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ES&H Development Activities for the W89 Warhead

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ABSTRACT

Environmental, Safety and Health (ES&H) issues became an important design consideration during the development of the W89 warhead for the SRAM II (Short-Range Attack Missile) missile. An action plan was developed to handle these issues at all the production agencies and at both the system and the component level. The main thrust was in the area of solvent substitution, in particular for solder flux removal. The cleaner d-limonene followed by an isopropyl alcohol rinse was selected for applications were the traditional cleaners were 1,1,1 trichloroethane or trichloroethylene. Compatibility testing rather than efficacy testing dominated the development effort. In addition to other solvent substitution applications, organic materials that were free of toluene diisocynate (TDI), and methylene dianiline (MDA) were explored for use in the W89.

ACKNOWLEDGMENT

A great many people throughout the DOE Nuclear Weapons Complex contributed to the work described in this report. Paul Longmire, SNL, Clint VanSickle and Al Bennett, KCD, had the management vision that began the solvent substitution for the MC4069 firing set assembly. At KCD, Dave Lowe, Gerry Meier, John Davis, Bob Sanders, Mark Benkovich, Mark Wilson, Fred Larson, Jim Tira, Dave Fossey, Tim Reno, performed the development and compatibility testing required to implement that substitution. The d-limonene residual gas analysis was performed by Dave Spieker, KCD. Ben Richardson, PX, contributed to the development of the technique. Stan Dubiel and Charlie Cook, KC, provided additional analytical support. Their work was supported by the activities of Sally Hoier, Paul Nigrey, John Sayre, Mike Oborny, Ed Lopez, and Rob Sorenson, Tommy Guess, Mark Stavig, Adra Baca, Jeff Reich, Pete Rand, Dave Zamora, Diane Peebles, and Ernie Correa at SNL. All SNL component engineers involved in the W89 project assisted in the compatibility studies. Tom Hand, KCD, supported the aqueous cleaning process implementation for metallic components. John DiChiaro and Bob Salarno, MD, developed the cleaning processes used for the DSSL, MC2949 and MC3753 actuators. Lisa Thompson, Y-12, developed the Solvent 140 and Water Chaser 140. Tom Gillespie developed the plasma wire stripping technique for magnetics at PP. Tom Neet, KCD, and Pete Rand, SNL developed TDI -free urethane foam encapsulant formulations. Ramona Myer, SNL, and Mark Wilson, KCD, developed TDI-free polyurethane adhesives. MDA-free epoxy formulations were developed by Ken Wishmann and Linda Domier, SNL, and George Dorsey, Y-12. Jim LeMay and Ing Chiu, LLNL, performed the Halthane compatibility studies, the partitioning experiment and modeling, and studies on other LLNL materials. Gas analysis for the DAAU systems test was performed by Lorelei Gross-Woods, PX. SNL management support for this work on the W89 was provided by Jack Jones, Dave Havlik, Russ Miller, and Ed Cull.

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INTRODUCTION

The W89 warhead was developed for use in the SRAM II (Short-Range Attack Missile) missile for the Air Force's Strategic Air Command. In November 1986, the W89 was assigned to the LLNL/Sandia design team during Phase 2a. Phase 3 development activities were begun in October 1987. Phase 4 pre production activities were started in June 1990. However, in September 1991, President Bush canceled the SRAM II program as part of a large scale reduction in American nuclear forces. At that time the First Production Unit (FPU) date for the W89 was scheduled for October 1994. Following the SRAM II program cancellation, W89 development efforts were redirected to concentrate on the SRAM A missile application. There was always a requirement for the W89 to be back compatible with the SRAM A missile with only minor modifications. However, in July of 1992, the DOD informed the DOE that there was no longer any re-warheading requirement for the SRAM A missile. The W89 was subsequently removed from the directive schedule. The FPU date at the time of cancellation was October 1995. Portions of the W89 development effort continue as an advanced technology demonstration program called Pit Reuse Enhanced Safety Surety (PRESS).

During the development of the W89, Environmental, Safety and Health (ES&H) became a very important issue across the Department of Energy. The original design of the W89 warhead was largely complete during Phase 3 when the compatibility test series, the Core Tests, was begun. The purpose the core tests was to prove out that there were no long term system level corrosion or compatibility problems confronting the W89 warhead. The initial intent was once the cores tests were designed and built, that no further materials changes would be made to the W89 design. These core tests began assembly in the Spring of 1989, and went into test at Pantex in October, 1989. That summer, after a review of environmental regulations, it was determined that Allied Signal Kansas City Division Plant (KCD) was not in compliance with air quality standards for a painting operation for B83 case parts. The result was a production stoppage on the B83 until a new, in-compliance paint and process could be identified and developed. In September 1989 Sandia held the first ever integrated workshop on waste minimization for non-nuclear components (Ref. 1). From the information presented at this workshop it was clear that warhead development efforts had new, additional constraints with which to deal with. It was expected that some new, very stringent environmental laws and regulations would take effect during the W89 production period.

The W89 program faced the unprecedented requirement of ensuring the warhead design could be produced in an ES&H compliant manner.

This has placed the W89 in the unique position of having a design that was nearly completed, with process and materials selections having been made and the compatibility core test series begun, and facing the prospect of changes forced upon it during production. Changes based upon ES&H requirements will be of a different character than usual design changes. Normally changes are made to improve the reliability of the design or facilitate production methods to improve the quality of the product and help reduce its cost. ES&H changes depart from this philosophy of continuous improvement and sometimes may require the elimination of materials and processes that were selected in the past for their excellent performance, successful stockpile history, and sometimes unique properties.

The goal for the W89 ES&H development activities was to avoid eleventh hour ES&H driven changes in production that have not been adequately characterized during development. This goal included minimizing the risks to warhead reliability due to ES&H

changes; to thoroughly characterize and document these changes during the warhead development phases, preferably before FPU; and ensure that production schedules are not jeopardized and avoid unexpected shut downs. Also, ES&H changes must not lead to any compromise to nuclear safety.

The purpose of this report is to document and describe these W89 development activities that were unique compared to traditional warhead designs. This report covers the W89 ES&H plan, development efforts on components and processes that involved cleaning, coating, adhesives, encapsulants, and system level compatibility testing. This work was conducted by a large number of participants at Sandia, the DOE production agencies and Lawrence Livermore National Laboratories working in a cooperative effort. Areas for future work are also described.

THE W89 WARHEAD CHARACTERISTICS

A complete warhead description is given elsewhere for the W89/SRAM II and the W89/SRAM A (Refs. 2,3,4). This section will describe characteristics of the warhead that are important from the perspective of ES&H development. For the most part, the ES&H issues for the W89/SRAM A are the same as for the W89/SRAM II. A complete environmental description is given in Reference 5. For ES&H compatibility, environments such as moisture levels and temperatures are important while other environments such as shock and vibration play a significantly lesser role. The stockpile life of the W89 for SRAM II was 20 years with a protected period of 27 years. One important feature of the W89 design is that most of the electronic subsystems of the Warhead Electrical System (WES) are in non-hermetic, unsealed housings. This design approach saves considerable weight, space and both development and production costs. It, however, means that all components share the same internal warhead environment. Chemical species from one component is free to migrate to any other component in the warhead. The warhead design must ensure that long term chemical compatibility exists between the materials of construction for all components as well as for any offgassing or migrating species that might form. With large scale materials and processing changes driven by ES&H concerns, ensuring that there were no adverse compatibility effects became the major effort in the ES&H development effort. In keeping with the open, non-hermetic design of the WES, the warhead uses desiccant to control the moisture level in the warhead. The moisture level requirement is expressed as a dewpoint. The maximum dewpoint temperature allowed at any time during the warhead stockpile life is -20^{0} F. Moisture in the internal warhead atmosphere comes from two major sources, water in components and water permeation through the environmental O-ring seals. To ensure that there is adequate desiccant available to absorb these quantities of water, each component was given a water budget which specified that maximum amount of water that could be brought into the warhead by that component. The moisture carried in by a component is largely governed by the hydroscopic nature of the materials within that component. If a material absorbs large amounts of moisture from, say, the humidity in the production plant atmosphere, then extraordinary handling techniques would be required to prevent excess moisture from entering the warhead. With ES&H materials changes there is a concern that some new materials may be significantly more hydroscopic than the traditional materials used.

Another method by which large amounts of moisture could enter components is if aqueous processes were used. An additional concern with aqueous processes is that water may be trapped in local areas and cause localized problems of corrosion or low resistance electrical pathways. As a result of these concerns, the W89 project imposed the following restrictions on any new ES&H or existing processes used to produce W89 components:

> Aqueous or water bearing processes must not be used unless the parts are not made of any organic or hydroscopic materials and are without trapping

joints. Where aqueous or water bearing processes are used it must be demonstrated that subsequent processing will remove all moisture from these parts or components. In any case, aqueous or water based processes should not be used for electronic subassemblies higher than the Printed Wiring Board (PWB) level.

For the W89/SRAM II, compatibility and moistures concerns were heightened because of the Built In Test (BIT) that was routinely planned for the MC4073 Programmer every time the SRAM II missile is powered up. This would occur at least once a day when the missile is on alert. The SRAM II was designed to be in the SAC bomber on runway alert for as much as 180 days a year. The Programmer BIT test would then exercise the electrical circuits routinely, providing an electrical potential that could accelerate any compatibility or corrosion problems that may exist.

Runway alert, captive carry in the bomber and missile free flight all contribute to large temperature extremes that the W89 warhead may experience. For the purposes of long term compatibility issues, the runway alert temperature extremes are of most concern. Also, the requirement for 720 hours of captive carry airborne alert has some effect. The temperature extremes within the warhead can vary depending upon the location of the component within the warhead and whether the warhead is in the SRAM II or SRAM A missile. SB 210447 (Ref. 5) lists thermal cycles for qualification tests to prove out the design and to demonstrate that the component will function during and/or after experiencing the worst case environmental limits. For long term temperature compatibility, these test cycles were expanded to include a simulation of the maximum extent chemical or diffusion related processes would proceed during the 20 year stockpile life. Because chemical processes occur rapidly at higher temperatures and slowly at low temperatures, only the high temperature extremes are considered. Ground alert temperature histograms were derived from taking 12 years of real weather data at Beale Air Force Base in Sacramento, California and normalizing upward to what the hottest base would be. For components in the region of the MC4069 Firing Set, 20 years of stockpile life could be simulated with 385 hours at 150° F for the SRAM II missile. For the SRAM A missile the simulation cycle would be 385 hours at 155° F. These thermal cycles would also apply to other forward WES components, for aft components, the temperature would be 5 degrees lower, while for components near the surface of the warhead, they would be about 3 degrees hotter (Refs. 6, 7, 8). Worst case cold temperatures would be -40° F for runway alert and -50°F for captive carry airborne alert. However, different system and component tests sometimes used various other thermal cycles based on other test purposes or as a basis of comparison for a standardized qualifying test method for similar components.

THE W89 ES&H PLAN DESCRIPTION

The W89 ES&H plan was published in April 1991 (Ref. 9) however the concept of ES&H activities for the W89 program was communicated to the production agencies as early as the fall of 1989. The purpose of this plan was to provide a framework of coordination for work in development and pre-production to achieve the goal of adequate characterization for ES&H changes within the time frame of the W89 program schedule. The plan also provides a framework to characterize ES&H changes that might be required after production has begun.

The Figure 1 is a flow chart that summarizes the ES&H plan for the W89. This flow chart starts with the activities begun in November 1989 with subsequent activities spaced out along a timeline that results in completion by FPU, which, at the time of the plan's publication, was January 1994. This plan was streamlined from previous versions in an effort to concentrate the technical resources into supporting the W89 development schedule while minimizing resource requirements for

coordination efforts. The plan is a four step process:
1) Identification of Hazardous Materials, 2) Sort, Prioritize and Schedule, 3) Develop Processes, Specifications & Handling Equipment; and finally 4) Implement into WR and Document Costs.

The first activity, identification of hazardous materials, was completed by each of the production agencies as a response to a DOE/AL request (Ref. 10). This activity was performed by the different production plants instead of the design agencies because the ES&H requirements will vary by local laws and regulations superimposed on the federal ones. In addition, ES&H compliance is an individual plant responsibility and is linked to the plant's DOE contract performance award.

1) Identification of Hazardous Materials:

Identification of hazardous materials for ES&H took place as a three step process. First a list of all materials that comprise a specific component or product was compiled. The materials were categorized as support materials, used in the fabrication processes for the product; or as production materials, the actual materials of construction. The materials listed, often as trade names, specification numbers and common product names then had to be examined to determine their actual material composition. Finally, these materials will be sorted according to the information in the Material Safety Data Sheets (MSDS's) as acceptable materials, materials that will be banned and therefore unavailable for future procurement, and materials whose ES&H characteristics require special handling and protective measures that make their continued use, while legally permissible, undesirable from a production agency standpoint. The end product of this activity was a component by component compilation of materials and processes which are ES&H concerns that must be addressed.

2) Sort, Prioritize and Schedule:

After completion of the materials identification, the ES&H issues were examined from two perspectives, at the component or MC level, and as a technical discipline of process technology. At the component level, it can be determined whether the functionality or reliability of the component could be at risk due to a change, and where the time factor for a change can best be assessed. Some parts, such as magnetics, must start PPI and other pre-production activities far sooner than others, such as metal case parts. Sorting by technical discipline, items would fall into several main categories: coating and plating, cleaning electronic assemblies, general metal cleaning, precision cleaning, polymeric encapsulants and adhesives. The likelihood of success in developing alternate materials and processes was be assessed in these activities. Then the decision was made whether a change could be developed for the production of the W89, or whether the materials and processes remain with the ES&H requirements fulfilled by implementing the necessary handling and protective procedures. In addition, there were a few circumstances where in-production changes were proposed. For all these issues, a schedule must be defined; either for development, characterization and testing of a new material/process, or the industrial engineering and procurement of the required handling facilities and equipment. An ES&H implementation plan was written for each plant ship entity to assure that all the varying ES&H issues are addressed on a timely basis for all W89 components. These decisions and implementation plans were made by the individual plant.

3) Develop Processes, Specifications & Handling Equipment:

To support FPU, all these activities will have to be developed, documented with written specifications prior to PPI. The activity is most stressing when development of new processes or materials is required. It should be noted however, that with the exception of a few materials, namely severe ozone depleters, most materials are not banned; it is their exposure to humans and discharge into the environment that is restricted. The documentation and control of these new materials and processes will often replace the current seven-digit (99000000) general specifications. For the time being, for application to the W89, these specifications were written as

SS specifications unique to the W89. Afterward, conversion to the seven-digit specifications should be considered.

4) Implement into WR, Document Costs:
The work to take these development activities and implement them into the WR production activities was expected to take the standard course through PPI, pre-production and TMS activities. However, the W89 has been under close cost control monitoring since the start of the program with the Decision Cost Study in Phase 2 and the Baseline Cost Study from Phase 2a through Phases 3 and 4. At every program review, the cost of each component was updated and compared to this baseline. However, the costing methodology used does not account for the large, out of scope changes that ES&H will have on W89 costs. Increased costs or capital equipment requirements resulting for ES&H changes are considered off baseline because ES&H compliance is a new requirement. These costs would not be unique to the W89, but rather would be incurred in any warhead design, new or old, being produced at the time of the W89 build. Another activity involved the ES&H impact of warhead costs. ES&H incurred costs, if large, would be important to distinguish from normal baseline component costs.
The costs for the ES&H activities needed be documented in a format separate from the W89 Baseline Cost Study. Because of the program cancellation, complete cost documentation was never rigorously pursued, but some general trends were noted.

Testing
In addition to these four steps it was felt that additional system level compatibility testing was necessary. This consisted of a second set of core tests CTU 11-15 that contained the new ES&H changes as well as a testing an accelerated aging unit (AAU) using one of the complete warheads built during Process Prove-In (PPI). These compatibility tests will be discussed later in this report.

This program plan was followed in so much as the progam schedule allowed. Several plants were unable to implement all portions of the plan prior to cancellation of the W89 program.

HAZARDOUS MATERIALS IDENTIFICATION

Each plant identified the major hazardous materials which would be used by traditional methods for the production of the W89:

Kansas City Division (KCD)
Initially KCD listed the following cleaning solvent for elimination or reduction (Ref. 11):
Dichrolomethane
Fluorinert*
Freon
Genesolv-D
M-Clene-D
Methylene Chloride
Trichloroethane
Trichloroetrifluoroethane (Freon 113)
This list was later expanded to include the following chemicals in materials of construction:
Methylene Dianiline (MDA)
Toluene Diisocyanate (TDI)
High Volatile Organic Compounds (VOC) Paint or Electrofilm
(> 3.5 lbs per gal emission)
Vinyl Cyclohexane Diepoxide (VCD)
*It should be noted that Fluorinerts are ozone safe and are not considered toxic. It was a mistake, probably due to confusion with CFC's, that Fluorinert was listed.

Pinellas Plant (PP)

General Electric Neutron Devices (GEND, now Martin Marietta Specialty Components) identified the following materials as actual or potential ES&H concerns by application to W89 components (Ref. 12).

W89 Product	Application	Chemical/Material
MC4135 Neutron Generator	Epon Curing Agent Z	Methylene Dianiline (MDA)
MC4190 Thermal Battery	"	"
Magnetics	"	"
MC4135 Neutron Generator	Mold Cleaning and Equipment Purging	Methylene Chloride
Magnetics	Wire Stripper (ISO Verre)	Hydrofluoric Acid
Magnetics	Bobbin/Coating (Bendix Reinforced Plastic)	Iron oxide, Fiberglass
General Use in Machine Shop and Chem. Clean Facility	Degreasing Operations "	Trichloroethylene "
General Application	Parts Cleaning Degreasing	Freons (CFC's) Methyl Chloroform

Pantex (PX)

For the non nuclear components for the W89 warhead (Ref. 13):

Description	Specification	Use	Hazard
Lubribond 220	MIL-L-23398	Warhead Screw Dry FilmLubrication	Suspect Carcinogen
Desiccant	MIL-D-3464	Various Areas	Suspect Carcinogen
White Ink	II-I-1795	Warhead Marking	Suspect Carcinogen

For Shipping Container coating:

Description	Specification	Hazard
Pretreatment Coating	MIL-C-8514	Carcinogen
Epoxy Primer	MIL-P-23377	Carcinogen

Rocky Flats (RFP)

The Rocky Flat Plant review for W89 hazardous materials identified some hazardous materials, but by the nature of the hazard these materials were judged not to require any replacement. (Ref. 14). Compared to "Traditional" cleaning methods, Rocky Flat has already changed from chemicals that would have required replacements to Oakite NST, an aqueous alkaline cleaner for metals.

Operation	Material Use	Hazardous Material	Action Required
Machining	Solvent	De-Solv-It	None
Welding	Solvent	Isopropyl Alcohol	None
Brazing	Solvent	Isopropyl Alcohol	None
Cleaning	Solvent	Isopropyl Alcohol	None
Inspection	Solvent	Isopropyl Alcohol	None
Radiography	Fixer	Ammonia Thiosulfate	None
Radiography	Developer	Potassium Sulfite	None

Mound (MD)

Mound identified the following materials for replacement (Ref. 15):

1. Chlorinated Hydrocarbons (CHC's)
   Chlorofluorocarbons (CFC's)
2. MDA materials.

Oak Ridge Y-12 Plant

While the Y-12 Plant does not make any Sandia components for the warhead, the JTA shares some components with LLNL for the telemetry housing. In that fashion, chemicals that Y-12 identified as hazardous materials that are used in the cleaning after metal machining operations apply to Sandia (Ref. 16):

Freon
Methyl Chloroform
Methylene Chloride
Perchloroethylene

DEVELOPMENT OF ES&H COMPLIANT ALTERNATIVES

The development of new acceptable processes evolved into development activities for specific processes and component applications, typically these activities were organized at each separate production agency. Coordination and communication of these activities, however, routinely occur within the DOE Nuclear Weapons Complex through several regularly scheduled meetings that discuss ES&H progress organized by technology. Table 1 summarizes the ES&H development areas for each production area. Table 2 summarizes the ES&H development areas required for W89 non-nuclear components. Much of the actual development work, as described in this report was performed at the plants as component or product based development.

Solvent Substitution:

A majority of the ES&H development was aimed at solvent substitution to eliminate the usage of CFC's or CHC's. These materials are either ozone depleters or suspect human carcinogens. Ozone depleters are part of a global environmental problem where these chemicals have been shown to deplete the protective ozone layer in the upper atmosphere. The use of these chemicals is regulated by an international agreement called the Montreal Protocol, to which the United States adheres. This set of regulations is the most stringent of all the environmental regulations affecting the W89. Currently, these ozone depleting chemicals are scheduled for complete elimination by the end of 1995. For the W89, any Freon product, Genesolv-D, or methyl chloroform (also known as 1,1,1 Trichloroethane, or TCA) are ozone depleters. Methylene Chloride, M-Clene-D, dichrolomethane, perchloroethylene, trichloroethylene (TCE) are all suspect human carcinogens and are coming under increasingly stringent regulations.

KCD

MC4069 Firing Set

At KCD, the firing set production traditionally used either Freon or TCE for cleaning solder flux from printed wiring board assemblies and other component assemblies (Ref. 17). Figure 2 shows the matrix of cleaning processes used for the firing sets. With the exception of process F which only uses isopropyl alcohol (IPA) for general cleaning and not intended for removing major amounts of solder flux, there is no qualified cleaning process without the use of Freon or TCE. In addition, these cleaning processes are frequently used. For the W89's MC4069 Firing Set, the original production flow used 49 different cleaning steps. (Many of these cleaning processes are before and after individual processing steps. Some of these cleaning steps are redundant to provide insurance against processing a part contaminated during handling, transport and storage. Another effort in a maturing ES&H program would be to examine the need for this redundancy and minimize waste generation by eliminating cleaning steps possibly by improving handling and storage conditions.)

The solvent substitution for the MC4069 Firing Set was one of the major ES&H development efforts in the W89 program (Ref. 18, 19). This effort actually started prior to the system level W89 ES&H plan. The objective was to select a new ES&H compliant cleaner that would clean as well as the traditional flux removal methods. This new cleaner (and process) would also have to effectively clean general contaminants from the MC4069 hardware. It also must be compatible with the existing MC4069 materials of construction and not cause any compatibility problems at the W89 warhead level.

The MC4069 Firing Set is a nearly ideal test vehicle for solvent substitution for warhead development. Firing sets contain a wide variety of components, materials and electrical functions including high voltage assemblies. The compilation of components and features in firing sets contain almost every type of component, material and electrical function found in every other type of warhead electrical component in the W89. The MC4069 Firing Set is also the most intricate physical layout in the W89 warhead. It is expected that if residual amounts of chemicals will be carried into the W89 warhead sealed environment, that the MC4069 would be the component that would most contribute to that residual level. Typical components included in the MC4069 are:

Printed Wiring Assemblies
Flex Cables
Various Connectors and Interconnects
High Voltage Assemblies encapsulated in glass microballoon filled epoxy
Low Voltage Assemblies encapsulated in rigid polyurethane foam
Capacitor Discharge Units, Sprytrons, Sprygaps, Plasmatrons, Hybrid Micro Circuits (HMC's), Stronglinks and Coded Switches.
Capacitors, Diodes, Zener Diodes (chips and packaged), Transistors, Optoelectronics, Magnetics, Pickups, Resistive Dividers, Resistors, Leadless Chip Carriers (LLC's), and miscellaneous Semiconductors.
Miscellaneous organic materials such as adhesives, encapsulants and wire sleeving.

Figures 3-6 show typical modules and assembly views (prior to encapsulation) of the MC4069. One of the final process steps is the encapsulation of the MC4069 with rigid polyurethane foam. This may increase the tendency to entrap residual chemicals.

Selection of Solvent Replacement

In activities prior to the W89 program involvement, Sandia personnel monitored the solvent evaluation studies conducted by several aerospace corporations and the IPC (Institute for Interconnecting and Packaging of Electronic Circuitry, a joint industry, military and EPA program to evaluate alternatives to CFC for printed board assembly cleaning). Table 3 lists the cleaners evaluated. Based on the results from the above tests DOE cleaner evaluation studies examined the following cleaners:

Petroferm: Bioact EC-7, a terpene based cleaner of d-limonene with added surfactants.
Exxon: Exxate 1000, based on octadecyl acetate.
Advance Chemical Technologies: ACT 100, an aqueous based cleaner with dimethylacetamide, diethanol methylamine and surfactants.
Kester Solder: Kester 5769, an aqueous based cleaner with monoethanolamine and surfactants.
Isopropyl alcohol (IPA)
Trichloroethylene (TCE), as the control.

As mentioned previously, the use of aqueous cleaners in the W89 is restricted to limited applications. For multiple printed wiring assemblies like the MC4069, aqueous cleaners are not suitable. After initial studies the aqueous candidates, ACT 100 and Kester 5769 were dropped from further considerations. Ultimately two other cleaners were evaluated, Petroferm: Bioact EC-7R, d-limonene with an addition of methyl laurate instead of surfactants, and finally Food Grade d-limonene.

Initial cleaning tests were performed to determine the ability to remove Kester 197 RMA Flux, a Rosin Mildly Activated flux with an amine hydrobromide as an activator. These tests were performed on substrates that mock up the materials in the firing set: Bare copper, copper dipped in 50/50 lead-tin solder, 17-4PH stainless steel (the firing set housing material), and polyimide glass (the printed wiring board material). The cleaning was performed by manual spraying with aspirated spray guns (80 psig, 45° spray angle), followed by isopropyl alcohol (IPA) spray rinse and dry nitrogen blow dry. The aqueous based cleaners also included a deionized water rinse prior to the IPA rinse. Because Bioact EC-7 contained surfactants this water rinse was also used, but was elmininated for EC-7R and d-limonene. Some of the first tests involved heating the cleaner to 110 or 120° F to determine if heating would improve the cleaning efficacy. It was determined that there was no discernable improvement, probably because once the solvent is sprayed, its temperature rapidly decreases to room temperature. The samples were sprayed until they appeared visually clean, or until it was apparent that the cleaner would not remove the contaminants.

After cleaning, the substrates were measured for cleanliness by several different methods:
-Visual inspection under room light and ultraviolet light.
-Omegameter to measure residual ionic contamination in an agitated IPA/deionized water solution.
-MESERAN (Measurement and Evaluation of Surfaces by Evaporative Rate ANalysis) to measure organic contamination.
-GAR-FTIR (Glazing Angle Reflectometer-Fourier Transform InfraRed) spectroscopy
-Water Drop Contact Angle Goniometry and water break tests.
- Surface analysis by AES (Auger Electron Spectroscopy) and XPS (X-ray Photoelectron Spectroscopy).

Many of these cleanliness measurements are complementary rather than redundant, and are sensitive in indicating the presence of only certain types of contaminants. However, to summarize a general indication of cleaning efficacy the Meseran results are presented here in figures 7-10. Reference 19 contains the complete data set. A Meseran number of 50 or lower indicates a very clean surface, 200 or less are moderately clean or sufficiently cleaned for in-process applications. Results greater than 200 indicate a grossly contaminated surface. Present along with the Meseran results is the spray times required to clean the surface for each solvent. Figures 7 and 8 show the removal of solder flux from the solder side of the solder dipped copper substrates. The data show that all the cleaners except ACT-100 and isopropyl alcohol easily remove the solder flux. ACT-100 gave two wildly varying results for its removal ability. Isopropyl alcohol showed better flux removal capabilities on some substrates however those results were experimental artifacts. Once the solder flux is heated in air as occurs in the soldering operation, it becomes partially polymerized and becomes much more difficult to remove with just IPA. Figure 9 and 10 show the efficacy of the RAM 225 silicone mold release removal. Figure 10 shows the removal on aluminum substrates instead of solder dipped copper. These results show that the two aqueous based cleaners are very poor at removing the mold release. Exxate 1000 requires much longer times to completely remove the mold release than the other effective cleaners.

Workers also tended to have difficulties working with Exxate 1000. It is a heavier and thicker liquid than most cleaners. As it is sprayed, and it usually required a long spray time, a fog would form in the fume hood that made visibility of the part difficult. It also appeared more difficult to rinse.

General Contaminants

In addition to the removal of flux and mold release required by the cleaning operation, the cleaning operation should also remove as many general contaminants as possible. In fact, some may view this aspect of the cleaning operation with equal importance. A final cleaning operation may be necessary for reliability even if future fluxless soldering processes are developed. A survey of the KCD Firing Set production area was made to compile a list of contaminants that are possible to find on production parts. These materials are listed in Table 4. Because all of these contaminents were organic, the cleanliness was evaluated by Meseran analysis. Aluminum samples were coated with the individual contaminant, allowed to air dry for 3 days, then spray cleaned until visually clean and finally rinsed in IPA, or for EC-7, rinsed in deionized water and then IPA. Overall, the cleaners shown to be effective for flux and mold release removal were equally effective against most types of contaminants. However, d-limonene and Bioact EC-7R were not as effective as the EC-7 formulation which included surfactants. In particular, d-limonene has some difficulty removing Epon 828 epoxy resin, OSR mold release and Versamid 140 epoxy curing agent. Only moderately clean surfaces were achieved in cleaning these contaminants, (Meseran numbers between 129 and 197). Also, d-limonene was very ineffective in removing Krylon acrylic overcoat, (Meseran number 1296). Table 5 is a compilation of these Meseran results. It should be noted that removal of Krylon may not an entirely desirable feature. While one would like overspray removal to occur, inadvertent removal of important product and component markings might occur. Experience has shown that Krylon is effectively removed from actual parts. The rigorous Meseran evaluation apparently used coatings considerably thicker than those applied in actual production practices.

Potential for Cleaner Residue

The cleaners used in traditional cleaning processes have very high vapor pressures. Subsequent to assembly of the warhead virtually all of these chemicals have evaporated and no residual is present. This was confirmed in the W89 system compatibility tests. The first series of core tests, CTU 1-10 showed no sign of residual CFC's or TCE in two years of atmosphere analysis. The full scale warhead assembly, DAAU-1, shows only traces of TCE. These new cleaner candidates have lower vapor pressures and some have additives. Figure 11 shows the thermogravimetric analysis of the candidate cleaners. This analysis, where a sample of the cleaner is allowed to evaporated and is then heated to evaporate as much as possible, gives a measure of how much residual cleaner may be left behind. Curve D shows that for Exxate 1000, only 20% evaporates unless it is heated above 71°C (160° F). It must be heated to 200° C in order to evaporate most of the solvent. Curves B and C for Bioact EC-7 and EC-7R retain most of their additives until the temperature is raised above 71°C. This temperature is significant because the vacuum bake out is limited to 71°C to prevent damage to components. The result is that only d-limonene, which has no additives, completely evaporated (Curve A). On subsequent evaluations, a coaxial wire cleaned with EC-7R had wicked up large amounts of methyl laurate, the EC-7R additive.

Final Selection of Cleaner Substitute

As a result of the cleaning efficacy studies and the thermogravimetric analysis the final cleaner selection was d-limonene. d-limonene may be purchased from many chemical suppliers because it is a generic chemical rather than a commercially blended product. This allows greater control over the composition of the cleaner. In fact, EC-7R was originally thought to be pure d-limonene. Curve A of figure 11 was actually purchased as EC-7R (Ref. 20). Later lots of EC-7R were found to have the methyl laurate additive, a composition change that occurred without notification from the manufacturer, Petroferm. d-Limonene is not as effective of a cleaner against some contaminants as some of the other cleaners. However, it cleaned moderately well with the exception of Krylon overcoat. It was decided that this modest cleaning ability on low probability contaminants was superior to the certainty of introducing residual surfactants or methyl laurate into the warhead environment.

d-limonene Characteristics

d-limonene is a terpene, similar to turpentine. It, is a byproduct extracted from citrus fruit peels, mostly oranges. d-limonene is non toxic, biodegradable, non corrosive, and has low toxicity. At this time it is not OSHA regulated and has no inhalation hazards (Ref. 21). Its flash point, however, is 122° F (as measured by the closed cup method). This classifies it as a combustible fluid. It is also considered a volatile organic compound (VOC). This VOC designation is somewhat ironic. Terpenes as a class of chemicals are emitted by many green plants. Worldwide, 500 million metric tons are emitted annually. Because d-limonene is a by-product of the citrus industry, the amount used for cleaning would otherwise find its way into the environment. Currently the world diverts 610 metric tons of these terpenes for cleaning (Ref. 128). Terpenes are also used in many household cleaning products like Lysol®, Pine Sol® and Spic & Span ®. d-limonene is also used as a flavoring agent in foods (Ref. 18). The purity level selected for d-limonene as a cleaning solvent is food grade. This grade is 95% d-limonene in a liquid that is 100% terpene. d-limonene itself is odorless, but food grade has a strong orange odor. This is due to the presence of mercene (Ref. 22). Higher purity grades of d-limonene (99%) are available that are odorless, but cost about 6 times as much as food grade. Because d-limonene is a combustible liquid, cleaning operations will be performed in an explosion proof fume hood. The ventilation in such a fume hood should prevent the orange-like oder from becoming a nuisance for production workers. Written comments on the acceptability of d-limonene were solicited from the Environmental Protection, Industrial Hygiene, Fire Protection, and Safety organizations at KCD. These organizations indicated that its use was acceptable, provided that appropriate engineering controls (dealing primarily with fire protection) as they specified were employed (Ref. 23).

d-Limonene also has some susceptibility to auto-oxidation in air. There is also the possibility of some impurities such as alcohols and aldehydes that will leave a sticky residue upon evaporation of d-limonene. This Non Volatile Residue (NVR) is soluble in IPA and should be removed by the rinse. Steps will be taken to ensure that the amount of NVR is controlled. The d-limonene purchased will be specified to have 1% maximum NVR content. Oxidation products will be controlled by procedures to use nitrogen purge in its storage containers. Small amounts, nominally 500 ppm, of the common anti-oxidant butylated hydroxy toluene (BHT) will be added to the d-limonene. Based on the results of long term NVR testing a 1 year shelf life is specified. This addition to d-limonene should not have the same concerns as the additions in the Bioact EC-7 and EC-7R formulations. First these additions are very small, 0.05% max. versus 10% in the Bioact products. BHT is a common antioxidant, and is in polyolefin sleeving that has been used in warheads for years. There is, therefore, considerable stockpile history of having BHT in warheads with no known adverse effects. KCD has written a materials specification for d-limonene, 4612140. This specification is included as Appendix A. A specification, SS392260, to detail the cleaning procedures for the use of d-limonene for the firing set has been written (Ref. 24).

Subsequent sections of this report will address the issues related to d-limonene residual amounts that may remain in the warhead. The vapor pressure information for d-limonene is given in Table 6 (Ref. 25).

Immediate Effects of d-limonene Cleaning

In addition to cleaning efficacy, the cleaning process must not have any adverse effects on the functionality of the firing set. Immediate effects include corrosion or corrosivity to exposed metal parts before and after cleaning, encapsulant adhesion to the metal surfaces, solderability to the cleaned surfaces and high voltage breakdown testing.

Two types of corrosion tests were performed (Ref. 26). The first used bare copper and copper coated with 63% tin, 37% lead eutectic solder. Coupons of these two materials were immersed in each cleaner for 8 days. Weight loss or gain measurements and visual observations were taken. In the second test, samples of the copper coupons were etched in concentrated sulfuric acid, and coated with the Kester 197 RMA solder flux. Half of these copper coupons were then dipped halfway in eutectic lead tin solder. The coupons were then cleaned by the respective cleaner and then given either no, partial (30 seconds) or full rinse (2 minutes). These samples were then exposed to a 40° C, 70& relative humidity environment for 1 month.

The immersion samples cleaned with TCE or IPA showed no attack. A very slight weight loss was shown for copper with EC-7. For the one month exposure tests, EC-7 and IPA showed the most corrosion. Corrosion with EC-7 was worse for the no and partial rinsed samples. Complete rinsing eliminated any residue problem. Moreover, all cleaners were judged to be acceptable by the amount of corrosion exhibited in this test. It was not known if the surfactants in the EC-7 or the d-limonene itself contributed to the corrosion. But because all the cleaners were judged to be acceptable, the tests were not repeated with d-limonene.

The MC4069 Firing Set, as most electrical assemblies, is encapsulated to help the circuit boards withstand mechanical environments such as shock and vibration. Adhesion to the parts surfaces contributes significantly to the mechanical support that the encapsulation contributes to the component. Typically the encapsulation is a rigid polyurethane foam. In small test specimens this foam tends to be weak and it is difficult to obtain good adhesion test data. A standard test method was developed using a hollow steel cylinder bonded to the cleaned substrate with an epoxy adhesive, Epon 828 with CTBN (Carboxyl Terminated ButadieneAcrylonitrile) and DEA (diethanolamine) hardener. A torque is applied to the cylinder until the adhesive bond fails in shear. Here the choice of adhesive is not the major variable, the bondability of the cleaned substrate surface is the test article. Substrates of solder dipped copper, polyimide glass circuit board material, and 17-4PH stainless steel were contaminated with the Kester 197 RMA flux and cleaned with the candidate cleaners, including Bioact EC-7 (essentially d-limonene), IPA, and TCE (as the control). In all cases, there were no significant differences between the shear strengths and the failure locations using the different cleaners (Ref. 27).

High voltage breakdown (or hi-pot) tests were performed using a polyimide glass printed wiring board specimen that had conductive paths with varying spacings down to a .010" minimum gap. The test board was contaminated with Kester 197 RMA solder flux or RAM 225 mold release, then cleaned with TCE, IPA or the candidate cleaner, then encapsulated in GMB/CTBN epoxy. They were then thermal cycled between -55° F and +165° F in a 4 hour cycle for 5, 10, 20 and 100 cycle durations. The modules were then tested in a vacuum of 0.3 torr to 7400 volts, the leakage current was measured and then the modules were tested to failure. For EC-7, the leakage current test was superior to TCE and there was no significant difference in the high voltage breakdown values (Ref. 28).

Solderability tests determined if subsequent soldering could be performed after cleaning operations. The solderability was assessed using the Sandia designed Wettability and Video Imaging System. Soldering was performed using the Kester 197 RMA solder flux and 63% tin, 37% lead eutectic solder. The results indicated that EC-7 would be acceptable based on comparable values with TCE (Ref. 29).

Compatibility of d-limonene: W89 Warhead Materials

The cleaning efficacy and practical aspects of using d-limonene are only a small part of the verification efforts required to achieve acceptance for use in WR (War Reserve) applications. Much compatibility work and long term testing is required to yield as close as possible the same assurance level of reliability as TCE has from many years of stockpile experience.

It is clear that the d-limonene cleaning process has no effect on metals and other inorganic materials. Organic materials, however, are known to have reactions to many cleaners, including the presently used TCE process. The W89 warhead components were broken down into lists of organic materials and functional components. All of these materials and components, or equivalent component families, were tested for compatibility to the d-limonene cleaning process. Appendix B lists the organic materials in the W89 warhead. This list is organized by materials in the MC4069 firing set and then other W89 components. If the organic material is present in the MC4069, its listing was not be repeated under other components. Materials within hermetically sealed packages are not listed. For the materials within the MC4069 firing set, an expected subseptibility to a response to d-limonene exposure is also listed.

If the W89 program had entered production, a comprehensive materials database would have been required (Ref. 30). Such a database will be needed in the future, for activites such as Alt's, Mod's, or SIPs (Stockpile Improvement Programs), in determining actions to be taken on materials that may be deemed hazardous in the future, and for determining dismantlement methods during Phase 7, retirement activities. Reference 30 is included in this report as Appendix C. All components in the W89 were required by the system control drawings (CD's) to minimize the use of hazardous materials, and to list those hazardous materials that will be used (Ref. 31).

Compatibility Screening and Testing Philosophy

It was recognized early on in the program that compatibility testing of materials would be required. A screening test was devised which was a deliberate overtest considering the extremes of a postulated cleaning process. This test involves immersion in d-limonene (or, for early tests, EC-7 or EC-7R) at 50° C (122° F) for 15 minutes, rinsing in IPA at room temperature and nitrogen blow dry. These were compared to a baseline exposure of a 15 minute immersion in TCE at room temperature followed by the same rinsing and drying steps. TCE's high vapor pressure prevent using it for elevated immersion testing. At one time, heating the alternate cleaners was contemplated but dropped from consideration. However, to maintain a consistent test database, all materials were tested using the 50° C/15 minute immersion, including new materials selections which were made later in the W89 program as part of design changes.

After the test exposure, the materials were measured for weight gain and physically examined for any visible or texture changes.

If the material appeared significantly changed or degraded by this simple screening test, a series of more complicated tests were performed that were overtests of a lesser margin. For materials that continued to show adverse changes, the additional testing was performed with less margin and more realistic warhead and manufacturing environments. Typically this involved first testing the material in a nitrogen atmosphere saturated with d-limonene vapor, then in a nitrogen atmosphere containing the equivalent concentration of d-limonene vapor that would exist if 8, 2, 1, or 0.5 gms of free d-limonene were present in the atmosphere. (Determination of the exact amount of d-limonene likely to be present in the W89 warhead is treated in detail in a later section of this report.) Initial materials tests were performed with bulk materials. If further testing was required, these materials would be placed into more realistic test configurations, in laminated form for example. In some cases materials were also examined while wet with the cleaning solvent then dried but with solvent absorbed into the material and then dried with the solvent desorbed to the extent that would be realistic in terms of the manufacturing conditions for the W89 warhead.

The organic materials listed in Appendix B were categorized by materials types. Screening tests were performed on all types of materials. The screening test data for materials that showed little response are summarized in Table 7. Where multiple tests were performed the datum with the largest weight change is shown. For the sake of completeness, materials that have been dropped from the W89 due to subsequent design changes are still shown. In general, epoxies and "plastics" known to be rugged engineering materials are highly resistant to d-limonene exposure. Some epoxies, that have less cross-linking show a slight response. Table 8 summarizes the screening test results for bulk materials that show a greater response to d-limonene (Refs. 19, 32, 33, 34, 35). Rubbers, acrylics and polyurethanes show moderate or severe response to d-limonene. Many of these responses are absorption and swelling that is reversible if the d-limonene is allowed to evaporate or is vacuum baked out of the material. While not presented in the data here, it should be noted that Exxate 1000 showed similar absorption and swelling effects. However, where a drying or vacuum bake out procedure removed almost all of the TCE and much of the d-limonene or EC-7, virtually all of the Exxate 1000 remained in those samples.

It is interesting to note that TCE in most cases causes a greater adverse response than does d-limonene. If the large amount of swelling does not cause mechanical damage in the material, however, the high vapor pressure of TCE allows virtually all of it to evaporate and return the material to its original state. TCE and similar chemicals were introduced into the stockpile over a number of years as their advantages became known: high vapor pressure, no residue, non-flammable, and (originally thought to be) non-toxic. Stockpile confidence in the use of TCE evolved of a period of years. However, if it were examined in an intensive program like this ES&H driven effort, as is being done with d-limonene, this phenomenon of large reversible swelling would probably have made its acceptance much more difficult.

Coatings

The different inks, stamp pad inks, epoxy inks, epoxy paints, Hysol DK17 conformal coating for components and epoxy coatings on capacitors were also exposed to the screening test. Because they are thin coatings, it is not possible to get meaningful weight change information from these tests. Visually, there was no change in any of these materials. The epoxy ink, however, was slightly softer, and some of the stamp pad ink could be wiped off after exposure to all the cleaners. These slight effects were judged not to be significant adverse effects.

Cables (Wiring)

The different types of cables were also exposed to the screening test. The cables are made of materials known to be inert to all the cleaners, yet some of the cables showed as much as 10% weight gain by all of the cleaners. In fact, analysis of the coaxial cable from the Trig 1 circuit showed large amounts of methyl laurate deposits (Ref. 37). This led to the decision of changing from Bioact EC-7R to d-limonene. It is clear that cleaner can get wicked up between the insulation and the metal wire or in metal braids and, unless this cleaner has a high vapor pressure, it will remain entrapped there, unable to be removed by rinsing. Additives such as those in EC-7 and EC-7R would worsen this effect. Vacuum bake out experiments showed that virtually all of the d-limonene can be removed. However this is much slower and less efficient for longer cable lengths i. e. 6 inches versus 3 inches. Moreover, single cables in a vacuum oven represent an ideal situation compared to entire firing set circuit modules or ECA's. It is reasonable to expect the potential exists for entrapment of residual d-limonene.

Functional Components

In addition to raw materials testing, numerous functional components were screen tested. These components would be exposed to d-limonene, aged and function tested according to the specifications for the specific components. In all of these tests, no deleterious effects or performance trends of the d-limonene or EC-7 exposure were seen. The following components were tested (Refs. 38-46):

SA3587 Mica Paper Capacitor
Capacitors: SA3687, SA3686, SA2827, SA3619, SA3184
MC3547 High Voltage Triaxial Connector
SA2404 Rack and Panel Connector
Metal Film Resistor Families (Epoxy Coated): 440015 , 440022
Wire Wound Resistor Families (Molded Silicone Coated): 440016, 440017,
Wire Wound Resistor Families (Dipped Silicone Coated):440010, 440065
NEMA MW1000 Wire used in Magnetics
MC4226 Programmer Clock
Diode Contact Assemblies of the SA3642 Filter Connector used in the MC4078 LAC

The materials in the Rack and Panel Connector are similar to materials in the LAC and JT connectors that would be exposed to the d-limonene atmosphere. Therefore the testing performed on the Rack and Panel Connector also pertains to the MC4078 (Lightning Arrestor Connector) LAC and JT type connectors. The insulation resistance of these connectors was also measured while the connectors were wet with d-limonene. While the resistance values dropped when wet, they were still well above the minimum specification values. The resistance values recovered their original values after the connectors were dry. The MC3547 has silicone rubber gaskets around each pin. The swelling of the silicone rubber caused these gaskets to come loose around the pins and fall out. They were replaced prior to continued testing; once dry there were no adverse effects. It should be noted that a similar experience with these rubber gaskets would have occurred with TCE cleaning.

Rolamites

Rolamites for the W89 program were scheduled to be produced by Raymond Engineering. There is also rolamite assembly work done at KCD in the same area as Programmer fabrication. There was concern that ultimately, as d-limonene cleaning was adopted by other components' production, that the production environment might contain d-limonene vapors. The susceptibility of all rolamites to this vapor was a potential concern. Rolamite testing was performed differently than for the other components. It was conducted more as an adhesive evaluation study than that of a component functional performance study (Ref. 50). Currently there are two types of rolamite designs that have been produced, one with glass cases and two Kovar end caps bonded with 3M SW2216B/A gray epoxy adhesive per 2180835, the other with DAP case and end caps bonded with A2/E hardener epoxy adhesive per 2140015. Glass, Kovar and DAP are all highly resistant to d-limonene. Rolamites of both types were immersed in d-limonene for 15 minutes or 2 hours, rinsed in IPA and air blown dry. Some rolamites were tested immediately, others were thermal cycled from -54 to +71° C for 100 thermal cycles, other rolamites were isothermally aged at 71° C for three months. Both the thermal cycled and isothermally aged rolamites were exposed to a saturated d-limonene environment during aging. All rolamites were tested by cutting in half, removing the rollers, and testing the end cap bond to failure by performing a "push out" test with a rod inserted into the rolamite case. All testing showed there were now adverse effects from d-limonene exposure. (However, as one might expect, the glass rolamite data show considerable scatter due to the brittle nature of the case material.)

Nylon Locking Elements for Threaded Fasteners

Many of the screws and threaded fasteners used to assemble the W89 warhead structure contain nylon locking elements to prevent the fasteners from becoming loose in vibration environments. The requirements for these self locking screws is defined by military specification (Ref. 51). NAS1352N-LL-06 (10-32 thread) screws were exposed to the d-limonene screening test conditions. Running torque tests were then performed. These tests were performed using a threaded standard specimen with an inspected 10-32 UNF Class 3B thread. The tests were performed using uncoated threads and threads sprayed with dry film lubricant, according to the W89 warhead assembly procedures. In all cases for uncoated, sprayed, exposed or unexposed screws, there was wide scatter in the data. However, in all cases the unexposed screws has slightly lower running torque values. Since there would only be cause for concern if exposure to d-limonene significantly reduced the running torque values, these slight increases were judged to be acceptable.

Rigid Polyurethane Foam Encapsulant

Rigid polyurethane foam encapsulant was examined both early in this development program and reexamined later on. It is very important to the environmental conditions in the warhead because there is a lot of it in the firing set and the programmer. It represents a major mass of organic material in the warhead. Also, it is hydroscopic. It is the major source of water brought into the warhead. Because this water is somewhat mobile, as is the carbon dioxide reaction product that forms the pores of the foam, it changes weight easily during compatibility experiments. It became important to know precisely what this foams interaction is with d-limonene. The initial work showed that its weight gain was small after 24 hours immersion, 1.5% for EC-7 compared to 1% for water and no change for TCE. After a 24 hour drying period weight losses were recorded, 0.2% for EC-7, 0.6% for water and 1% for TCE. These small changes were judged to be insignificant - no visual or texture changes were noted (Ref. 47). In the second study, foam samples were desiccated for 7 days at 71° C to ensure they were dried out and most of the carbon dioxide had diffused out. After exposure to saturated d-limonene vapor, again for 7 days at 71° C, the foam showed a weight gain of 0.44%. Compression tests were also performed that showed no significant differences between foam that was exposed or not exposed to d-limonene (Ref. 48). While the weight gain is small, considering there is 335 gms of foam in the warhead, its potential for d-limonene absorption is considerable. The fact that the foam will be unharmed by the d-limonene is indeed fortunate.

Because this foam contains free TDI, it is also on the list of ES&H materials to be eliminated. Progress on that effort is summarized later in this report. Those new materials will also have to be examined for their interaction with d-limonene.

Molded Desiccant Foam

In order to control the dewpoint of the warhead internal environment and to meet some mechanical requirements, a relatively new material is used in the warhead. The Collar is made of Heat Resistant Rigid Urethane ("PAPI") Foam per specification # 2170674, filled with Molecular Sieve, 10 angstrom units per specification # 8500041. This, again, is a massive part, 1500 gm. At LLNL (Ref. 49), samples of this molded desiccant foam were immersed in d-limonene at 22 and 75° C for both 65 and 162 hours. Only weight gains of 0.6 to 1.6% were measured. The were no textural changes observed or detectable dimensional changes. This molded desiccant foam was not activated so that only the absorption of the urethane matrix was being measured. Some chemists have speculated that the desiccant material itself may absorb d-limonene. Careful studies with activated molded desiccant foam (Ref. 127) have shown that bulk samples weighing 40 grams would pick up 0.125% d-limonene, although if the material were powdered it would pick up much more (1%). In the actual bulk configuration, it is highly doubtful that the molded desiccant foam will absorb any d-limonene.

O-rings

The EPDM (Ethylene Propylene Diene) Rubber material is used for the o-rings that make the primary environmental seal for the W89 warhead. The screening tests showed a large reaction for this material. Additional testing was performed on .125" thick test slabs of EPDM that were obtained as a requirement for each lot of o-rings produced (Ref. 52). Specimens of this material were again exposed to the d-limonene screening test environment. Compression set and hardness testing was then performed as required by the material specification (SS455508). However, the compression set test requires the material be heated at 125° C for 70 hours before testing. Because this drives off the d-limonene in the material, a series of compression set tests were repeated after 70 hours at room temperature and then 3 to 18 days after the first compression set test. The results of this testing is shown in Table 9. These results show that while the EPDM is initially softened and swollen, it recovers all of its properties after 18 days. The reaction to d-limonene is a swelling effect only and is not permanent even under overtest conditions. However, in actual use o-rings would only be exposed to the d-limonene vapor that is residual to the warhead. Functional o-rings test samples were designed and fabricated that mocked up an actual o-ring application in the W89 warhead. The sample mocked up the J1 or MC4078 LAC connection to the warhead. The actual Environmental O-rings, LAC (P/N 455522) procured in Phase 3 development were used. These specimens were vacuum leak tested, exposed to saturated d-limonene vapor while thermal cycled from -54 to +71° C for 100 thermal cycles, or isothermally aged at 71° C for three months. After aging they were leak tested again. In all cases, the leak rate was less than 2.5×10^{-8} cc std He at STP/sec. There was no difference between exposed o-rings and the unexposed, control group. The W89 warhead was designed with an allowance of 1×10^{-6} cc std He at STP/sec. Upon disassembly of these test specimens it was discovered that while in the saturated vapor canisters, the test units must have been tipped because the specimens were exposed to liquid d-limonene for a short period. In view of this being an increased overtest, it is clear that EPDM o-rings are perfectly acceptable in a warhead environment that experienced d-limonene cleaning.

Polystyrene

Of all the materials that reacted to d-limonene, polystyrene had the worst reaction. TCE, though, would produce a similar reaction. Where polystyrene is exposed, only IPA can be used for cleaning. This cleaning process will provide much less cleaning efficacy than do the other cleaning processes. Polystyrene is used for the encapsulant of the CAP (Code

Activated Processor). While the CAP is not hermetically sealed in a rigorous fashion, it is solder sealed and then given a gross leak test to ensure the leak rate is less than 10^{-4} std cc of He at STP/sec. Two mock-up CAPS were tested by 24 hour immersion into EC-7 and rinsed with IPA. No solvent leakage occurred into the CAP housing. The polystyrene was analyzed afterward - confirming this. As a result, d-limonene cleaning was judged to be acceptable to the CAP component engineers (Ref. 36).

While polystyrene does not pose a greater incompatibility with d-limonene or EC-7 than with traditional TCE cleaning, designers must realize it is not to be directly exposed to a d-limonene cleaning process.

Pyralux and Flex Cables

Pyralux is acrylic film adhesive that is used in flex cable circuits. Early on, it showed a susceptibility to d-limonene in the bulk form. However, Pyralux is always used as a laminate between layers of Kapton and copper. In the Kapton/Pyralux/Kapton configuration the Pyralux showed almost no susceptibility to EC-7. Because flex cables are a critical functional component of the W89 WES, it was decided to do extensive functional testing on flex cable material. Flex cables specimens were fabricated with an interdigitated copper pattern, Figure 12. These specimens were then stressed by mounting them in a fixture. Samples were placed in a d-limonene saturated vapor atmosphere for 3, 6 and 12 months of isothermal aging at 71° C or 100 thermal cycles between -54° and +71° C. The specimens were measured for weight gain , peel strength, surface resistance and voltage breakdown (Ref. 53). Weight gain studies showed that after being exposed to the screening test conditions, that samples had 0.2 to 0.7 % weight gain. After 1-5 days at room temperature, much of this weight gain was lost. From 5-10 days, there was little change. For both TCE and EC-7, there was a final weight gain of 0.1%. There was no difference in peel strength for specimens cleaned in TCE, EC-7 or uncleaned control specimens. Between the control specimens and the d-limonene exposed specimens, there was no significant differences before or after aging or thermal cycling for peel strength, insulation resistance or voltage breakdown (Ref. 54).

Adhesives

Particular attention was paid to adhesives. Adhesives used in the Firing Set were bonded in standard lap shear specimens made of either aluminum or epoxy glass printed wiring board (PWB) laminate material. The substrates were cleaned in TCE or EC-7R. Specimens that had substrates cleaned with EC-7R were also exposed to a saturated d-limonene vapor for 3, 6 and 12 months of isothermal aging at 71° C or 100 thermal cycles between -54° and +71° C. For adhesives that showed some response with aging, a second set of specimens were isothermally aged in a nitrogen atmosphere at 71° C for 3 months as a control (Refs. 55, 56, 62). The adhesives tested were:

Epibond 104 Epoxy
MB-185 Epoxy
Epon 828/GMB/CTBN/DEA Epoxy
Polysulfide
MVP-11 Acrylic
Y9469 Pressure Sensitive Adhesive (PSA) Acrylic Tape

Where the substrates were cleaned with TCE or EC-7R, there were no differences in the zero time strengths of any of these adhesives. After 100 thermal cycles, the Y9469 (PSA) Acrylic Tape had only half its original strength while all other materials showed no degradation. Two other adhesives showed degradation during the isothermal exposure testing. Table 10 summarizes this data. MVP-11 and Y9469 show that, similar to Pyralux, that acrylics are sensitive to d-limonene exposure. Polysulfide is also affected. However, these adhesives are used only as assembly aids in the firing set. Retention of their mechanical properties over stockpile life is unimportant. What is important for these materials is that their response to d-limonene does not form or release harmful reaction products into the warhead atmosphere. Gas analysis was performed on the atmospere of the canisters that were used to store these specimens during aging. While traces of sulfur, methacrylate, toluene and sulfur dioxide were detected, these gas traces were previously known to exist in warhead atmospheres and stockpile history indicates that they will not be a problem in these applications. Polysulfide, however, warrants additional attention. While only an assembly aid in the firing set, it is a unique adhesive because of its high elasticity. It is used in other applications in the W89 and other warheads. It strength degradation was accompanied by an observed loss of elasticity. The 3 month aging data in nitrogen looked strange, perhaps due to a different lot of polysulfide that may have cured differently. A second set of more controlled polysulfide tests has now been started to look more closely at its response to aging with and without d-limonene exposure (Ref. 57, 132, and 134). Lap shear specimens with aluminum and stainless steel substrates were isothermally aged up to one year at 71 ^{o}C (160 ^{o}F) or thermally cycled up to 200 cycles from -60 to +160 ^{o}F while exposed to varying concentrations of d-limonene vapor representing 2 to 8 grams of residual d-limonene in the warhead. The results showed that, compared to control samples, that the shear strength of the polysulfide significantly decreased from an initial shear strength of approximately 300 psi, to 100 psi, while control aged samples showed a strength of 200 psi. These strength reductions were seen after 6 months of isothermal aging. The reductions after both 100 and 200 thermal cycles were not as severe. However, the 12 month isothermal aging data was not significantly different than the 6 month data. This indicates that while there is a substantial reduction in the strength of polysulfide bonds, that the degradation does not continue indefinitely, but reaches some level of stability. Polysulfide is used primarily for its elasticity and compliance, not as a high strength structural bond. The remaining strength of the polysulfide bond after exposure to d-limonene should be sufficient for the applications where it is used.

Threadlocker adhesives were evaluated in a separate study (Ref. 58). Locktite 271, the highest strength blend of the 3 Locktites -271, 242 and 222 -used on the W89, was used in the study. In this study, threaded specimens were immersed for 2 hours in d-limonene, then vacuum or air baked out for 2 hours at 160^{o} F. No degradation was noted, and none would be expected for the lower strength grades of Locktite, either.

Other adhesives in the warhead were given the screening test and measured for weight gain. One adhesive, Halthane 88-3 showed a severe response. It will be discussed in a later section. Explostick 473, Epon 828/Versaimid 140, and LST are all epoxy based and showed no weight change (Ref. 55). APC-2.5 showed a significant increase, 6.67% weight gain. Similar to acrylics, silicone rubber adhesives are affected by d-limonene, yet their only uses in the W89 are as assembly aids. There response is a simple swelling mechanism and is not a worry in their applications in the W89 warhead. Silicon rubber components with structural requirements will be discussed in another section of this report.

HVLI

The High Voltage, Low Inductance (HVLI) connector is used in both the neutron generator and the main CDU circuits. It uses a silicon rubber molded part to hold the copper contacts and provide a high voltage standoff barrier. Silicone rubber will swell to large weight gains when in contact with liquid d-limonene. Dielectric strength testing was done to determine the electrical function effect of d-limonene on silicone rubber, Dow Corning 747U. Samples were allowed to swell by soaking up solvent and were measured in the swollen condition. Even with weight gains of up to 11% (for d-limonene and 13% (for TCE), there were no changes in the dielectric strength values (Ref. 63). A series of evaluations of the HVLI were performed where the HVLI was exposed to realistic amounts of d-limonene for the W89 warhead. HVLI units assembled in Main Contact and Seal Assemblies electrically functional test units were exposed to 100 thermal cycles between -54° and +71° C in saturated d-limonene vapor. Other samples were isothermally aged at 71° C in the canisters with d-limonene cleaned firing sets and pulled for electrical testing at 1, 3, and 6 months (Ref. 59). The test units were measured for contact resistance and vacuum Hi-Pot tested at 10 kVolts and 2.5 Torr vacuum. The test data show no difference between the control at time zero or any difference after 6 months exposure (Ref. 60). The samples were visually examined when the test units were disassembled, no adverse conditions were noted.

Cellular Silicone Cushions

Silicone rubber is also used in the LLNL designed cellular silicone cushions. These cushions have the critical function of accommodating differential thermal expansion stresses so that the main charge high explosive components are not damaged. Samples of the cellular silicone material, Silicone Foam M9750, showed signs of severe swelling in the presence of saturated d-limonene vapor. A series of experiments were devised that exposed the pad material in the same configuration as expected in the warhead, that is, exposed only at the edges and under 32% compression. The amount of d-limonene was also limited. The d-limonene was varied in series of tests up to the equivalent of 12 grams of d-limonene in the warhead. This equivalent was based on the ratio of total cushion mass in warhead to test specimen mass. At the time of this testing it was thought that the maximum amount of residual d-limonene in the warhead would be 6-8 grams. The results from this test series, that exposed the specimens for up to 6 months at 71° C, is given in Table 11 (Ref. 61). Performance data is presented in terms of the load deflection test. It should be noted that there are significant changes in the response over time for the control samples as well. These data show that there are no differences in the thickness changes or the load deflection between the d-limonene exposed and the control specimens. It also must be kept in mind that TCE liquid and saturated vapor exposure also swells the cushion material (although, in stockpile no TCE exposure would be expected from the warhead environment).

LLNL reviewed this data in the light of other test results and concurred with the opinion that d-limonene does not pose a compatibility problem for their cellular silicone cushions in the W89.

Polyurethane Encapsulants and Polyurethane Sleeving

Polyurethane encapsulants and sleeving are used as insulation material in high voltage areas of the firing set. Large weight gains of both d-limonene and TCE are possible in these materials, although bake out experiments have shown that, under ideal conditions, anyway; virtually all of these solvents can be removed (Ref. 64). The dielectric strengths of the EN-7 and RN1501 were measured while the d-limonene and TCE were absorbed into the specimens. No significant degradation of the dielectric strength was noted. Where EN-7 was exposed to d-limonene and dried, the dielectric strength also remained unchanged. In addition, spectroscopic studies on EN-8 indicate that there is no chemical reaction between the absorbed d-limonene and the EN-8 (Ref. 66). Therefore as a class, polyurethanes are expected to suffer no degradation of electrical properties from d-limonene.

Polyolefin Sleeving

Polyolefin also shows weight gain from d-limonene exposure. Because it is used as shrink tubing, it was considered to have two forms, as-received, and shrunk. Similar to polyurethane materials, vacuum bakeouts completely remove d-limonene from polyurethane (Ref. 66). Also dielectric strength with d-limonene absorbed, or after cleaning and drying, remain, unchanged (Ref. 65). Mechanical testing (modulus, tensile strength, and ultimate elongation) of the polyolefin was performed (Ref. 67). It was shown that d-limonene has no effect on either type of tubing after 1 minute or 24 hours of immersion.

Halthane 88-3

Halthane 88-3 is a polyurethane adhesive developed by LLNL and Y-12. It does not contain any TDI and uses a curative (Asilamine) that is not considered a suspect human carcinogen. Halthane 88-3 is used by LLNL in the bonding the secondary assembly and is used by Sandia in several areas as an encapsulant. Halthane 88-3 specimens were placed in thermal cycling and isothermal aging chambers with saturated d-limonene vapor and were found to have fallen apart in 3 days or less. LLNL embarked on an evaluation study to carefully determine what amount of d-limonene effects the Halthane 88-3 bond strength (Ref. 68). Specimens whose total mass equaled the mass of Halthane in the LLNL bond were exposed to from 0.5 to 2.0 grams of residual d-limonene. This determined how much d-limonene the bond would actually absorb in the warhead environment. With this range of d-limonene, weight gains from 0.25% to about 8% were observed. Standard lap shear bonds were then exposed to d-limonene to absorb this same amount and tested. The results showed that Halthane is still sensitive to d-limonene in these amounts. As shown in Figure 13, the bond exhibits a detectable loss in strength form 2-3% weight gain and substantial losses (about 50%) with only a 5% weight gain. Using a high temperature cure for the Halthane does not significantly improve its resistance to d-limonene. The actual amount of d-limonene in the warhead is vital in determining the effect on this bond. Also the amount of d-limonene absorbed by other materials is important.

It should be noted that this appears to be another swelling phenomenon. d-Limonene is also rapidly desorbed out of Halthane 88-3. It appears the d-limonene swells the bond until the interfacial stresses between the Halthane and the substrate are nearly as large or larger than the Halthane's adherence strength to the substrate. If three dimensional restraint effects are present they may restrict Halthane from absorbing d-limonene. This effect was seen when, armed with the knowledge of d-limonene's effect on Halthane, an assembly was exposed to d-limonene to debond and reuse an expensive assembly. Debonding did not occur after one month of saturated vapor expposure and liquid immersion did not rapidly speed the process. This effect was also noted in that lap shear specimens absorbed slightly less d-limonene than bulk samples.

Additional testing was performed to determine what the long term effects might be when the Halthane 88-3 absorbed some amount of d-limonene. LLNL performed an isothermal aging study where lap shear speciments were allowed to absorb from 2 to 4% d-limonene and aged at 70 ^{O}C for up to 6 months (Ref. 134). The results, shown in figure 14, indicate that no strength degradation occurs with time for Halthane 88-3 bonds.

The LLNL application of the Halthane bond does not perform a critical structural load during stockpile, but its strength is important during assembly and disassembly operations. Halthane 88-3 is a very attractive material for use in warheads based on its ES&H compliant nature and other attributes. Additional applications in warhead designs looks encouraging, provided that exposure to large amounts of d-limonene during manufacturing can be can be avoided.

Firing Set Testing

A test plan was developed to expose functional Firing Set hardware to d-limonene and perform performance testing afterward. The MC4069 Firing Set is composed of modules of different circuits and functions. Modules were cleaned with Bioact EC-7R, (prior to the switch to d-limonene) or TCE as a control. One group of four EC-7R cleaned module sets and one TCE cleaned module set was given 100 thermal cycles between -54° and +71° C, removed and electrically tested, placed in isothermal aging at +71° C, and removed for electrical testing after 1, 2, 4, and 6 months. A second group of modules was similarly cleaned and aged except that the 6 month isothermal aging and electrical tests at 1, 2, 4 and 6 months as performed before the thermal cycling and testing. For both these groups, the EC-7R cleaned modules were exposed to 0.05 volume % d-limonene/nitrogen atmosphere for a saturated vapor environment. Five complete firing sets were also built. At this level, four were cleaned with d-limonene and one with TCE. Two firing sets were aged and tested similar to the first group of modules (thermal cycling first). The TCE cleaned and the remaining two firing sets were aged with the thermal cycles last. For the complete firing sets, there was no additional d-limonene added. It was felt that since the MC4069 Firing Set was expected to bring the most d-limonene into the W89 warhead, that additions of d-limonene would be an overtest and that no addition would be the most realistic test. The HVLI testing described above was done with some of these firing set canisters. Table 12 depicts the aging matrix. For testing, the firing sets are function tested according to component acceptance criteria. Visual examination and gas analysis were also performed. The gas analysis did not indicate anything unusual in any of the canisters. Virtually all of the electrical testing indicated no problems with d-limonene. There were electrical failures, however the causes of all of these failures have been positively identified as problems due to immature or non-WR quality hardware that had nothing to do with d-limonene. Visually one transformer supplied for firing sets by the Pinellas Plant showed some swelling. Its appearance was exaggerated because it was close to the surface of rigid polyurethane foam encapsulation. The unit is electrically functional. The condition of the transformer was reviewed by the component engineer and judged not to be a problem.

H.E. Materials

The LLNL high explosive materials were evaluated (Ref. 70). LX-17 IHE was not a concern, but LX-16 and ultrafine TATB were. In saturated vapor experiments, LX-16 would have approximately 1.5% weight gain, ultrafine TATB, 2.5% -probably as a surface area effect. LX-13 and ultrafine TATB were also shown to rapidly desorb d-limonene. For critical components, such as for what these materials are used for, formulation changes of greater than 0.1% require extensive requalification. Functional testing was planned on the W89 components made of LX-16, ultrafine TATB and LX-13. This testing would have exposed the materials to the equivalent of 8 grams of residual d-limonene in the warhead. In addition, the system level tests, DAAU-1 and the second core test series, CTU 11-15, will also evaluate these parts in units that used d-limonene cleaning. The results of the residual d-limonene analysis, described below, apparently alleviated some of the concerns regarding HE compatibility. Upon cancellation of the W89 program, the HE component tests and CTU 11-15 tests were never completed, and analysis of the DAAU-1 test has not been performed.

Sandia energetic materials were also evaluated (Ref. 71). Thermal Analysis tests were performed on $TiH_{1.65}/KClO_4$, CP, PETN, $Ti/KClO_4$, HNS, HMX, and Barium Styphnate. Chemical Reactivity tests were also performed for $TiH_{1.65}/KClO_4$, CP, and PETN. With the exception of HMX, which is not used on the W89 and is known for its compatibility problems in other systems, no adverse effects of EC-7 were observed. The HMX only had a slight reaction, considering its reactivity with other materials, it is not expected to have a problem with d-limonene, either.

Partition Experiment:

Because of some of the compatibility issues with organic materials, it became clear that more sophisticated testing would be necessary for some materials. It is important to know the amounts of d-limonene that will actually be absorbed by each organic material. Most evaluations were conducted assuming an amount of d-limonene in the warhead and further assuming that the sensitive material under test would absorb all of that amount. A test was configured to look at all of the d-limonene absorbers together to determine which materials would absorb d-limonene in favor of others that might desorb it. These tests were performed at LLNL to include the energetic materials (Ref. 72). The partitioning experiments were performed using the quantities of each d-limonene scaled to the proportions found in the W89 warhead. The free volume of the test canisters was also adjusted with the addition of inert aluminum blocks to scale properly to the W89 warhead. The proportions for the W89/SRAM A application were used for this experiment. Four d-limonene quantities were used representing 0, 2, 4, and 8 residual grams of d-limonene in the warhead. Lesser quantities were also desired, but the scale of the experiment does not allow for meaningful weight gain measurements for additions of less than the equivalent of 2 grams of d-limonene. The containers were heated from room temperature to 155^{o} F in 3 days, held at 155^{o} F for 16 days to mock up diffusion controlled transport for the equivalent of a 20 year stockpile life (Ref. 73), cooled to room temperature in 18 hours and allowed to equilibrate at room temperature for one week. While the molded desiccant foam was activated, and all materials were kept as dry as possible, it was necessary exclude the contributions of the rigid polyurethane foam and the molded desiccant foam from the analysis on the assumption that water weight gain significantly affected these two materials. Figures 15 and 16 show the normalized weight changes for all materials. Figure 16 displays the data only for the energetic materials. Based on this data, the LLNL d-limonene partitioning model was revised. It now predicts a weight gain for LX-16 of 0.01%.

Residual d-limonene Analysis

In addition to the partitioning quantities of d-limonene between the various absorber materials in the W89 warhead, the actual quantity of residual d-limonene present in the warhead is most important for the compatibility evaluations. d-Limonene is thought to come from two sources in the warhead. For a component like the MC4069 Firing Set it will come in as absorbed into those particularly absorbing organic materials directly exposed to the cleaning process. Secondly, in areas where the d-limonene will wick up by capillary action and not be rinsed out. This second effect made it impossible to calculate the amount of d-limonene in a MC4069. Weight gain measurements were made (Ref. 74, 75, 76), but because of the large mass of the part, the in-process weight changes such as evaporation of the solder flux vehicle, and the addition of the lead-tin solder, the resolution of these data made them meaningless. These weight gain changes lead to an estimate that about 6-8 grams of residual d-limonene may be possible. A "fluxless solderless" firing set was built (Ref. 77). This was an attempt to eliminate large changes in mass during a mock module and firing set build up with all the realistic cleaning operations being performed. Weight changes were recorded immediately after cleaning, and 1 hour after cleaning, for both d-limonene cleaning and IPA only cleaning. The data show that immediately after cleaning there are large weight gains, more for IPA than for d-limonene. After a 1 hour wait, the d-limonene cleaning process actually totals a loss, 0.40 grams, while the IPA process total is a gain of 1.43 grams. The total weight gain 1.03 grams. From this data it is hard to say what mass exchanges are actually occurring, but a high end figure of 2.0 gram residual d-limonene for the warhead was assumed for further compatibility testing purposes.

This confusion over residual d-limonene quantities lead to an entirely different approach. Whole firing set units were built for the purpose of baking out and analyzing the quantity of d-limonene evolved for the unit. This approach would not only directly measure the quantity of d-limonene, it would also measure the amount of d-limonene that would evolve from the unit and be available for reaction with other components in the warhead. Three MC4069 firing set units are available for this purpose: one from the firing set aging studies for functional testing, one built to the ECA level but not encapsulated, and the DLA-1 unit, built as close as possible to typical production manufacturing, handling and storage methods. These last two units were placed in canisters, and baked out to mock the 20 year stockpile life of the W89 (Ref. 73), then the atmosphere in the canister was analyzed for the total quantity of d-limonene. Between these two units, it will be possible to determine if longer diffusion distances caused by the presence of the encapsulation have an effect on the amount of d-limonene available to the warhead environment.

In addition to the Firing Set, a DLA-1 MC4073 programmer has also been built. This unit was originally built to the ECA level cleaning with TCE. It was then cleaned twice at the ECA level with d-limonene (once to mock up the cleaning at the PWA level, once to mock the cleaning at the ECA level). The procedure is acceptable because there is much more access in the geometry of the programmer compared to the MC4069 Firing Set. After fabrication at the ECA level, the programmer in not cleaned again, but it does undergo a chip burn in process, which is a long time, elevated process prior to encapsulation. It is expected to hold much less d-limonene than the MC4069.

An analysis technique to quantify the d-limonene had to be developed. First experiments showed that is may be difficult to collect all of the d-limonene. Yields of as low as 11% were achieved in the first attempts. That yield has improved to greater than 90% by improvements to the sampling apparatus and the use of higher vacuum levels to provide for d-limonene transfer (Ref. 78). Pantex plant had developed a technique for analyzing for d-limonene in the DAAU-1 warhead test unit. This analytical method is quite sensitive, with a delectability limit of 96 milligram/liter. However, it was also discovered that d-limonene transfers quickly at a vacuum pressure of 0.2 Torr, but does not readily transfer above 0.6 Torr (Ref. 79). Using this information and additional refinements it is expected that the analytical procedure for d-limonene developed at KCD has a limit of delectability of 0.1 milligrams or less and a quantitative accuracy of approximately 20% (Ref. 129).

The first MC4069 firing set unit, actually used to prove out the system without risk to the two specially built units was from the functional firing set test programs aged in canisters at KCD. Recall that test intentionally added d-limonene to the canister atmosphere. This unit was not baked out but vacuum sampled:

1 trial sample: 11. milligram
2 trial sample: 25. milligram
3 trial sample: 3.9 milligram
4 trial sample: 3.9 milligram
5 trial sample: 0.53 milligram

The two sample firing set units were baked out and sampled, not once but three times This represents the equivalent of 3 stockpile lives, i.e. 60 years.

Unit 544 Unencapsulated Unit
1st sampling: 1.26 milligram
2nd sampling: 1.7 milligram
3rd sampling: 1.08 milligram

Unit 527 DLA-1, encapsulated unit
1st sampling: 2.75 milligram
2nd sampling: 0.31 milligram
3rd sampling: None Detected

MC4073 Programmer DLA-1 Units
After 3 samplings: None Detected

It can be seen that the amount of d-limonene in the firing sets is far less than the 2-12 grams estimated by the previous methods. It also shows that a component with a simpler form factor like the programmer has almost no potential for retaining d-limonene. It is clear however that foam encapsulant will restrict the mobility of any retained d-limonene. To determine that actual amount of d-limonene residing in the firing sets, as opposed to the mobile d-limonene that was measured here, an additional test has been proposed using supercritical carbon dioxide extraction. This test is pending, but it requires highly specialized equipment and additional analytical development. Results are not expected for some time.

DAAU-1 d-limonene Analysis

The DAAU-1 test unit was a full scale W89 warhead. The MC4069 Firing Set for this unit had the modules cleaned with TCE, but the final ECA level assembly was performed using EC-7R cleaning. The MC4073 programmer was cleaned with d-limonene. At the time of assembly of the DAAU-1 warhead unit, no citrus odor was detectable coming from either of these two components. The complete description of the DAAU-1 testing cycle is described later in this report. Gas analysis for DAAU-1 is available for the first 6 months aging (Ref. 80), as well as the complete post test analysis (Ref. 130). No d-limonene has been detected in any of the interim gas samples. The post test analysis took a much larger gas sample and only a very minute peak of d-limonene was detected (smaller that what lends itself to quantiative estimates, despite the high sensitivity of the analysis technique.) For all practical purposes, there was no mobile d-limonene in the DAAU-1 atmosphere.

Waste Minimization

As a final note in the development of d-limonene cleaning, waste quantities were examined. Because d-limonene has a flash point less than 125° F, it must be treated as a hazardous waste and a combustible liquid. The situation is the same for the IPA rinse. KCD plans are to send this waste out of the plant for commercial incineration. The current cost for this is \$0.3/lb (or \$2.10 per gallon of d-limonene). The use of d-limonene with an IPA rinse was compared to the traditional use of TCE and IPA. KCD disposes of this by commercial vendor (incinerates) also at \$2.10/ Gallon. It was found that for the MC4069 Firing Set, 3.74 gal d-limonene and 7.93 gal IPA would be used. This is approximately 2.6 times as much d-limonene as TCE and 2.2 times as much IPA (Ref. 69). It should be noted that there is also an accompanying specification change. Initially, the d-limonene cleaning specification called out the minimum time required both for d-limonene cleaning and IPA rinse. It was noted in this study the IPA rinse time specified is considerably longer than what was typically used when the earlier cleaning specification, which merely stated to rinse until clean, applied. (The minimum times have since been deleted from the specification as a requirement, but average spraying time for the d-limonene process hasn't changed.) While these amounts are higher, the overall ES&H benefit outweighs the added waste. Furthermore, in future studies, ways to optimize the cleaning and minimize the waste volume could greatly improve upon these quantities.

Passivation of the Firing Set Castings

The MC4069 is housed in a 17-4 PH stainless steel casting. Upon receipt of this casting from the vender, it is machined to provide additional features and establish more accuracy for existing features. After these operations it is necessary to clean and passivate the casting. The passivation process, actually a nitric acid cleaning process used to get stainless steel parts very clean, requires pre-cleaning steps to assure the gross organic, i.e.

oil, contamination is removed. Traditionally, the parts were cleaned after machining in the machining area at KCD, Department 95, then moved to the chemical cleaning area, Department 97 where the part is again precleaned and passivated. In both areas the preclean consisted of using TCE, followed by an aqueous cleaning operation called descaling. TCE cleaning was the step that needed to be changed. Originally, Department 95 switched to high flash point mineral spirits (Safety-Kleen 140). However, there were numerous worker complaints about fumes. While mineral spirits are non-toxic, the odor problems for the workers were judged to be serious enough to require changing.

Actrel 4493L was selected. This is a highly purified mineral spirits with a high flash point and very low odor (Ref. 81).

For the total cleaning process, three different processes were evaluated.

1) The traditional process:
    In Dept. 95:
    TCE pre clean
    Aqueous Cleaner (Branson Industrial Cleaner)
    Transfer to Dept. 97:
    Clean with TCE
    Alkaline Cleaner (Oakite 90)
    Descaler Cleaner (Wyandotte Ferlon 500)
    Nitric Acid Passivate
    Descaler Cleaner (Wyandotte Ferlon 500)

2) The alternate process:
    In Dept. 95:
    Actrel 4493L
    Aqueous Cleaner (Branson Industrial Cleaner)
    Transfer to Dept. 97:
    Clean with Actrel 4493L
    Alkaline Cleaner (Oakite 90)
    Descaler Cleaner (Wyandotte Ferlon 500)
    Nitric Acid Passivate
    Descaler Cleaner (Wyandotte Ferlon 500)

3) A preferred alternate process:
    In Dept. 95:
    Actrel 4493L
    Aqueous Cleaner (Branson Industrial Cleaner)
    Transfer to Dept. 97:
    Descaler Cleaner (Wyandotte Ferlon 500)
    Nitric Acid Passivate
    Descaler Cleaner (Wyandotte Ferlon 500)

It was thought that in addition to the elimination of TCE, that the cleaning processes of the Actrel and Aqueous cleaner do not need to be repeated in Dept. 97.

The cleaners were evaluated against 5 contaminants that are processing fluids to which it is likely that the parts will be exposed:

Tapping Fluid - Energy Release Tapping Oil (97022000)
Cimstar 3700 - Water Base cutting fluid (97022340)
Van Straaten 5487C - Chlorinated and sulfurized cutting oil (97022418)
EDM Fluid - Exxon Mentor 28 (97022418)
Dye Penetrant - Zyglo ZL56

Initial evaluations were performed using AES (Auger Electron Spectroscopy). This is a very sensitive surface analytical tool. The results, shown in Table 13, indicate that all three processes get the housing material extremely clean (Ref. 82).

Follow-on tests were conducted to ensure that other compatibility problems did not arise. These included weldability (Ref. 83), bondability, using the same epoxied rod torque test (Ref. 84) technique as before (Ref. 27), corrosion and machining. The machine tests involved the use of tapping fluid in an actual tapping operation to mock up a high heat machining operation to determine if cutting oils could be baked on and become more difficult to clean. None of these follow up studies have shown any problems (Ref. 85). The preferred alternate process has become baseline for W89 firesets and Actrel 9943L in now in use for all Dept. 95 cleaning operations that once used TCE.

Other ES&H Activities at KCD:
In order to coordinate other ES&H activities of the W89 at KCD an implementation plan was written. This plan, orignally presented at the W89/SRAM II Phase 4 Readiness Review in June 1991, gives all materials and processes an ES&H rating. In turn all W89 product entities were given a rating based on the production and support materials used in its manufacture. Action plans for replacement materials and processes were organized by both part and manufacturing department. These schedules are updated and the ES&H status of each part is presented at the W89 KCD program reviews.

MC4073 Programmer Cleaning

Cleaning processes for the programmer will largely follow the steps of the MC4069 Firing Set. d-Limonene has been used in development hardware starting with units delivered in November 1991. The cleaning is performed in the firing set production area, a d-limonene cleaning facility in the programmer production area at KCD (Dept. 53) became operational. The printed wiring boards (PWB's) have switched from the traditional photoresist process that used TCE, to using aqueous processing using a water soluble solder mask material, Vacrel 8140. PWB's however are sensitive to absorbed water during subsequent soldering operations. After the aqueous process, the PWB's are baked out to a very low moisture content, below the dewpoint requirements of the W89 warhead (Ref. 86). The system compatibility tests, DAAU-1 and the second set of core tests, CTU 11-15, have programmers built using d-limonene cleaning. The programmer housing consists of a cast aluminum baseplate and a cover of formed aluminum sheet. Traditional cleaning of sheet metal parts used TCE or Freon, this will be replaced by a hot (120 - 150^{o} F) aqueous detergent cleaning process. The cleaner will be Castrol Clean 3625, a non foaming alkaline detergent that is high volume, high pressure (to 50 psi) sprayed, water rinsed and air blown dry.

W89 Cable Cleaning

The W89 cables consist of either round wire or flex cable circuits soldered to connectors. The soldered areas are traditionally cleaned with TCE, so d-limonene will be used as a replacement. The soldered areas are molded into backshells or potted shapes using Adiprene Cyanacure per 9927104. Additional materials were looked at using the d-limonene screening test developed for the firing set (Ref. 87). One material, nylon cord showed significant weight gain in d-limonene, but all was removed after a vacuum bake out at 71^{o} C for 40 min. This nylon cord, similar to the Pyralux adhesive layers, will be edge exposed to the d-limonene cleaning process at the ends of the flat flex cable. The connectors were previously shown to be d-limonene compatible. The Adiprene Cyanacure potting would absorb d-limonene, but it will not be directly exposed to it. The d-limonene would also easily desorb from the Adiprene Cyanacure. Six cables have been built for long term testing. These cables were built using d-limonene cleaning and are in a saturated d-limonene environment for 10,000 hours at 71° C . So far, from the data after two months (Ref. 88) and six months (Ref. 131) aging, there appears to be no problems after periodic electrical testing. Two W89 cables sets have been built with d-limonene for use by the component engineers. These cables were electrically function tested and Hi-pot tested. No problems have been seen. All subsequent cable production are planned to be cleaned with d-limonene. For the system compatibility tests, DAAU-1 had all cables cleaned with the traditional process. The second set of core tests, CTU 11-15, will have samples of round wire and flat flex cables soldered to connectors, cleaned with d-limonene and potted with Adiprene Cyanacure. A d-limonene cable cleaning specification, SS 398964, has been written, patterned after the d-limonene cleaning specification for the MC4069 firing set.

JTA Cleaning

The W89 JTA (Joint Test Assembly) is produced using the telemeter from the Telemetry Engineering Department at KCD. Traditionally they use TCE or AP-20 (80% perchloroethylene, another carcinogen needing replacement, and 20% amyl acetate) and are examining the use of d-limonene for cleaning. Components to be screen tested with d-limonene were listed. A review of the components that are used in telemetry units was conducted. Over 12,000 part numbers were identified. However these part numbers were found to relate to 5,000 purchased components which in turn could be reduced to 35 different groups, or types, of electronic packaging that needed to be evaluated. The evaluation used was different and more conservative than used for the firing set screening tests. It was felt that the telemetries are larger, more complicated units and components could see many more cleaning operations. The screening test used was a 24 hour room temperature immersion exposure to the solvent. A rating system from 1 (for completely compatible) to 5 (for totally incompatible) was used to assess each component. For intermediate ratings of compatibility a second test of 1 hour room temperature immersion was used. System level tests to determine long term aging effects are expected to be performed. These tests will be defined in detail by each project group. The Telemetry Project Department 134 at KCD intends to eliminate the use of AP-20 and TCE. An extensive list of compatibility ratings for components has been compiled for the purposes of conducting those assessments (Ref. 89).

Stronglink Cleaning

The MC4063 Single Stronglink Assembly (SSA) is located in the MC4069 Firing Set. It is a hermetically sealed unit so that is was not evaluated in the firing set cleaning studies. However, it uses the CFC blends Freon T-WD602 and Freon TMS for either ultrasonic cleaning or vapor degreasing (Ref. 90). The parts cleaned are mostly small mechanical piece parts and some mechanical subassemblies with welds. The SSA's are assembled in a clean room. Parts leave the clean room area to be welded and are returned as subassemblies These subassemblies must be cleaned in large part to remove any particulate matter that may have been formed during the welding process.

The development work was divided into 2 phases, in Phase 1 cleaning for stainless steel piece part and subassemblies with no trapping joints were changed to an ultrasonic detergent cleaning process and dried in a nitrogen purged oven. HEPA filters were installed in the oven to ensure the nitrogen drying process did not cause particulate contamination. The cleaning process used, P1251904, uses a mixture of Dirl-lum 603, an alkaline detergent with ammonium hydroxide. This cleaning was performed on 90% of the parts of the SSA. Two units of this type were built and evaluated after aging to greater than the equivalent of 20 years stockpile life (Ref. 91). No problems were experienced with these two units.

In Phase 2 the remaining 10% of parts and assemblies were to be cleaned with new alternate processes. These parts had either trapping joints, were coated with lubrication or could possibly be attacked by the detergent cleaning process. Initially, HCFC-123 was proposed for these applications. HCFC-123 is a hydrogenated chlorofluorocarbon with a low ozone depletion potential (0.02) slated to replace CFC-113, i.e. Freon. (Ref. 92). However, in June 1991, the results of toxicological testing in rats showed that tumor formation was so severe that the producers of HCFC-123 immediately pulled the product off the market for cleaning purposes (Ref. 93). The backup process selected is ultrasonic and vapor degreasing with IPA. Because it has a low flash point, fire protection with IPA ultrasonic and vapor cleaning is a concern. Until a UL approved cleaning system for production is procured, the cleaning is being performed with IPA using laboratory scale equipment. For some parts where only water based coolants are used, high pressure deionized water cleaning is being used.

For cable cleaning, to ensure there is no contact loop resistance problem, a Freon cleaning process was used. This has been replaced with a dry powder soda blast (using sodium bicarbonate) followed with an ultrasonic acetic acid cleaning step (to remove any powder traces), IPA rinse and blown dry (Ref. 120).

Structural Parts Cleaning

Cleaning of metal parts for the W89 structure does not have the same compatibility concerns as does electrical parts and organic materials. Aqueous alkaline detergent cleaning with Branson Industrial Cleaner is used. This cleaning is enhanced with heating, to about 150° F, and ultrasonic agitation. No problems should be encountered so long as the parts are rinsed and dried quickly. Most machining is performed using water base cutting fluids. However, where cutting oils are used, the Actrel 4493L, first used on the firing set housings, is used. The experience with Actrel 4493L has been excellent. There are no problems with its ability to clean parts and it has eliminated the problems with odor previously encountered with Safety-Kleen 140. Sheet metal formed parts, such as the Programmer Cover,will be cleaned with a hot (120 - 150° F) aqueous detergent cleaning process using Castrol Clean 3625. It is sprayed, water rinsed and air blown dry. The TCE vapor degreaser that was traditionally used has been removed from the production area. In the interim, sheet metal parts are being cleaned like machined parts, ultrasonically in Branson Industrial Cleaner.

Mound

MC4237 Initiator and MC3753 Actuator Cleaning

The MC4237 Initiator and MC3753 Actuator are electro-explosive devices (EED's) built at Mound for the W89 program. The MC3753 actuator is a multi-program part; it is shown in Figure 17. Mechanically, both these parts are identical. There are differences, however, in the type and amount of energetic materials in each of them. The headers for these parts were traditionally cleaned with methylene chloride, a suspect human carcinogen. The initial candidate replacement was n-methyl pyrrolidinone (NMP), however, one manufacturer of NMP had to make a "Significant Risk" notification to the EPA because toxicological testing showed NMP to be embryo-lethal (Ref. 94). After this notification, work on NMP at Mound was halted. Other candidate cleaners that were evaluated were Arco DPM, Bioact EC-7, and Ethyl Lactate. These cleaners were evaluated using XPS surface analysis .

Based upon these results, shown in Table 14, Bioact EC-7 was selected. The total cleaning process used at Mound is called the 4 Step Process (Ref. 95). It involves the following steps.:

-Ultrasonic clean in cleaner for 2 min., repeat (with fresh solution)

-Ultrasonic clean in 4 step solution for 5 min.:

	Parts
IPA	600
D. I. Water	150
Triton N-101	1.5
Span 80	.38

- D. I. Water Rinse
- Ultrasonic Clean in D. I. Water for 2 min.
- D. I. Water Rinse
- Ultrasonic Clean in IPA for 2 min.
- Heat Lamp Dry for 15 min.

However further evaluations showed that after each cleaning operation, the parts get no cleaner (Ref. 96), as shown in figure 18. Therefore, using Bioact EC-7, the cleaning was modified to what is called the 2 Step Process:

- Ultrasonic clean in EC-7 for 5 min., repeat
- Rinse in D. I. water at 75° C
- Ultrasonic clean in D. I. water at 75° C for 2 min., repeat twice
- Rinse in IPA for 1 min., repeat
- Argon blow dry

The MC3753 is a high reliability device. In the W89 WES design, its reliability value directly affects the warhead reliability statement. In order to assure that the MC3753 retains its high reliability, an extensive compatibility program was planned. As mentioned previously, compatibility studies were performed with EC-7 and all the Sandia energetic materials. An aging study for MC3753 and MC4237 components is underway. Table 15, shows the test matrix. Units are isothermally aged or thermal cycled at Pantex using the W89 DAAU-1 thermal cycle. After aging, the units are either disassembled for chemical analysis or performance (V.E.C.) tested. To date, 6 month and 1 year isothermal aging have been completed and tested without any observed problems.

Other portions of the MC3753 and MC4237 that are made in the Parts Machining Area will be ultrasonically cleaned in d-limonene after machining. Traditionally they would have been cleaned with Freon 113. All these metal parts will ultimately be cleaned again during later fabrication steps with the Bioact EC-7 in the two step process. The energetic materials loading used Freon and alcohol, now they will be cleaned using only alcohol.

DSSL Cleaning

The DSSL (Detonator Safing StrongLink) is similar in function to the MC4063 SSA. At Mound the piece parts made in the Part Machining Area will be ultrasonically cleaned with d-limonene instead of Freon. In the Surface Finishing Area, where plating is done, perchloroethylene has been traditionally used. In the E building general assembly operations, cleaning would be performed with either TCE or the methylene chloride 4 Step Process. These two areas will now use the EC-7 2 Step Process described for the MC3753. The EC-7 cleaning with the 2 Step Process should result in a very clean product. The cleaning actions of the EC-7 with the surfactant additives should be enhanced with the following water rinse and ultrasonic steps. At the same time, the water steps will ensure there is no residue from the (water soluble) surfactants. Because at the time that cleaning is performed the DSSL contains only inorganic piece parts, no residual d-limonene will be in the DSSL.

MC4295 Timer Cleaning

The MC4295 Timer of the MC4296 Timer and Slapper Assembly will be cleaned in the Parts Machining and Surface Finishing Areas at Mound. (There are no ES&H issues at the MC4296 part level.) As with the MC4237, MC3753, and DSSL, the Part Machining Area would traditionally use Freon 113, and the Surface Finishing Area would use perchloroethylene. As with the other components, the Parts Machining Area will now use d-limonene and the Surface Finishing Area will now use the EC-7 2 Step Process . In fact, the W89 ES&H development efforts at Mound have led to the complete removal of Freon 113 from any plant operation in the Part Machining Area. Mound is now free of all ES&H targeted cleaners for all operations, largely due the effort for the W89 Program.

PINELLAS PLANT

Wire Stripping for Magnetic Components

In the production of magnetic components, the insulation of the transformer wire must be stripped. These wires are typically very fine (Figure 19). Wire stripping is performed using the commercial product Iso-Verre. This product contains hydrofluoric acid, phenol, and methylene chloride. This stripper is a very active chemical that has severe fume problems and is a major cause of worker burns at the Pinellas Plant. Laser stripping, plasma stripping, UV curable resins, and direct welding were originally evaluated. Plasma stripping was initially selected. This process uses an atmosphere of 8% tetrafluoromethane in oxygen at 0.5 Torr. The insulation is stripped using a plasma at 400 watts for 45 minutes (Ref. 97). The process requires separate fixturing for every specific component design (Figure 20). Because these fixtures were complicated, a second alternative was developed where heat strippable wire is used (Ref. 98). The wire, NEMA Standard # MW28-C Class 130, has a polyurethane insulation coating with a nylon overcoat. Either solder on the solder iron is used to burn off the insulation or, for finer wires, the insulation is burned off during the solder operation. There will be some charred residue on the wire where the insulation has been burned off and some blistering of adjacent insulation may occur. These minor problems are resolved by using one additional wire wrap on the solder post so that wire coming from the post will be undisturbed, intact wire.

LAC Cleaning

The LAC connectors are currently cleaned with CFC's. Replacement with an aqueous cleaning process is being evaluated. The cleaning of connector shells was evaluated using the cleaner Oaktite NST (SS390941, per 46A100662, Schedule "G"). XPS analysis was used to examine the surface cleanliness of the connector pins, glass and interior metal shell. This evaluation showed that Oakite was an effective cleaner that left no contamination behind (Ref. 99). For other stages of the LAC fabrication, the geometrical configuration will make surface analysis impossible. Functional testing methods will be used prove out the cleaning method.

Heather Cleaning

The MC3318 has many different processing steps that use methylene chloride, TCE, or Freon for cleaning. Pinellas Plant is following the lead of the Rocky Flats Plant in using some of the alternate cleaners that have been implemented on gas transfer structural metal parts (Ref. 100). Procedures using Oakite NST cleaners are being evaluated at Pinellas for many of these similar cleaning applications (Refs. 101, 102). Oakite Citradet is being evaluated for use as a non aqueous cleaner in some of the other applications. Citradet is similar to Bioact EC-7, it is a d-limonene based cleaner with surfactant additions. However, Oakite changed the Citradet formulation (without notification) to include a new addition, hexaleneglycol, which is a toxic that is an ES&H disposal problem (Ref. 103) The old formulation will still be commercially available, however. A new Citradet specification, SS394027, has been written to assure the proper, older formulation is procured.

MC4135 Neutron Generator Cleaning

The Neutron Generator group first is looking at elimination techniques before starting development of cleaning replacements (Ref. 104). Neutron Generators are encapsulated using epoxy formulations. The molds and tooling for these encapsulation operations require large amounts of methylene chloride for equipment cleaning. A PTFE (i.e. Teflon-like material) impregnated nickel plating has been evaluated as a surface coating for molds. This prevents the epoxy from sticking to the molds, so excess is easily pulled off without the need for solvents. This coating, however, does not work as well on some of the tools used in the encapsulation process. Here a strippable plastic, Techtron PPS, is being evaluated as a tooling material. Also, disposable stainless steel and plastic liners are being evaluated. After an encapsulation operation, the whole liner would be discarded. This amount of non-toxic solid waste would actually be smaller and more easily handled than the methylene chloride liquid waste presently generated. Plastic bead blasting is being evaluated as a backup cleaning method. The Instrumentation Post in the neutron generators uses a substrate that is alumina filled epoxy. The epoxy impregnation operation also needs to use methylene chloride for cleaning. The attempt to avoid this cleaning is to change the substrate material to alumina. The cleaning of the high voltage ceramic elements uses Freon 113. It is thought that this cleaning process may be unnecessary. Elimination of this cleaning operation will be evaluated. Plasma cleaning is the back up operation.

The neutron tubes are composed of many piece parts. These are cleaned with TCE or methylene chloride vapor degreasing. These cleaning operations will be replaced with Alconox, a mildly alkaline detergent cleaner using an IPA or 3A Alcohol rinse to dry the parts. Some of the other parts such as the neutron tubes get cleaned with a Freon spray before testing. These parts are tested in Freon baths that are easily contaminated, hence the need for the Freon spray cleaning. The testing baths are being changed to Fluorinert FC72. This material is much less reactive than Freon and doesn't pickup contamination. This has eliminated the need for the Freon spray cleaning. A similar approach has been taken at the vendor where the voltage bar and current stack ceramic elements are made. Peanut oil is used in an operation that could use Fluorinert FC43 instead, eliminating the need for cleaning of the peanut oil. Ceramic cutting operations are conducted at the vendors using mounting waxes. Traditionally, these waxes are cleaned off with TCE. Pinellas Plant has developed the Citradet cleaning operation for similar in-house applications. They intend to transfer this cleaning process to the vendor.

General Cleaning

For most cleaning applications, Pinellas Plant is starting with recommendations from Sandia based upon work done at Sandia or in the KCD firing set program. The machining areas are using the Actrel series of mineral spirits, included Actrel 4493L. For non-aqueous cleaning d-limonene or terpene based cleaners are being evaluated.

Y-12

Metal Cleaning

Sandia shares some structural metal components with LLNL at Y-12 for the JTA. Y-12 has identified two alternate cleaning solvents to replace their usage of Freon, Methyl Chloroform (1,1,1, Trichloroethane), Methylene Chloride, and Perchloroethylene. Solvent

140 is a mineral spirits product with a high flash point, above 140° F. The second is Water Chaser 140, a blend of Solvent 140 and a polar solvent for use in applications were water based cutting fluids were used. Both cleaning efficacy and compatibility issues were examined before adopting these two cleaners (Ref. 105). These cleaners are now in use at Y-12; in fact, the LLNL Halthane 88-3 studies included Solvent 140 cleaning of the aluminum substrate specimens.

Organic Materials Substitution

At the beginning of the W89 development effort, organic materials substitution was not considered. The timeframe for development of the W89 warhead and the time required to qualify organic materials replacement was thought to be too short for employment in the W89. Furthermore, the organic materials needing replacement are not the same rigorous schedule as are ozone depleting chemicals used in cleaning. However, as the W89 scheduled was delayed and progress on substitutes was made, this development was monitored by the W89 program to determine if some of these new materials could be used. There are two materials in particular for which substitutes are needed: TDI, Toluene Diisocynate, and MDA (Methylene Dianiline). Both are considered suspect carcinogens. MDA is also considered a liver toxin. OSHA now has regulations in effect to limit MDA occupational exposures . TDI is present in many polyurethane encapsulants such at EN-7, EN-8, and Adiprene. It is also in the rigid polyurethane foam.
MDA is used as a curing agent in epoxies. It is also used in the polyimide printed wiring board material.

TDI- Free Foams

Presently both the MC4069 Firing Set and the MC4073 Programmer use TDI bearing rigid polyurethane foam for encapsulation. The MC4226 Clock for the Programmer built at Pinellas Plant also used TDI Foam. Pinellas eliminated this material by switching from the TDI foam encapsulation to an epoxy case.

KCD is evaluating the use of BKC44037 foam for component encapsulation (Ref. 106, 107). This is a sucrose based resin cured with a polymeric isocyanate (PMDI). While many of the mechanical properties are similar to TDI foam, the ductility of this new foam is much less. Measurements at Pinellas indicate that this new foam formulation absorbs about twice as much water as does the current foam. Because these encapsulants are used in large quantities, such an increase in residual water into the W89 warhead would make dewpoint control difficult.

Sandia has been developing a foam based on polymeric isocyanate and the polyol that is presently used in the current TDI foam in the hopes of maintaining good ductility. However, flow is worse, especially at the higher foam densities. Characterization of these foams will continue with a decision of which, if either, formulation should be selected to develop for WR production.

At the present time the TDI foam is safely and legally handled with engineering controls. That is the plan for W89 production if neither of these foams can be developed. However, the production plants feel strongly that wherever possible, that less hazardous materials should be developed and used instead of using engineering controls to work with toxic materials (Ref. 108).

TDI Urethane Encapsulants

TDI polyurethane encapsulants are used in the fireset for encapsulating areas to prevent high voltage breakdowns. The molded backshells on many of the W89 cables also contain small, but legally significant amounts of TDI in the Adiprene materials (>0.1% TDI). The MC4161 and MC4162 Optoelectronics at Pinellas Plant used TDI encapsulant but switched to epoxy to avoid the TDI issue. The DSSL at Mound uses Halthane 88-3 as encapsulant in the Monitor Assembly to avoid the use of TDI encapsulant. A second use of Halthane 88-3 was as a potting in the actuator electrical connector backshell. However diodes inside the potting were being damaged by the thermal stresses generated by the Halthane 88-3. This material was changed to a polysulfide conformal coat and Epoxy, Epon 828/GMB/DEA Epoxy. However, this problem would have occurred with any polyurethane, not only with Halthane 88-3.

The development is further complicated by the fact that American Cyanamid decided to no longer produce the curing agent Cyanacure. This is a business decision unrelated to ES&H issues. Cyanacure is used in the W89 cables. At the present time, with the lower production levels, there is a large stockpile of Cyanacure adequate for W89 production. There are also plans for alternate producers for Cyanacure, but no commitments have been made. Prudence dictates that new curing agents be identified for the new TDI-free polyurethane materials.

A low TDI polyurethane, PET-90A, EN-21, a non TDI polyurethane, and Halthane 88-3 were evaluated. EN-21 was found to have a high glass transition temperature, 21^{o} C. Because this means it would be unstable in the range of stockpile temperatures, it was dropped from further consideration (Ref. 109). PET-90A and Halthane 88 were also evaluated with Cyanacure and Ethacure 300 curing agents. The PET 90A with either Cyanacure or Ethacure 300 looks to be a promising replacement for Adiprene. Adhesion and d-limonene compatibility testing are planned. Both materials showed good mechanical and electrical properties. Halthane 88-3 has a short pot life but might be suitable for certain applications. Halthane 88 with Cyanacure or Ethacure 300 has either a very slow reaction rate or a very long pot life, unsuitable for room temperature curing applications (Ref. 110). Another system being looked at is Isonate 143L/Polamine 100. These systems will also be evaluated for suitability as replacements for EN-7 and EN-8. (Ref. 111).

Ethacure 300 looks good as the replacement for Cyanacure (Ref. 112). However, there is some concern over the future of Ethacure 300. Ethacure 300 is 80% 3,5,dimethythio-2,4-toluenediamine and 20% 3,5,dimethythio-2,6-toluenediamine. While toxicological data to date have been "very good", it is similar in structure to MDA and other suspect carcinogenic substances. Ethyl corporation has plans to perform a two year animal feeding study on Ethacure 300.

Similar to TDI foams, the current baseline design of the W89 is to use TDI encapsulants with safe handling until these new materials are adequately characterized for WR use.

MDA Free Printed Wiring Boards

Work is being performed at both Sandia and KCD to evaluate non MDA polyimide and cyanate ester based printed wiring boards. The properties characterization work on the candidate materials in nearly complete. So far, the best candidates are Hitachi 67N and Norplex PY260 for non MDA polyimide and Fortin T-80 and Nelco 8000 for cyanate ester. Some of the concerns are shrinkage in non MDA polyimides and moisture compatibility with cyanate ester. Some of these effects will not be able to be assessed until quantities of actual printed wiring assemblies are built. The Programmer would then build development units using this material. Again, the baseline design at present would be to use the existing MDA bearing materials and perform production safely and legally with engineering controls.

MDA Epoxy Curing Agent

MDA has some unique properties it imparts to epoxies. It has a low coefficient of thermal expansion, it is crack resistant, it has a high service temperature, a moderate curing temperature, and has good flow and filling properties. MDA is the major component in a commercial product Shell Curing Agent "Z". It is used as encapsulation in magnetics at Mound and the Pinellas Plant and in large quantities in neutron generators at Pinellas. It should be noted that once cured, the MDA is fully reacted and the epoxy no longer is a hazard with MDA.

An extensive development program was performed at Pinellas to prove in an alternate material, Ethacure 100, Diethyltoluenediamine or DETDA for magnetics, (Figure 21). This development was sucessfully completed (Ref. 113) and planned for production in the Pinellas Plant magnetics production line in February of 1991. Just at this time, unfortunately, Ethyl Corporation, the makers of Ethacure 100 announced that tumors were discovered in a two year rat feeding study (Ref. 114). Because, with this new information, Ethacure 100 was judged no safer than MDA, it was decided to still use MDA with the existing MDA handling facilities and not take any technical risk with the process change. (The Pinellas Plant has built an extensive facility and handling system for MDA usage.)

Mound has the responsibilities for the solenoid magnetic coils for the DSSL. It also uses MDA in the MC4295 PXD used for timer certification (required for production but not a component in the W89 warhead). Mound decided to switch to Ethacure 100 from MDA. The DSSL solenoid coil has become a vendor part. The vendor, however, has had trouble with the use of Ethacure 100 and has MDA handling facilities. The baseline design is for the DSSL solenoid coil to be made with MDA at the vendor.

Neutron generators use two types of epoxies. The neutron tube portion of the unit uses Epon 828 with glass microballoon filler and Diethanolamine (DEA) hardener. DEA is in wide use and has no known carcinogenic effects. The power supply region uses Epon 828 with alumina filler and Z hardener. Work on replacing the Z with Ethacure 100 looked promising. More than twenty units in which Ethacure 100 was used to replace both the DEA and the Z passed functional testing. But this work was stopped after the Ethyl Corporation announcement regarding health effects. Replacing the Z with DEA was attempted. Twenty seven neutron generator units with DEA were successfully function tested after 100 thermal cycles over the temperature range of -51° C to +74° C (Ref. 116). In addition, temperature gradients were also used during curing to help reduce voids at the interface of the two epoxies and to minimize encapsulation stresses (Ref. 115). Ten units were built with this method and thermal cycled. Eight of these units were tested and all eight had output level failures of the power supply. These failures have put the application of DEA hardener under scrutiny because of tests years ago with DEA that also had failed. (Those failures are thought by some to have been caused by bad ceramic components.) The current 10 units however, were cured directly after preheating for the encapsulation process. That is the procedure used for gradient cure on electronic neutron generators in production for other programs. However, ferroelectric neutron generators, the type used on the W89 typically are cooled to room temperature after pouring and before curing. It is thought that elimination of the room temperature rest may have damaged the power supplies. A new series of testing is being performed using DEA where 3 units will be gradient cured without the room temperature rest and 28 additional units will be made with the gradient cure and the room temperature step. These units will be thermal cycled and tested. This should determine whether DEA itself or the lack of a room temperature step caused the recent power supply failures.

New Epoxy Systems

There are several new epoxy systems under development for replacing MDA. The most developed is ANH2 (previously known as HA 48). This is a 4 part anhydride cured system (Ref. 117):

	Parts per weight
EPON 828-CTBN	50
HHPA (Hexahydophthalanhydide)	40
NIAX 1025 (Polyol)	15
EMI-24 (Imadozole)	2.0

All of the characterization of this epoxy, in terms of mechanical and physical properties is complete. Work is being conducted to determine if the 4 parts can be combined in a fashion to make a two part system that will be easier (and more familiar) to work with in production. Evaluations of a typical magnetic component (the 377806 family) are now underway at Pinellas Plant. Initial results look good. There is also some work examining the use of MHHPA (Methylhexahydophthalanhydide) instead of HHPA. It may offer some processing advantages and have less of a production concern with water pick up.

A total of 21 neutron generator units made with ANH2 are in process now. A test series of 12 ultimate dielectric strength power supply test units is also in process. There are some production concerns with ANH2 in terms of higher viscosity and shorter pot life than with MDA.

A second promising epoxy is Formula 456 based on an aliphatic amine formulation (Ref. 118):

	Parts per weight
Epon 826	50
Dow XU-71790.04	25
Jeffamine D-230	12.5
Ancamine 2049	12.5

This formulation does not have all its physical and mechanical properties characterized yet. Efforts are now in process to attempt to measure those properties as quickly as possible. It appears to have some advantages over other MDA substitutes, particularly lower coefficient of thermal expansion and viscosity. There is some confusion over the availability of DOW XU-71790.04. Whether or not it will be commercially marketed is a position that has been changing in the recent past. At the present time is looks like it will be available. There are two backup formations, #'s 426 and 428 that replace the XU-71790.04 with CTBN. These formulations have somewhat higher viscosities, though (Ref. 119). After the property characterization is complete, evaluations with components will be performed for Formula 456.

As with TDI, the current baseline design of the W89 is to use MDA with safe handling until these materials are adequately characterized for WR use.

Dry Film and Bearing Lubricants

Dry film lubricants are paint or spray coatings with molybdenum disulfide to lower and control friction and prevent metal galling. They are used on sliding metal surfaces in MC4063 SSA and the DSSL. They are also used on structural parts such as tape joint tapes, bushings and threaded fasteners. Their use has increased on threaded fasteners for the W89 because many of these fasteners are made of stainless steel instead of cadmium plated high strength steel. Cadmium plating imparts galling resistance but it is a severe heavy metal poison and its use has been avoided on the W89. The commercial dry film lubricants contain CFC's, methylene chloride or high levels of VOC's (Volatile Organic Compounds) as vehicles for the lubricant powders. Many different formulation alternates have been looked at, IPA based coatings, molybdenum disulfide sputtered coating, and diamond or diamond-like amorphous carbon coatings. These have proved unsuitable. Recently Mound has identified a water base product, Everlube 9000 (Ref. 121). This material has been evaluated for the DSSL cables and after repeated tests appears as good or better than the original material (Ref. 122). DSSL cables is one of the most stressing applications for the performance of dry film lubricants. Everlube 9000 is now in the baseline design of the DSSL. Samples of DSSL cables with this material will be put into the second series of core tests, CTU 11-15.

An additional material was also identified, T-81. This is a vendor applied material in an aqueous based binder with a polyamide-imide resin. The lubricants are fluorinated ethylene propylene and molybdenum disulfide (Ref. 123). It has been decided to use Everlube 9000, however T-81 may be more suitable for other appliciations.

KCD is also evaluating the use of Everlube 9000. At the present time, for fastening hardware for valves and stronglink applications, they are using the "AS Mix", a high VOC formulation that is prepared in house (SS397168). Currently KCD has an exemption from the EPA for VOC emissions because of an activated charcoal canister system used in the plant to trap VOC emissions.

One disadvantage to Everlube 9000 is that it requires a bake out process (300° F for 2 hours or 375° F for 1 hour) to cure the coating. This makes it difficult for its use where threaded fasteners with nylon locking elements are used for warhead assembly. Conversations with the manufacturer of Everlube 9000 indicate that there is not going to be a replacement for air-dried dry film lubricants in the foreseeable future. Pantex has changed from aerosol spray to a liquid dip of the present material, Lubribond A, to help limit air emissions.

Both the DSSL and the MC4063 SSA used Vydax as a bearing lubricant. Vydax is a DuPont product that is a slurry of fine Teflon powder in a Freon liquid. Attempts to replace the Freon with water, alcohol or another liquid has been unsuccessful. After further review, its purpose in the DSSL, to prevent fretting corrosion, was deemed unnecessary and the use of Vydax was dropped. For the SSA, the use of silicon nitride ceramic bearings in place of stainless steel ones is being evaluated. Initial results look good. Units for evaluation have been built using the ceramic bearings in two bearing applications. (Ref. 120).

Paint, Powder Coating

Paints for the H1556, the W89 shipping and storage container, can contain either CFC's or high VOC's. One alternative that has been identified is using a powder coating instead of paint. The powders are applied to the unit using no liquid. An electrostatic charge holds the powder onto the unit until it is fused into a smooth coating in a baking oven. There are no ES&H problems with powder coatings. They are durable and do not easily chip. Their drawbacks are that they may be hard to touch up for repair and rework. They also use a high curing temperature, 400° F instead of a maximum of 180° F for the paint bake ovens at KCD (Ref. 124). The coating selected is a TGIC (Triglycidal Isocynurate) polyester material called Skywhite by Fuller O'Brien.

Plating

At Mound, electrical contacts for the MC3753, actuator, MC4237 initiator, and the MC4107 slapper cable are plated with gold. Traditionally, these plating operations used a cyanide process. Because of its toxicity, the cyanide has been replaced with a gold sulfite process for the MC3753 and MC4237. However the Tape Processing Lab built for the production of the MC4107 cannot use the gold sulfite process. This is more of a mass production facility and the plumbing associated with it has problems with precipitation in the equipment. This facility would still use the traditional cyanide process.

At KCD, the structural aluminum parts use electroless nickel plating. A comparative evaluation of different coatings for the case parts was done early in the program. Electroless nickel was compared to PTFE impregnated electroless nickel, chromate coating (Alodine) and anodize. For the combination of durability, corrosion protection and conductivity needed, electroless nickel was the best coating. While nickel solutions are considered hazardous waste, the use of electroless nickel is still acceptable. It will remain in the baseline design for the W89.

Chromate conversion coatings are not ES&H compliant because they contain hexavalent chromium, a carcinogenic material. These coatings are very thin, on the order of thousands of angstroms, so there is actually very little hexavalent chromium present. At the present time, there are not suitable replacement coatings for WR use. The chromate conversion coating is still used in areas where there are threaded holes in the aluminum case parts. There is no practical method of coating the threads with electroless nickel and maintaining proper thread dimensions. One test unit without chromate conversion coating on the threads was exposed to thermal cycling and 100% humidity for the length of time equivalent to 10 years of stockpile life. The corrosion of the aluminum, due to the galvanic effects of the nickel plating and stainless steel hardware, was unexpectedly severe. This indicates the need for chromate coating in these areas. Traditionally, all the aluminum parts in the warhead were chromate coated as a matter of general practice. This includes the neutron generator container and clamp, the programmer baseplate and cover, and various sheet metal aluminum parts. Because the W89 interior environment will be controlled to a low dewpoint, there should be no corrosion problems requiring that these parts be chromate coated. Therefore, the coating was eliminated on all aluminum parts inside the environmental seal. Still, there is a need for electrical conductivity on these parts for proper electrical grounding in the warhead. The original core test units, CTU 1-10 contained parts that were not chromate coated. Conductivity measurements were made on the neutron generator container and clamp for units that were aged for 1 year. There was no degradation of conductivity seen on these parts.

System Compatibility Testing

The system level compatibility effects of the ES&H driven changes are being evaluated using DAAU-1 (Development Accelerated Aging Unit), and a second series of Core Tests. DAAU-1 is a full scale W89/SRAM II warhead unit. All the Sandia electrical components are fully functional. As mentioned previously, this unit was built with the MC4069 Firing Set cleaned at the ECA level with EC-7. The MC4078 Programmer was completely cleaned with d-limonene. All the LLNL were cleaned with Solvent 140 where applicable. All aluminum parts inside the warhead were bare, i. e. no chromate coating. The DSSL was also cleaned the Bioact EC-7. DAAU-1 was assembled at LLNL's Site 300, which does not have humidity control. However, the unit was bagged and desiccated during times when not being actively worked on. The unit also saw two purge and backfill operations for evaluation purposes. DAAU-1 was aged for one year using the thermal cycle shown in Figure 22. The unit undergoes periodic gas sampling, dew point measurement and analysis, as shown in Figure 22. A more complete gas analysis was done after aging was completed. The unit should be disassmbled and the components functionally evaluated, when programatic resources become available..

With the exception of the elimination of chromate coating on aluminum parts, the first core test series, CTU 1-10, used traditional manufacturing materials and processes. These ten units were either isothermally aged at 30, 50 or 70° C or thermally cycled as shown in Figure 23. The aging lasted for 6 months, 1 or 2 years, with the exception of the 30° C isothermally aged unit which will be aged for at least 3 years, instead of 2 years. The second set of core tests, CTU 11-15 was planned to be built specifically to test for ES&H induced changes. These five units will either be aged at 70° C or thermally cycled to the same cycle as used for CTU 1-10. The units will be aged for 6 months, 1 year, and, with just the thermal cycle, for 2 years. The units, as before, with have neutron generators with Z-Hardener, the DSSL's will include Halthane 88-3 in the monitor assembly, and th aluminum parts will be without chromate coating. All electrical components, the firing set and programmer and cable samples will be cleaned with d-limonene. The DSSL will be cleaned with Bioact EC-7 and there will be samples of the mechanical cables with water based dry film lubricant. The Firing Set Housings will have been cleaned twice, once with mineral spirits as part of the pre-cleaning process, and then with Actrel 4493L. The MC4237 initiators and the MC3753 actuators will be cleaned in Bioact EC7. All the Sandia components for these units have been fabricated, however, program resources prevented the assembly and testing of these units. However, these units sttill remain available for follow on testing.

ES&H METRICS

A system of metrics was devised to show the progress that the W89 program has made in terms of ES&H changes. The performance metric that was chosen was the number of hazardous materials applications, or the number of hazardous materials used on a ship entity. The W89 B-document and the Summary Items listed in W89 program review meeting minutes at each plant were used to compile a list of Ship Entities that were considered significant items for the purposes of measuring ES&H activities. For each item, the number of different hazardous materials used in the production of that item was counted. For example, if 1,1,1 Trichloroethane, Perchloroethylene, Trichloroethylene (3 CHC's) and Freon PCA, and Freon 113 (2 CFC's) were used in making a ship entity, thc total number of hazardous materials applications was counted as 5. If there is a long and complicated process to make that particular ship entity and the use of any of the 5 chemicals is repeated, the total number of hazardous materials applications was still considered to be 5. If the same process and hazardous material was used on a different ship entity, that would be counted as an additional application of a hazardous material, even if the two ship entities were similar. Hazardous materials usage at vendors was not included. Figure 24 depicts the summary of the ES&H metric. The horizontal time axis shows: "Traditional" production methods, before ES&H became an issue; current procedures used at "Present", what is done now for development hardware builds; the projected procedures for "FPU", our goal for procedures expected to be qualified for WR at the start of production; and then ultimately in "Post-FPU" it is thought that ES&H acceptable materials could be found for all applications.

FUTURE WORK

Continued qualification development work is needed for alternate organic materials. Component evaluations will be necessary for TDI and MDA replacements. The same is true for plating and coating alternates. There also needs to be follow through on the system compatiblity level, with DAAU-1 and the second series of core tests.

These should also be back up candidates evaluated in case some of the new materials become identified as hazardous. ES&H goals are sometimes a moving target. During this program three selected candidates: Ethacure 100, HCFC-123 and NMP had to be dropped after new, adverse toxicological information became available. In the case of Ethacure 100, significant investments in WR qualification were made before it had to be dropped. A backup is particularly needed for Ethacure 300 because of its chemical similarity to Ethacure 100. The specter of a viscous cycle looms, where the DOE's WR qualification work on a candidate material is done concurrently to toxicological feeding studies. When both are completed, the candidate material must be abandoned. One backup non-aqueous cleaner should be examined for a d-limonene back up. Two materials, Ethyl Lactate and Tetrafural Alcohol have been identified for this purpose. It is important to limit the candidates, however. There are many new commercial cleaners entering the market as different companies try to take advantage of the "Green Movement". Many of these are slight variations and cleaners already evaluated. Evaluation of every cleaner would be a waste of valuable resources.

Hydrofluorocarbons (HFC's) and Perfluorocarbons (PFC's) are now coming on the market at near "drop-in" replacements for traditional vapor degreasing using CFC's, TCA, or TCE. These chemicals have limited cleaning ability and commercial development is focusing on co-solvent identification for making blends that are effective cleaners. For some of the current W89 applications, such as for the MC4063 SSA assemblies, where particulate removal it the main goal, the existing chemicals may be adequate. This could replace the IPA vapor degreasers and alleviate the fire protection concerns of those devices. Yet, PFC's may represent another moving target. Currently the EPA is considering restrictions on PFC's based on global warming potential. Global warming is one new area of environmental concern that the W89 ES&H program did not address. It is the author's opinion that global warming is a phenomenon that is far less demonstrated than ozone layer depletion. The logic of limiting relatively small quantities of cleaner compared to the huge CO_2 quantities generated by worldwide combustion is difficult to reconcile.

A continuous improvement program based on waste minimization should be in place for established cleaners like d-limonene. Automated equipment that is approved for combustible liquids should be developed. Recycle and reuse of d-limonene should be closely evaluated. This effort should not be underestimated, however, because it is fundamentally different than the once-through cleaning performed now. Now only fresh, clean d-limonene contacts all the surfaces of the assemblies, the contaminants are dissolved, removed and rinsed off. With reuse, even clean surfaces are now exposed to contaminants in the d-limonene. The process must be closely scrutinized to make sure that clean surfaces are not contaminated by materials moved around and re-deposited by reused, contaminant loaded d-limonene. The issue of auto-oxidation creating non volatile residues must also be examined in the context of reuse.

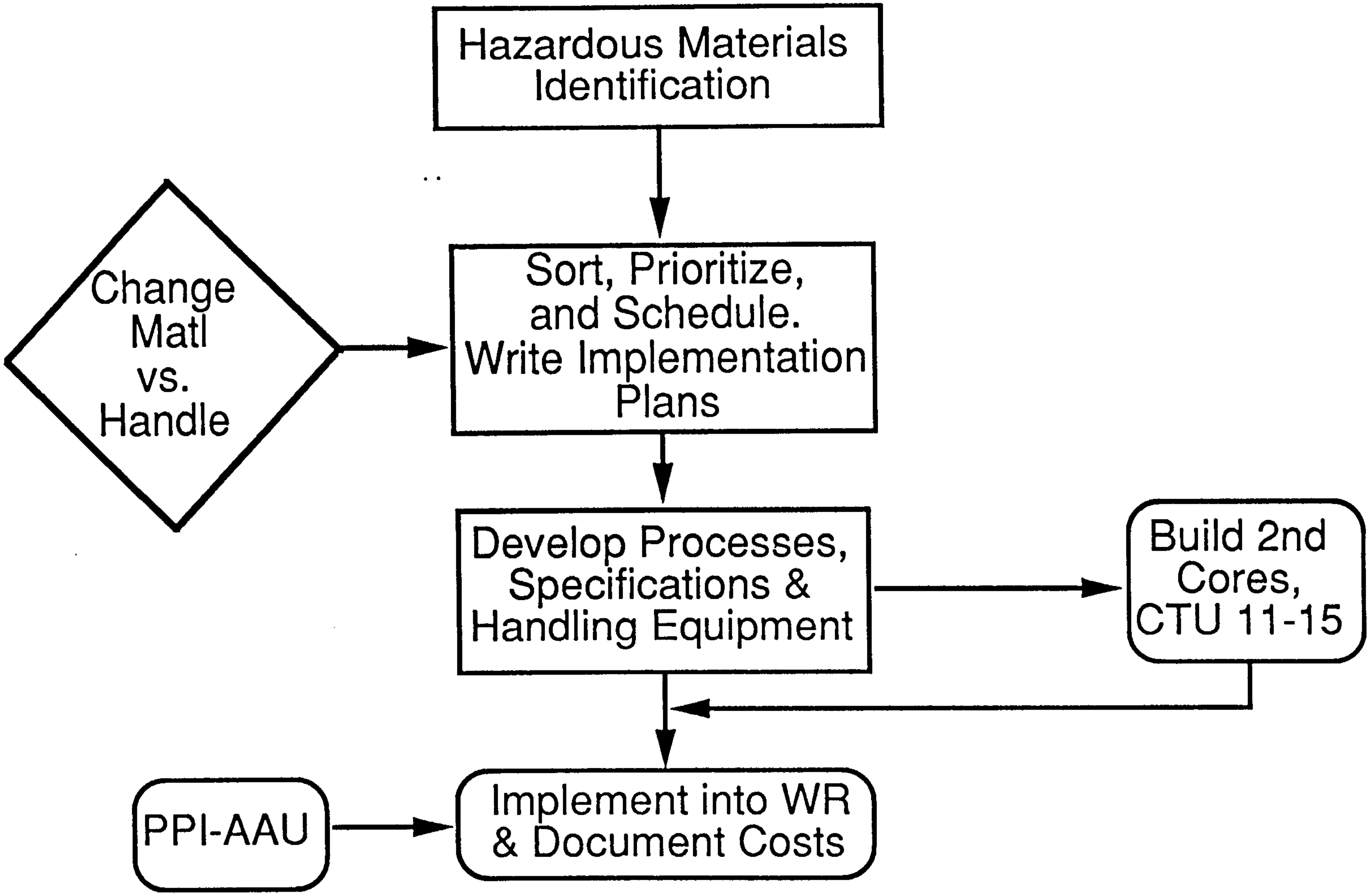
Future systems will also use more surface mount technology in electrical components. These will have smaller stand off distances to the circuit board, .003 to .005". Liquid cleaners will have difficulty entering and cleaning these small spaces.

SUMMARY

The W89 was a unique warhead development program in that it had to face ES&H changes midway in the development program. With large amounts of development efforts, particularly in the area of compatibility, the W89 program successfully met all ES&H requirements. The major changes had to do with solvent substitution for cleaning. Compared to traditional cleaners, the new ES&H cleaners are more application specific. For flux removal and many other applications, d-limonene was successfully used. For others cleaning applications, highly refined mineral spirits, Actrel 4493L and aqueous cleaners were qualified. More work needs to be done for organic materials and plating substitutions.

A proactive approach in ES&H development is vital. The success of the d-limonene solvent substitution program was largely due to the firing set cleaning program that was started before the ES&H issues were felt at the warhead level. All continuing warhead development should be viewed with a similar ES&H prospective.

Figure 1.
THE W89 ES&H PLAN



Sandia
National
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Figure 2. Traditional Cleaning Processes for Firing Sets

Process	TCE Spray	IPA Spray	Freon TE35 Spray	Freon TF Spray	N2 Blow Dry	Vacuum Bake
A	★	★		★	★	★
B	★	★			★	
C		★		★	★	★
D			★		★	
E	Refer to Process B					
F		★			★	

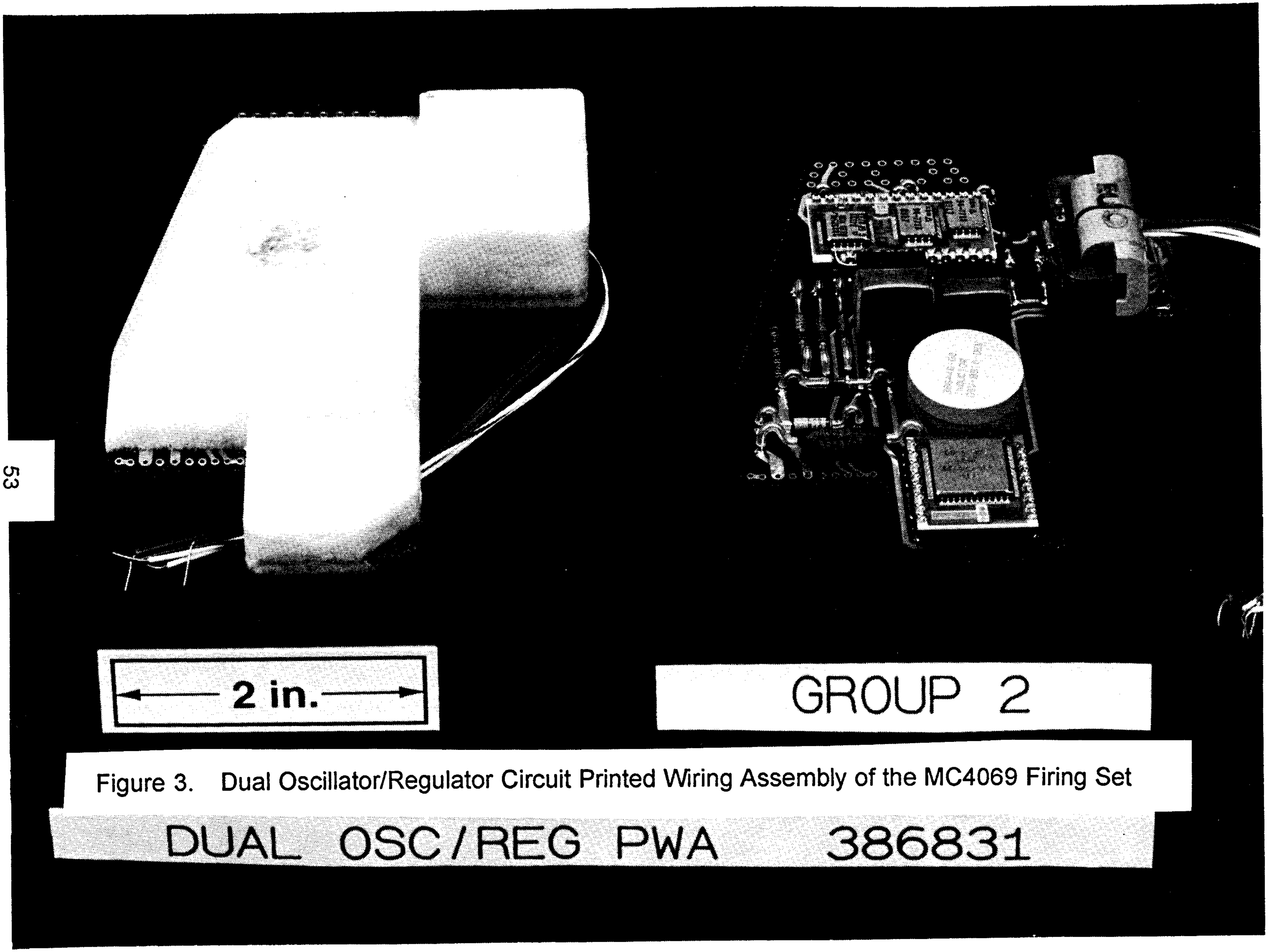


Figure 3. Dual Oscillator/Regulator Circuit Printed Wiring Assembly of the MC4069 Firing Set

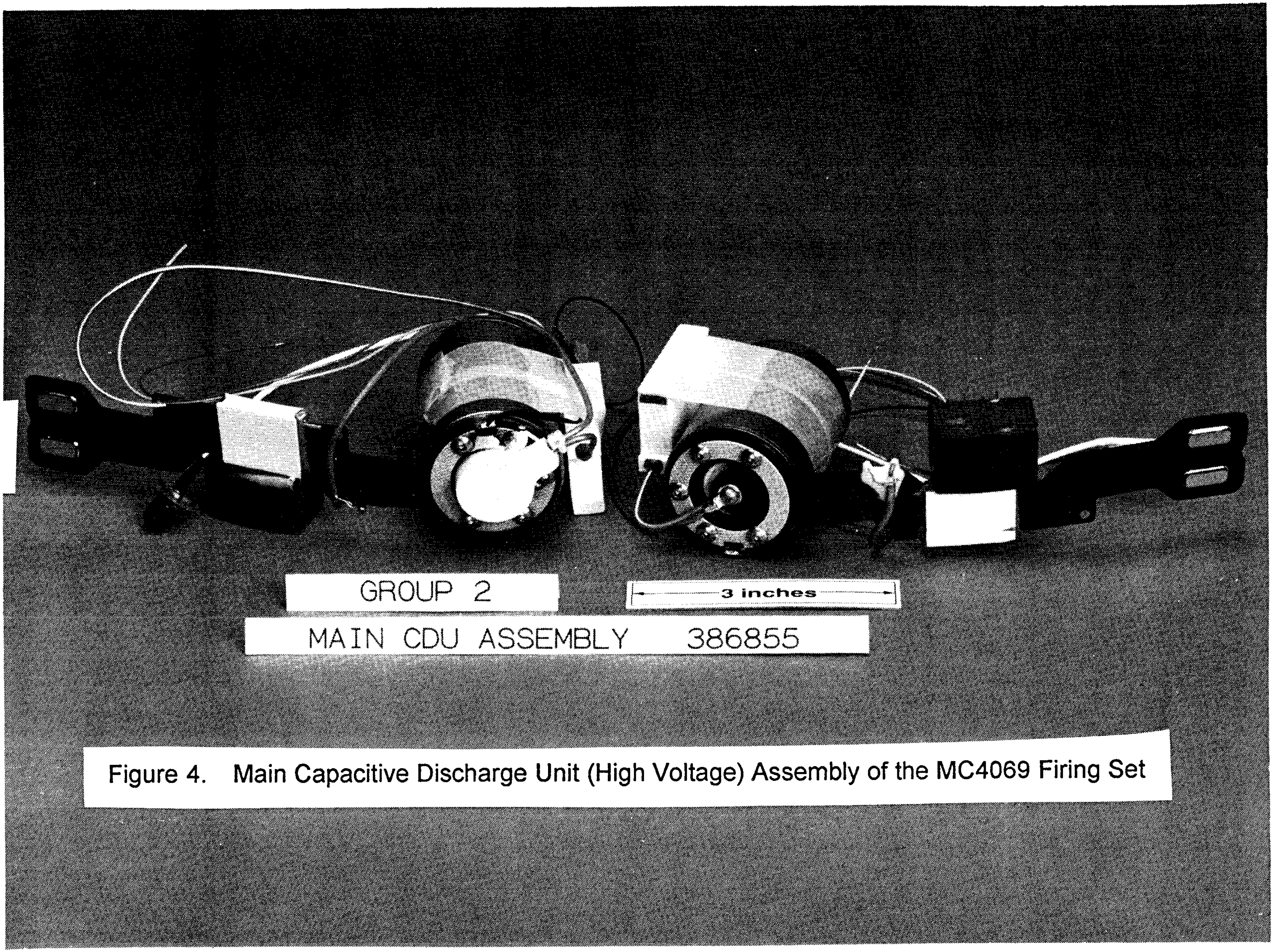


Figure 4. Main Capacitive Discharge Unit (High Voltage) Assembly of the MC4069 Firing Set

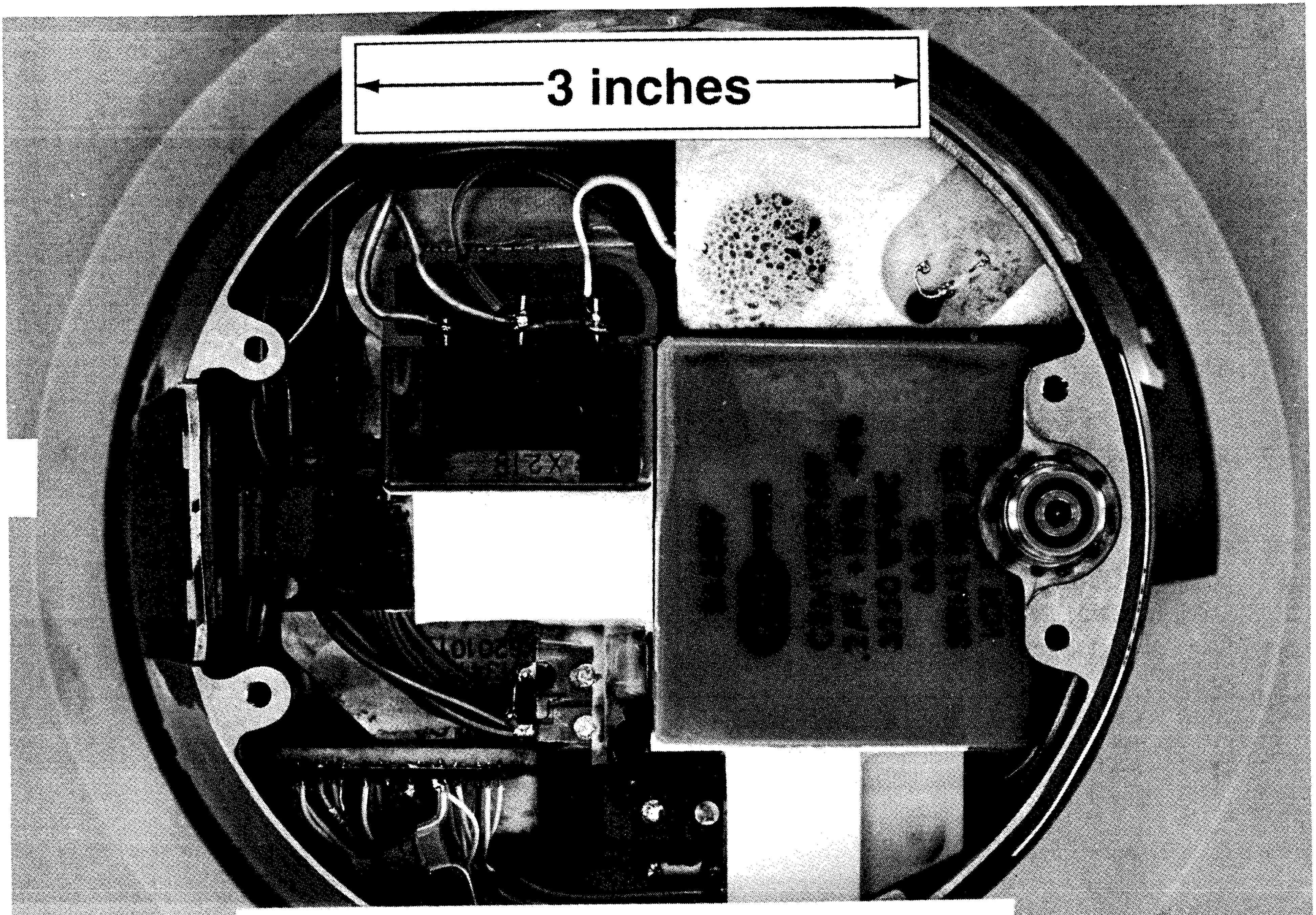
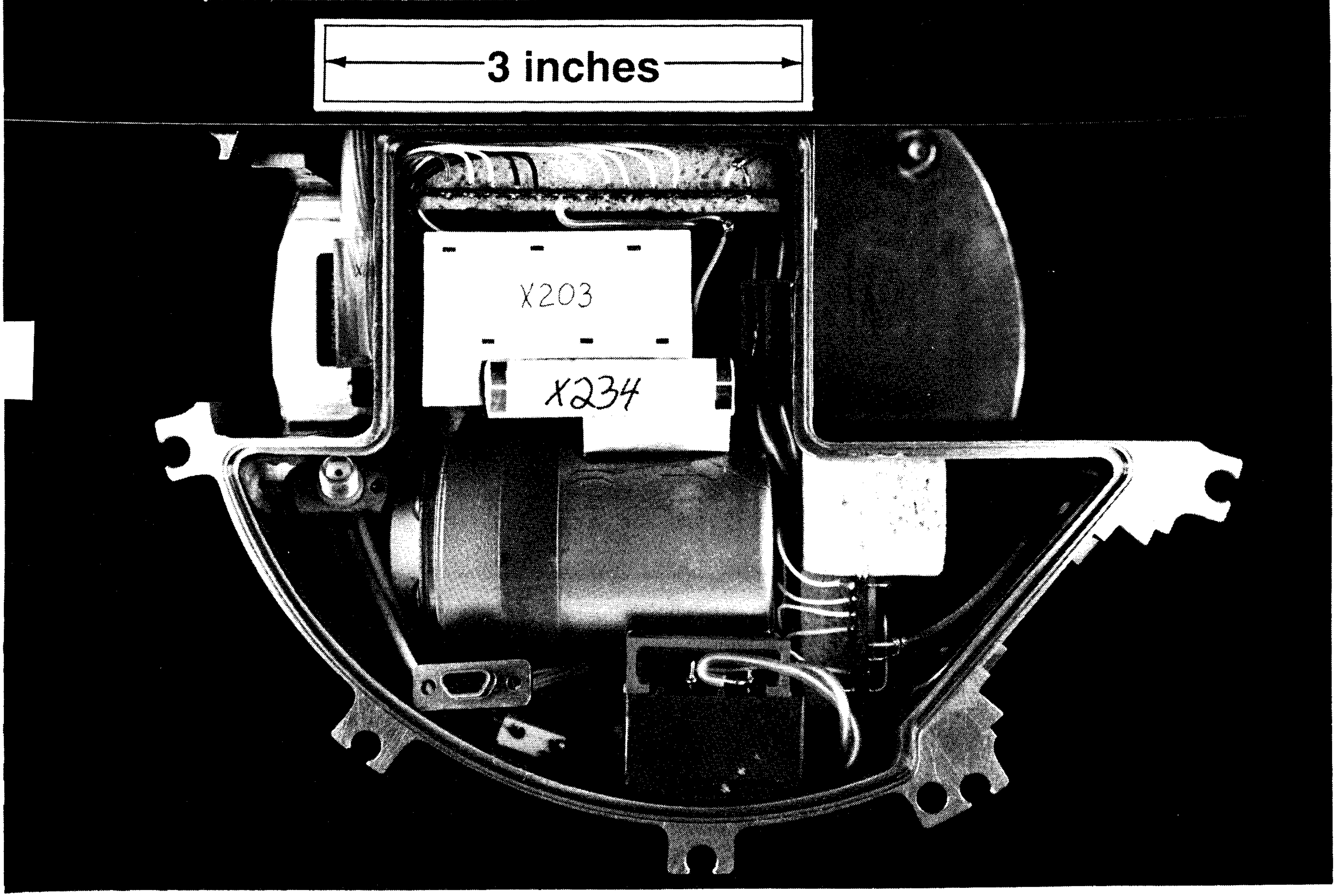


Figure 5. Forward View of the Electrical Circuit Assembly Level (ECA) of the MC4069 Firing Set

Figure 6. Aft View of ECA of the MC4069 Firing Set Prior to Encapsulation



W89 FIRESET CLEANING EVALUATION

KESTER 197 SOLDER FLUX REMOVAL FROM SOLDER DIPPED COPPER, (SOLDER SURFACE)

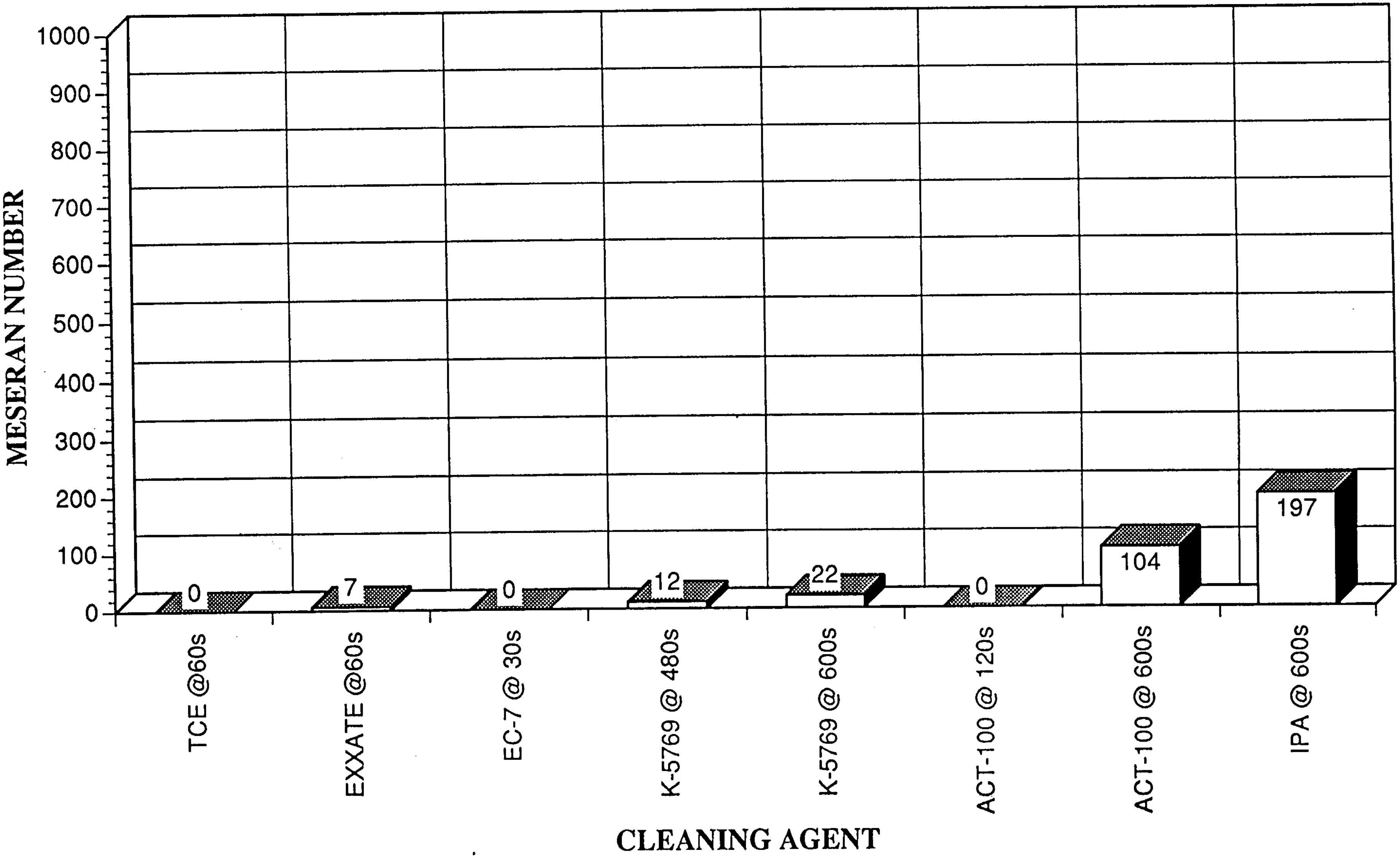


Figure 7. Meseran Results for Flux Removal of Solder Dipped Copper Surfaces

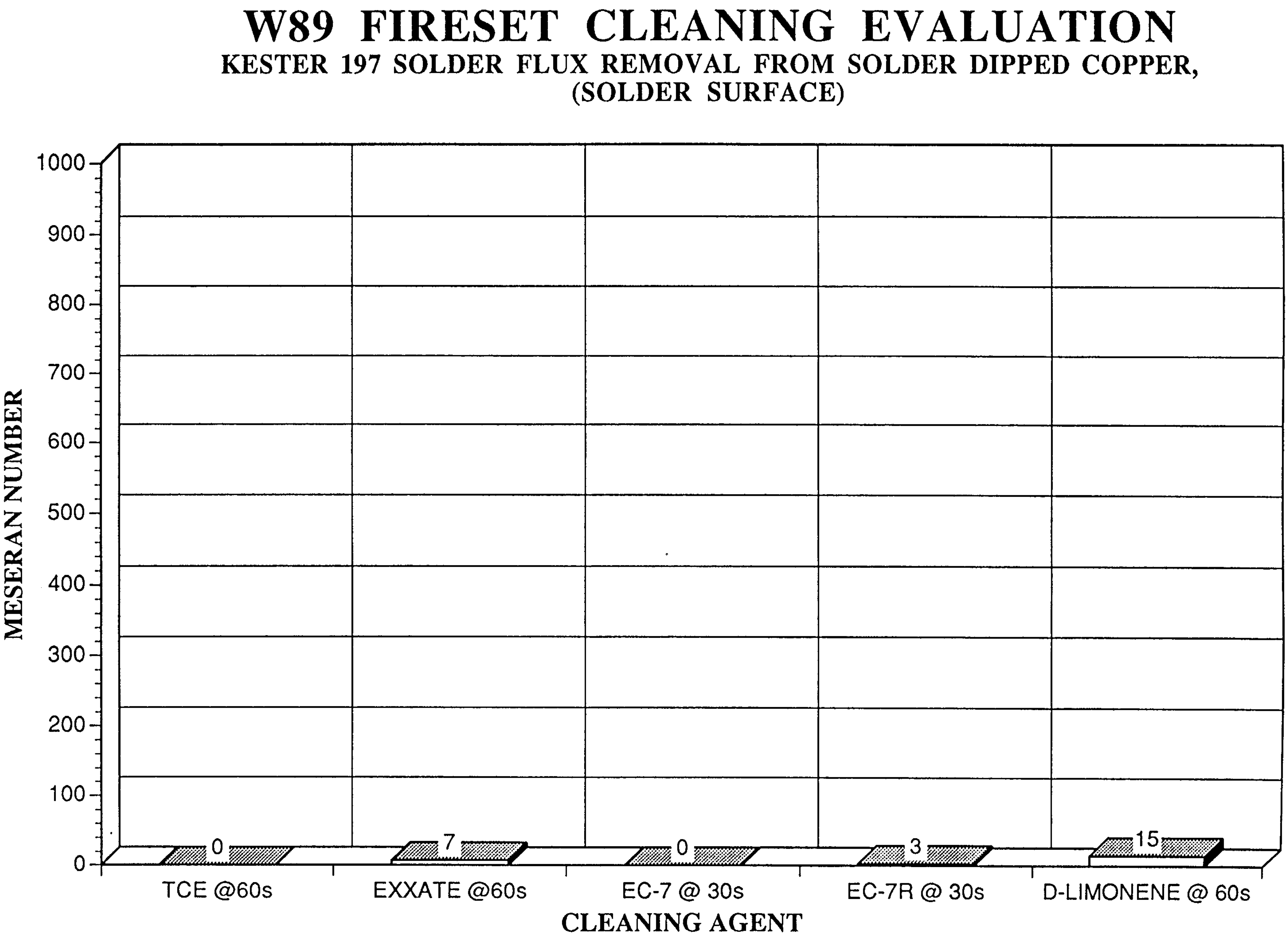


Figure 8. Meseran Results for Flux Removal of Solder Dipped Copper Surfaces, Comparison of Effective Cleaners with EC-7R and d-Limonene

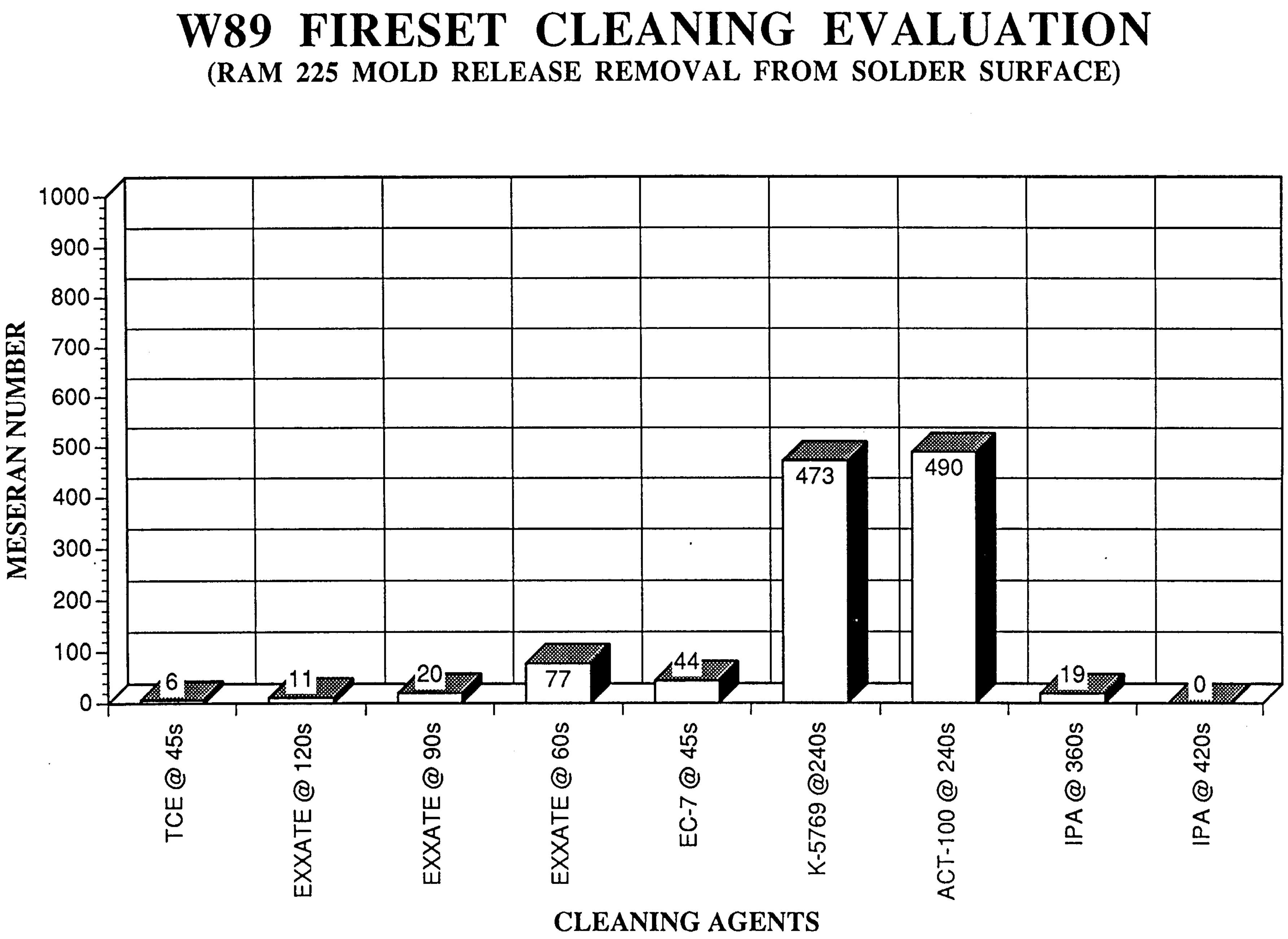


Figure 9. Meseran Results for RAM 225 Mold Release Removal of Solder Dipped Copper Surfaces

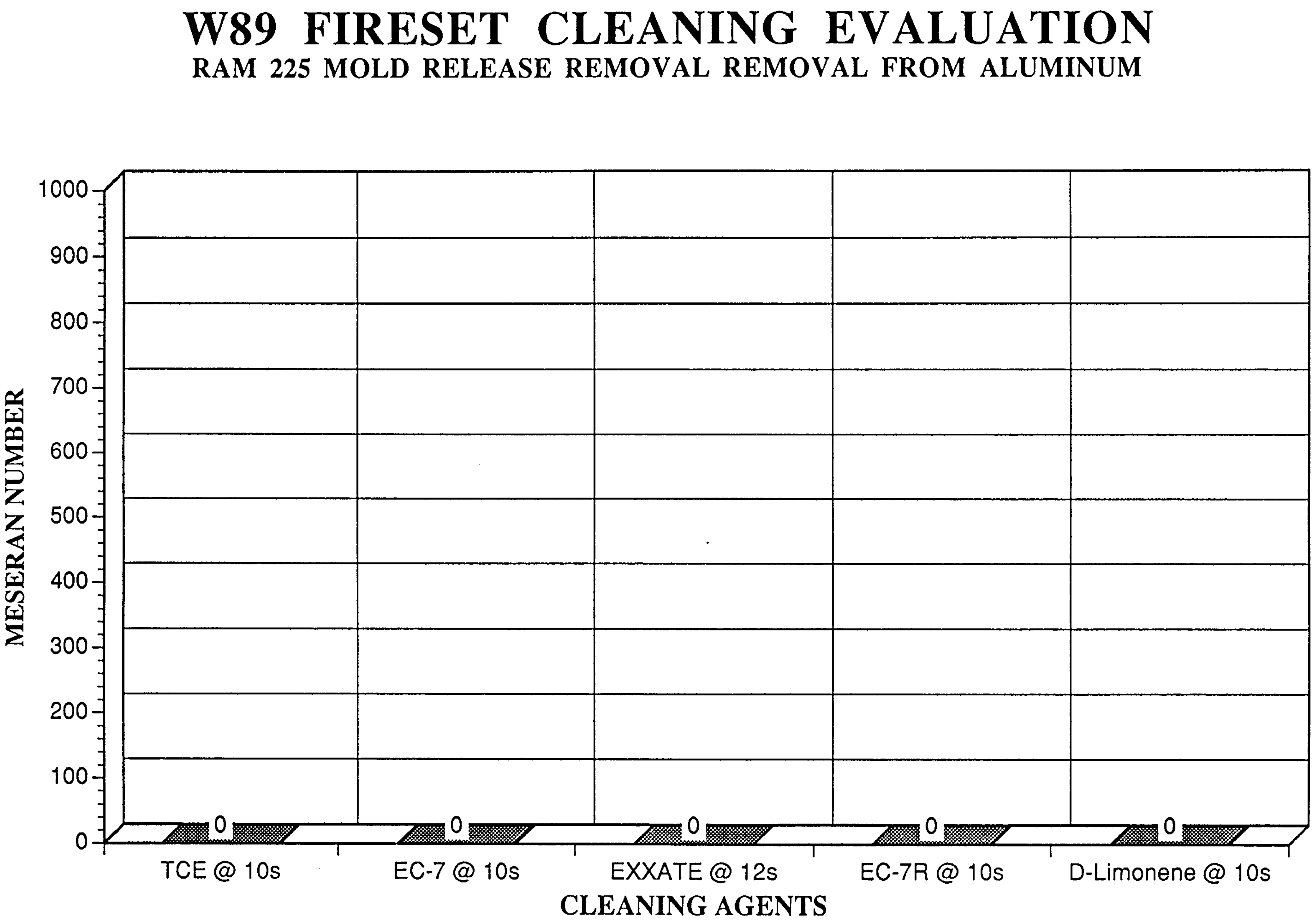
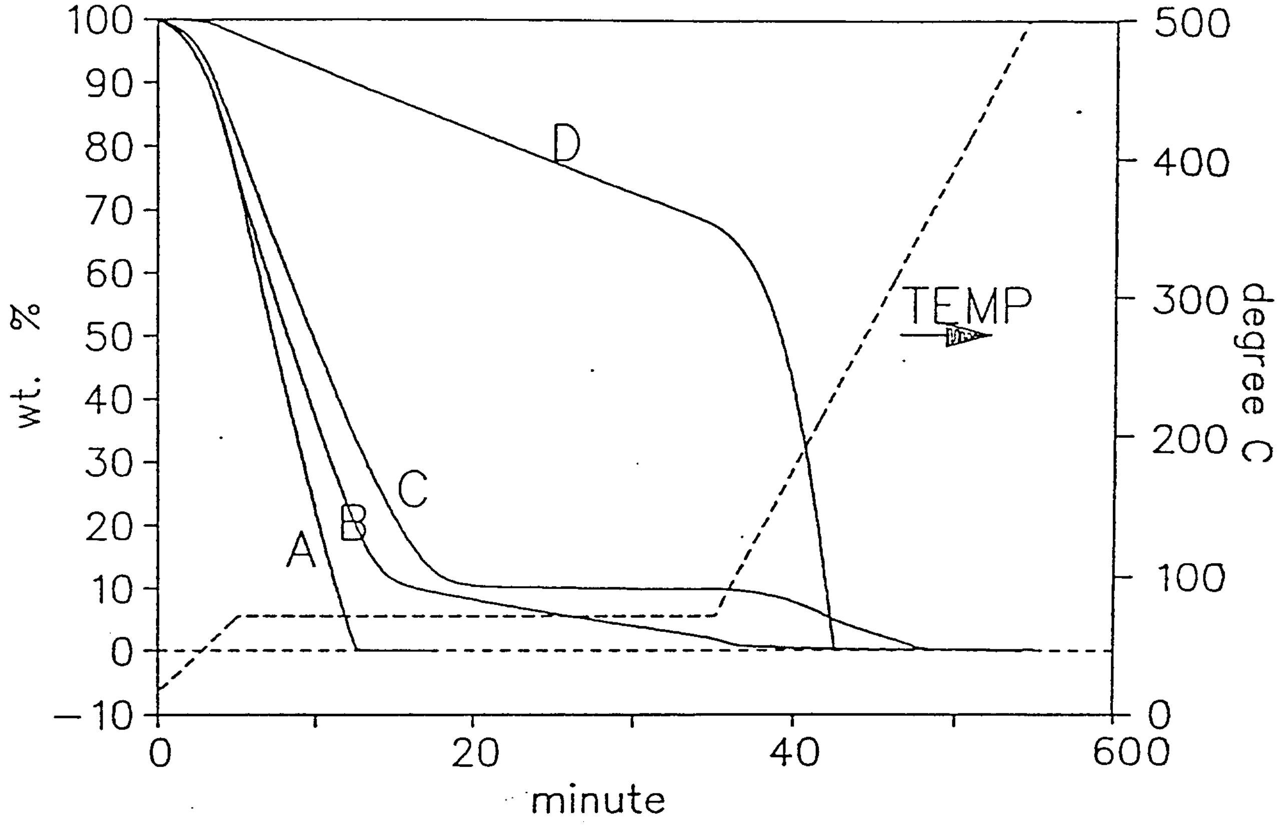


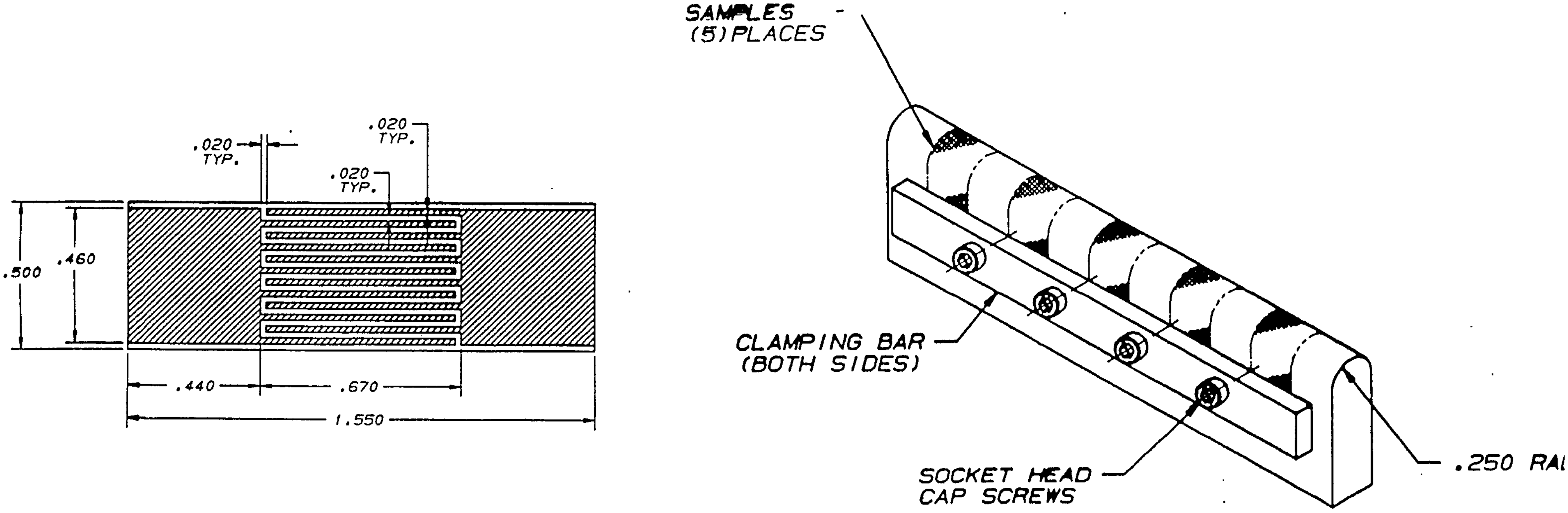
Figure 10. Meseran Results for RAM 225 Mold Release Removal from Aluminum Substrate Comparison of Effective Cleaners with EC-7R and d-Limonene

Figure 11. Thermogravimetric Analysis of Candidate Cleaners



A = d-Limonene
B = EC-7-R
C = EC-7
D = Exxate 1000

Figure 12. Flex Cable Specimens and Test Fixture



SOLVENT AGING TEST SAMPLE

AGING FIXTURE FOR SOLVENT TESTING

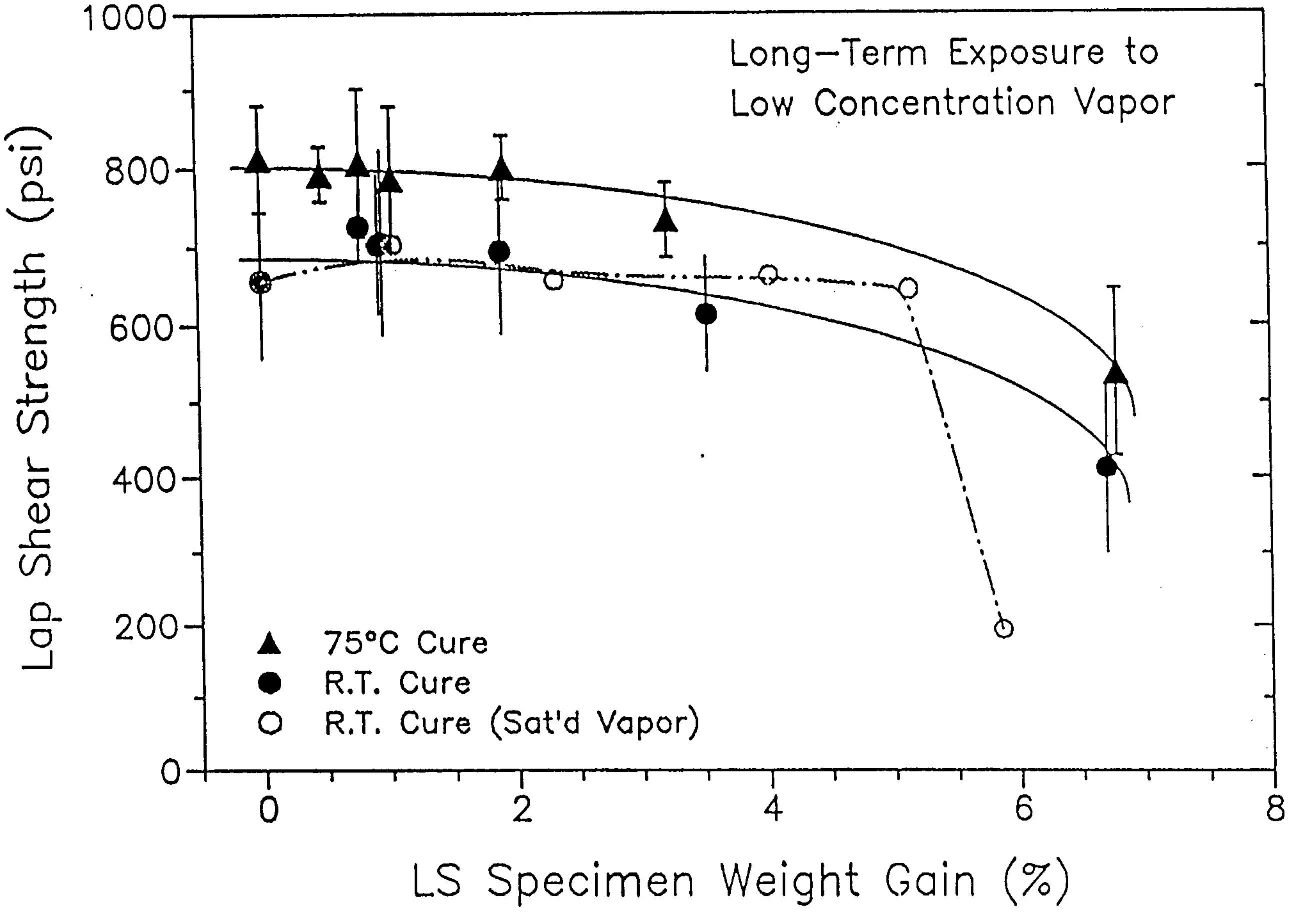
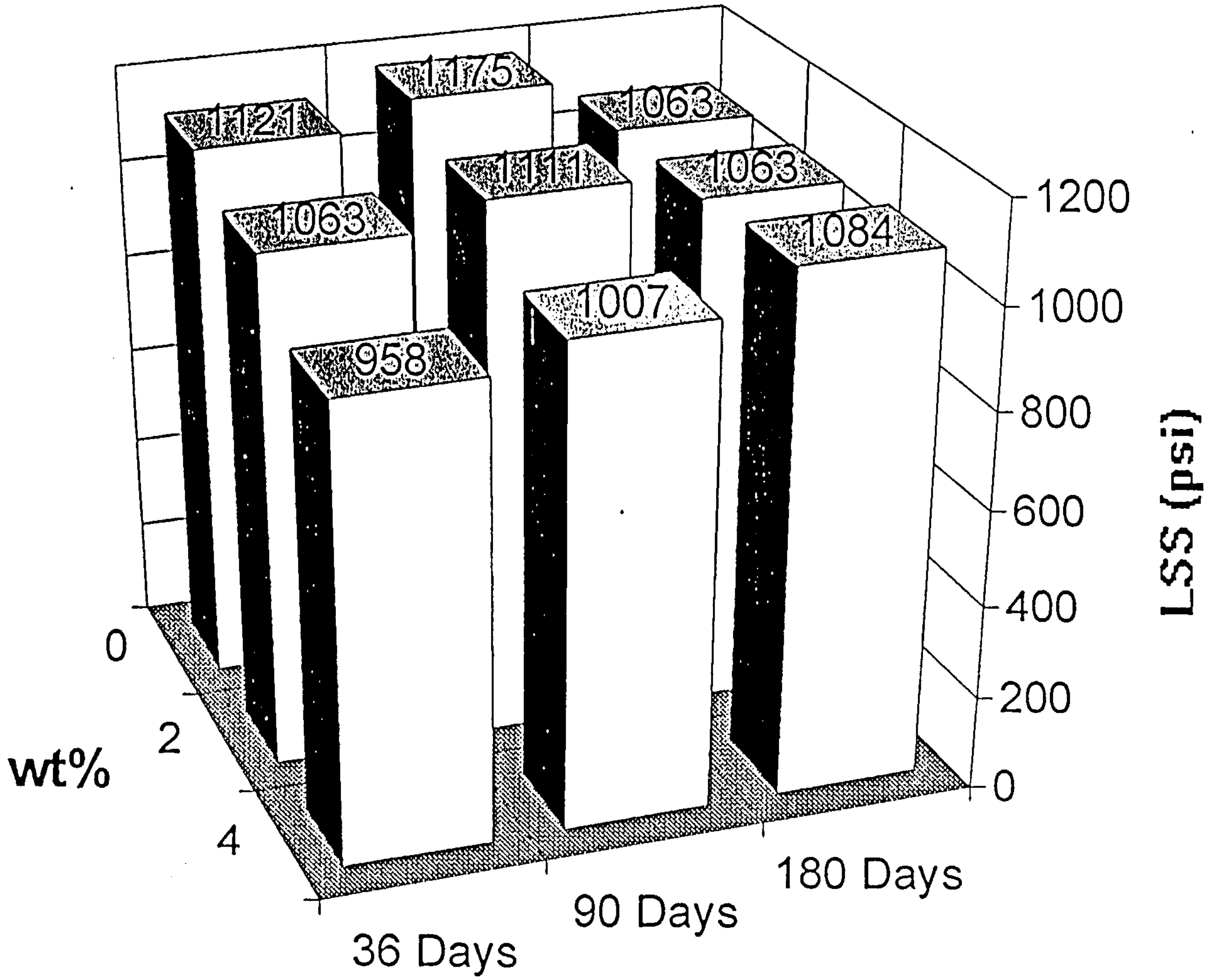


Figure 13. Halthane 88-3 Lap-Shear Strength in Low Concentration d-Limonene Vapor

Figure 14. Halthane 88-3: Results of Long-Term d-Limonene Exposure Study



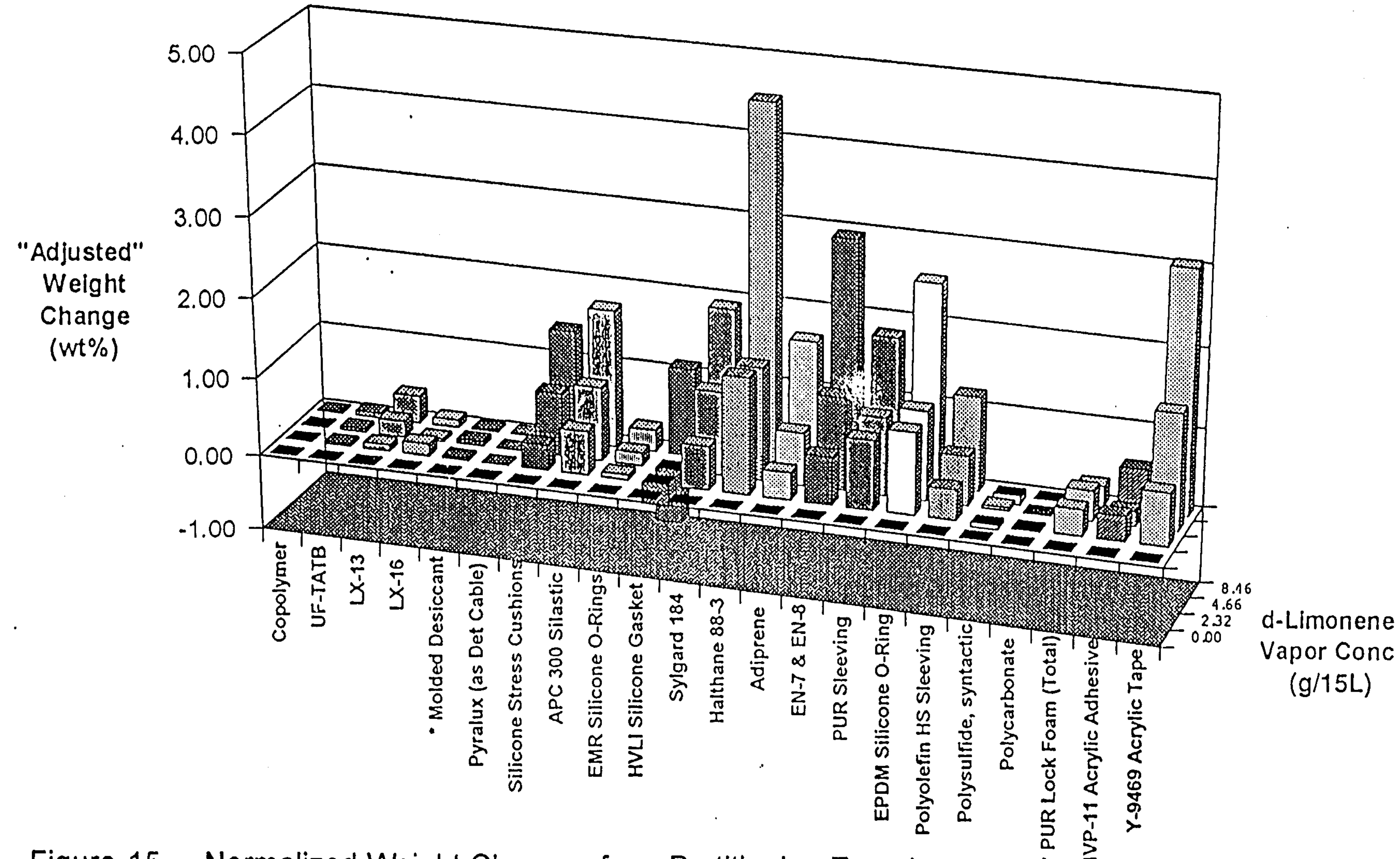
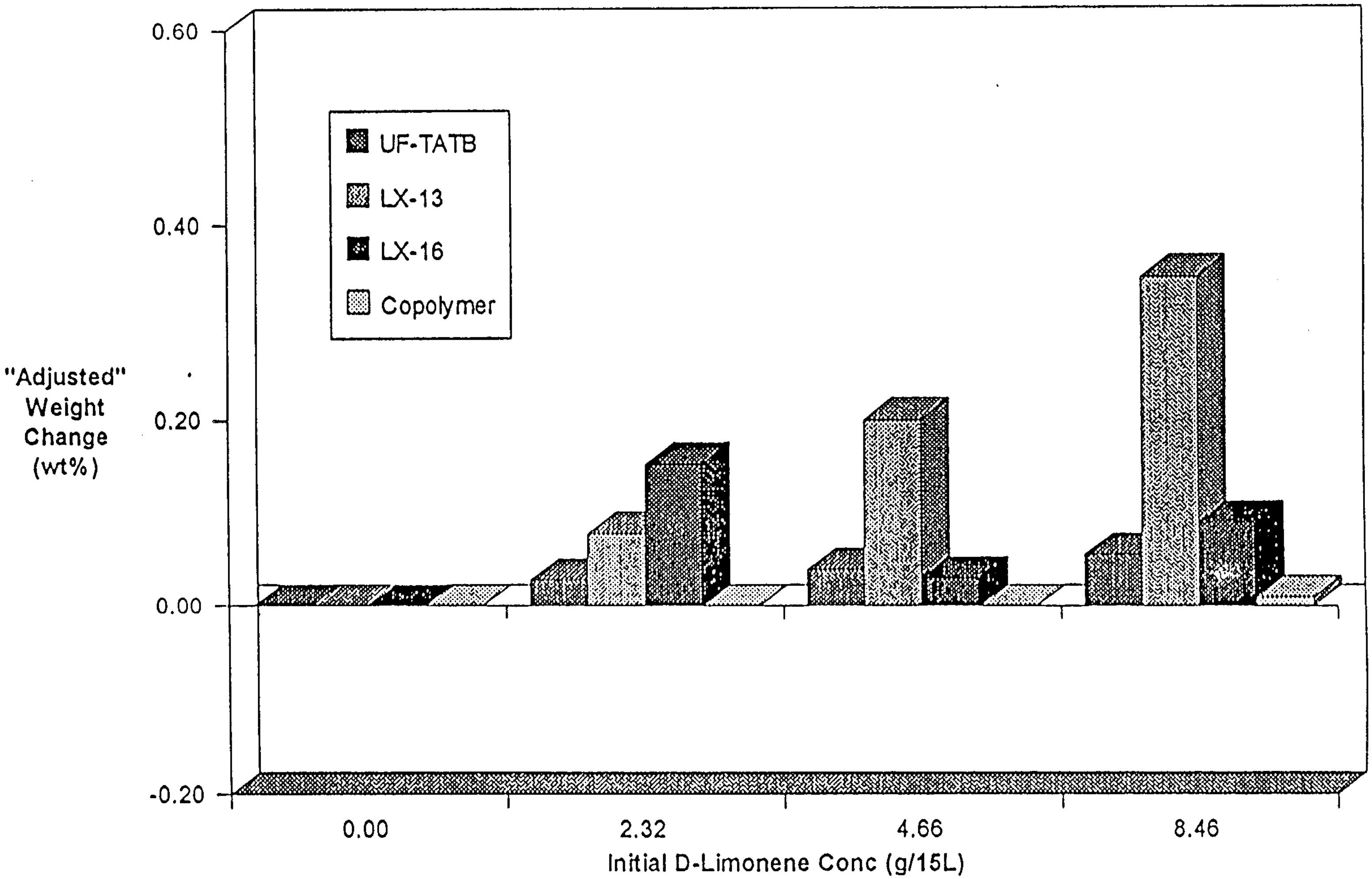


Figure 15. Normalized Weight Changes from Partitioning Experiment

Figure 16. Energetic Material: Normalized Weight Changes



MC3753

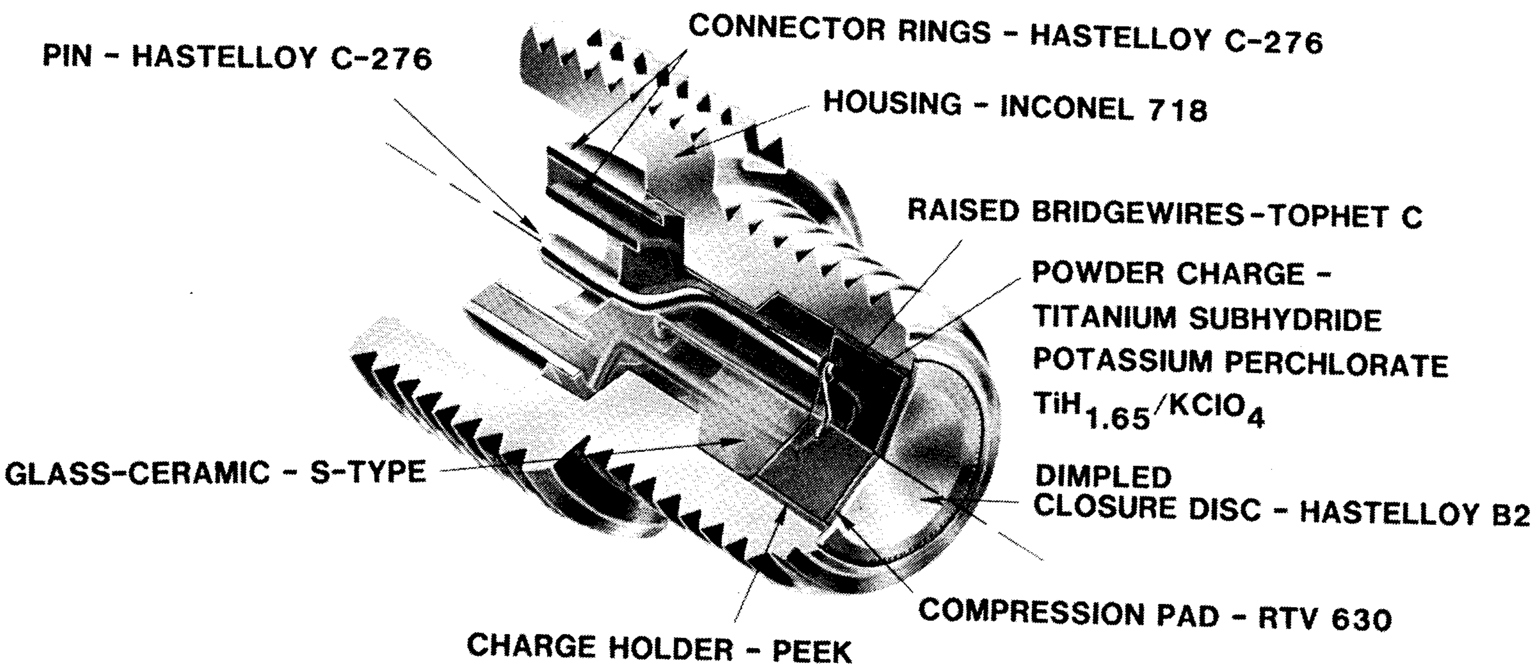
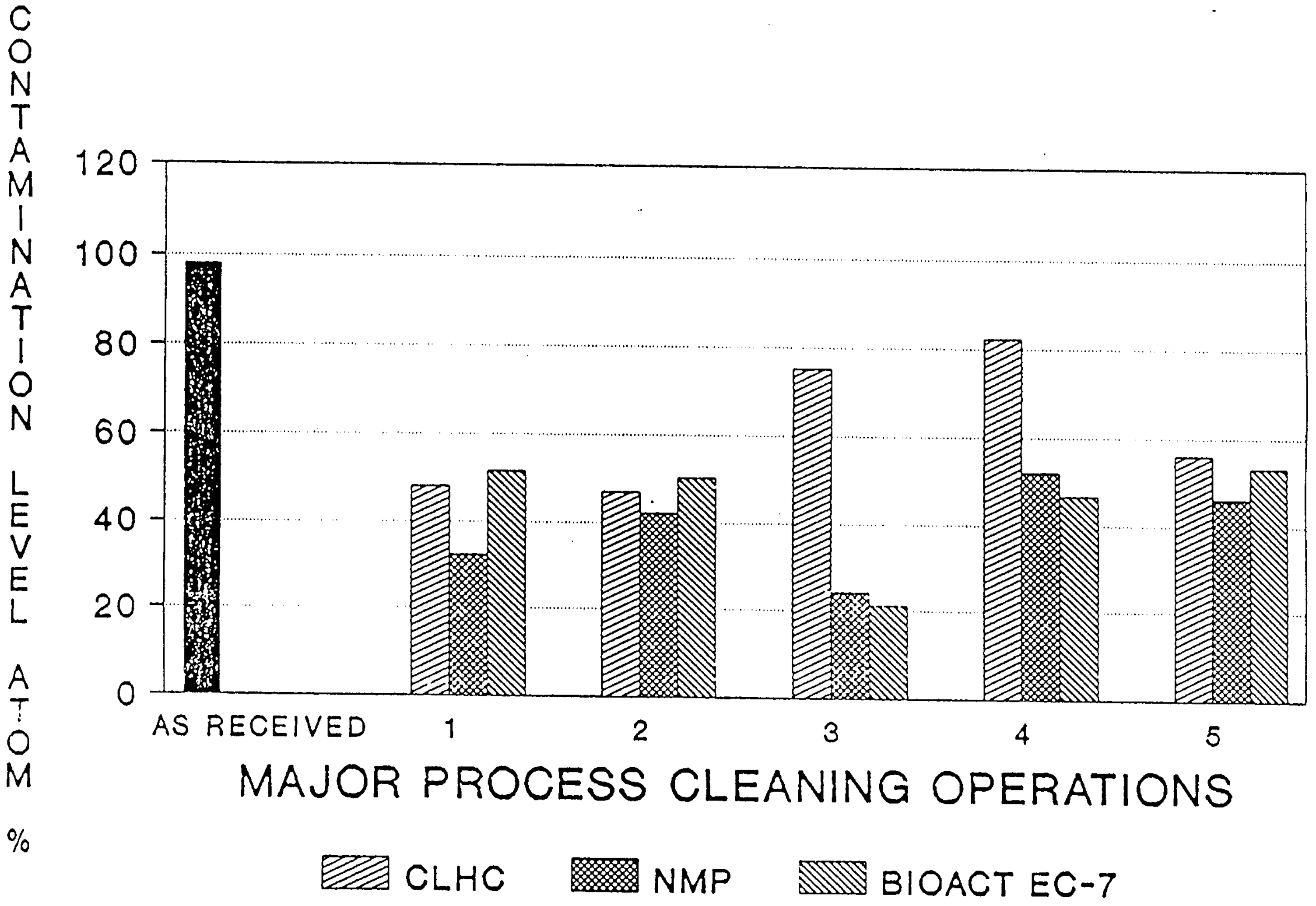
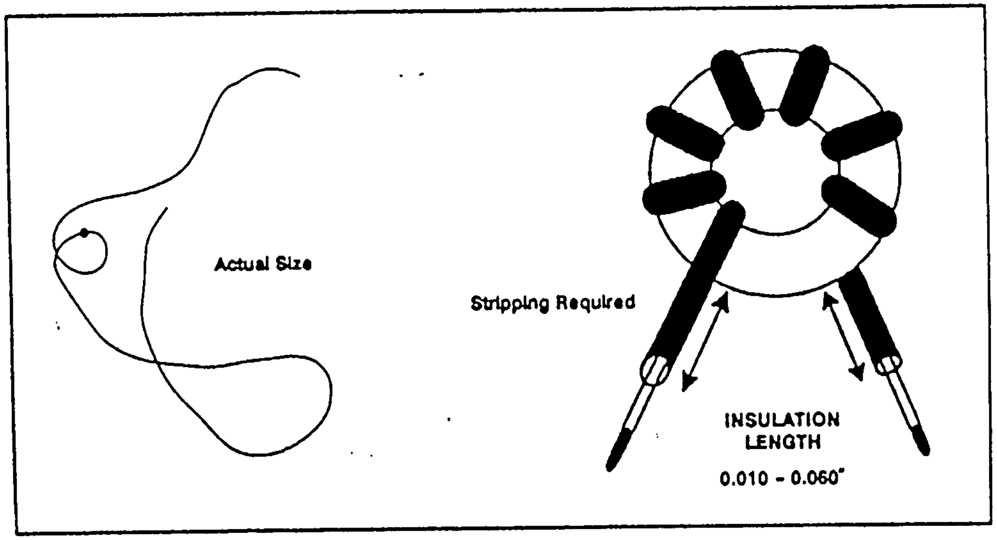


Figure 17. MC3753 Actuator

Figure 18. Cleanliness After Each Operation in the "4 Step Process"

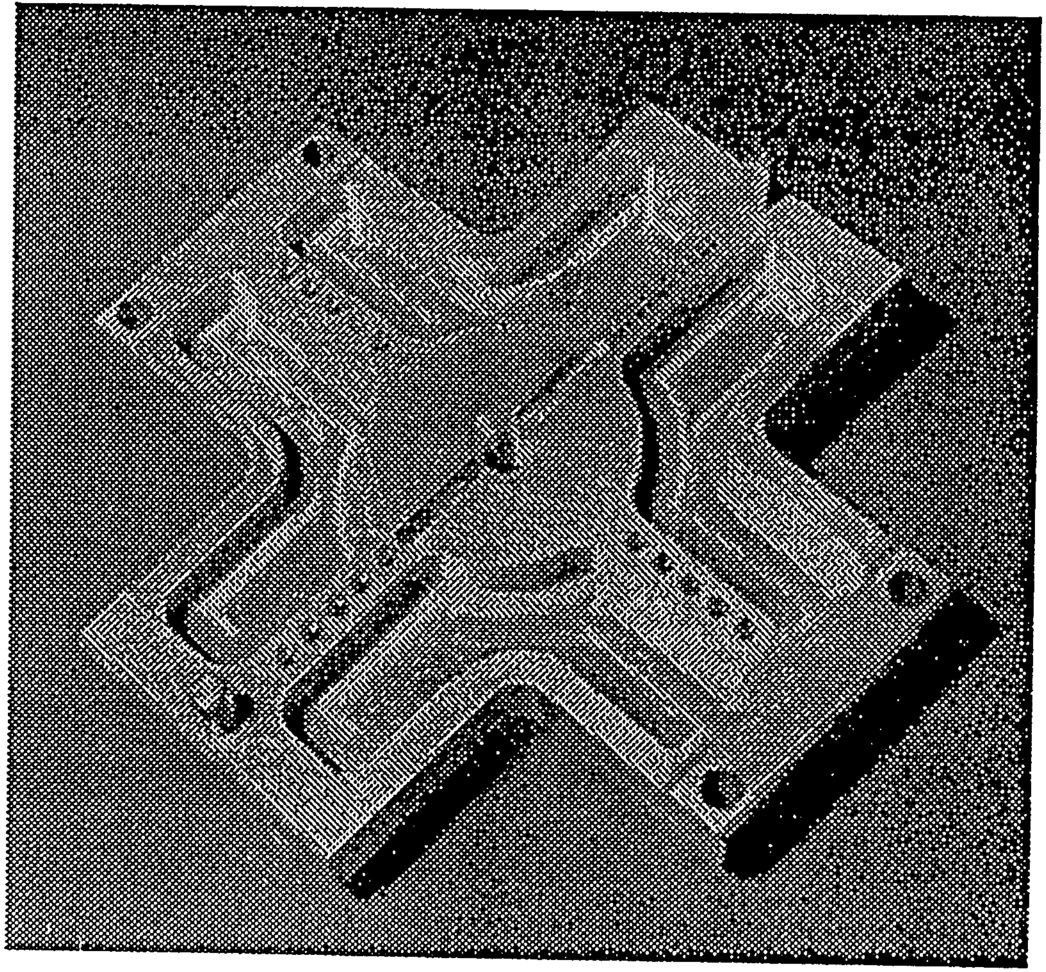




Flyspec Inductor

Figure 19. "Flyspec" Inductor

Figure 20. Stripping Fixture for the Flyspec Inductor



Stripping Fixture for Flyspec Inductor

Producibility
Engineering

CURING AGENT Z SUBSTITUTE
EVALUATION PLAN

02-04-91

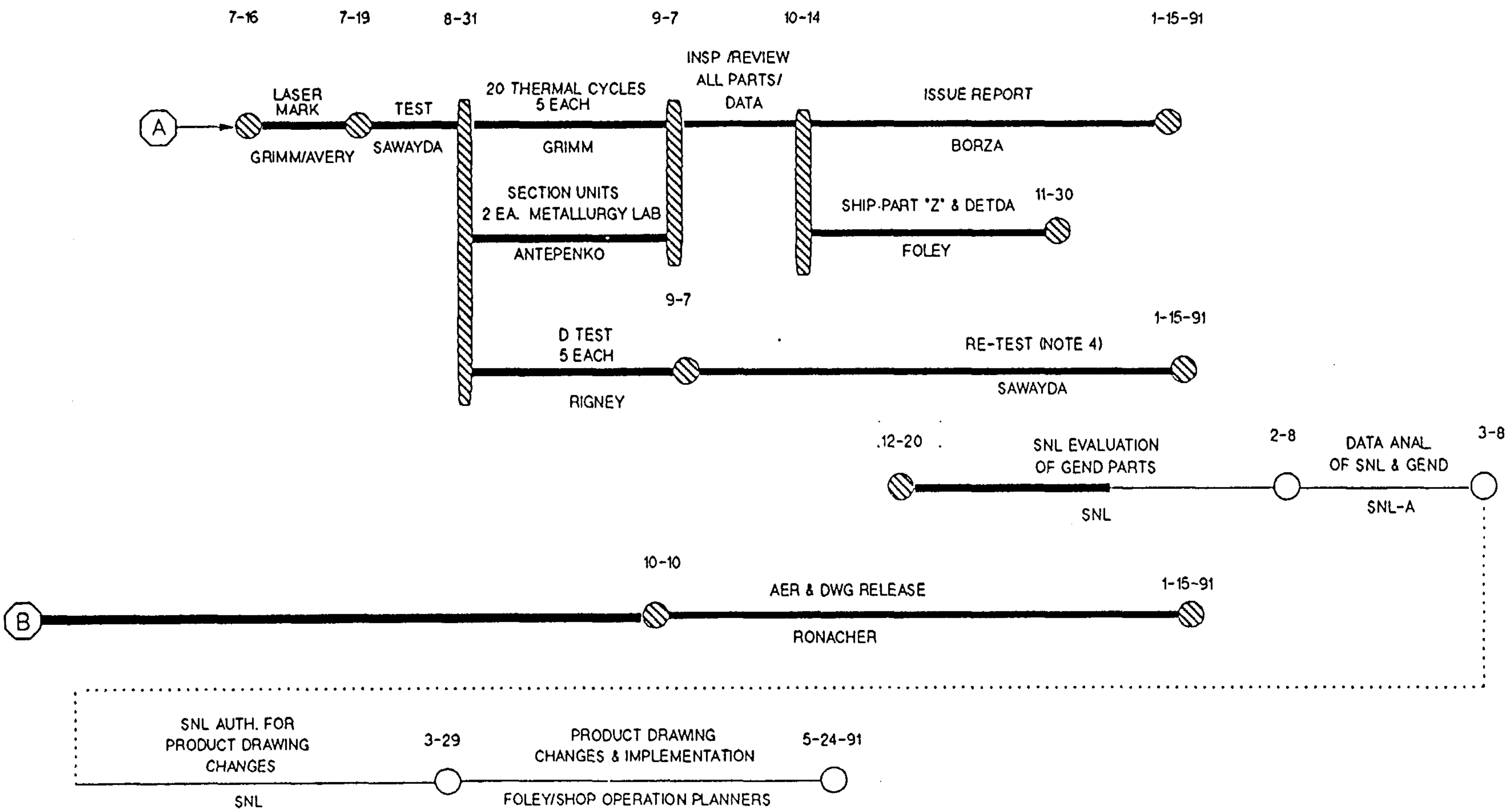


Figure 21. MDA Substitution Plan for Magnetic Products at Pinellas Plant

Page 2 of 2

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Figure 21. MDA Substitution Plan for Magnetic Products at Pinellas Plant (cont'd)

Producibility
Engineering

CURING AGENT Z SUBSTITUTE
EVALUATION PLAN

01-15-91

NOTE 1: 15 PARTS EACH

377839	378123
378045	379808
378118	380059
380044	379824

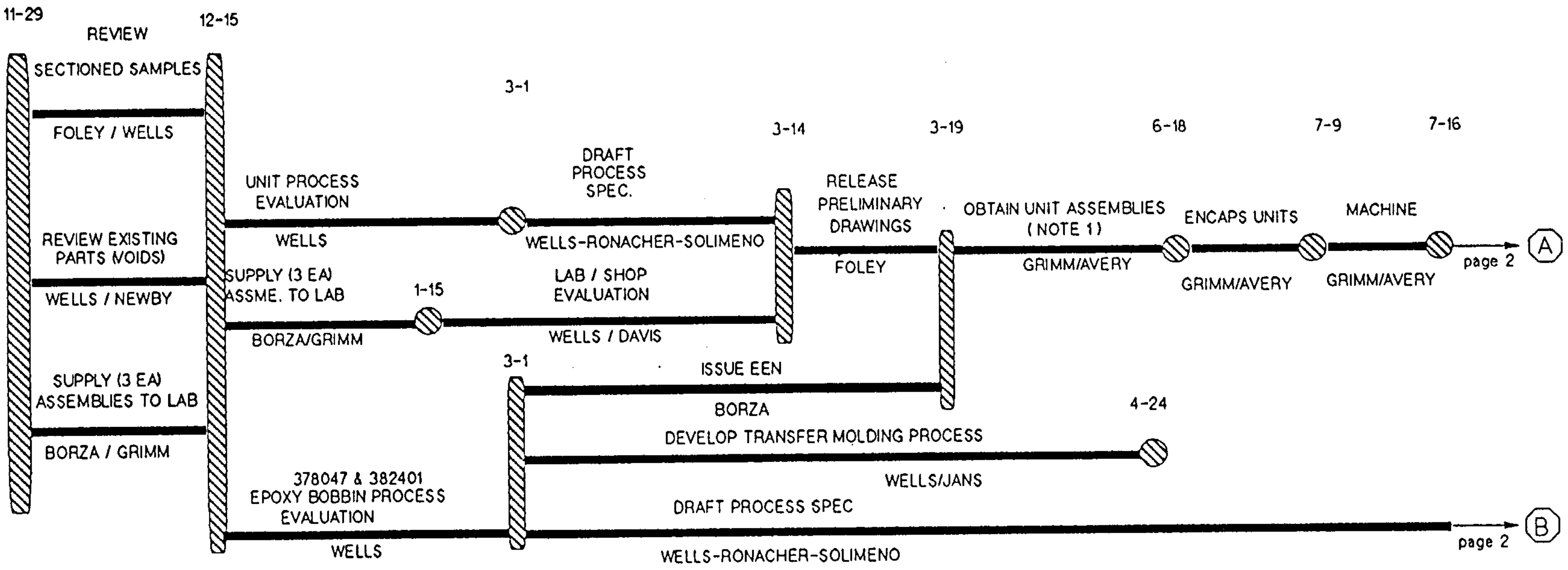
377941 OR 378124

NOTE 2: CURING AGENT SHALL BE DETDA

NOTE 3: FULL IMPLEMENTATION IS PREDICATED UPON N/A COMPATABILITY & AGING STUDY

NOTE 4: RETEST OF 379824 ON HOLD - TESTER PROBLEMS.

NOTE: 5: REVISED 01-15-91



Attendance: C. Adams
F. Borza
J. Foley
W. Garrett
T. Gillespie
E. Jorn
J. Ronacher
B. Wells

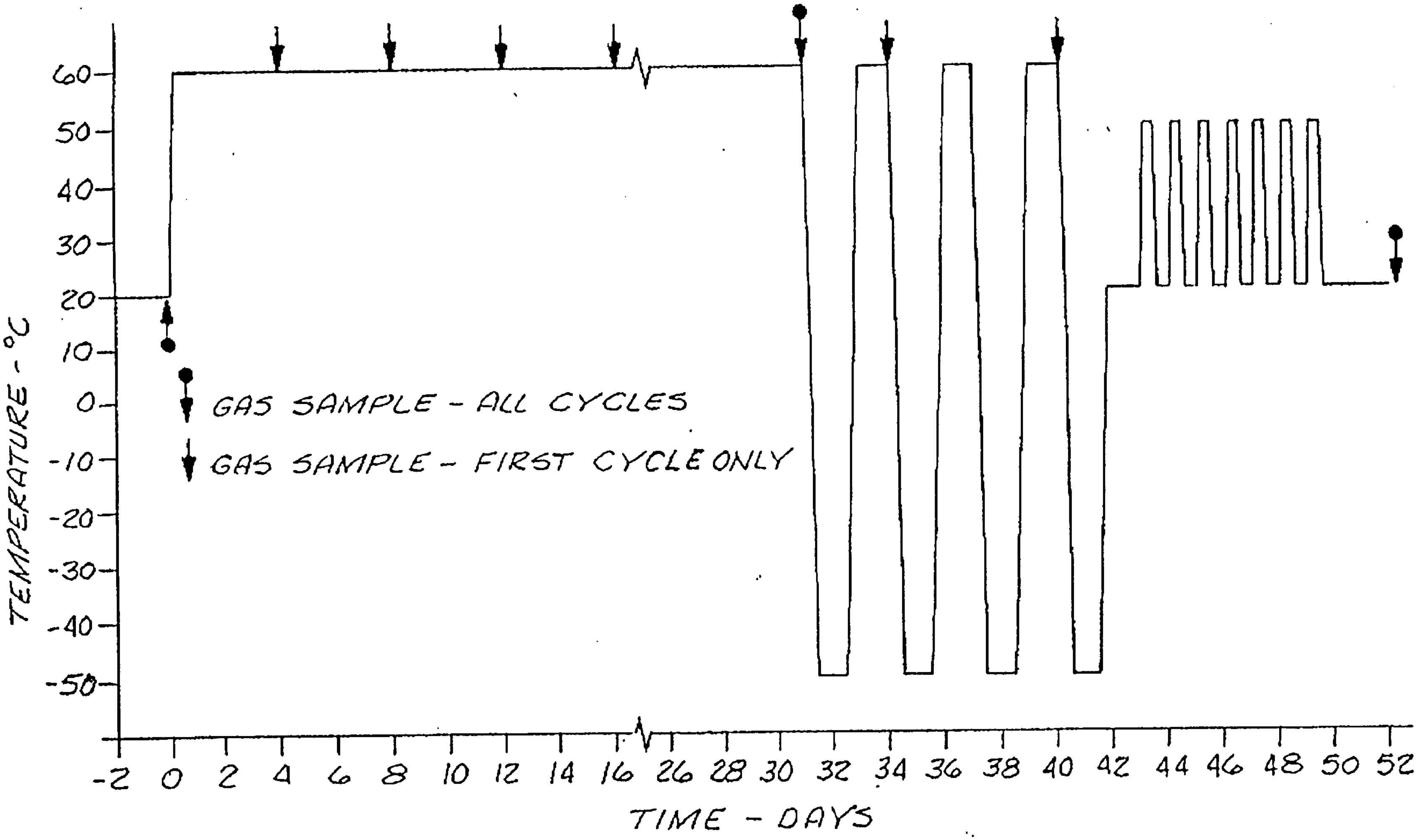
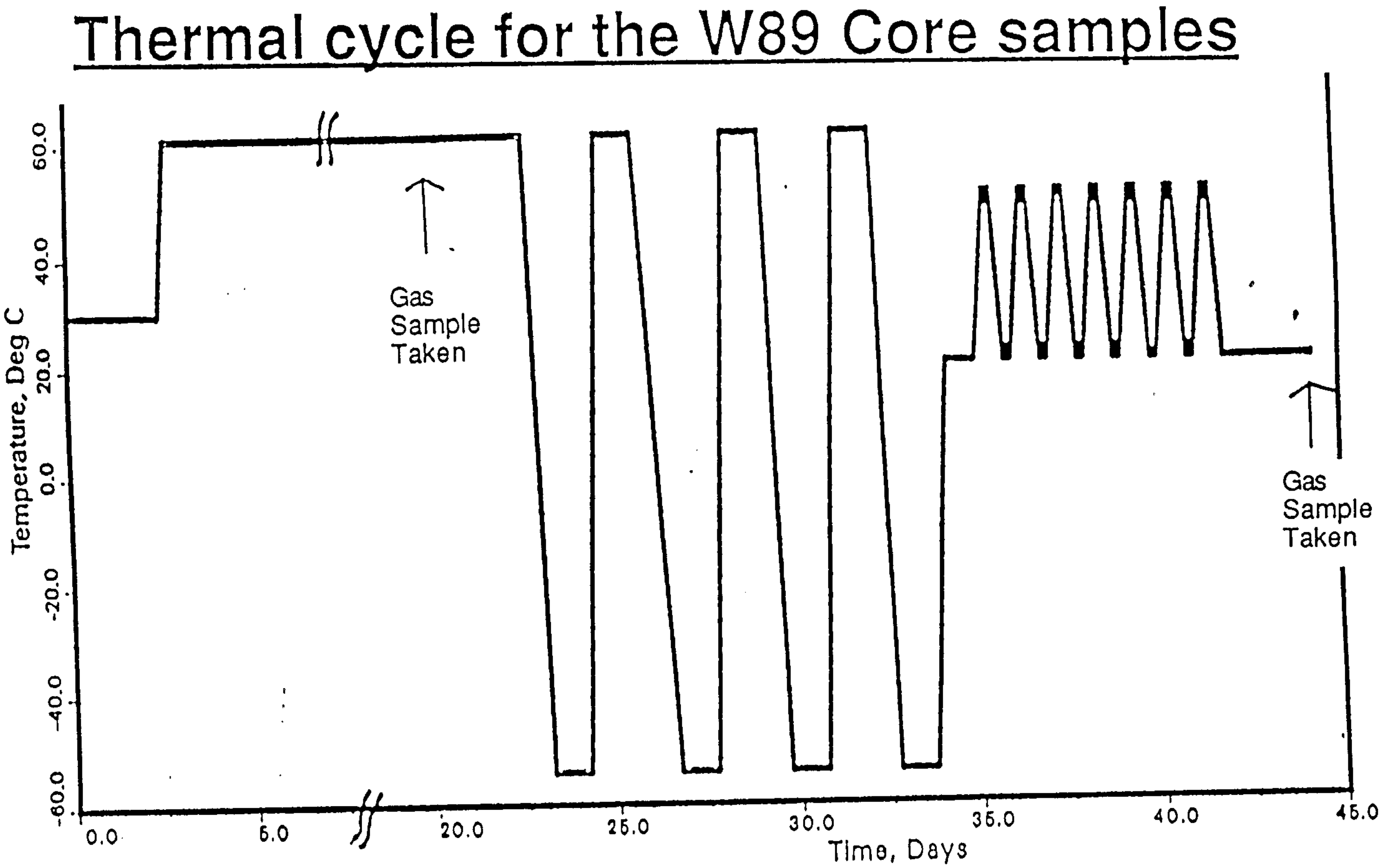


Figure 22. DAAU-1 Thermal Cycle

Figure 23. Core Test Unit Thermal Cycle



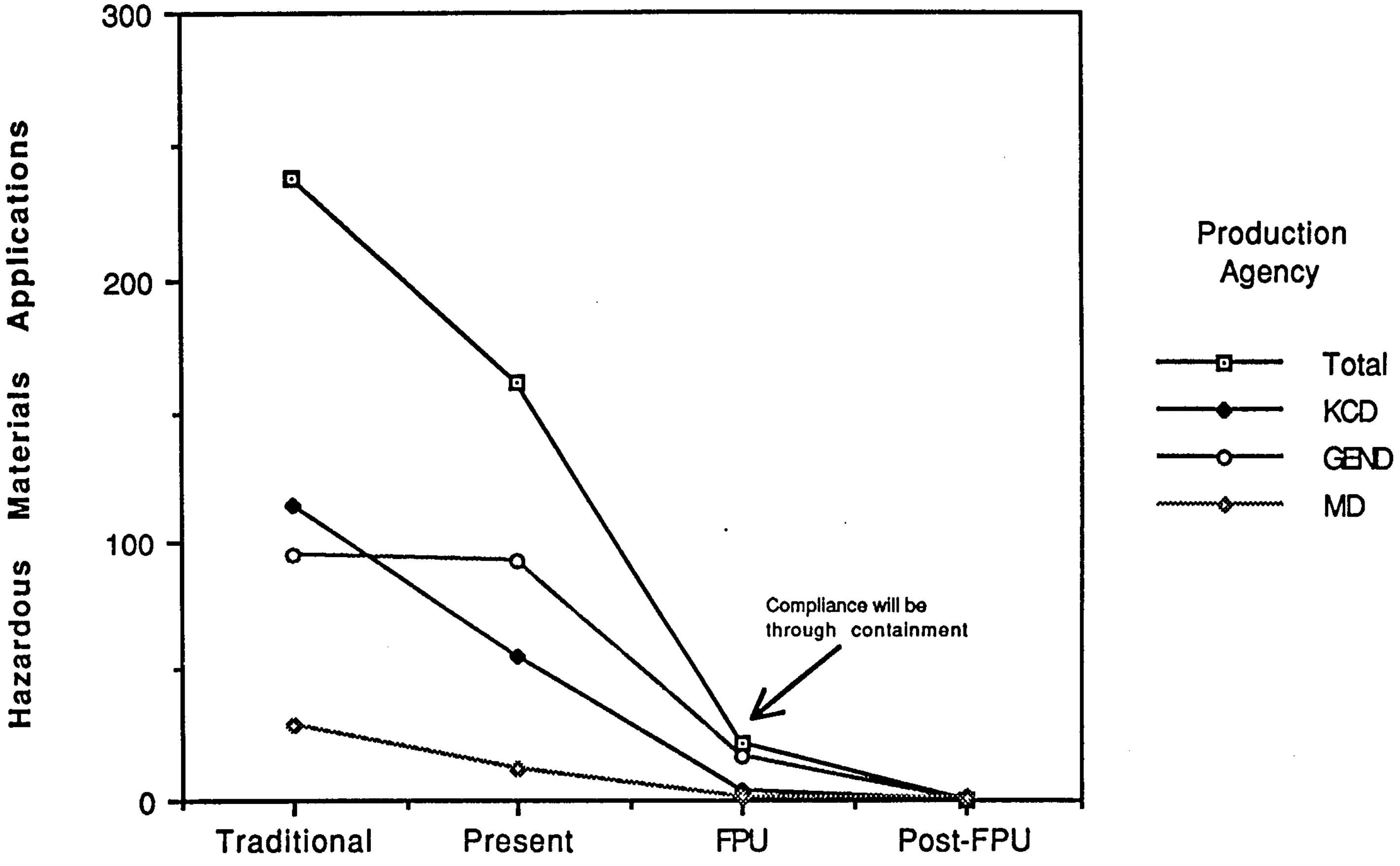


Figure 24. W89 ES&H Status

Table 1 W89 ES&H Development Areas for Production Agencies

	Cleaning	Coating	Adhesives	Encapsulants	Printed Wiring Boards
KCD	X	X	X	X	X
MD	X	X	X	X	
PP	X			X	
Y-12	X				
PX	X	X			

Table 2 Development Areas for W89 Non-Nuclear Components

	Cleaning	Coating	Adhesives	Encapsulants	Printed Wiring Boards
MC4069 Firing Set	X		X	X	X
Firing Set Housing	X				
MC4073 Programmer	X		X	X	X
MC4063 Stonglink	X	X	X	X	X
Cables	X			X	
Rolamite	X				
JTA (Joint Test Assy)	X				
Structural Parts	X	X			
MC4237 Initiator	X	X			
MC3753 Actuator	X	X			
DSSL	X	X	X	X	
MC4295 Timer	X	X	X	X	
Magnetics	X			X	
LAC	X	X			
MC3318 Heather	X				
MC4135 Neutron Gen.	X			X	
Shipping Containers		X			

Table 3 Early Candidate Cleaners in Solvent Substitution Studies

Associated Chemists: Templex 873
Brulin: 815QR, 815GD
Chemical Ways: KC270
Grace: Daraclean 282
Quaker: 624GD
Pennwalt: Turco 3878 LF-NC, Turco 4215 NC-LT (also with diethy glycol monobutyl ether additive), Turco 6719, Turco Plaudit
Rochester-Midland: SE353, SE354, SE358, SE358BA, SE373, SE374
World Enzymes: Environzyme
3D: Citragold
3M: Citra-Solv
Petroferm: Bioact DG-I, Bioact DG-III, Bioact EC-7
Tri TCN: Hemo-sol, Hemo-Sol E, Hemo-Sol W
Oakite: Okemclean
Mirachem: Mirachem 100
Unitech International: Unitech CV-250
ICI Chemicals: Porpaklone
Kester Solder: Kester 5121 and 5769
Martin Marietta: Marclean
Alpha Metals: Alpha 565 and 2110
ByPAS of Toledo: ByPAS
Chemtech International: CT-1, CT-2

Table 4 General Contaminents in the KCD Firing Set Production Area

Contaminent	Uses
Apiezon grease M	Vacuum grease
Carnuba Wax	Mold Release
Dioctyl phthalate	General contaminant, vinyl plasticizer
Epon 828 resin	Epoxy adhesive resin
Krylon spray acrylic	Cover coat for ink markings
OSR Mold Release	Mold Release
Starrett M-1 Oil	Penetrating oil and lubricant
Thalco 550	Mold Release
Vaseline	Lubricant, hand lotion ingredient and plasticizer
Velva Sheen	Dust mop treatment
Versamid 140	Epoxy curing agent
WD-40	Penetrating oil and lubricant
Zinc stearate	Mold Release

Table 5 MESERAN Results for the Removal of General Contaminents

	Cleaner Meseran #/Cleaning Time, seconds				
Contaminent	TCE	EC-7	Exxate 1000	EC-7R	d-limonene
Apiezon Grease M	0/10 s	0/6 s	0/10 s	0/5 s	0/5 s
Carnuba Wax	0/10	0/17	0/45	10/45	0/30
Dioctyl phthalate	0.5	0/5	0/5	0/5	0/5
Epon 828 resin	0/10	0/12	0/10	48/240	172/240
Krylon spray acrylic	0/15	0/240	0/150	1097/240	1296/240
OSR Mold Release	0/75	0/105	26/240	0/210	197/240
Starrett M-1 Oil	0/10	0/13	0/25	0/15	0/10
Thalco 550	0/60	0/102	11/65	0/ 60	29/30
Vaseline	0/10	0/8	0/15	11/9	3/9
Velva Sheen	0/5	0/7	0/10	0/8	0/8
Versamid 140	0/40	0/100	32/360	0/240	129/240
WD-40	0/10	0/8	0/10	0/8	10/10
Zinc stearate	0/ 2	0/3	18/5	1/3	12/3

TABLE 6

PROPERTIES OF d-LIMONENE

TEMP, °C	VAP PRESS mm Hg	VAP DEN g/lit	LIQ DEN g/cc	LIQ VOL, %
0	0.256	0.00205	0.8692	0.000236
10	0.570	0.00440	0.8606	0.000511
20	1.180	0.00880	0.8520	0.00103
30	2.30	0.0166	0.8434	0.00197
40	4.23	0.0295	0.8348	0.00353
50	7.43	0.0503	0.8262	0.00609
60	12.5	0.0820	0.8176	0.0100
70	20.2	0.129	0.8090	0.0160
71	21.2	0.135	0.8081	0.0167

Table 7 Screening Tests Results of Bulk Organic Materials with Low Response

Material	Weight Gain % TCE	EC-7	d-Limonene	Changes
Polycarbonate	4.4		0.03	Cracked+chalky in TCE
TPX	1.4		0.5	
Kynar (Polyvinlidene Fluoride)	0.03		0.01	
Kapton	0.33	0.3	0.7	
Polyimide/Glass (PWB)	0.1		0.15	
Polysulfide	5.5	0.2	0.3	Volume increase with TCE
Mylar	0.4		0.2	
Parylene C Coating	1.5		<0.1	Softer in TCE
DAP	0.01	0.05	0.04	
Polyetherimide	0.06	0.36	0.7	Crazed in TCE
PEEK	0.74	.044	0.26	
PPS (Fortron)	0.46	.034	0.44	
Phenolic Cotton	0.02	0.5	0.3	
TFE Rod	0.04	0.1	0.05	
Nylon Rod	0.07	0.18	0.18	
ZYTEL (screw locking element)	6.6		2.8	
EPON 828/Versimid 140	5.1	0.53	0.7	
FM123, Epoxy/Glass	8.1	1.8	1.4	
Scotchcast 8	5.1	0.38	0.4	Softer in TCE
Epon 828/U	0.02	0.07	0.01	
Green HYSOL Epoxy, Sprygap	0.02	0.007	0.004	
Epoxy/Mica/Z, Red	0.005	0.01		
Epoxy glass cloth	0.08	0.09	0.034	
A2 Epoxy	0.014	0.002	0.002	
Epoxy Molding/ Glass Fiber, Brown	0.01	0.01	0.014	
Epoxy/Mica/Z, brown	0.02	0.28		
GMB Potting	0.07	0.02		
GMB Quick Patch	0.05	0.22		

Table 8 Screening Tests Results of Bulk Organic Materials with Moderate to High Response

	Weight Gain %			
Material	TCE	EC-7	d-Limonene	Changes
Poly styrene Foam	dissolved	diss'd	diss'd	Unchanged in IPA
Polysulfone	8		0.2	Cracked in TCE and d-lim. dissolved in TCE
Polysulfone, stress relieved	"insignificant"		insig.	no cracking
EN7 Polyurethane	32.7	16.3		softer in both
EN7w/1.5 Hr 160° F vac. bake out	.14	8.7		
EN8 Polyurethane	31	15.6	13.5	swelled and softer in all
EN8w/1.5 Hr 160° F vac. bake out	4.8	9.6	5.6	
Polyurethane sleeving			31.5	swelled and softer
Adiprene L100/Cycnacure	16		4.4	swelled in TCE
Lacquer Covercoat	22	3	5.2	swelled and softer in all
Sylgard 184/GMB	6.7	5	5.6	swelled and softer in all
Cellular Silicone	126	208	198	swelled and tears easily in all
Cell. Sil. after 22 hrs at R. T.	0.4	15	5	
Silicone, red	39	26	37	swell and more rigid in all
" ,after 22 hrs at R. T.	0.1	5	9	
Silicone 747 U	1.4	14.7	22.6	swelled in EC-7 and d-lim. more rigid in all
Flourosilicon O-ring	6.6		2.8	
EPDM O-ring	47		26	swelled and softer in both
Polyolefin Sleeving, As Received	13.9	24.4	24.3	
Polyolefin Sleeving, As Received, 1.5 Hr 160° F vacuum bake out	1.0	-0.8	-0.8	
Polyolefin Sleeving, Shrunk	16	22	25	
Polyolefin Sleeving, white As Received	2.7	11	14.3	
Polyolefin Sleeving, black, As Received	6	13	16.1	
Pyralux	46.9	8.0		Glass transition temp lowered by both, but TCE sample recovered
Pyralux, after 1 hr desiccation	17.7			
Pyralux, after 1 day desiccation	3.7	5.1		
Pyralux, after 3 days desiccation	3.9			
Kapton/Pryalux	0.6	1.3	1.2	
Kapton/Pyralux/Kapton	1.5	.27	.62	
Transkote KR			30	Adhesive layer appears dissolved off and reprecipiated
Transkote KR/Kapton			3	No change

Table 9 EPDM O-ring Test Results After d-Limonene Exposure

Condition	Swell Weight %	Hardness Shore M	Compression Set, % after 70 hrs at 125°C
Before Soak	-----	75	5.9
After Soak	13.5	67	7.2
After Compression Test	-0.9	74	-----

Condition	Swell Weight %	Hardness Shore M	Compression* Set, % after 70 hrs at R. T.
Before Soak	-----	75	6.4
After Soak	14.8	68	-17.2
Immediately After Compression Test	6.8	73	-----
3 Days After Compression Test	2.3	-----	-9.5
8 Days After Compression Test	0.6	-----	-3.5
18 Days After Compression Test	0.0	-----	0.9

* Based on original thickness, swelling causes number to be negative

Maximum allowable compression set = 12%
Hardness requirement is 73-83 Shore A

Table 10. Adhesives Degraded by d-Limonene

EFFECT OF ENVIRONMENTAL EXPOSURE ON SHEAR STRENGTH

EXPOSURE	MVP-11	POLYSULFIDE	Y9469 PSA
BASELINE (NO EXPOSURE)	3632 PSI	240 PSI	195 PSI
3 MONTHS IN NITROGEN ATM. @ 71 C.	4056	353	138
3 MONTHS IN d-LIMONENE ATM. @ 71 C.	1224	108	91
6 MONTHS IN d-LIMONENE ATM. AT 71 C.	1468	120	68
12 MONTHS IN d-LIMONENE ATM. AT 71 C.	1028	14	104

WET BLASTED ALUMINUM CLEANED WITH EC-7R

Table 11. Cellular Silicone/d-Limonene Compatibility Study

SOLVENT MASS (g)	WEAPON EQUIV SOLVENT MASS (g)	EXPOSURE TIME (months)	AVG CHANGE IN THICKNESS (%)	AVG CHANGE IN LOAD (%)
0.000	0.0	1	0	-9.2 +/- 0.6
0.012	2.0	1	0	-9.7 +/- 0.1
0.036	6.0	1	0	-8.7 +/- 2.5
0.072	12.0	1	1.3 +/- 1.8	-11.4 +/- 0.6
0.000	0.0	2	0	-19.3 +/- 0.5
0.012	2.0	2	0	-18.5 +/- 2.1
0.036	6.0	2	0	-18.6 +/- 1.3
0.072	12.0	2	1.3 +/- 1.8	-19.3 +/- 1.2
0.000	0.0	6	-3.8 +/- 1.8	-32.8 +/- 0.4
0.012	2.0	6	-2.6 +/- 0.0	-29.5 +/- 5.7
0.036	6.0	6	-3.8 +/- 1.7	-27.2 +/- 3.2
0.072	12.0	6	-3.8 +/- 1.8	-29.5 +/- 1.2

W89 FIRESET AGING PROGRAM
FIRESET AND MODULE EVALUATION PLAN

CAN NO.	CLEANING SOLVENT	ISOTHERM THERM CYC	THERM CYC ISOTHERM	MONTH 1			MONTH 2			MONTH 4			MONTH 6			MONTH 7		
				GAS	ELE	VIS	GAS	ELE	VIS	GAS	ELE	VIS	GAS	ELE	VIS	GAS	ELE	VIS
1	TCE	X		X	X	X	X							X	X		X	X
2	D-LIMONEN	X		X	X	X											X	X
3	D-LIMONEN	X					X							X	X		X	X
4	D-LIMONEN	X		X	X	X											X	X
5	D-LIMONEN	X					X							X	X		X	X
0A	TCE/HVLI	X		X	X	X	X							X	X		X	X
0	D-L/HVLI	X		X	X	X	X							X	X		X	X
6	TCE		X	X	X	X	X	X	X								X	X
7	D-LIMONEN		X		X	X	X	X	X								X	X
8	D-LIMONEN		X	X	X	X				X	X	X					X	X
9	D-LIMONEN		X		X	X	X	X	X								X	X
10	D-LIMONEN		X		X	X				X	X	X					X	X
11	D-L/HVLI		X	X	X	X		X	X		X	X					X	X
12	TCE/HVLI		X		X	X		X	X		X	X					X	X
	TCE	X			X	X								X	X		X	X
	D-LIMONEN	X															X	X
	D-LIMONEN	X												X	X		X	X
	D-LIMONEN		X		X	X					X	X					X	X
	D-LIMONEN		X					X	X					X	X		X	X

Table 12. W89 Fireset Aging Program

Table 13. Firing Set Housing Cleaning: AES Results

CONTAMINENT	CARBON/IRON PEAK HEIGHT RATIOS CLEANING PROCESS METHODS TRAD.	ALTERNATE	PREFERRED ALT.
TAP FLUID	.737	.944	.761
Cimstar 37OO	.845	.453	.645
Van Straaten 5487	.732	.670	1.095
EDM FLUID	.868	1.063	.806
DYE PENE	.721	.963	1.203

- AS RECEIVED CARBON/IRON PEAK HEIGHT RATIOS = 55.20

Table 14 XPS Carbon Analysis for Header Cleaning

CLEANER	XPS CARBON (AT. %)
AS-RECEIVED	36.0
METHYLENE CHLORIDE / 4 STEP CLEANING	27.0
EC-7 / 4 STEP CLEANING	15.7
NMP / 4 STEP CLEANING	14.7
DPM / 4 STEP CLEANING	38.4
ETHYL LACTATE / 4 STEP CLEANING	20.0

	METHYLENE CHLORIDE (CONTROL)		BIO ACT EC-7 2-STEP PROCESS		BIO ACT EC-7 4-STEP PROCESS	
	MC3753	MC4237	MC3753	MC4237	MC3753	MC4237
6 WEEK ELEVATED			CHEM ANAL: 2 VEC: 3	CHEM ANAL: 1 TEST: 2	CHEM ANAL: 2 VEC: 4	CHEM ANAL: 2 TEST: 3
6 MONTH CYCLIC		CHEM ANAL: 2	CHEM ANAL: 2	CHEM ANAL: 1	CHEM ANAL: 2	CHEM ANAL: 2
1 YEAR CYCLIC	CHEM ANAL: 2	CHEM ANAL: 2	CHEM ANAL: 2 VEC: 12	CHEM ANAL: 2 TEST: 2	CHEM ANAL: 2 VEC: 13	CHEM ANAL: 2 TEST: 4
2 YEAR CYCLIC	EXTRA: 1	EXTRA: 2	CHEM ANAL: 2 EXTRA: 1	CHEM ANAL: 1 EXTRA: 1	CHEM ANAL: 2 EXTRA: 2	CHEM ANAL: 2 EXTRA: 1
1 YEAR ELEVATED		CHEM ANAL: 2 EXTRA: 1	CHEM ANAL: 2 EXTRA: 1	CHEM ANAL: 1 EXTRA: 1	CHEM ANAL: 2 EXTRA: 1	CHEM ANAL: 2 EXTRA: 1

ELEVATED INDICATES CONSTANT 70°C.

Table 15. Long-Term Compatibility Test Plan for Methylene Chloride Replacements for Header Cleaning

Appendix A. 4612140, d-Limonene, Materials Specification

Released 8-24-92

CAGE CODE 14061

F. N. Larsen, KC837 F. N. Larsen 8/21/92
A. T. Oravecz, SA2833
C. Anderson, SL8274

D-LIMONENE (U)

Page	1	2	3	4	5	6
Issue	C	C	C	C	C	C

Drawing Callout: D-Limonene per 4612140-(1).

(1) Insert Control Number Suffix.

Design Agency Control Number	Issue	Release/ Change Number	Date
4612140-00	A		9/89
	B	911750KC	5/91
	C	922527KC	8/92

1. GENERAL.

1.1 Scope. This specification defines the requirements for d-Limonene, a terpene solvent derived from natural citrus products and suitable for cleaning and other solvent uses. Chemically this material is designated as: (R)1-methyl-4-isopropenyl-1-cyclohexene. The product shall be relatively pure containing at least 95% d-Limonene and stabilized against oxidation by the addition of Butylated Hydroxy Toluene (BHT) antioxidant. The material shall be a clear water-white, low viscosity liquid with a mild to very light orange odor.

1.2 Definition. /M/ is a symbol placed adjacent to a section or paragraph to designate requirements or information pertinent only to KCD, it suppliers and subcontractors.

2. DOCUMENTS.

None.

3. REQUIREMENTS.

3.1 Approved Products. The following are approved products:

Product Designation	Manufacturer
D-LIMONENE, Food Grade, MF	Florida Chemical Co.

3.2 Properties. The material shall conform to the following requirements:

Property	Requirement	Test Method
d-Limonene, %	95% minimum	Gas Chromatography per Section 4.3
*Non-Volatile Residue	1% maximum	Residue on Evaporation per Section 4.4
*Optical Rotation, Degrees/Decimeter	+100.0 minimum	Optical Polarimetry per Section 4.5

***NOTE:** These requirements are the responsibility of the Buyer only and NOT the Seller.

3.3 Fourier Transform Infrared Spectroscopy. A Fourier Transform Infrared (FTIR) spectrum shall be run by the Buyer, per 4.6, on each lot of material submitted for acceptance testing. A report of conformance or nonconformance is required.

3.4 Antioxidant. The material shall be inhibited against oxidation by the addition of Butylated Hydroxy Toluene (BHT) antioxidant at a concentration of 400 to 600 parts per million. Buyer shall confirm this requirement by gas chromatography analysis per 4.7 on each lot of material submitted for acceptance testing.

4. QUALITY ASSURANCE PROVISIONS.

4.1 Lot Definition. A lot shall be a homogeneous quantity of material.

4.2 Lot Inspection and Testing. A representative sample from each lot, submitted for acceptance, shall be inspected and tested for conformance with the requirements listed in Sections 3.2 through 3.4.

4.3 Gas Chromatography Analysis. The percent d-Limonene shall be determined by any suitable gas chromatography method that utilizes a silicone coated capillary column and a flame ionization detector and adequately separates the many terpene, aldehyde, alcohol and ketone impurities. A technique such as a wet needle sample injection and peak area normalization is acceptable in lieu of an analytical standard. A sample chromatogram shall accompany the analytical report.

4.4 Residue On Evaporation. A Non-Volatile Residue (NVR) value shall be determined by the following procedure:

a. Weigh a dry 80 x 45 mm No. 3180 Pyrex evaporating dish to the nearest 0.1 mg.

b. Add by volumetric pipet, ten milliliters of the sample solvent.

c. Reweigh dish and sample. Calculate the weight of sample.

d. Allow the sample to evaporate at room temperature overnight. A fume hood should be used for volatile removal. A flowing dry nitrogen blanket should also be used to speed evaporation and minimize oxidation.

4.4 Continued.

e. Place the dish in an air circulating oven at 71°C (160°F) for four hours.

f. Allow dish and residue to cool and reweigh.

g. Calculate the percent residue by dividing the weight of the residue by the sample weight and multiply by 100. Report as NVR.

4.5 Optical Polarimetry. The optical rotation of the material shall be measured to confirm that this product is derived from natural citrus products and not from pine wood products. The measurement is made using an optical polarimeter, such as a Model SR-6 Polarimeter with sodium lamp (full circle scale, 0-180° right and left) available from Polyscience, Niles, Illinois. A 100 mm sample tube is used for the measurement. The requirement is for the specific rotation which is obtained by dividing the measured optical rotation by the d-Limonene density (0.84 at 25°C), when using a 1 decimeter (100 mm) length cell.

4.6 Fourier Transform Infrared Spectroscopy. An FTIR spectrum shall be run by the Buyer on each lot of material submitted for acceptance. The conditions for analysis should typically include: a capillary film between KBR plates, a range of 4000 to 400 cm^{-1} scan, and a resolution of 4 cm^{-1}. A typical spectrum for reference is shown in Figure 1.

4.6.1 Report. An evaluation and judgement of conformance or nonconformance shall be made with respect to the intensities and position of the various absorption bands given in the reference spectrum.

4.7 BHT Analysis by Gas Chromatography. The BHT antioxidant content shall be measured by any suitable gas chromatography method capable of isolating the BHT peak from the solvent and its impurities and quantitating at the parts per million level. A silicone coated capillary column and a flame ionization detector are recommended. A calibration curve shall be prepared from analytical standards of BHT over the range of 0 to 1000 ppm and used to quantitate the BHT content of the sample submitted for acceptance.

5. /M/ PACKAGING, HANDLING AND STORAGE.

5.1 Protective Packaging. The packaging container shall be glass or coated metal cans new (never used before), clean, sealed from moisture and shall be capable of being easily resealed.

5.2 **Handling. This material should always be protected from exposure to air (oxygen) in storage and handling. Container should always be flushed and blanketed with dry nitrogen and sealed tightly to prevent oxidation and to extend the shelf life.**

5.3 Package or Container Identification. Each individual container of material shall be marked with at least the following information:

Material Specification Number
Product Designation
Name of Manufacturer
Manufacturer's Lot Number
Date of Receipt
Storage Condition: B1-7.
Shelf Life: 1 Year from Date of Receipt

5.4 Safety and Health Precautions. Undiluted liquid may be irritating to skin and eyes. Inhalation of vapor is irritating to throat and lungs. This material has only very slight acute toxicity and is noncarcinogenic.

5.5 Shelf Life. The shelf life of this material is one year from date of receipt from the manufacturer. Any material that reaches this age before use shall be reinspected for conformance to the requirements of Sections 3.2 and 3.3. If it fails to meet any requirements, it shall be scrapped. If it meets all of the requirements, it shall be retained. The material shall be retested every 12 months to the same requirements until it is consumed or scrapped.

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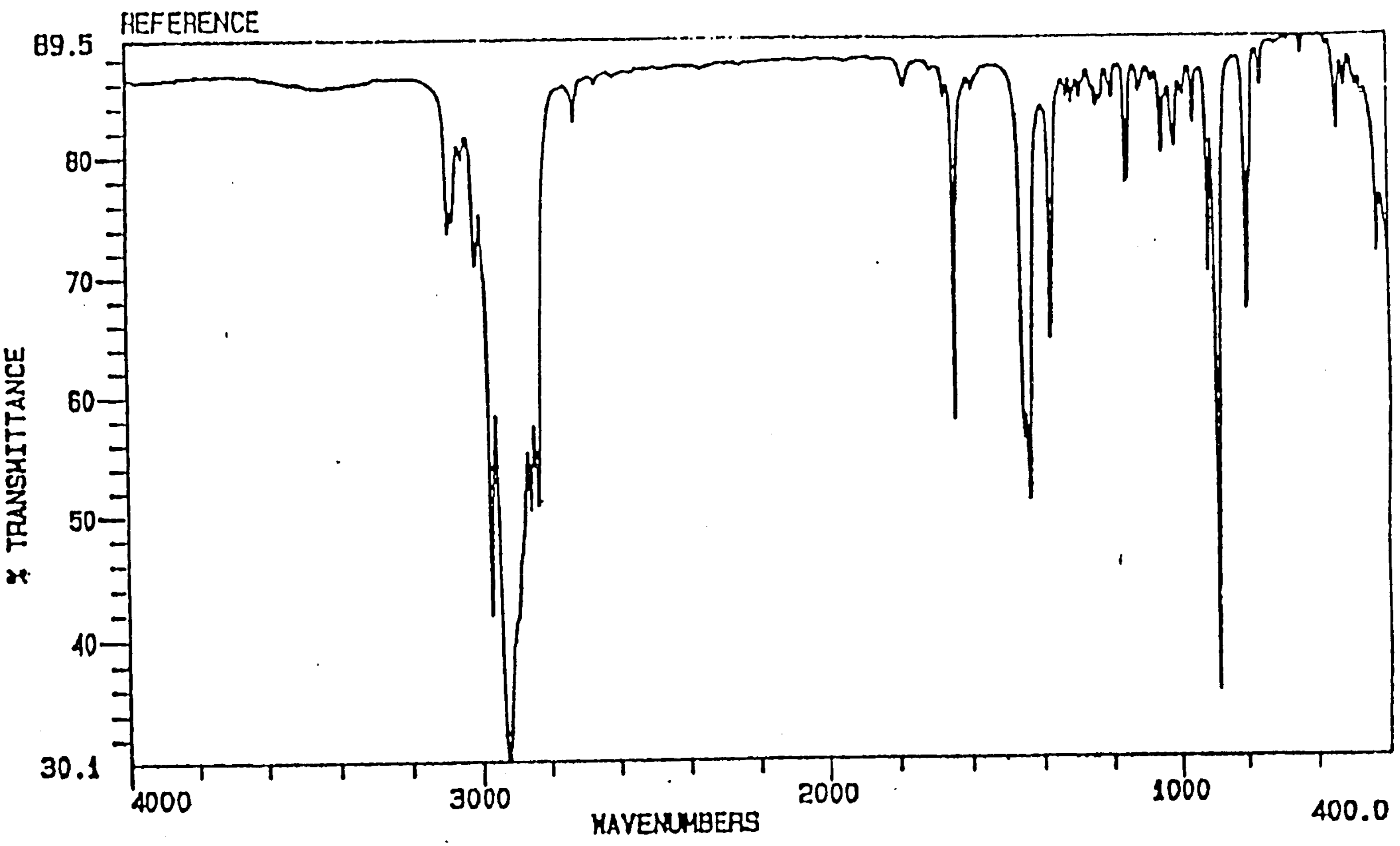

ALLIED SIGNAL

d-LIMONENE,
CAPILLARY FILM BETWEEN KBR PLATES
SCANS : 32 RES : 4.00

Figure 1

Appendix B. Organic Materials List for the W89

MC4069 Firing Set:

Material	Specification or Part Number	Expected Suseptibility
Covercoat Laquer	2170116	High
Soldermask	4702901	Medium
Ink, stamp pad	5093162	Medium
Ink, Epoxy	5096951	Medium
Hysol MH8F0188	0026	Low
Polyolefin sleeving	2211506	High
GMB Encapsulant	0003	Low
GMB Quick Patch	0004	Low
Phenolic	2030199	Low
Polyphenylene Sulfide (PPS)	2225027	Low
Polyethylene Terethlat (PET)	0062	Medium
Polystyrene (PS) Beads	1463424	Medium
FM 123 Epoxy	2180935	Low
Epon 826/Diethylenetriamine(DET)	0006	Low
Epoxy Paint	0015	High
EN8 Polyurethane Encapsulant	2519605	Medium
Epon 828/Versamid 140	2201225	Medium
Hysol DK17	0017	Low
EFTE Wire, black	8230600	Low
Polyolefin sleeving, black	2211516	Medium
Polyetherimide	MIL-P-D4618	Medium
Phenolic Covercoat	0052 & 0059	Low
Teflon sleeving	221115XX	Low
Epoxy glass cloth	2141004	Low
Cable, RG196	8275196	Low
TFE Rod	ASTM-D-1710	Low
Diallyl Pthalate (DAP)	MIL-M-14G	Medium
Epoxy	21801071	Low
Teflon wire	82209XX	Low
Mica filled epoxy with Z hardener	0001	Low
Phenolic Linen	0059	Low
EN7 Polyurethane Encapsulant	2519603	High
Polyolefin sleeving	2211556	Medium
Hysol Case	0022	Low
Epoxy Impreg	0025	Low
Kapton	0023	Low
Scotchcast 8	2140606	High
Nylon	ASTM-D-4066	Medium
EFTE Wire, white	8230609	Low
Epoxy mold	2144087	Low

DSSL:

- Polyolefin heat shrink tubing
- Halthane 88-3 potting
- Syntactic, Polysulfide (9905106)
- Epoxy, Epon 828 glass microballoon filled with DEA hardener per (9927085)
- Kapton
- Phenolic (connector shell)
- Fiberite E264H (2144087) Glass Filled Epoxy
- DAP
- Insulation, Tubing, PTFE per MIL-I-22129C
- Everlube 9000, Molybdenum disulfide solid film lubricant

Adhesives:

- Pryalux .001"thick
- Epoxy per 2181026 (EC2214)
- Epon 828 Epoxy Resin per 2140285
- Epoxy per 2181065
- Epoxy per 2181086
- Curing Agent (Ethacure) per 4604047
- Silicone RTV:
- Uniglobe Kisco SHIN-ETSU KE3497W (per MIL-A-4616A, Type II) or Dow Corning 3145 or Dow Corning 3140
- Epoxy (unspecified - in Cannon "Nano" connector)

Connectors:

- Fiberite E264H (2144087) Glass Filled Epoxy
- Fiberite E2748 Mineral and Glass Filled Epoxy
- Fiberite E3938C Glass Filled Epoxy
- Silicon Rubber, Class 2 (ZZ-R-765)
- Teflon insulators
- Polyetherimide (GE Ultem 2300)
- DAP, RX1310, Type SDG (Mil-M-14)

Adhesives:

- Silicone RTV:
- Uniglobe Kisco SHIN-ETSU KE3497W (per MIL-A-4616A, Type II) or Dow Corning 3145 or Dowcorning 3140
- Polyimide-imide coating, Amaco AI-10 powder (with acetone and N-MethylPyrrolidone as solvents)
- 3M AF110

HVLI Gasket:

- Silicone Rubber, Dow Corning DC747 (per SS391245)

C.S.A.(Contact Seal Assembly):

- PEEK (per 2221007)

MC4078 LAC:

- Fiberite E3938C Glass Filled Epoxy
- Polyamide (Nylon), unknown spec or Teflon
- Polyimide per SS306045
- Polysulfone, Mineral Filled, Union Carbide Mindel M-825, BCO Spec #9-7843
- Epoxy Resin Encapsulation, BCO Proprietary, BCO Spec #9-4954-2

	Encapsulation per 9927085, 828 CTBN/ GMB 40 (or GMB 32)/DEA
	Non-hermetic Microsemi diodes in SA3642
Adhesives	Epoxy Adhesive per 46A102300P001, Hysol Epoxy Patch 151

Cables:

	Polyimide	.002" per 2330722
	Polyimide	.001 per IPC-FC-233/A
	Polyimide	.004" per 2322952
	Polyimide	.002" per 2322952
	Nylon Fabric per 2204257	
	Polyurethane per 2170456, extruded sleeving	
	Zytel 101 Nylon,	
	Adiprene Cyanacure per 9927104 (Encapulant)	
Adhesives:	Pryalux	.002" Adhesive per 2181044
	Pryalux	.001" Adhesive per 2181043
	Pryalux	.001" per 2330722
	Pyralux	per 2330724
	Adhesive	.002" per 2322952 (probably Pryalux)
	Adhesive	.001 per IPC-FC-233/A (probably Pryalux)
	Epoxy per 2181034 (Fortin)	

MC4196 Cable and Timer:

	Kapton
	Transkote KR, Polyester with polyethylene adhesive copolymer
HE:	HNS-FP
	HNS
Adhesives:	Pyralux
	Fortin Epoxy
	Silicone RTV per SS384888
	Anhydride cured glass microballoon filled epoxy per SS390353
	Adhesive per 2240055 rapid setting (assembly aid)
	RTV silicon primer per 2526631

MC4135 Neutron Generator Assembly:

	Lexan polycarbonate
	Kapton per 2322921-54
	Sylgard 184 (fused glass, cabosil or glass microballoon fillers)
	Sylgard 184 - glass microballoon filled potting
	Epon 828 GMB encapsulant (glass microballoon filled) with DEA hardener
	Epon 828 Alumina filled encapulant with "Z" hardener
Adhesives:	Loctite 222
	Epoxy film per 2181033 or 218034, .001 or .002" thk ECC031 or ECC032 (Fortin) or FM-10310R (American Cyanamide)
	Epon 828 with Versimid 40 hardener (or 140?)
	Rapid Bonding Adhesive per SS284668 (alpha cyanoacrylate type)

Programmer:
Fiberite E264H (2144087) Glass Filled Epoxy
Polyurethane (foam), 4 lb per 2170327
Teflon wire insulation per 8220701 thru -08
Nylon Braided Tape (waxed) per 2202168
Teflon tape 9922040
Polyolefin Heat shrink sleeving per 2211516,
2211526
2211549
Sleeving per 2270103 (Polyolelfin?)
Polyimide Glass per 2141954
Plastic Sheet, Polyimide/Glass Fabric per 2141953
Prepreg Glass Fabric,Polyimide Resin per 2191011

Adhesives: Devon F, Al filled epoxy per 2181085
Epoxy Kit, EA934NA, per 2181086
Epoxy, Devcon per 2181050
Marking:
Stamp Pad Inks: 5093162, 5093262, 5093762, 5093153, 5093350, 5093243, 5093201, 5093004
Covercoat Lacquer Krylon Spray, 5040400
Covercoat Epoxy, 5084403

Components:
Capacitors: SA3184 Epoxy particle hand painted and thermal fused to 2-5 mil coating with HYSOL DK17 or AVX P504G (same formulation different supplier)
SA3613,3614,3616,3617,and 3619: Epoxy molded or Epoxy case and then epoxy backfilled. Epoxy type HYSOL MG8F-0341
Core Memory #391020:
Epoxy, Mica filled encapsulant with "Z" hardener
DAP
Polyester, insulation
Epoxy coating (unknown type) on Magnetic core (vendor)
Within coating: Fiber cover, material unknown
Relay, MC3594 (Babcock is vendor):
Polyimide per Mil-P-46112, Type 1 for bobbin
Polyimide insulation per JW1177/15 Class 220,Type M
Kapton sleeving, per Mil-P-46112B Type II, Grade A.
Teflon oversleeve
Silicone elastomer tubing per Mil-Std-104A.
Ryton R4, (High temperature polyphenylene sulfide, glass filled, productiof Phillips Chemical.)
Fiberglass impregnated teflon yarn (Dodge Fike, Type R Spec #E701-390)
Clock, MC4226:
Polyurethane TDI foam with covercoat
Cover coat: UVR 6110: a UV cured Cycloaliphatic epoxide
Adhesive: Epon 828 w/Versimid 140
Encapsulant: Epon 828, Mica Filled, "Z" Hardener per 9927020-01

Resistors:
440015 Metal Film: Pheolic Varnish, Borden Chemical SE1008 Epoxy coating (particles heated into coating),Dexter DK-18-0715
440016,17 & 440060, 61,65: Silicon coating (type unknown)

ESD:

DAP glass fiber filled per 2081077
Silicon F Fluid, 2 centisoke per 6500503-01, PX318689
Polyolefin shrink tubing
Fiberite E264H (2144087) Glass Filled Epoxy

Adhesives: Epoxy "A2" per 2140015-02
Epon 828-GMB (glass micro ballon filled) with hardener.
Note: Hardener not specified but "Z" is prohibited, DEA is usually used.

Misc:
LX-13
PETN
EPDM O-rings per SS455508
Krytox 240AD O-ring grease
Heat Resistant Urethane Foam, per 2170674, 1.36 +/-.03 gm/cc
Polyamide (6/6)Elastomer Modified, Zytel ST301 per 2204290
Zytel 101,per Mil-F-18240 Screw Locking Element or Zytel 42, Nylon-610, Nylon #11 or Nylon Fed. Spec. L-P-410A, Type 6
Rulon J - Polyimide loaded Teflon
Lexan Polycarbonate
Dexter Hysol Epoxy EPK 9340
Locktite 242 per 2502329
Solid Film Lubricant, Lubribond Type A, per 6530914-01
Gore-Tex expanded PTFE sleeving, nylon fabric
Epon 828/Versmid 125

LLNL Components:

TPX - polymethylpentene
Silicon Foam M9750
Lexan polycarbonate
Kapton

HE's: LX-17-1
LX-13
LX-16
HNS
HNS-FP (fine particle)
Ultrafine TATB

Adhesives: Halthane 73-18 per RM266191: Halthane 73 prepolymer, amine cured
Halthane 88-3:Halthane 88 prepolymer cured with Asilamine 4854
Spec. RM255172 for Hathane 88-2 will b revised
LST (low surface tension epoxy adhesive) replaces:
Explostick, but may still be used in oneapplication:.
Explostick Adhesive 473
Epoxy per RM255734
APC 2.5 per RM253987 (Silicon resin base)
APC 300 (Silicon resin base)
RTV 732 (1,2 silastic)
Epon 828 -Versamid 140 Epoxy
Pyralux .002" thick
Fortin Epoxy

Appendix C. Sandia National Laboratories Policy on Weapon Program Responsibilities from Design through Retirement

SANDIA NATIONAL LABORATORIES POLICY ON WEAPON PROGRAM ENVIRONMENT, SAFETY, AND HEALTH RESPONSIBILITIES FROM DESIGN THROUGH RETIREMENT

A basic principle of the Sandia National Laboratories environment, safety, and health policy is to ".....design products and conduct operations with the highest regard for the protection and preservation of the environment, safety, and health of its personnel, contractors and the public." Activities involving weapons in all phases including development, stockpile, and retirement shall adhere to this policy constantly and diligently.

Weapons now in development, such as the W89 and W91, incorporate the most benign materials and processes possible in order to respect the environment throughout the development/production/stockpile/retirement. Future weapon component and system development programs will not only utilize the most benign materials and processes possible, but also design and conduct the development, testing, and production start-up such as to generate minimal waste and by-products.

To assure safety and full compliance with environmental regulations during disassembly and disposal, complete documentation of all materials in a weapon system shall be an integral part of its development program. We will provide materials data for components and systems in stockpile and retirement phases to the agencies performing those activities. Each weapon project organization shall be responsible for maintaining its materials information so that composition, amounts, and locations of materials under question can be easily retrieved.

Sandia National Laboratories will also support the DOE activities in the development and application of treatment and disposal technologies to achieve permanent, safe disposal of all hardware and materials from weapon retirement.

At Sandia National Laboratories, quality, safety, and protection of the environment are primary in all phases of our mission.

G. N. Beeler, 5100 (A)

Joan Woodard

J. B. Woodard, 6600

E. E. Ives, 8100

July 1, 1991

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