mls	5	2 min	142.7	pass	
	15 mls	9	3 min	173.3	sl fail BL	ultra sonic 1 min. hot soapy H2O
			4 min	165.9	sl fail BL	ultra sonic 1 min. hot soapy H2O
			4 min	155.9	sl fail BL	ultra sonic 1 min. hot soapy H2O
			4 min		sl fail BL	ultra sonic 1 min. hot soapy H2O
			4 min	147.7	pass	ultra sonic 1 min. hot soapy H2O
	15 mls	13	3 min	164.3	pass	ultra sonic 1 min. hot soapy H2O
	15 mls	15	3 min	162	pass	water rinse only
	15 mls	2	3 min		fail BL,WB	water rinse only
	15 mls	6	3 min	133	fail BL,WB	water rinse only
		6	3 min	139	fail BL,WB	
		6	6 min		fail BL,WB	
OIL CONTAMINATION	oil added	coupon #	stirring	temp F	result	notes
EP-921	15 mls	3	2 min	70	fail	10 % remaining BL
		3	3 min		pass	
	15 mls	18	3 min	74	fail	oil coating, ultrasonic 1 min soapy H2O

Figure B-6 Loading Test Data
page2

LOADING TEST

			4 min		fail	oil coating, ultrasonic 1 min soapy H2O
	30 mls	14	3 min	79	fail	oil coating, ultrasonic 1 min soapy H2O
	30 mls	6	3 min		fail	10 % oil coating, ultrasonic 1 min soapy H2O
		9	3 min	80	fail	50 % oil coating, ultrasonic 1 min soapy H2O
		9	3 min		fail	50 % oil coating, ultrasonic 1 min soapy H2O
		9	3 min		fail	50 % oil coating, ultrasonic 1 min soapy H2O
Formula 815GD	15 mls	4	2 min	153.6	pass	
	15 mls	10	3 min	169.2	fail V,BL	ultrasonic 1 min soapy H2O
		10	4 min	162.3	fail V,BL	ultrasonic 1 min soapy H2O
		10	4 min	152	fail V,BL	ultrasonic 1 min soapy H2O
		10	4 min	134	fail V,BL	ultrasonic 1 min soapy H2O
		10	4 min	146.6	pass	ultrasonic 1 min soapy H2O
	15 mls	14	3 min	175.4	pass	ultrasonic 1 min soapy H2O
	15 mls	16	3 min		sl fail WB	ultrasonic 1 min soapy H2O
	15 mls	1	3 min		sl fail WB	ultrasonic 1 min soapy H2O
	15 mls	7	3 min		fail WB BL	ultrasonic 1 min soapy H2O
		7	3 min		fail WB BL	ultrasonic 1 min soapy H2O
		7	3 min		fail WB BL	ultrasonic 1 min soapy H2O
		7	6 min		fail WB BL	ultrasonic 1 min soapy H2O
OIL CONTAMINATION	oil added	coupon #	stirring	temp F	result	notes
Freon	15 mls	1	2 min	68	fail BL	
			3 min		pass	
	15 mls	20	3 min	71	fail	ultrasonic 1 min soapy H2O
			4 min		fail	ultrasonic 1 min soapy H2O

Figure B-6 Loading Test Data
page3

LOADING TEST

		13	3 min	73	sl fail	ultrasonic 1 min soapy H2O
	30 mls	6	3 min	75	fail	ultrasonic 1 min soapy H2O
		11	3 min	75	fail	ultrasonic 1 min soapy H2O
		11	3 min		fail	ultrasonic 1 min soapy H2O
JPX	15 mls	5	2 min	74	fail BL	
		5	3 min		sl fail BL	
		5	3 min		fail BL	
	15 mls	19	3 min	77	fail BL	
			4 min		pass	ultrasonic 1 min soapy H2O
	15 mls	15	3 min	83	pass	ultrasonic 1 min soapy H2O
	30 mls	6	3 min		fail BL	ultrasonic 1 min soapy H2O
		7	3 min	86	sl fail BL	ultrasonic 1 min soapy H2O
		7	3 min		pass	ultrasonic 1 min soapy H2O
	30 mls	2	3 min		pass	ultrasonic 1 min soapy H2O
	30 mls	18	3 min		pass	ultrasonic 1 min soapy H2O
	30 mls	9	3 min		pass	ultrasonic 1 min soapy H2O
	30 mls	4	3 min		pass	ultrasonic 1 min soapy H2O
	30 mls	5	3 min		pass	ultrasonic 1 min soapy H2O
OIL CONTAMINATION	oil added	coupon #	stirring	temp F	result	notes
Partsprep	15 mls	4	2 min	76.8	fail	oil floating on surface and in vortex
	15 mls	8	3 min	76	fail V BL	1 min ultrasonic hot soapy water
		8	4 min	78.5	fail V BL	1 min ultrasonic hot soapy water
		8	4 min	79.3	fail V BL	1 min ultrasonic hot soapy water
		8	4 min		fail V BL	1 min ultrasonic hot soapy water

Figure B-6 Loading Test Data
page4

		15	3 min		pass	1 min ultrasonic hot soapy water
	15 mls	17	3 min	79.3	fail WB	Hot H2O rinse only
	15 mls	3	3 min		fail WB	1 min ultrasonic hot soapy water
		8	3 min		fail V WB	1 min ultrasonic hot soapy water
		8	3 min		fail V WB	1 min ultrasonic hot soapy water
		8	6 min		fail V WB	1 min ultrasonic hot soapy water
Trichloroethane	15 mls	2	2 min	71	fail WB	
		2	3 min		pass	
	15 mls	21	3 min	75	pass ?	
		21	4 min		pass	
	15 mls	12	3 min	75	pass	1 min ultrasonic hot soapy water
	30 mls	6	3 min		pass	1 min ultrasonic hot soapy water
	30 mls	10	3 min	76	fail BL	1 min ultrasonic hot soapy water
		10	3 min		fail BL	1 min ultrasonic hot soapy water
DRILUBE CONTAMINATION	All tests started with 300 mls of cleaning solution.					
	oil added	coupon #	stirring	temp F	result	notes
Biogenic Regent	none	9	3 min	70.6	fail WB	
	none	9	3 min	73.5	fail WB	
	none	9	3 min		fail WB	
	none	9	3 min		fail WB	

Figure B-6 Loading Test Data
page5

LOADING TEST

BioAct 280		16	3 min	70.5	pass	
	15 mls	15	3 min		pass	
	15 mls	19	3 min		pass	
	15 mls	11	3 min		fail	
		11	3 min		pass	
	15 mls	13	3 min		sl fail	
		13	3 min		pass	
	15 mls	6	3 min		sl fail	
		6	3 min		pass	solution remained clear
Citrex		17	3 min		pass	
	15 mls	4	3 min		pass	
	15 mls	3	3 min		pass	
	15 mls	2	3 min		fail	
		2	3 min		pass	
	15 mls	20	3 min		fail	
		20	3 min		fail	
		20	3 min		fail	
DRILUBE CONTAMINATION	oil added	coupon #	stirring	temp F	result	notes
Daraclean 282		5	3 min	129	pass	
	15 mls	21	3 min		fail	
		21	3 min	140	pass	
	15 mls	16	3 min	132	fail	
		16	3 min		fail	
		16	3 min		fail	
		16	3 min		fail	

Figure B-6 Loading Test Data
page6

LOADING TEST

EP-921		8	3 min	70.1	pass	
	15 mls	1	3 min		fail	
		1	3 min		fail	
		1	3 min		fail	
		1	3 min		fail	coupon surface coated with oil
Formula 815GD		15	3 min	152	pass	
	15 mls	12	3 min		sl fail BL	
		12	3 min	140.6	pass	
	15 mls	1	3 min	132.3	fail	
		1	3 min		fail	
		1	3 min		fail	
		1	3 min		fail	
Freon		7	3 min	71.2	fail	
		7	3 min		fail	
		7	3 min		fail	
		7	6 min		fail	
DRILUBE CONTAMINATION	oil added	coupon #	stirring	temp F	result	notes
JPX		14	3 min	68.6	fail	
		14	3 min		fail	
		14	3 min		fail	
		14	3 min		fail	Drilube not affected
Partsprep		18	3 min	76.1	pass	
	15 mls	6	3 min		pass	
	15 mls	23	3 min		fail	

Figure B-6 Loading Test Data
page7

LOADING TEST

		23	3 min		fail	entire surface coated with oil
Tricloroethane		10	3 min	69.8	fail	
		10	3 min		fail	
		10	3 min		fail	
		10	6 min		fail	

Figure B-6 Loading Test Data
page8

APPENDIX C
ANTI-SEIZE TEST RESULTS

Figure C-1

ASTM A193 Grade 8 Class 2					
			Torque (ft-lbs)		
load (lbs)	Fel-Pro C5A (Existing)	Fel-Pro C5A (Replacement)	Anti-Seize Special	Loctite A-S 767	Dow Molykote 1000
0	0	0	0	0	0
5000	67	72	64	67	70
10000	137	145	123	136	146
15000	195	211	179	195	190
20000	245	290	239	279	253
25000	280	353	300	348	330
30000	343	400	372	445	415
load(lbs)	Fel-Pro C5A (Existing)	Dow Molykote 1000	Dri-Lube Type 822		
0	0	0	0		
5000	67	70	74		
10000	137	146	153		
15000	195	190	242		
20000	245	253	332		
25000	280	330	397		
30000	343	415	450		

Figure C-1 Bolt Force vs Torque Tests
ASTM A193
Page 1

CLAMPING FORCE vs TORQUE

BOLT DESCRIPTION								
MATERIAL: ASTM A193 GRADE 8 CLASS 2								
ANTI-SEIZE MATERIAL USED: FEL-PRO C5-A (NEW)								
BOLT LOAD	TORQUE APPLIED (FT-LBS) #1	TORQUE APPLIED (FT-LBS) #2	TORQUE APPLIED (FT-LBS) #3	TORQUE APPLIED (FT-LBS) #4	FRICTION FACTOR #1	FRICTION FACTOR #2	FRICTION FACTOR #3	FRICTION FACTOR #4
0	0	0	0	0	0.00	0.00	0.00	0.00
5,000	72	71	67	61	0.23	0.23	0.21	0.19
10,000	145	132	134	128	0.23	0.21	0.21	0.20
15,000	211	183	188	177	0.22	0.19	0.20	0.19
20,000	290	238	254	225	0.23	0.19	0.20	0.18
25,000	353	298	325	277	0.22	0.19	0.21	0.18
30,000	400	400	405	358	0.21	0.21	0.21	0.19
BOLT DESCRIPTION								
MATERIAL: SAE GRADE 8								
ANTI-SEIZE MATERIAL USED: FEL-PRO C5-A (NEW)								
BOLT LOAD	TORQUE APPLIED (FT-LBS) #1	TORQUE APPLIED (FT-LBS) #2	TORQUE APPLIED (FT-LBS) #3	TORQUE APPLIED (FT-LBS) #4	FRICTION FACTOR #1	FRICTION FACTOR #2	FRICTION FACTOR #3	FRICTION FACTOR #4
0	0	0	0	0	0.00	0.00	0.00	0.00
5,000	51	55	61	47	0.16	0.17	0.19	0.15
10,000	118	107	125	101	0.19	0.17	0.20	0.16
15,000	170	160	178	143	0.18	0.17	0.19	0.15
20,000	220	210	243	203	0.17	0.17	0.19	0.16
25,000	270	270	312	252	0.17	0.17	0.20	0.16
30,000	320	340	390	295	0.17	0.18	0.21	0.16
BOLT DESCRIPTION								
MATERIAL: ASTM A193 GRADE 8 CLASS 2								
ANTI-SEIZE MATERIAL USED: FEL-PRO C5-A (ORIGINAL)								
BOLT LOAD	TORQUE APPLIED (FT-LBS) #1	TORQUE APPLIED (FT-LBS) #2	TORQUE APPLIED (FT-LBS) #3	TORQUE APPLIED (FT-LBS) #4	FRICTION FACTOR #1	FRICTION FACTOR #2	FRICTION FACTOR #3	FRICTION FACTOR #4
0	0	0	0	0	0.00	0.00	0.00	0.00
5,000	67	56	56	55	0.21	0.18	0.18	0.17
10,000	137	106	109	110	0.22	0.17	0.17	0.17
15,000	195	152	156	160	0.21	0.16	0.17	0.17
20,000	245	200	212	215	0.19	0.16	0.17	0.17
25,000	280	239	279	270	0.18	0.15	0.18	0.17
30,000	343	302	342	350	0.18	0.16	0.18	0.19
BOLT DESCRIPTION								
MATERIAL: SAE GRADE 8								
ANTI-SEIZE MATERIAL USED: FEL-PRO C5-A (ORIGINAL)								
BOLT LOAD	TORQUE APPLIED (FT-LBS) #1	TORQUE APPLIED (FT-LBS) #2	TORQUE APPLIED (FT-LBS) #3	TORQUE APPLIED (FT-LBS) #4	FRICTION FACTOR #1	FRICTION FACTOR #2	FRICTION FACTOR #3	FRICTION FACTOR #4
0	0	0	0	0	0.00	0.00	0.00	0.00
5,000	57	53	53	47	0.18	0.17	0.17	0.15
10,000	120	100	107	91	0.19	0.16	0.17	0.14
15,000	175	135	143	142	0.19	0.14	0.15	0.15
20,000	220	190	195	210	0.17	0.15	0.15	0.17
25,000	290	238	250	255	0.18	0.15	0.16	0.16
30,000	355	300	308	295	0.19	0.16	0.16	0.16

Figure C-2
Friction Factor, Bolt Force Test
Page 1

CLAMPING FORCE vs TORQUE

BOLT DESCRIPTION								
MATERIAL: ASTM A193 GRADE 8 CLASS 2								
ANTI-SEIZE MATERIAL USED: ANTI-SEIZE SPECIAL								
BOLT	TORQUE	TORQUE	TORQUE	TORQUE	FRICTION	FRICTION	FRICTION	FRICTION
LOAD	APPLIED	APPLIED	APPLIED	APPLIED	FACTOR	FACTOR	FACTOR	FACTOR
	(FT-LBS)	(FT-LBS)	(FT-LBS)	(FT-LBS)	#1	#2	#3	#4
	#1	#2	#3	#4				
0	0	0	0	0	0.00	0.00	0.00	0.00
5,000	64	56	55	40	0.20	0.18	0.17	0.13
10,000	123	100	98	97	0.20	0.16	0.16	0.15
15,000	179	150	152	146	0.19	0.16	0.16	0.15
20,000	239	205	245	208	0.19	0.16	0.19	0.17
25,000	300	268	277	273	0.19	0.17	0.18	0.17
30,000	372	348	360	340	0.20	0.18	0.19	0.18
BOLT DESCRIPTION								
MATERIAL: SAE GRADE 8								
ANTI-SEIZE MATERIAL USED: ANTI-SEIZE SPECIAL								
BOLT	TORQUE	TORQUE	TORQUE	TORQUE	FRICTION	FRICTION	FRICTION	FRICTION
LOAD	APPLIED	APPLIED	APPLIED	APPLIED	FACTOR	FACTOR	FACTOR	FACTOR
	(FT-LBS)	(FT-LBS)	(FT-LBS)	(FT-LBS)	#1	#2	#3	#4
	#1	#2	#3	#4				
0	0	0	0	0	0.00	0.00	0.00	0.00
5,000	56	53	57	52	0.18	0.17	0.18	0.17
10,000	112	108	117	115	0.18	0.17	0.19	0.18
15,000	157	165	178	170	0.17	0.17	0.19	0.18
20,000	220	220	240	215	0.17	0.17	0.19	0.17
25,000	276	290	310	258	0.18	0.18	0.20	0.16
30,000	350	352	380	300	0.19	0.19	0.20	0.16
BOLT DESCRIPTION								
MATERIAL: ASTM A193 GRADE 8 CLASS 2								
ANTI-SEIZE MATERIAL USED: LOCTITE A-S 767								
BOLT	TORQUE	TORQUE	TORQUE	TORQUE	FRICTION	FRICTION	FRICTION	FRICTION
LOAD	APPLIED	APPLIED	APPLIED	APPLIED	FACTOR	FACTOR	FACTOR	FACTOR
	(FT-LBS)	(FT-LBS)	(FT-LBS)	(FT-LBS)	#1	#2	#3	#4
	#1	#2	#3	#4				
0	0	0	0	0	0.00	0.00	0.00	0.00
5,000	67	66	67	58	0.21	0.21	0.21	0.18
10,000	136	130	127	120	0.22	0.21	0.20	0.19
15,000	195	180	179	169	0.21	0.19	0.19	0.18
20,000	279	250	231	230	0.22	0.20	0.18	0.18
25,000	348	320	291	289	0.22	0.20	0.18	0.18
30,000	445	400	360	345	0.24	0.21	0.19	0.18
BOLT DESCRIPTION								
MATERIAL: ASTM A193 GRADE 8 CLASS 2								
ANTI-SEIZE MATERIAL USED: DOW MOLYKOTE 1000								
BOLT	TORQUE	TORQUE	TORQUE	TORQUE	FRICTION	FRICTION	FRICTION	FRICTION
LOAD	APPLIED	APPLIED	APPLIED	APPLIED	FACTOR	FACTOR	FACTOR	FACTOR
	(FT-LBS)	(FT-LBS)	(FT-LBS)	(FT-LBS)	#1	#2	#3	#4
	#1	#2	#3	#4				
0	0	0	0	0	0.00	0.00	0.00	0.00
5,000	70	57	50	51	0.22	0.18	0.16	0.16
10,000	146	96	101	99	0.23	0.15	0.16	0.16
15,000	190	147	145	140	0.20	0.16	0.15	0.15
20,000	253	200	205	215	0.20	0.16	0.16	0.17
25,000	330	259	260	278	0.21	0.16	0.17	0.18
30,000	415	340	338	310	0.22	0.18	0.18	0.16

Figure C-2
Friction Factor, Bolt Force Test
Page 2

CLAMPING FORCE vs TORQUE

BOLT DESCRIPTION								
MATERIAL: SAE GRADE 8								
ANTI-SEIZE MATERIAL USED: LOCTITE A-S 767								
BOLT LOAD	TORQUE APPLIED (FT-LBS) #1	TORQUE APPLIED (FT-LBS) #2	TORQUE APPLIED (FT-LBS) #3	TORQUE APPLIED (FT-LBS) #4	FRICTION FACTOR #1	FRICTION FACTOR #2	FRICTION FACTOR #3	FRICTION FACTOR #4
0	0	0	0	0	0.00	0.00	0.00	0.00
5,000	47	50	52	50	0.15	0.16	0.17	0.16
10,000	97	97	97	102	0.15	0.15	0.15	0.16
15,000	149	137	137	140	0.16	0.14	0.14	0.15
20,000	200	180	175	200	0.16	0.14	0.14	0.16
25,000	245	230	228	249	0.16	0.15	0.14	0.16
30,000	300	300	322	305	0.16	0.16	0.17	0.16
BOLT DESCRIPTION								
MATERIAL: SAE GRADE 8								
ANTI-SEIZE MATERIAL USED: DOW MOLYCOTE 1000								
BOLT LOAD	TORQUE APPLIED (FT-LBS) #1	TORQUE APPLIED (FT-LBS) #2	TORQUE APPLIED (FT-LBS) #3	TORQUE APPLIED (FT-LBS) #4	FRICTION FACTOR #1	FRICTION FACTOR #2	FRICTION FACTOR #3	FRICTION FACTOR #4
0	0	0	0	0	0.00	0.00	0.00	0.00
5,000	41	40	37	45	0.13	0.13	0.12	0.14
10,000	81	80	86	97	0.13	0.13	0.14	0.15
15,000	130	127	137	147	0.14	0.13	0.14	0.16
20,000	190	192	200	212	0.15	0.15	0.16	0.17
25,000	250	245	260	267	0.16	0.16	0.17	0.17
30,000	342	330	321	331	0.18	0.17	0.17	0.18
BOLT DESCRIPTION								
MATERIAL: ASTM A193 GRADE 8 CLASS 2								
ANTI-SEIZE MATERIAL USED: DRI-LUBE TYPE 822 MMSN306A								
BOLT LOAD	TORQUE APPLIED (FT-LBS) #1	TORQUE APPLIED (FT-LBS) #2	TORQUE APPLIED (FT-LBS) #3	TORQUE APPLIED (FT-LBS) #4	FRICTION FACTOR #1	FRICTION FACTOR #2	FRICTION FACTOR #3	FRICTION FACTOR #4
0	0	0	0	0	0.00	0.00	0.00	0.00
5,000	74	92	125	82	0.23	0.29	0.40	0.26
10,000	153	175	177	167	0.24	0.28	0.28	0.27
15,000	242	257	270	250	0.26	0.27	0.29	0.26
20,000	332	320	350	340	0.26	0.25	0.28	0.27
25,000	397	395	478	430	0.25	0.25	0.30	0.27
30,000	450	455	600	540	0.24	0.24	0.32	0.29
BOLT DESCRIPTION								
MATERIAL: SAE GRADE 8								
ANTI-SEIZE MATERIAL USED: DRI-LUBE TYPE 822 MMSN306A								
BOLT LOAD	TORQUE APPLIED (FT-LBS) #1	TORQUE APPLIED (FT-LBS) #2	TORQUE APPLIED (FT-LBS) #3	TORQUE APPLIED (FT-LBS) #4	FRICTION FACTOR #1	FRICTION FACTOR #2	FRICTION FACTOR #3	FRICTION FACTOR #4
0	0	0	0	0	0.00	0.00	0.00	0.00
5,000	57	65	60	62	0.18	0.21	0.19	0.20
10,000	122	125	118	118	0.19	0.20	0.19	0.19
15,000	188	180	168	170	0.20	0.19	0.18	0.18
20,000	270	235	240	235	0.21	0.19	0.19	0.19
25,000	338	300	308	290	0.21	0.19	0.20	0.18
30,000	420	378	330	333	0.22	0.20	0.17	0.18

Figure C-2
Friction Factor, Bolt Force Test
Page 3

SALT FOG DATA

TEST ITEM DESCRIPTION
BOLT OR FITTING MATERIAL: ASTM A193
BOLT OR FITTING SIZE/DESCRIPTION: 3/4" Stud

INITIAL RUNNING TORQUE (in-lbs)	FINAL TORQUE (ft-lbs)	SEPARATION TORQUE (ft-lbs)	RUNNING TORQUE (in-lbs)	ANTI-SEIZE MATERIAL USED
0	300	200	0	A-S Special
0	348	230	0	Loctite 767
0	330	200	0	Dow Molykote 1000
0	350	230	0	Fel-Pro C5A (new)
0	380	190	0	Fel-Pro C5A (original)

TEST ITEM DESCRIPTION
BOLT OR FITTING MATERIAL: 340 Stainless Steel
BOLT OR FITTING SIZE/DESCRIPTION: 1" AN Union

INITIAL RUNNING TORQUE (in-lbs)	FINAL TORQUE (ft-lbs)	#1 SEPARATION TORQUE (ft-lbs)	#1 RUNNING TORQUE (in-lbs)	#2 SEPARATION TORQUE (ft-lbs)	#2 RUNNING TORQUE (in-lbs)	ANTI-SEIZE MATERIAL USED
0	100	85	0	90	0	A-S Special
0	100	70	0	85	0	Loctite 767
0	100	85	0	90	0	Dow Molykote 1000
0	100	70	0	90	0	Fel-Pro C5A (new)
0	100	90	0	75	0	Fel-Pro C5A (original)

TEST ITEM DESCRIPTION
BOLT OR FITTING MATERIAL: 340 Stainless Steel
BOLT OR FITTING SIZE/DESCRIPTION: 2" AN Union

INITIAL RUNNING TORQUE (in-lbs)	FINAL TORQUE (ft-lbs)	SEPARATION TORQUE (ft-lbs)	RUNNING TORQUE (in-lbs)	ANTI-SEIZE MATERIAL USED
0	400	280	0	A-S Special
0	400	360	0	Loctite 767
0	400	380	0	Dow Molykote 1000
0	400	270	0	Fel-Pro C5A (new)
0	400	240	0	Fel-Pro C5A (original)

TEST ITEM DESCRIPTION
BOLT OR FITTING MATERIAL: SAE GRADE 8
BOLT OR FITTING SIZE/DESCRIPTION: 3/4" Bolt

INITIAL RUNNING TORQUE (in-lbs)	FINAL TORQUE (ft-lbs)	SEPARATION TORQUE (ft-lbs)	RUNNING TORQUE (in-lbs)	ANTI-SEIZE MATERIAL USED
0	275	200	0	A-S Special
0	245	160	0	Loctite 767
0	250	160	0	Dow Molykote 1000
0	270	170	0	Fel-Pro C5A (new)
0	290	160	0	Fel-Pro C5A (original)

Figure C-3
Salt Fog Data
page 1

THERMAL CYCLE

TEST ITEM DESCRIPTION
BOLT OR FITTING MATERIAL: 304 Stainless Steel
BOLT OR FITTING SIZE/DESCRIPTION: 2" Union

INITIAL TORQUE (in-lbs)	SEPARATION TORQUE (Break away value)	ANTI-SEIZE MATERIAL USED
400	1/6th turn, then galled	A-S Special
400	able to separate but galling	Loctite 767
400	separated and threads are free running	Dow Molykote 1000
400	1/3 turn then galled	Fel-Pro C5A (new)
400	inseparable	Fel-Pro C5A (original)

TEST ITEM DESCRIPTION
BOLT OR FITTING MATERIAL: 304 Stainless Steel
BOLT OR FITTING SIZE/DESCRIPTION: 1" Union

INITIAL TORQUE (in-lbs)	#1 SEPARATION TORQUE (lb/ft)	#1 RUNNING TORQUE	#2 SEPARATION TORQUE (lb/ft)	#2 RUNNING TORQUE	ANTI-SEIZE MATERIAL USED
100	210	immediate galling	340	5 ft-lbs then galled	A-S Special
100	180	20 ft-lbs then galled	250	10 ft-lbs then galled	Loctite 767
100	210	0 ft-lbs	270	0 ft-lbs	Dow Molykote 1000
100	210	5 ft-lbs, then galled	200	5 ft-lbs, then galled	Fel-Pro C5A (new)
100	280	0 ft-lbs	260	0 ft-lbs	Fel-Pro C5A (original)

Figure C-4
Thermal Cycling Test Results
page 1

APPENDIX D
PROPELLANT COMPATIBILITY TEST RESULTS

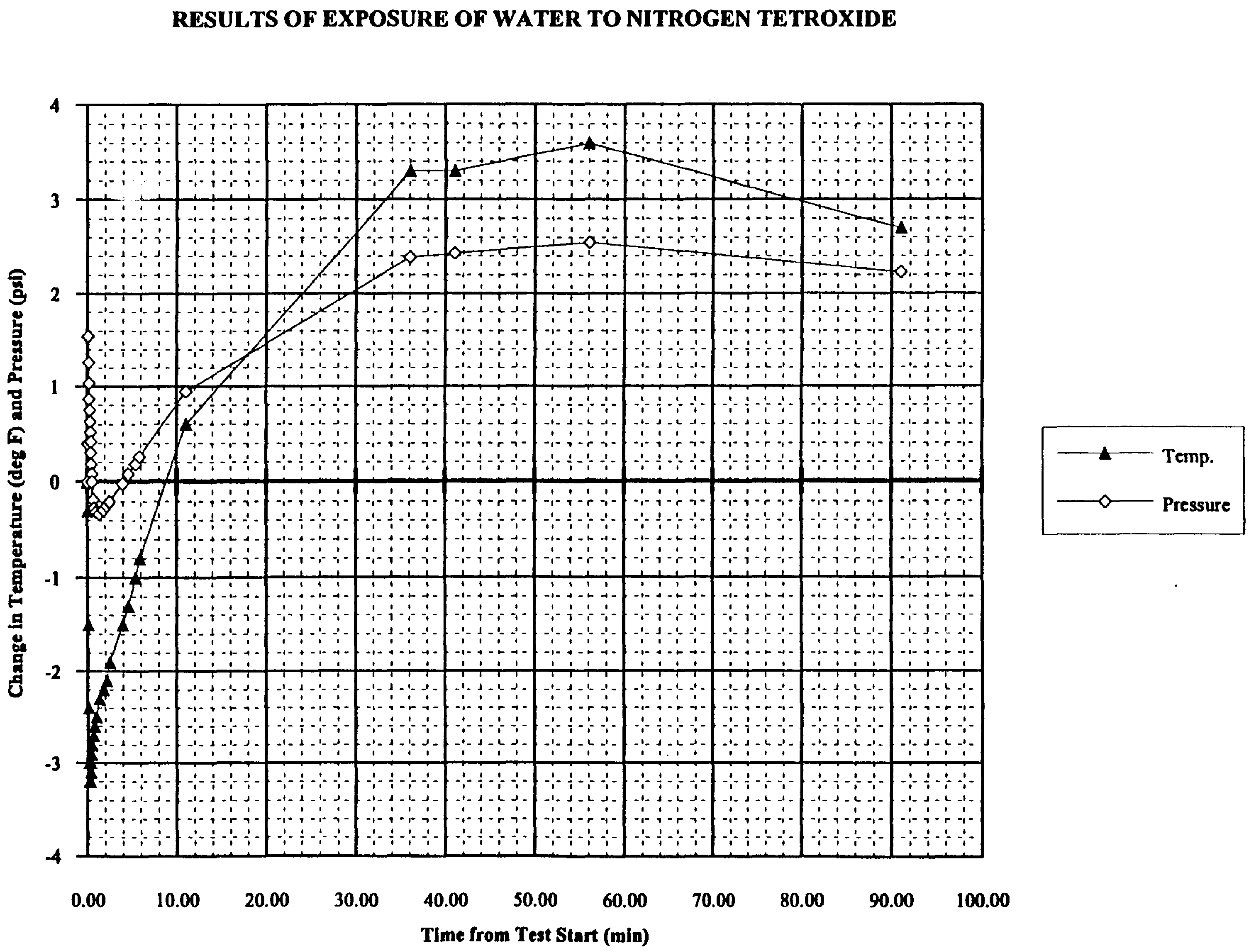

Figure D-1 Results, w[illegible] and nitrogen tetroxide

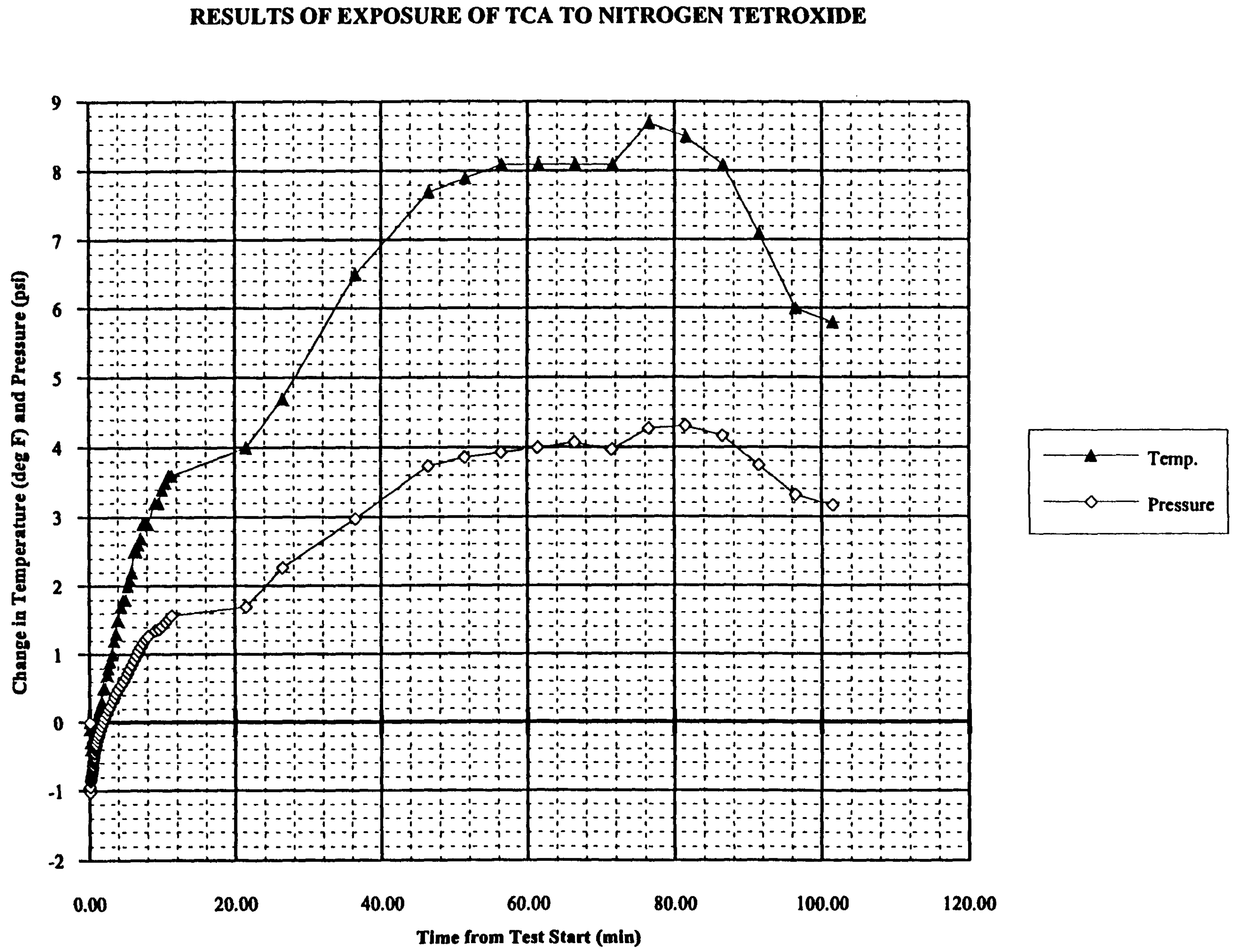

Figure D-2 TCA and nitrogen tetroxide results

RESULTS OF EXPOSURE OF CFC-113 TO NITROGEN TETROXIDE

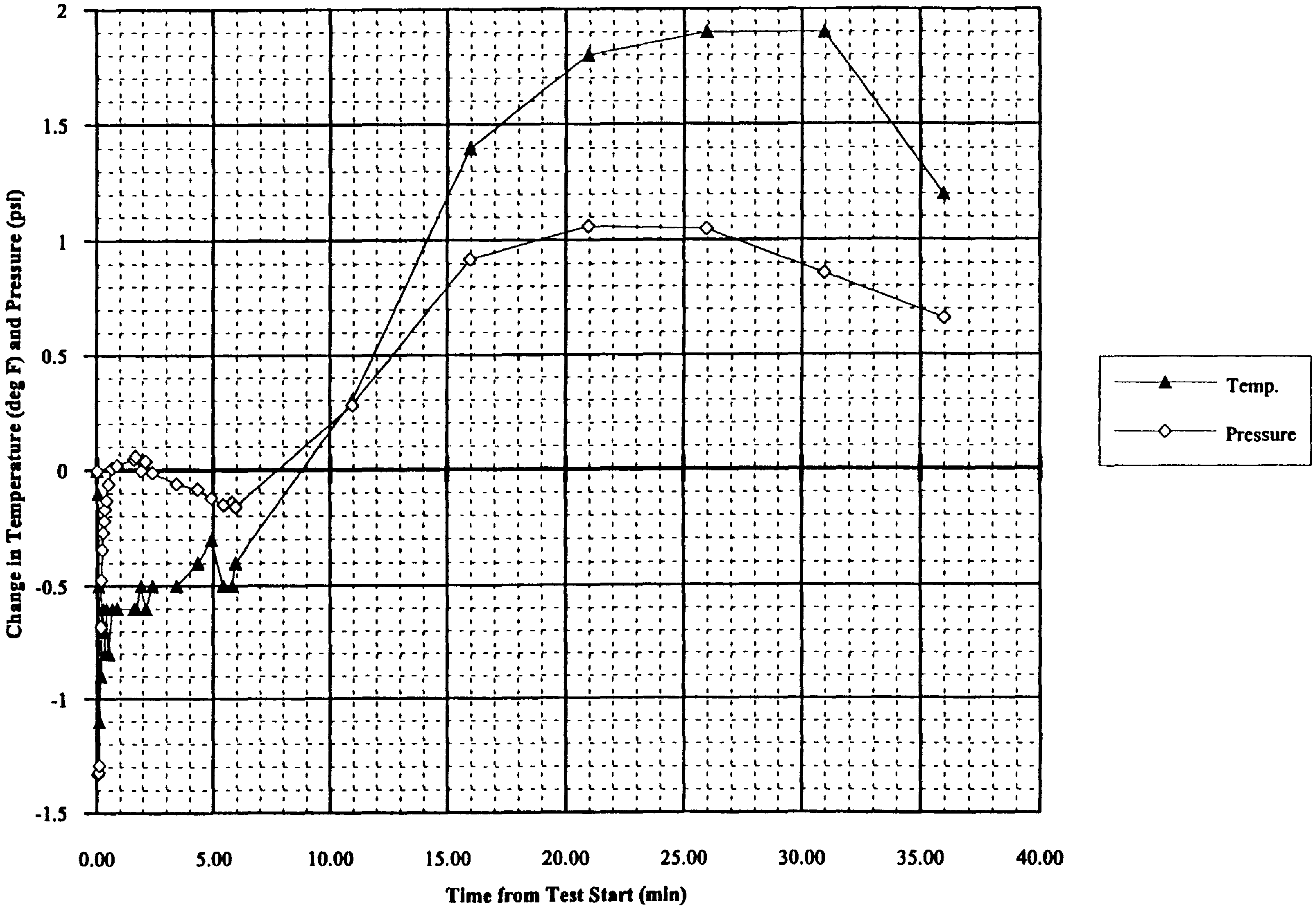

Figure D-3 CFC-113 + nitrogen tetroxide

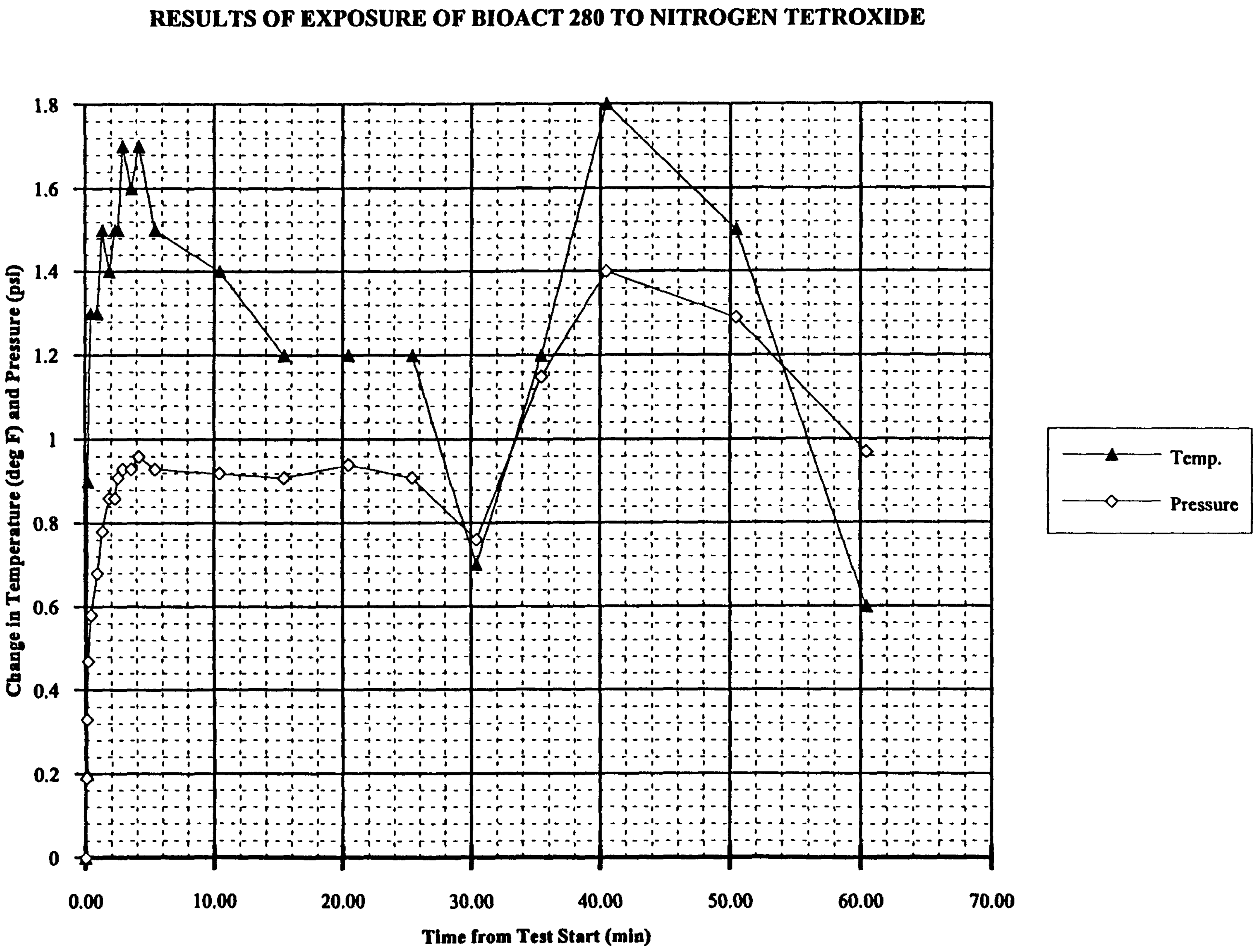

Figure D-4 Bioact 280 and nitrogen tetroxide

RESULTS OF EXPOSURE OF BIOGENIC REAGENT TO NITROGEN TETROXIDE

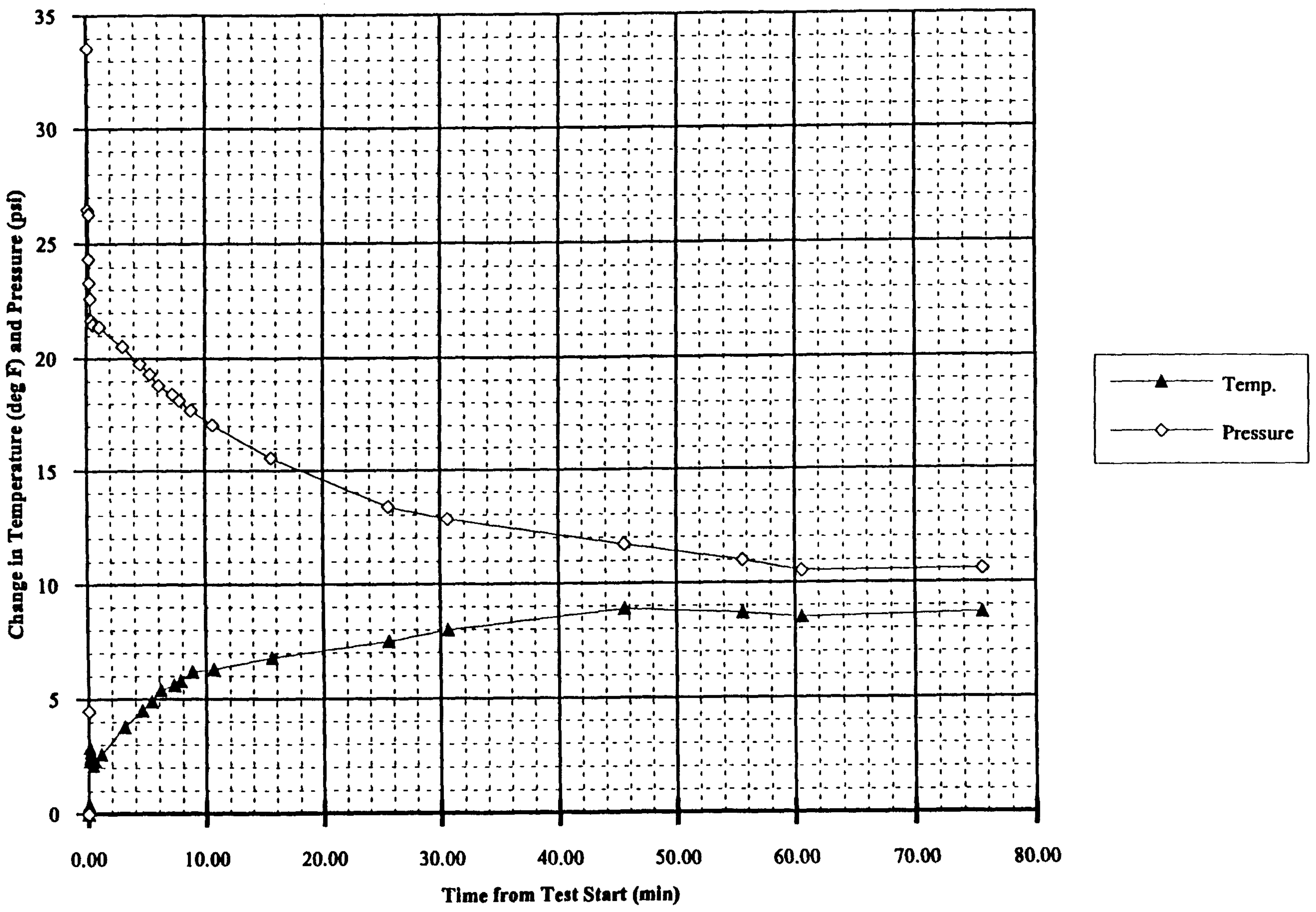

Figure D-5 Biogenic Reg[illegible] and nitrogen tetroxide

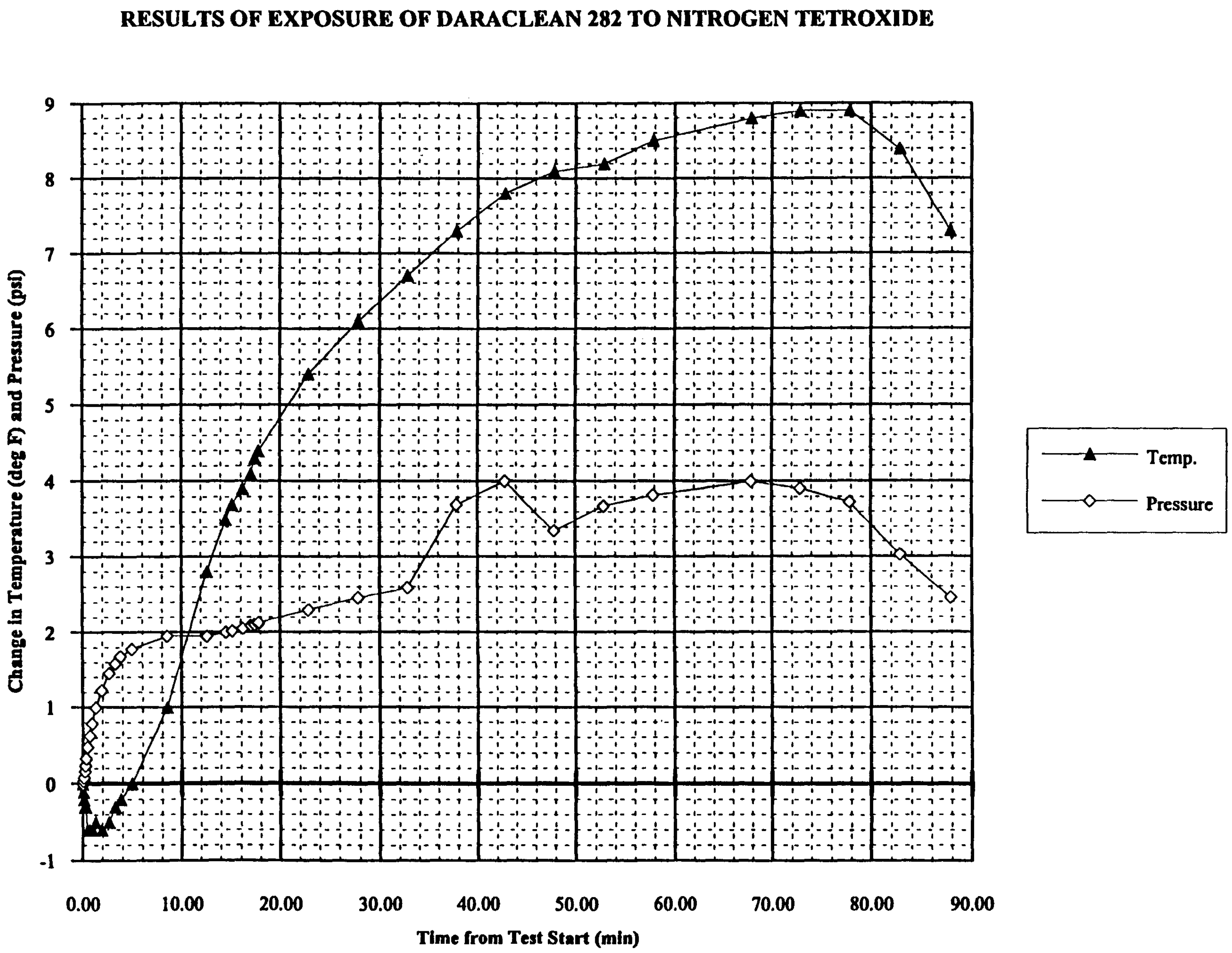

Figure D-6 Daraclean 282 and nitrogen tetroxide

RESULTS OF EXPOSURE OF EP-921 TO NITROGEN TETROXIDE

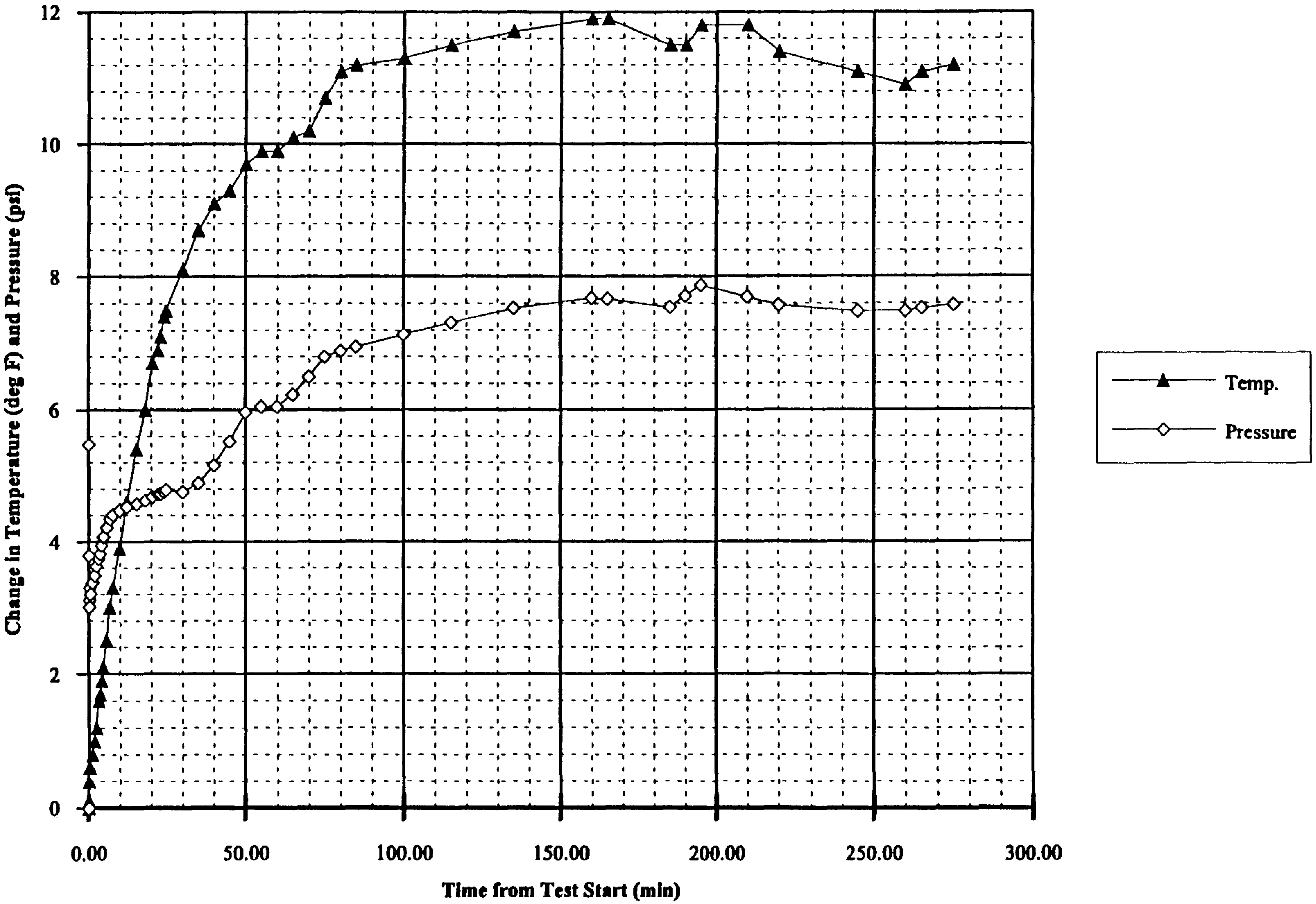

Figure D-7 EP-921 nitrogen tetroxide

RESULTS OF EXPOSURE OF FORMULA 815 TO NITROGEN TETROXIDE

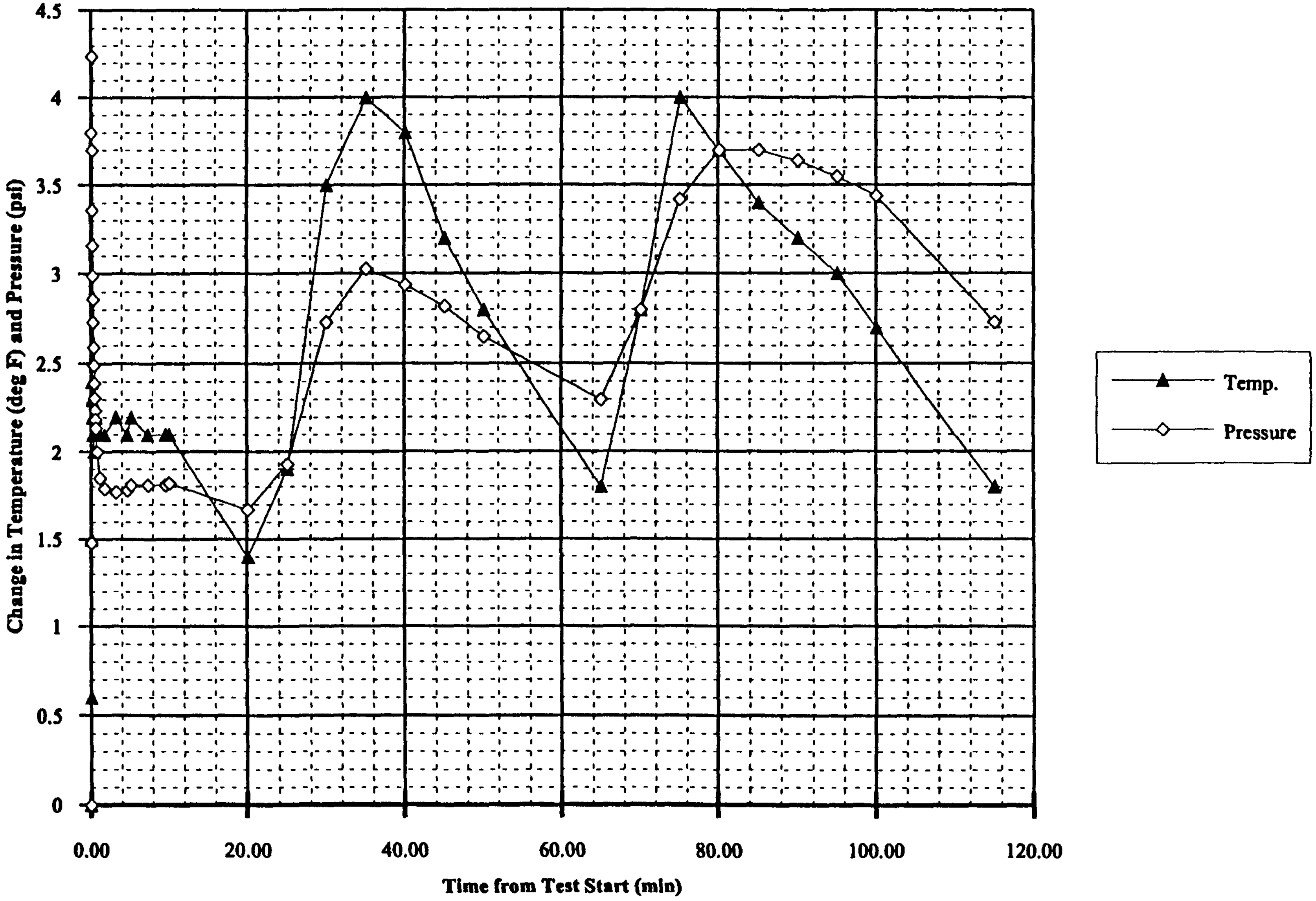

Figure D-8 Brulin 815GD and nitrogen tetroxide

RESULTS OF EXPOSURE OF PARTSPREP TO NITROGEN TETROXIDE

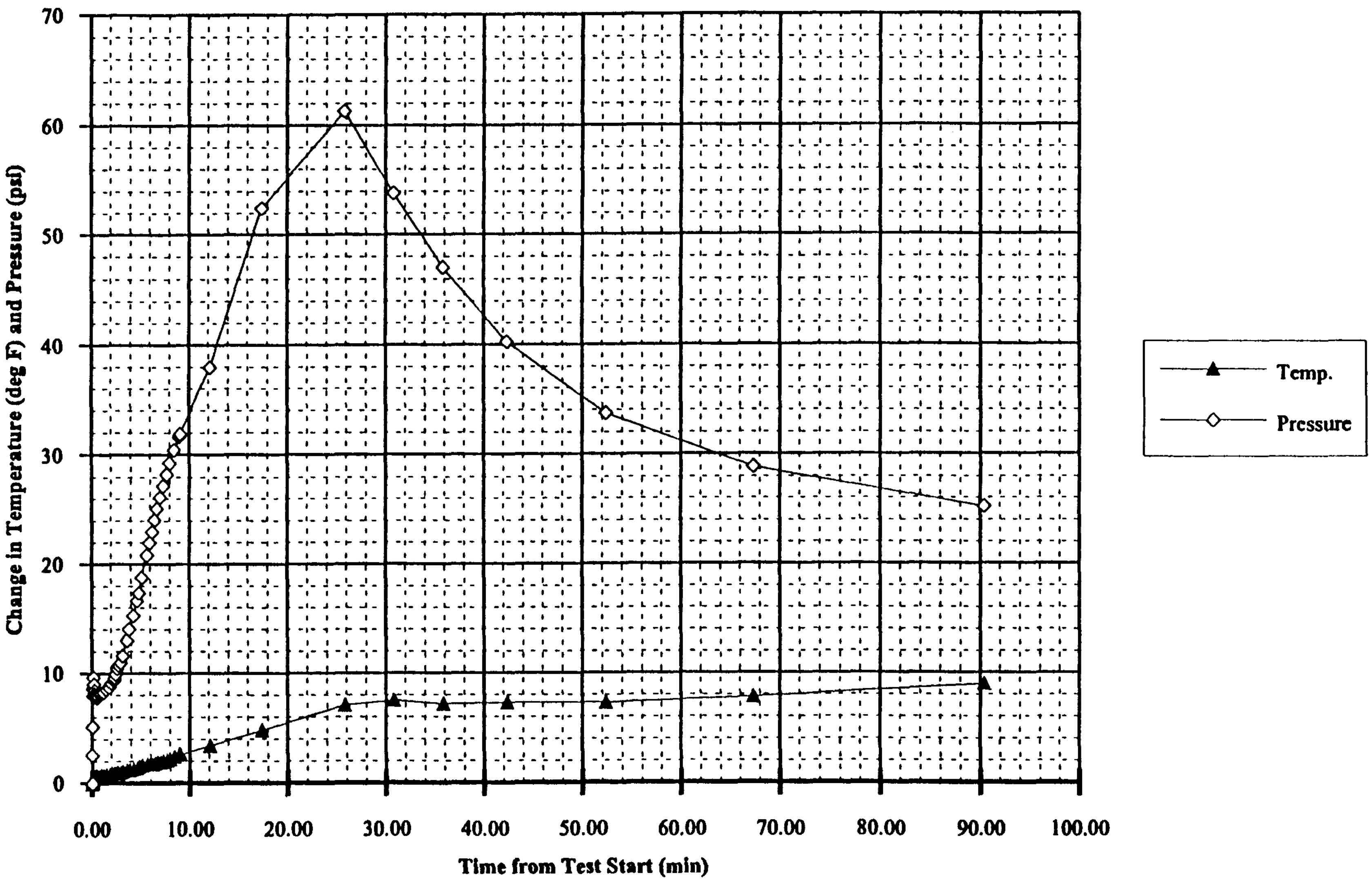

Figure D-9 Parts Pre d nitrogen tetroxide