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**THE MASSACHUSETTS  
TOXICS USE REDUCTION INSTITUTE**

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**ELIMINATION OF OZONE  
DEPLETING CHEMICALS  
AT M/A-COM, INC.**

Technical Report No. 9

1993

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University of Massachusetts Lowell

# Elimination of Ozone Depleting Chemicals at M/A-COM, Inc.

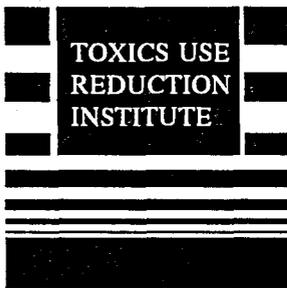
Edward Surette  
Principal Investigator

M/A-COM, INC.  
Lowell, Massachusetts

## The Toxics Use Reduction Institute Matching Grants Program

August 1993

The Toxics Use Reduction Institute  
University of Massachusetts Lowell



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## **Toxics Use Reduction Institute Matching Grants Program**

The Institute annually provides direct funding to Massachusetts industries on a matching basis for toxics use reduction (TUR) feasibility and technology studies. The Matching Grants Program was initiated in FY93 to facilitate the development and use of innovative techniques that reduce the use of toxic chemicals or the generation of toxic byproducts in Massachusetts businesses. Grants are awarded on a competitive basis for companies to conduct TUR studies at their facilities. Recipients prepare project reports which assist in transferring toxics use reduction technologies and methods to other companies. The opinions and conclusions expressed in this report are those of the authors and not necessarily those of the Toxics Use Reduction Institute.

M/A-COM, INC.  
401 Edgewater Place, Suite 560  
Wakefield, MA 01880  
Vendor Code: 042-090-644-000-7  
Requisition #2803  
Trust Purchase Order #S-040007

## TOXICS USE REDUCTION INSTITUTE (TURI) DELIVERABLES

Final Report December 1992 and June 1993

Traditionally, cleaning steps in fabrication and assembly have often relied upon chlorinated solvents and chlorofluorocarbon solvents for the removal of flux residues. With the identification of chlorofluorocarbon 113 (a.k.a. Freons) as a contributor to the ozone depletion in the stratosphere, there is an expedience to reevaluate cleaning and to find ODC free alternatives that at a minimum are at least technically equal to existing methodologies. Industry has been looking at all cleaning media to evaluate their health potential to operators who use them, to the air we breath, to the water we drink and to the stratospheric ozone which protects us from harmful ultraviolet radiation. Solvent cleaning and aqueous/semi-aqueous cleaning must meet stringent guidelines when introduced in a manufacturing environment. New cleaning materials have been developed in an attempt to meet the needs of the industry and to provide safe alternatives.

### Phase I

In February of 1992, the M/A-COM Executive Council authorized the development of a task force which would be responsible for the elimination of ozone depleting chemical usage throughout the corporation.

In constructing the task force, the Executive Council reviewed the major ozone depleting chemical users (by division) and selected individuals who collectively are knowledgeable about their product lines with emphasis on overall cleaning involvement.

The Task Force developed a program with the stated objective of promoting the reduced use of ozone depleting chemicals through material substitution, process modification and best management principles.

Internal divisional surveys were conducted to aid the task force in identifying the specific types of ODCs used within the product bases throughout the corporation. With this information the task force would be able to target the ability of each facility to efficiently and effectively begin to eliminate ODCs. Sixteen divisions were targeted in the survey. From the results obtained it was evident that the majority of ozone depleting chemicals was used for cleaning electronic assemblies. Machine parts was the next largest user which represented a different cleaning enigma, therefore the ODC steering committee formed two distinct cells. These cells allowed the Task Force to focus on two major components of ODC usage within the corporation. It also allowed each task team to schedule specific research, testing and implementation of alternative cleaning systems.

In order to develop standards to evaluate alternative cleaning systems, the ODC Elimination Task Team focused on the following specific criteria. The cleaning system must:

- a) Be non-ozone depleting
- b) Accomplish effective cleaning
- c) Not impair the quality of our product by chemical degradation or mechanical damage
- d) Be both mechanically and chemically safe for our products
- e) Enhance work place safety (i.e. low toxicity, low flash points etc.)
- f) Be approved by IPC and the U.S. Department of Defense (DOD)
- g) Be commercially or readily available
- h) Have some on-site history within the industry
- i) Minimize generation of waste by-products, with zero wastewater discharge as a goal
- j) Reduce air emissions of volatile Organic Compounds (VOCs) to specified levels which will avoid the need for air handling permits

Being environmentally responsible requires that wastes be disposed of in a responsible manner and that waste generation be minimized. In a practical manner, when this policy is applied to ODC replacement cleaning methodologies, it means that the rinse water and or cleaning chemicals will need to be recycled or "closed looped". Accumulated wastes would be placed in containers and shipped from company premises by approved waste handlers for environmentally safe disposal.

The objective of the ODC Elimination Task Force is to develop a "risk Free" non-ozone depleting cleaning process for electronic assemblies and machined parts. This necessitated the team to divide the project into two phases.

The goal of the phase I initiative was to identify chemistry candidates that had gone through testing developed and sponsored by the International Printed Circuit Board Association (IPC) and the Department of Defense Phase II program. Once the candidates were identified the ODC Task Team developed and followed a controlled test procedure, which allowed the team to evaluate the chemistries and cleaning equipment in contact with the piece parts to be cleaned.

Non-ozone depleting cleaning systems include the entire cleaning process, which is the selection of the cleaning chemistry, cleaning equipment, tooling/fixtures, and a rinse water recycling capability. The intent of our test plan was to find systems that offer qualitative advantages over our present chlorinated solvent processes in both cleaning performance and environmental impact.

In the system approach to cleaning, the plan as stated included a mechanism to handle wastewater. Wastewater treatment must include the separation of contaminants from process water followed by recycling of the purified water back to the cleaner. Contaminants are concentrated and removed from the system separately. A Reverse Osmosis Membrane (RO) system will be used to remove these contaminants and return DI water to the cleaning system. This closed loop system becomes an integral part of the Equipment.

The task team developed and implemented a screening program to identify cleaning equipment that could use the cleaning chemistries established. Equipment manufacturers were invited to M/A-COM in order to present their equipment credentials. An in-depth questionnaire evolved to insure that each equipment supplier would be asked to supply answers to like questions. After carefully weighing the results of twelve or more equipment interviews, seven were chosen for actual testing.

The actual testing, due to logistics, was accomplished in the field. The test plan orchestrated performances at the seven suppliers of cleaning equipment using the three pre-established cleaning chemistries. The baseline control group, as well as all test samples were manufactured from one lot of material, using the same operator (s), following identical procedures and equipment using a single set-up and immediately stored in clean static free bags for subsequent testing. Baseline specimens were cleaned in vapor degreasers using either Trichloroethane 1,1,1 and or Freons TMS, TMC and other specific proprietary cleaning processes. The cleaning systems, that were evaluated for all electronics applications, are identified as semi-aqueous. Each independent semi-aqueous system (equipment/cleaner) was evaluated using a detailed piece part distribution which included the following:

108 specially designed test samples

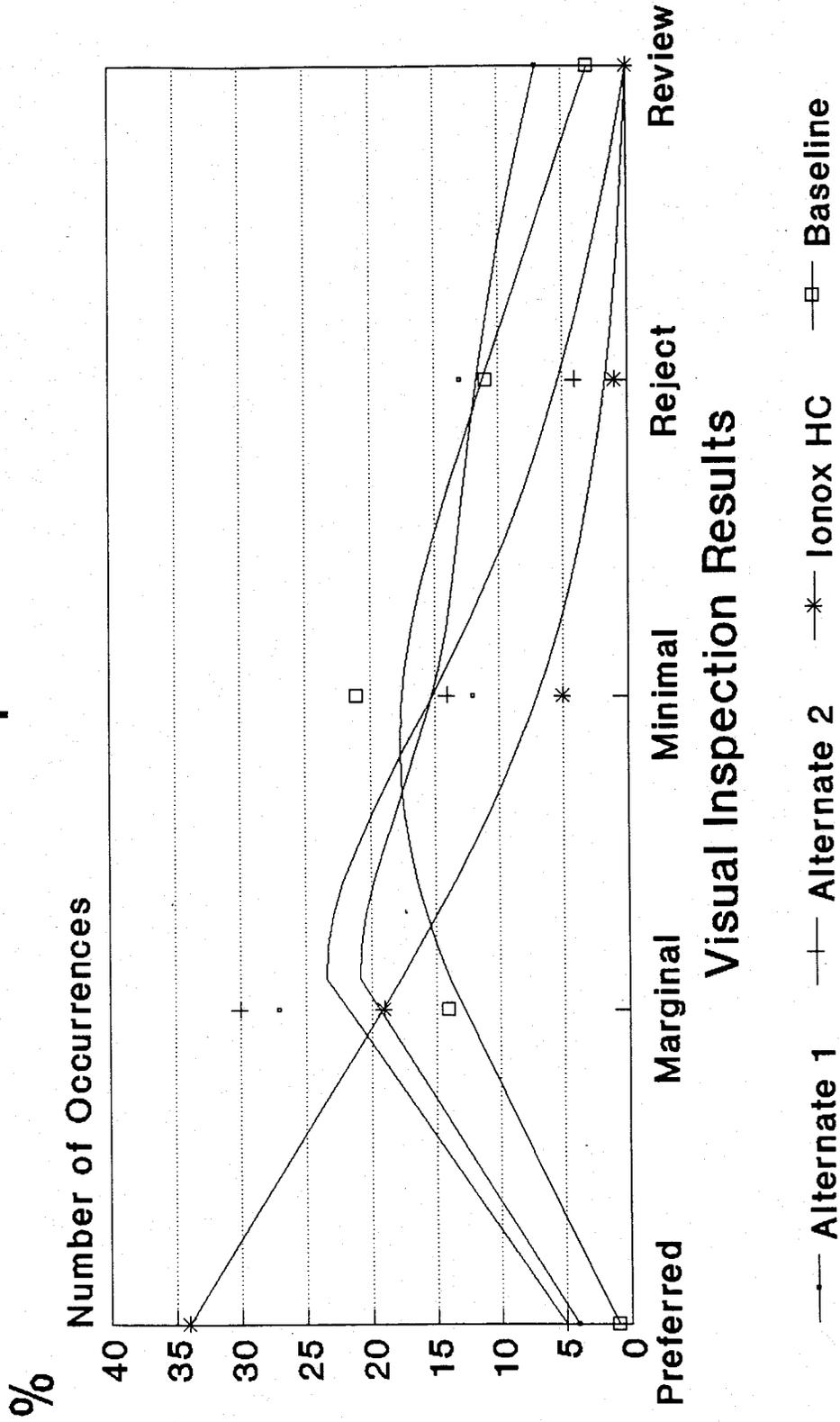
- \* Epoxies (15 families of conductive and non-conductive pastes and sheets),
- \* Gold wire bonds (.0007inch to .001inch diameter wedge, ball and ribbon thermosonic/thermocompression bonding),
- \* Gold plated machined sub-assembly housings,
- \* Fully constructed hybrid micro-electronic components (switches, amplifiers, mixers, phase modulators etc.),
- \* Conformal coating on circuit boards,
- \* Flux applications,
- \* Furnace, hot plate, and hand soldered gold plated substrates (using Sn-96, Sn-60, Sn-62, and 80/20 gold/tin solders) and
- \* Glass slides with various oils and other process contaminants.

In total, 2,268 test samples were processed for the electronic contingent through the chosen semi-aqueous cleaning systems. An additional 108 test pieces identified as the **control lot** were cleaned using baseline materials (Trichloroethane 1,1,1, Freon TMS, Freon TMC) in on-line vapor degreasers.

Immediately upon the completion of each cleaning cycle a visual inspection was accomplished. The inspection was a thorough 100% visual at a magnification of between 10X and 30X. All inspections were performed by the same inspector, wearing finger cots or gloves, to reduce the human factor. The results were recorded, as to degrees of cleaning against an internal inspection document used throughout normal product manufacturing and based on Military Standard's. Figures #1 and #2 show the composite results of the three cleaning chemistries evaluated plus the baseline results with two of the cleaning machines tested. In both figures the preferred category denotes materials cleaned above reproach, the marginal status equates to material acceptable with minor imperfections, the material identified as minimal, relates to borderline clean which may require minor touch-up cleaning prior to the product being moved along in production, the reject category demands product be sent back through the cleaning cycle as a rework (rework always includes additional cost implications) and finally the review category depicts material that showed no signs of cleaning and will be evaluated as to why this phenomenon occurred.

After each piece part inspection was completed, the inspector packaged and identified, by serial number, the component for further testing. Other tests performed included Ionic Resistivity, Wire bonding, Solderability, Surface Scanning Microanalysis, and Water Absorption (epoxy and boards).

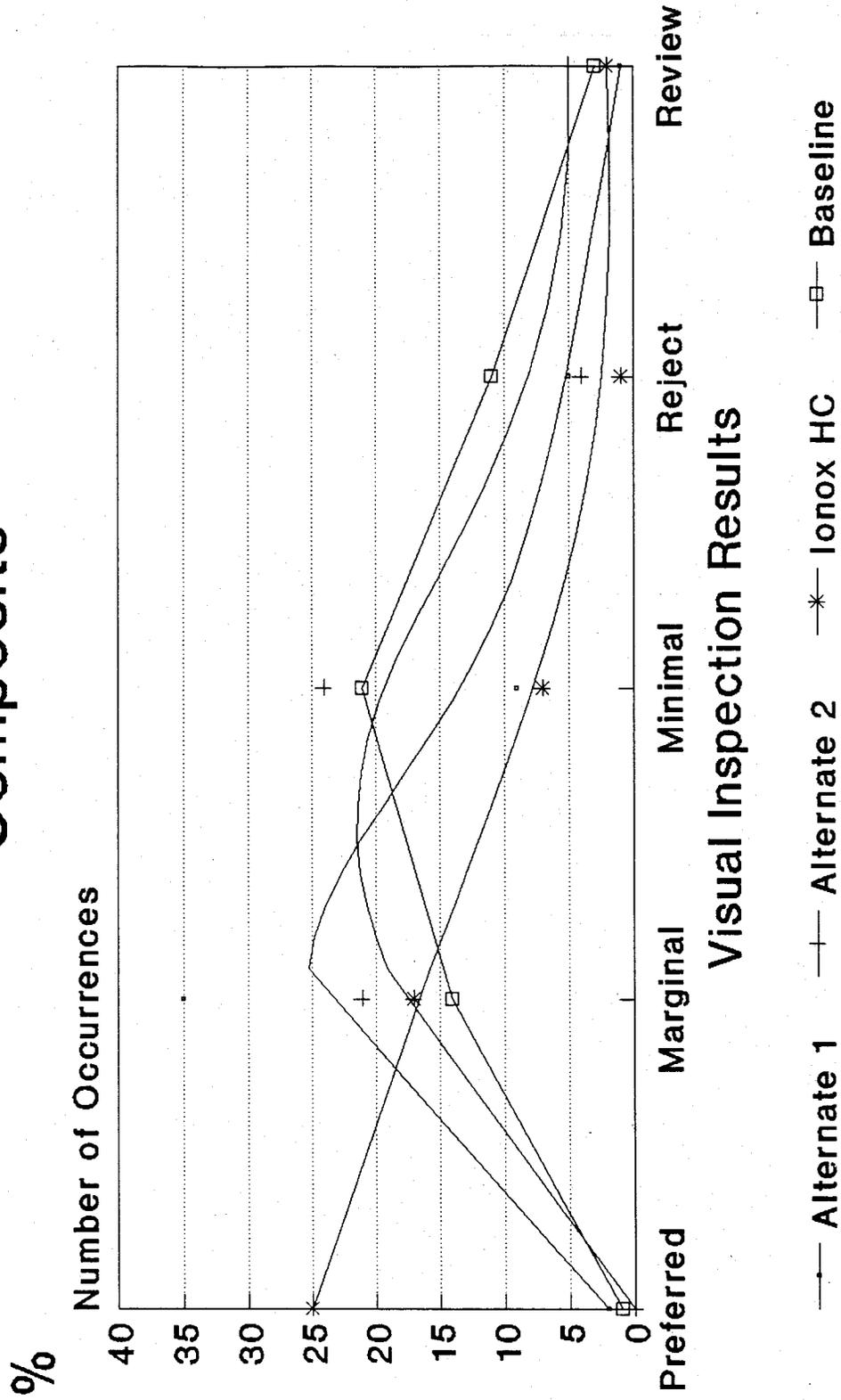
# ECD 6307/9300 Composite



Visual Inspection

**Figure #1**

# Accel Microcel "2" Composite



Visual Inspection

## Figure #2

The Ionic Resistivity test was conducted on the Omegameter 600R. In this test a mixture of isopropyl alcohol and water is flushed over the test unit. The difference in conductivity between the before and after is thought to be directly proportional to the contamination removed. The Ionic Contamination Composite depicting how the various chemistries perform throughout all the equipment tested is shown in Figure #3. To further show how each equipment option relates to the cleaning chemistry used see Figure #4. These results reinforce the data accumulated in other tests, that is, all three alternative chemicals proved to be superior to the baseline cleaning methodologies. The Ionox HC chemistry clearly appeared, as a result of this test, to be the optimum cleaner.

The methods used to evaluate wire bonding throughout this program was the pull test. While the pull test is valid for wedge bonding, it is necessary to use a shear test to evaluate ball bonding. The theory and applications of the above tests are fully documented in both Military Standards and ASTM methods. The program test results of ball bond shear testing are shown in figure #5. All recorded shear test data shows a distinct similarity however, the ECD and Accel equipment do exhibit a slight advantage.

The test results from the Scanning auger microanalysis (SAM) are shown in Figure #6. A correlation between visual inspection and SAM data was realized, test parts that were cleaned with alternative chemistries have a surface (approximately six thousand angstroms thick) cleanliness that exceeds all baseline cleaning results. The Ionox HC again is seen as the chemistry of choice.

One additional analysis was accomplished at the end of each cleaning system test, prior to changing chemicals. This test consisted of identifying the rinse water composition after cleaning our test samples. The analysis included:

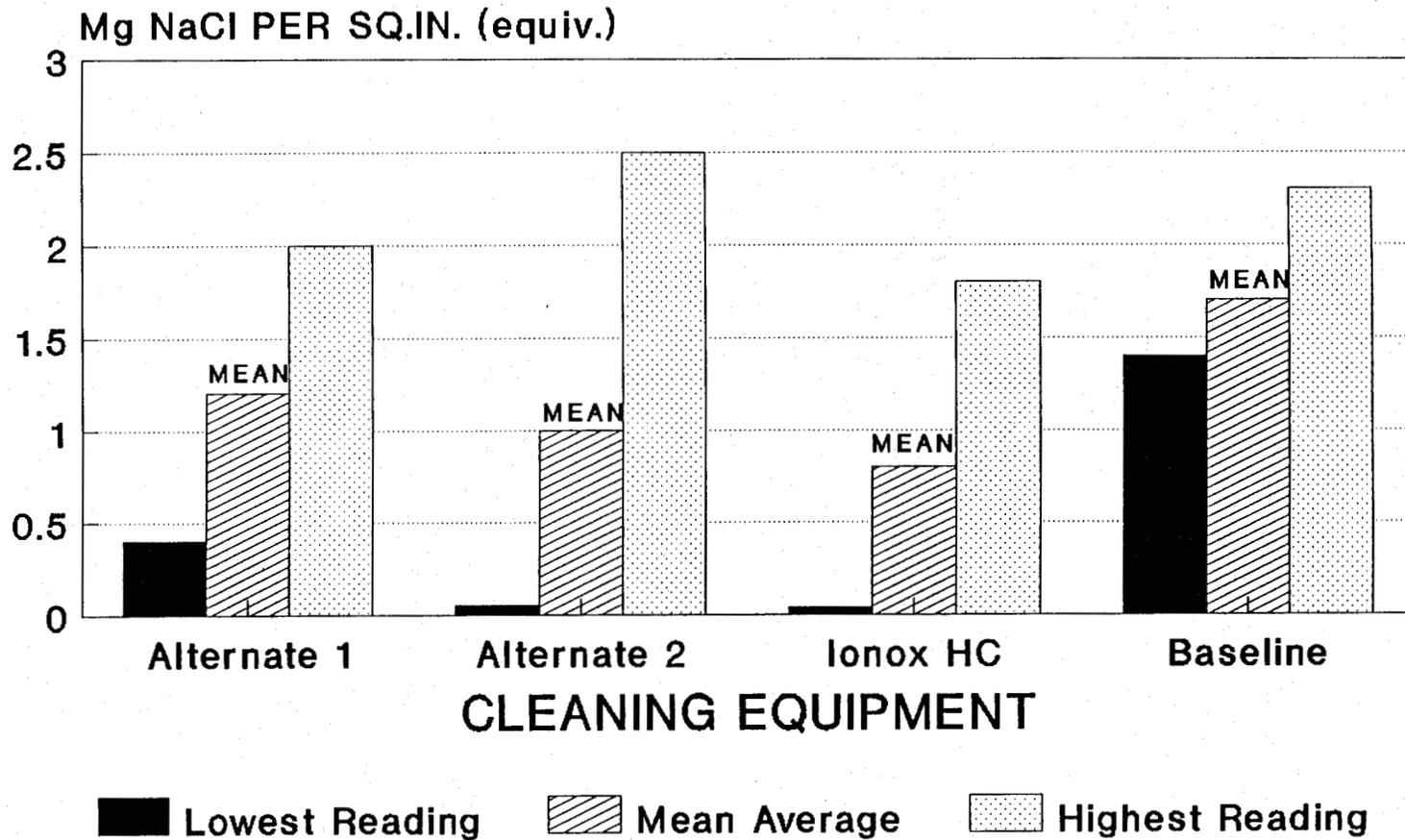
- \* Purgeable Organics VOA,
- \* A/BN Extractables in water,
- \* BOD,
- \* COD,
- \* Iron,
- \* Flash Point,
- \* Total Metal Content,
- \* Nickel,
- \* Oil and Grease by IR,
- \* Lead,
- \* Tin,
- \* Total Suspended Solids and
- \* Zinc

The individual rinse water results, from this analysis, will be used in determining the requirements for the cleaning system's closed loop design.

### Phase I Conclusions

Throughout the ODC Elimination Test Program, that was accomplished in the field, to determine an adequate alternative to the existing Chlorofluorocarbon cleaning systems, one chemistry continued to surpass all other cleaning chemistries evaluated. That chemistry was the Ionox HC, manufactured by Kyzen Corporation, Nashville, TN. However, as learned during this test program, one cleaning system will not clean all products. Where the Ionox HC is superior for cleaning flux and like residues, a second cleaning system is required to clean oils from machined parts.

# IONIC CONTAMINATION COMPOSITE



All Equipment Tested

Figure #3

# IONIC CONTAMINATION COMPARISON WITH IONOX HC

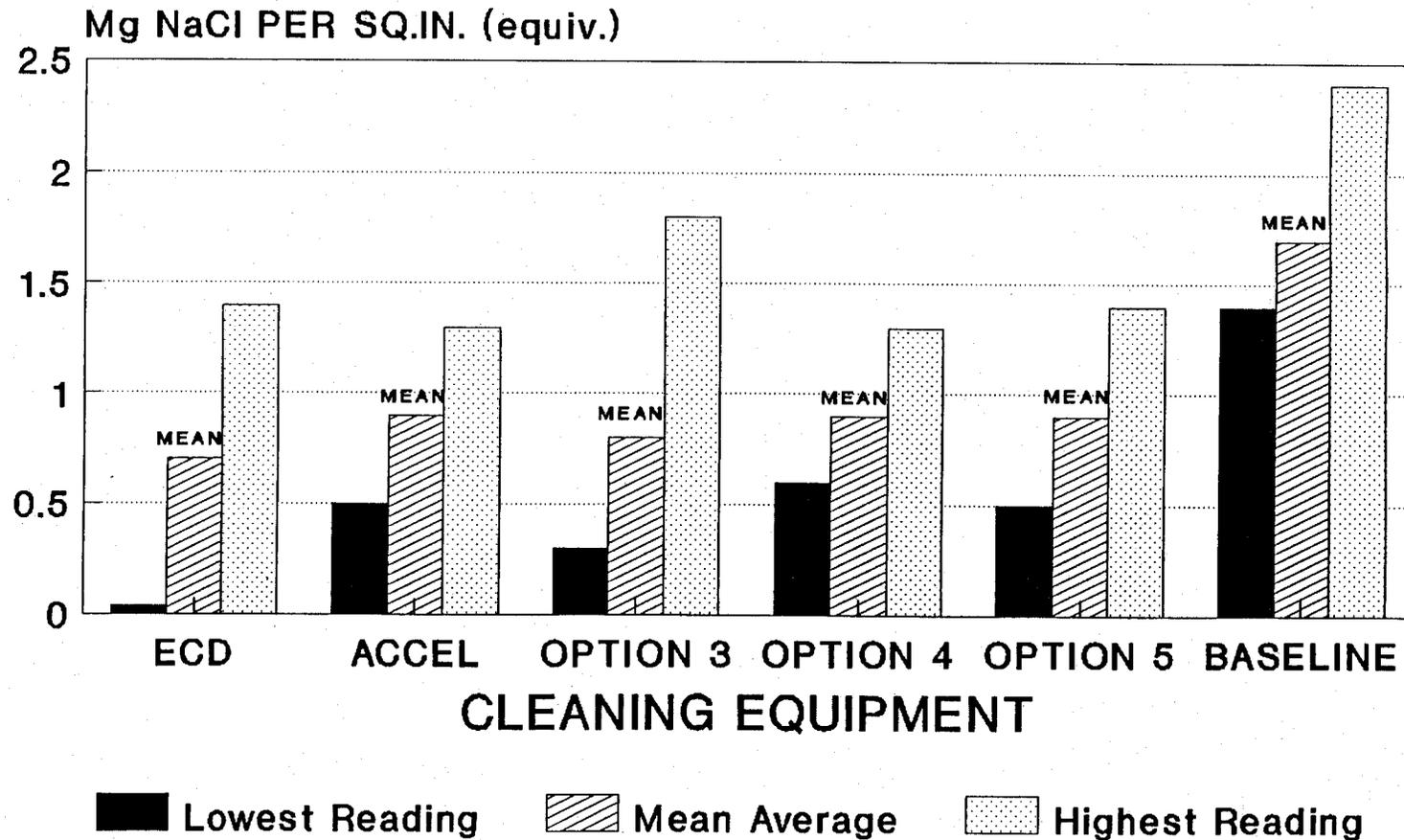


Figure #4

# WIRE BOND SHEAR TEST

## EQUIPMENT COMPARISON USING IONOX HC

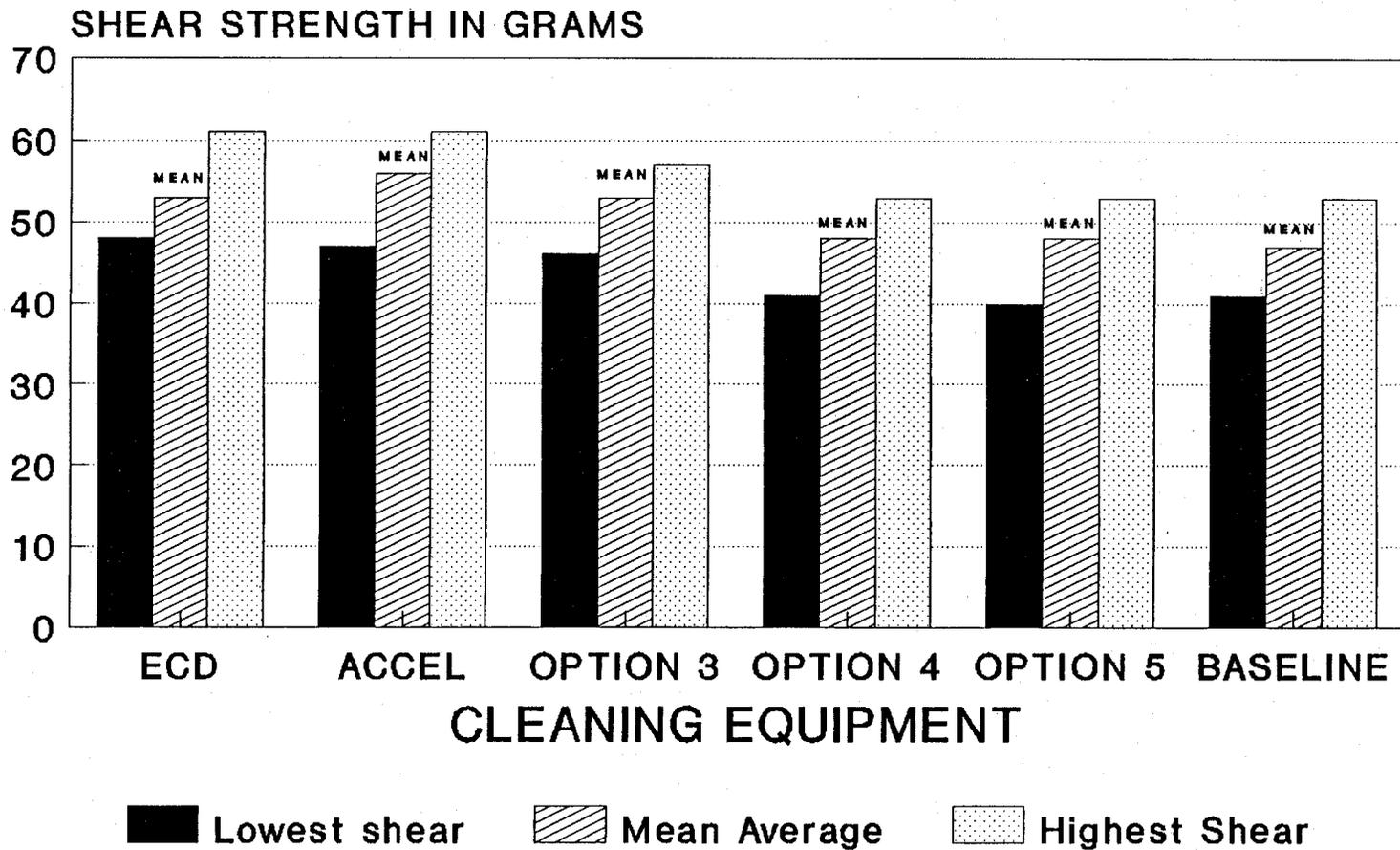
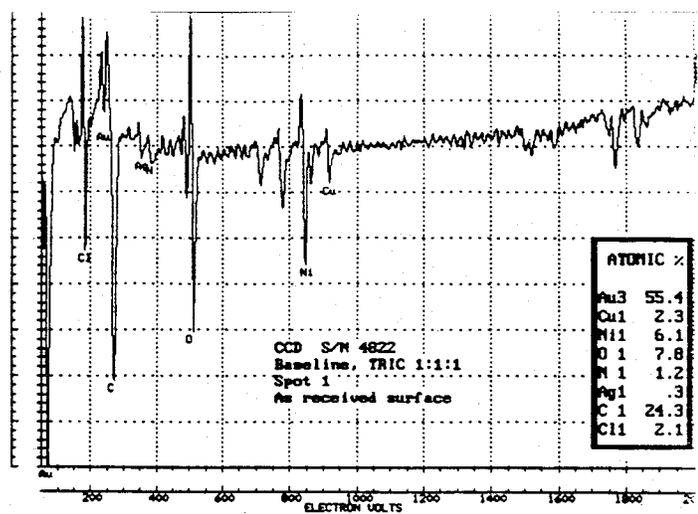
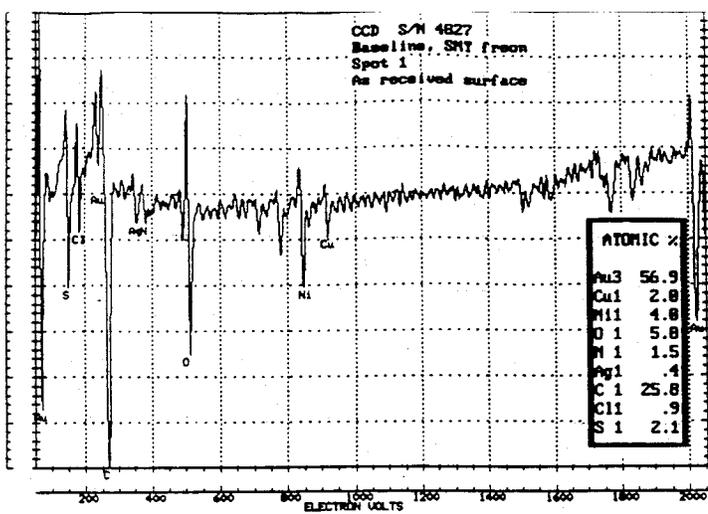


Figure #5



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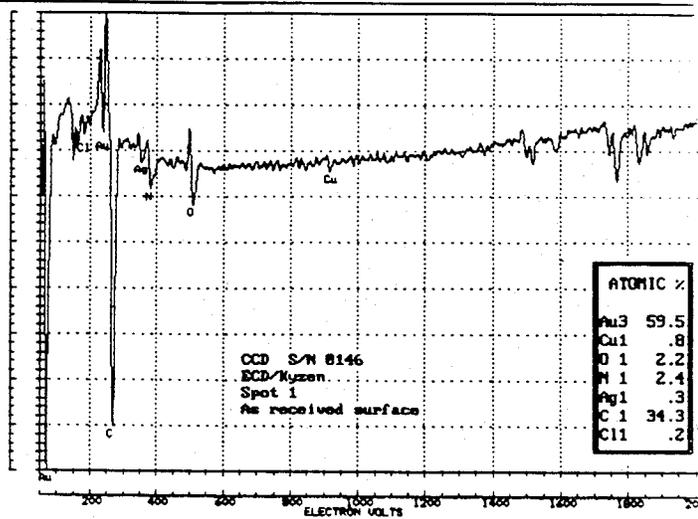
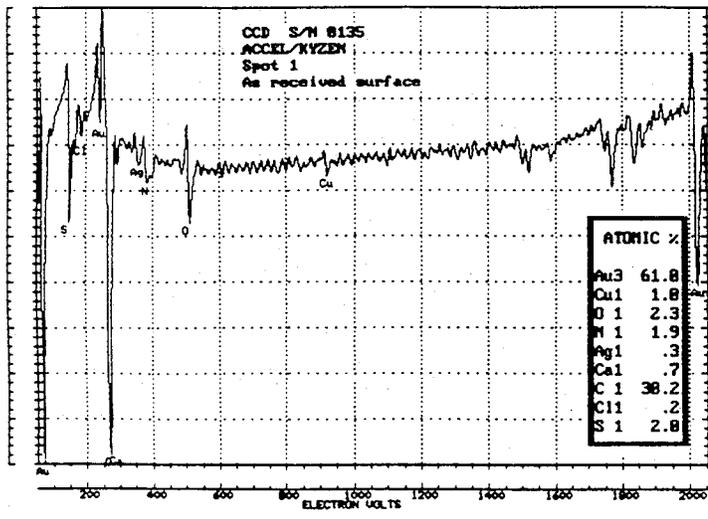
ACQUISITION PARAMETERS

SPECTRUM TYPE: AUGER SURVEY      COLLECTION TIME: 4.4  
 PRIMARY VOLTAGE: 5000      POINT NUMBER: 1  
 DERIVATIVE ORDER: 5      SMOOTH ORDER: 9  
 QUANTIFICATION TYPE: Peak-Peak      QUANTIFICATION UNITS: Atomic %

FILENAME: \AUGER\2416A4.OBJ      DATE: 09-24-1992      JOB #: 22

ACQUISITION PARAMETERS

SPECTRUM TYPE: AUGER SURVEY      COLLECTION TIME: 5.86  
 PRIMARY VOLTAGE: 5000      POINT NUMBER: 1  
 DERIVATIVE ORDER: 5      SMOOTH ORDER: 9  
 QUANTIFICATION TYPE: Peak-Peak      QUANTIFICATION UNITS: Atomic %



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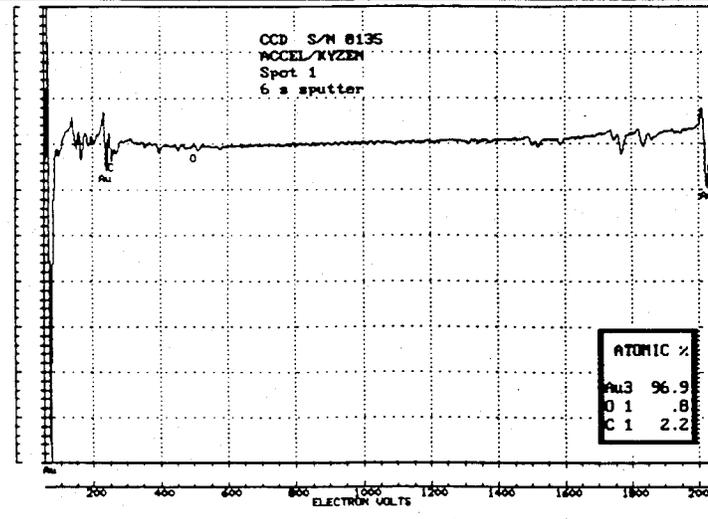
ACQUISITION PARAMETERS

SPECTRUM TYPE: AUGER SURVEY      COLLECTION TIME: 11.73  
 PRIMARY VOLTAGE: 5000      POINT NUMBER: 1  
 DERIVATIVE ORDER: 5      SMOOTH ORDER: 9  
 QUANTIFICATION TYPE: Peak-Peak      QUANTIFICATION UNITS: Atomic %

FILENAME: \AUGER\2416C9.OBJ      DATE: 09-24-1992      JOB #: 224

ACQUISITION PARAMETERS

SPECTRUM TYPE: AUGER SURVEY      COLLECTION TIME: 7.33  
 PRIMARY VOLTAGE: 5000      POINT NUMBER: 1  
 DERIVATIVE ORDER: 5      SMOOTH ORDER: 9  
 QUANTIFICATION TYPE: Peak-Peak      QUANTIFICATION UNITS: Atomic %



FILENAME: \AUGER\2416J2.OBJ      DATE: 09-25-1992      JOB #: 22416

ACQUISITION PARAMETERS

SPECTRUM TYPE: AUGER SURVEY      COLLECTION TIME: 11.73  
 PRIMARY VOLTAGE: 5000      POINT NUMBER: 1  
 DERIVATIVE ORDER: 5      SMOOTH ORDER: 9  
 QUANTIFICATION TYPE: Peak-Peak      QUANTIFICATION UNITS: Atomic %

The most effective semi-aqueous chemistry, for Freon replacement, was tested to be the DuPont Axarel 52. The Axarel, as determined from FTIR testing, did remove more oil from piece parts tested than existing baseline chemistries. This alone does not prove necessarily that axarel cleans better than Freon. However, it indicates that Axarel is capable of cleaning the parts tested to a level equal to or better than what is now acceptable. It does indicate that Axarel clearly outperformed all other chemistries tested.

The Kyzen, Ionox HC, alcohol/water based semi-aqueous cleaner has shown throughout Phase I testing, to have reduced ionic residue levels which were typically one-half to one-fourth of those corresponding levels achieved after cleaning with our baseline chemistries. The Ionox exhibits high chemical solvency and penetration properties inherent of semi-aqueous cleaning agents which enable cleaning to be performed at lower temperatures and low spray pressures.

The Kyzen, Ionox Hc is a water based material formulated with a proprietary alcohol. This chemistry is formulated with a series of polar and non-polar solvents, surfactants and activators to provide a cleaning solution which is non-combustible while maintaining effective cleaning characteristics. This chemistry did provide the proper balance of ingredients which was able to clean a broad range of fluxes through our vast product mix.

The DuPont Axarel 52 is a proprietary hydrocarbon cleaning agent for semi-aqueous cleaning processes. It is formulated to give the proper balance of selective solvency, high flash point (210 degrees F) and low toxicity. In addition, Axarel 52 is designed to promote rinsing of cleaned parts and allow separation of the cleaning agent and dissolved soils from the rinse water. Its solvent-soluble rust inhibitor protects most metal parts from flash rusting during cleaning and rinsing.

Cleaning/System Conclusions

The cleaning system is not one dimensional, it is not just the ability to find a cleaning alternative and a means to support its use. It is several key elements that alone are insignificant without all the other ingredients in place. A single cleaning system consists of the following:

- \* The cleaning solvent,
- \* The cleaning equipment,
- \* The piece part holding fixtures, (which will allow the work to be cleaned at the correct angle to the spray nozzles), and
- \* The water recycling system, to accomplish closed loop rinse water recycling

The test data has distinguished three suppliers of cleaning equipment. Each cleaning system will perform a specific cleaning task as shown in table I. These cleaning systems have been partially qualified, for their respective use, as a result of Phase I testing.

TABLE I

Equipment/Supplier	Chemistry Used	Product Cleaned
Accel Microcel 2 centrifugal cleaning system Plano, Texas	Ionox HC	Semiconductor wafer fab, fine wire applications
ECD Model 6307/9300 batch cleaner Milwaukie, Oregon	Ionox HC	General Assembly cleaning, solder & fluxes
J.M. Ney Co. Model PCS-35-US Ultrasonic batch cleaner Bloomfield, Connecticut	Axarel 52	Machined parts (oils and heavy contaminants)
Kyzen/Zenon Closed Loop rinse water recycling Ontario, Canada	Ionox HC/Axarel 52	Used on all equipment, for all products

### Recommendations

The recommendations, derived from Phase I testing, are necessary to implement a "Risk Free" process change and chemical substitution. A second Phase, for ODC elimination, will be required to optimize the cleaning system for all products manufactured.

A full scale system analysis using Kyzen Ionox HC and Axarel 52, with the equipment defined in Table I, must be accomplished to insure the integrity of the products cleaned. To insure product reliability and performance standards, that are consistent with existing cleaning practices, further process testing, by divisional product application, is being performed.

Single pieces of equipment and chemistries, that were determined as a result of Phase I testing, has been purchased for immediate installation at two Beta sites. Phase II testing will allow for detailed engineering evaluations and qualifications on all M/A-COM product presently being manufactured.

Design of Experiments (D.O.E.) will be the key tool in the design and optimization of the cleaning process, more specifically, analysis of the effects of control factors on process output. It will also be used to effectively verify the physics of the cleaning system. The use of DOE will allow for many control variables to be studied simultaneously, providing a more accurate understanding of the process when compared to the number of variables within the system. The intent of using DOE for cleaning process optimization will be to develop answers to the following:

- \* Which control factors have significant effect on the output response?
- \* What is the relationship between the control factors and the output response?
- \* Where control levels should be set to achieve optimum output response?

### Phase II

The Phase II evaluations are being used to optimize and eliminate any and all risks to the cleaning system process. This is accomplished by developing a plan whereby all system variables are identified and analyzed for their specific or critical roles in the cleaning process. Each cleaning system will require a distinct individualized program to address differences in equipment. Using sequential design of experiments techniques, a scientific approach is taken toward building, running, and analyzing all system tests. By using this approach a mathematical model can be built from an experiment that uses only a fraction of the cleaning process experimental combinations. The model will then define, with a high degree of confidence, what the results will be in areas of the experimental matrix that are not run.

The Phase II plan outlines the sequential DOE approach used for developing the parameters necessary to clean specific circuits and other piece parts. Phase II is using robust engineering practices to focus on a smaller number of critical factors (equipment cycle times, cleaning/rinse temperatures, chemistry specific gravity measurements and rinse water resistivity) in order to better understand them. After eliminating some of the less critical factors from the experiment, an evaluation test run will be used to focus on critical process factors. With fewer factors, a more accurate model can be built. Upon system qualification completion, a verification run will be accomplished using the defined optimized settings to achieve desired results.

The final steps of Phase II testing will be to determine process capability. This will consist of running several lots of material through each of the M/A-COM divisional manufacturing facilities, with the same parameters that were established, with them, during their portion of the Beta site verification runs. Run data will be compiled and monitored to determine the capability of the process.

The three pieces of equipment that will be used in Phase II testing have been delivered. The electronic products equipment, Accel and ECD has been delivered to Lowell Ma., while the Ney machine parts processing equipment has been delivered to the Watertown Ma. Beta site. The Beta Sites have been facilitated with exhaust, electrical and plumbing. As of May 3, 1993 the ECD equipment has been tied into the Kyzen/Zenon KZ100 closed Loop DI water recycling system. The other two evaluation machines will be installed by May 7, 1993.

An Installation consists of the equipment manufacturers completing final assembly and plumbing between their respective equipment and the closed loop Kyzen/Zenon system.

The development of the Phase II process optimization plan and procedure, by division, has been ongoing for the past four weeks. The first division to undergo product/process qualification will be an assembly group from the Microelectronics division based in Lowell, Ma. The balance of the schedule which will determine the order of divisional testing is being developed for release by 5/10/93.

The development and testing of the Kyzen/Zenon KZ100 closed loop DI water purification system has been one of the highlights of defining a total cleaning system which will enhance pollution prevention. This portion of the cleaning system will allow for DI water on demand while eliminating the waste rinse water from being sent to drain. The system being developed will service multiple cleaners within a single division. We are working with Serial Number #0001.

### **Phase II Goals and Objectives**

In order to implement a "Risk-Free" cleaning process to each division of M/A-COM, the ODC Elimination Task team will use statistical methods and analysis techniques to establish optimum process parameters and overall capabilities.

The statistical methods that will be used include:

- Robust Design to include the development of the baseline statistical plan,
- Data collection,
- Experiment Analysis using statistical methods,
- Variability Reduction and process optimization,
- Process capability and statistical analysis,
- Qualification, confirmation and
- Final acceptance for implementation

The results of this analysis will be used by each division to establish process settings, introduce on-line statistical process control, document cleaning procedures, define training, and develop site specific tooling.

### Robust Design

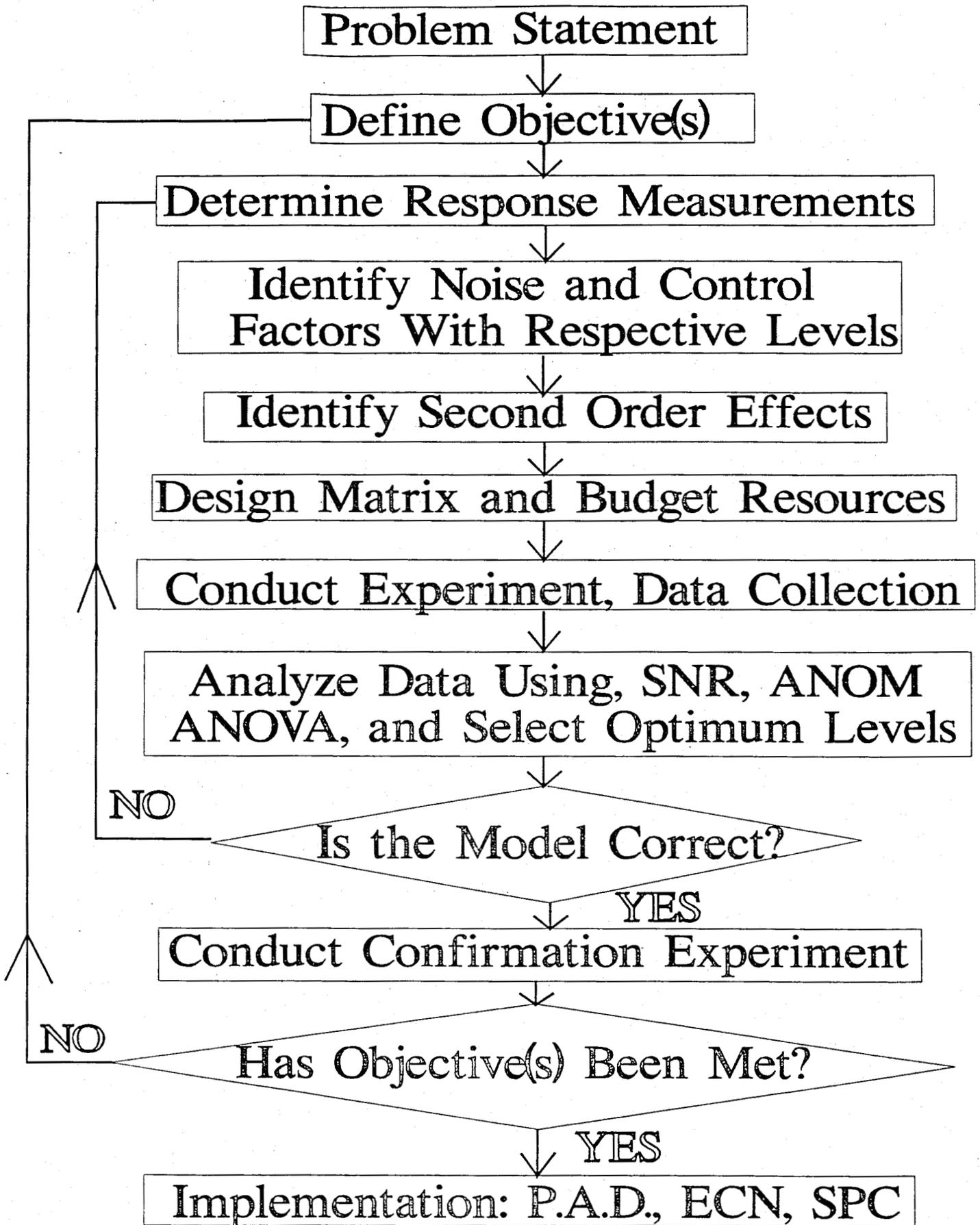
The concepts of robust design and variability reduction are used here to reduce or eliminate the sources of unnecessary start-up costs that are present whenever a new process or new design is introduced to a M/A-COM production line. Rework and manufacturing down time occur because production operations produce parts that do not meet specific requirements, either because of tight specifications or production process variability. Using robust designs, the need to have narrow specification limits is eliminated and the product will be cleaned satisfactorily within a wide production process variability. The indicator of this optimum ratio of design specification to production variability is the process capability index which will be the final out-put of this statistical analysis.

Robust design is a variant on the design of experiments (DOE) methods used to optimize products and processes for performance enhancements. It is a mix of several tools that have been developed for critical process analysis, its elements include; brainstorming, design of experiments, orthogonal arrays, analysis of variance, loss function and signal-to-noise ratio (this is an indicator of variability and has no relationship to a similar term used in electrical engineering).

In conducting this robust design experiment we involved many of the tools used in Total Quality Management (TQM). The success of the cleaning system development is dependent on focusing, optimizing and measuring the proper characteristics, and following the guidelines of the methodology. see figure (7). The steps used in performing robust engineering on alternative cleaning systems chosen in phase I herein, are as follows:

1. **Problem Definition:** The first task in performing this robust design experiment was to outline the goals of the experiment and to define the characteristics of the cleaning systems to be optimized. Creation of the boundaries of the process to be optimized is important. The experiment should not be constrained to a small part of the cleaning equipment or solvent or fixturing. On the other hand, the experiment should not be all encompassing in attempting to optimize every variable. Some early decisions were made to only define critical process variances.
2. **Team Creation:** A project task team was selected to conduct experimentation and analysis of division product vs cleaning systems under test. The task teams that were chosen came from a nucleus of original ODC Elimination people as well as manufacturing operations/engineers from each of the specific product lines at each M/A-COM division. It was not necessary for all of them to have in-depth technical understanding of the cleaning technology, but the team members must have experience in the cleaning process and its required results. Knowledge in statistical methods, and in particular robust design techniques was supplied by Bob Spinazzola, a specialist in this field and a M/A-COM manufacturing engineer.
3. **Parameter Selection:** A Cause and Effect Diagram was constructed by the project task teams which consisted of Manufacturing, Design, Quality Assurance Engineers, Production Supervisors, Operators, and Inspectors. See figure (8) and (9). The diagram is important in describing and defining the problem graphically. As shown, it categorizes the problem in four main areas: Machine, Material, Process, and design. This approach served as a tool to obtain everyone's input as to what variables are associated with cleaning as they relate to specific operations. The main purpose of these diagrams was to provide a tool which was used to guide the discussions within the task team as to what factors and issues to include in selecting critical parameters.

FIGURE 7



Flow Chart For Design Of Experiments

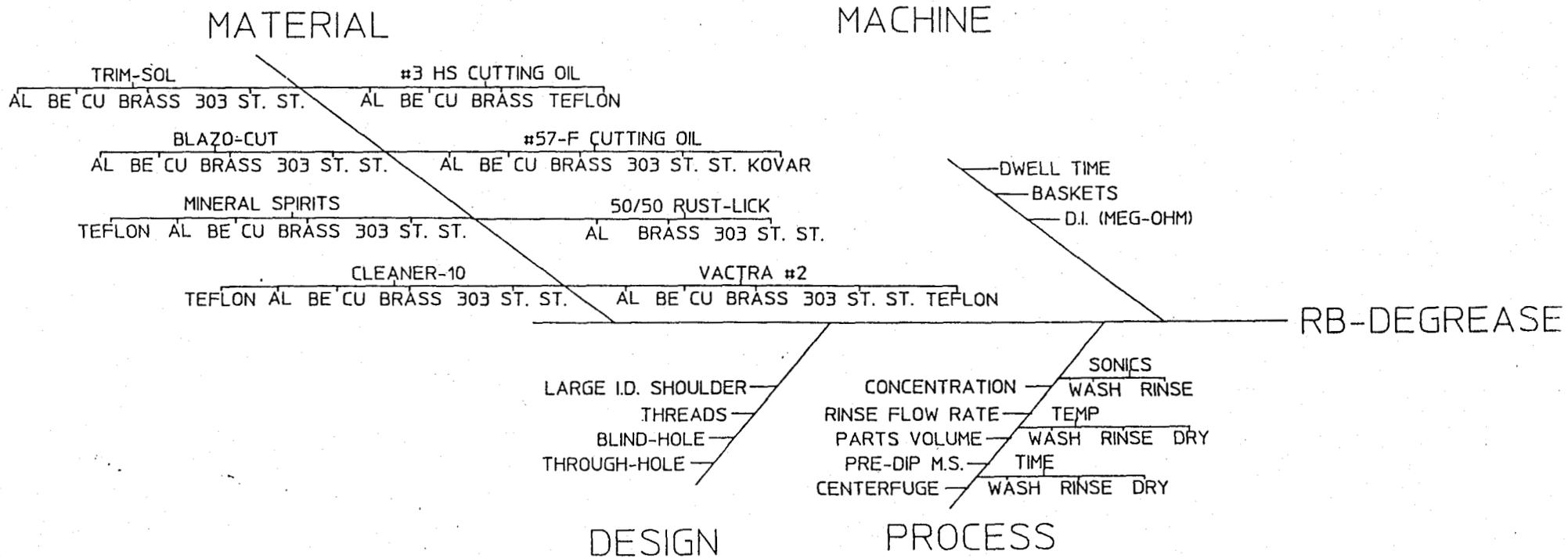


FIGURE 8

Mark Darga

MATERIAL

MACHINE

TRIM-SOL  
AL BE CU BRASS 303 ST. ST. #3 HS CUTTING OIL

BLAZO-CUT  
AL BE CU BRASS 303 ST. ST. #57-F CUTTING OIL

MINERAL SPIRITS  
TEFLON AL BE CU BRASS 303 ST. ST. 50/50 RUST-LICK  
AL BRASS 303 ST. ST.

CLEANER-10  
TEFLON AL BE CU BRASS 303 ST. ST. VACTRA #2

DWELL TIME  
BASKETS  
D.I. (MEG-OHM)

AXAREL-52

Cleaning Process

SONICS  
WASH RINSE FREQ.  
RINSE FLOW RATE TEMP  
PARTS VOLUME WASH RINSE DRY  
PRE-DIP M.S. TIME  
CENTERFUGE WASH RINSE DRY

LARGE I.D. SHOULDER  
THREADS  
BLIND-HOLE  
THROUGH-HOLE

DESIGN

PROCESS

Clean Criteria

- Visual
- Black Light
- FTIR
- Cycle Time

- Saturation pts of each oil
- Measurement technique of Saturation
- Schematic of equipment with all controls identified

FIGURE 9

4. **Design of Experiments (D.O.E.)** is a key tool in the design and optimization of processes or, more specifically, the study of the effects of control factors on process output. It was also used effectively to verify the physics of the system, and to explore new system operating conditions. One key advantage of DOE is that many control factors may be studied simultaneously, providing a more accurate analysis of the process as compared to the traditional studies of varying one control factor at a time. Specifically, results obtained from this experimental design was used by the experimenter to tell when there were any significant advantages in changing the control factor and/or the control factor level from one to another. With the use of Analysis of Variance (ANOVA), the control factors that make a significant contribution to the output response were determined and studied carefully. As we performed this robust design, DOE answered these questions about the cleaning systems under test:

- a) Which control factors have significant effect on the output response?
- b) What is the relationship between the control factors and the output response?
- c) Where control levels should be set to achieve optimum output response?

In traditional process studies, the output response is the yield, the number of the percent defective, strength, etc. and the objective of experimental design is to maximize or minimize the output response. This leads to the traditional assumption that accepting an output response within the quality specification is equally good, and an output response falling outside the quality specification is bad.

Alternatively, the intent of this cleaning system analysis was to utilize Taguchi's Loss Function concept. Taguchi defines the loss function as; "the financial loss imparted by the process due to a deviation from its desired target value". The loss function is a quadratic expression estimating the cost of deviating from target caused by variability of the process characteristic in terms of the monetary loss due to product non-compliance in the eyes of the customer.

The principle function of the DOE methodology used in this study was to improve efficiency obtained from the use of multifactor process designs, this afforded us the opportunity to study all possible interactions between the process variables (factors). Two factors are interacted if the behavior of one factor depends on the particular level of the other factor. Thus it was not possible to study process interactions with one factor at a time experimentation, and the undefined presence of an interaction could lead us to erroneous conclusions. In order to fully utilize this factorial analysis we estimated all interactions, if none were present, we allowed the effect of every factor to be evaluated as if the entire experiment were devoted entirely to that factor.

5. **Analysis of Data:** Once the experiment was performed, the data was analyzed to determine the optimal settings of the parameters and there levels for adjusting the mean of the design to target and for minimizing the process variability. The significance test is only a statistical determination of the probability that the effect of the particular parameter on the process characteristic measurement is not due to chance alone. Although there are techniques for improving the significance levels of parameters, graphical presentation of the data is sufficient to determine the best parameter setting in order to adjust the process mean to target and reduce process variability.
6. **Prediction and Confirmation of experiments:** One of the unique elements of robust designs is to be able to predict the characteristic value to be achieved, especially in performing experiments for increasing the quality of designs. In addition, a range of the predicted characteristic values can be calculated, which increases the confidence of achieving the target.

After performing the experiments and conducting the data analysis, a choice of parameter levels was made to compromise between setting the design characteristic mean to the target value and reducing variability.

Once all the choices and predictions of the robust design experiment was made, a confirmation run was accomplished before final adoption of the process decision which verified the theoretical analysis. This confirmation tested out the entire robust design process before full process release and implementation was able to take place.

7. **Baseline Statistical plan** was developed using the principles of robust design as shown above. The software package chosen for this program study is identified as "Catalyst/DOE", it has the capability of creating the following statistical designs:

- Full Factorial
- Fractional Factorial
- Taguchi's Orthogonal Arrays
- Central Composite
- Optimal Designs

The optimal Designs was preferred. The reason being, that an optimal design can be calculated for any number of runs and any combination of levels of inputs. It was preferable to match the test plan to the specific requirements of the products and cleaning systems being tested rather than to distort system requirements to fit any of the standard statistical designs.

The test plan depicted here (see figure 10 A-D), was developed to satisfy the parameters of the ECD cleaning system with respect to the large scale integration products manufactured by the MFA operation within the MED division. Each other test plan developed will be for a specific cleaning system and a divisional product being manufactured.

### Phase II Data Collection

Once the test plan was developed the most time consuming portion of the program was to obtain the sample product. The samples that were used for qualification testing, of the cleaning systems, were actual good product taken from the manufacturing lines. This material was cleaned as directed, tested and returned to the production lines to continue processing. Where customer specifications dictate that there be prior approval of any process changes, the product tested was sent back through the original cleaning systems prior to proceeding with production. The use of actual product rather than simulated product, as was developed for phase I testing, creates several logistic problems however, the data generated will best define the process in terms of actual production as viewed by each specific product line.

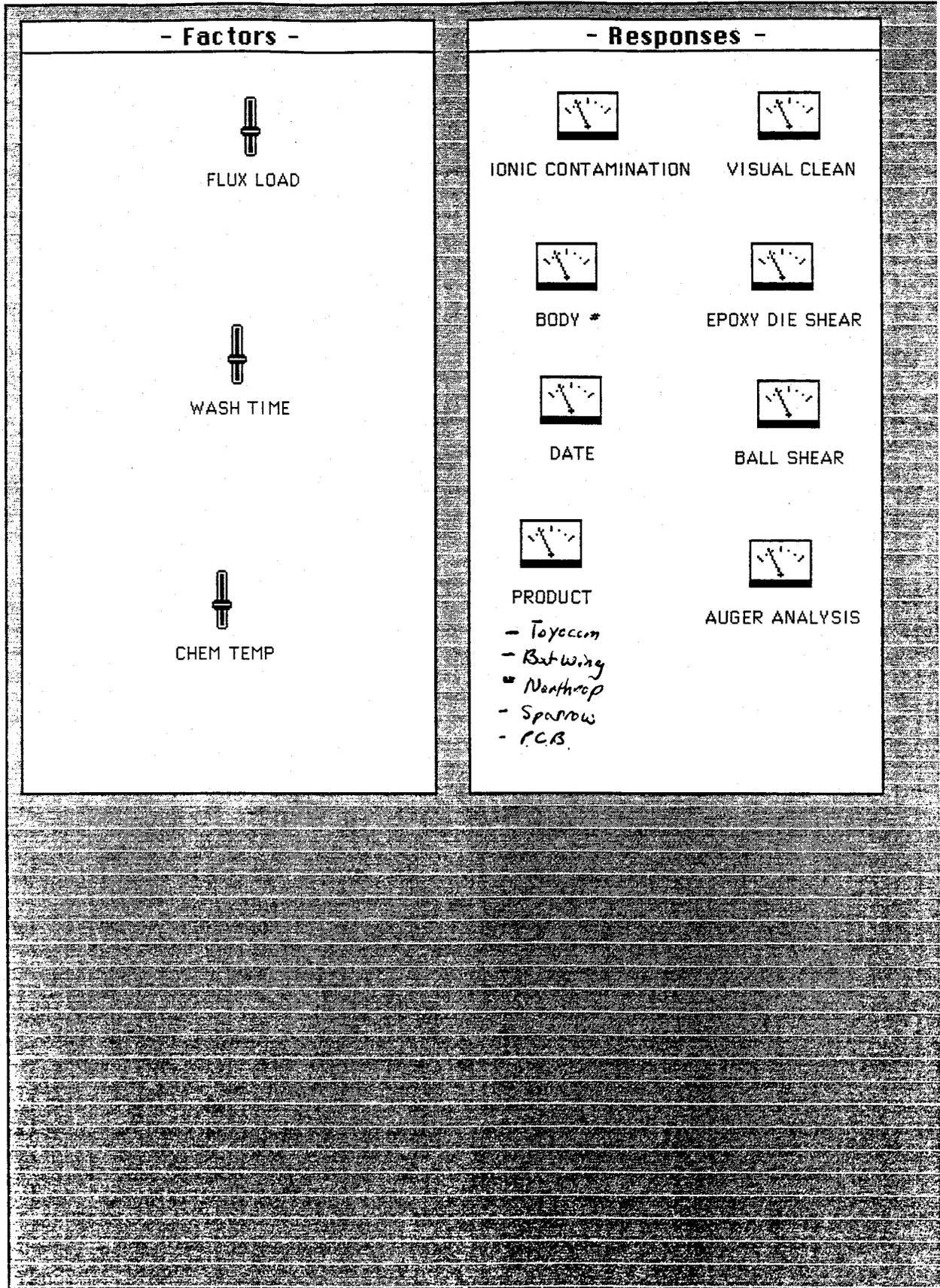


FIGURE 10-A

	Name	Symbol	Low	High	Units	Increment	Current
			Value	Value			Setting
1	WASH TIME	W.TIME	3	11	MINUTES	1	5
2	FLUX LOAD	FLUX L.	0	14	FLUX %	0.1	0
3	CHEM TEMP	C.TEMP	78	135	DEG.F	1	78

FIGURE 10-B

	Name	Symbol	Goal	Lower Limit	Target Value	Upper Limit	Units	Average	Spread	Increment
1	VISUAL CLEAN	CLEAN	Smaller	-	0	1.5	1 GOOD-10 BAD	0	0	0.1
2	BALL SHEAR	BALL	Larger	8.0	∞	-	GRAMS	0	0	0.1
3	EPOXY DIE SHEAR	EPOXY	Larger	5.0	∞	-	GRAMS	0	0	0.1
4	AUGER ANALYSIS	AUGER	Smaller	-	0	10.0	%CONTAMINANTS	0	0	0.1
5	IONIC CONTAMINATION	IONIC	Smaller	-	0	10.00	MICRO-G/IN**2	0	0	0.01
6	BODY #	R 6	Target	0	50	100		0	0	1
7	DATE	R 7	Target	0	50	100		0	0	1
8	PRODUCT	P	Target	0	50	100		0	0	1

FIGURE 10-C

Run No.	WASH TIME MINUTES	FLUX LOAD FLUX %	CHEM TEMP DEG.F	VISUAL CLEAN 1 GOOD-10 BAD	BALL SHEAR GRAMS	EPOXY DIE SHEAR GRAMS	AUGER ANALYSIS %CONTAMINANTS	IONIC CONTAMINATION MICRO-G/IN**2	BODY #	DATE	PRODUCT
1	3	0.0	78								
2	11	0.0	78								
3	3	0.0	78								
4	11	0.0	78								
5	7	0.0	106								
6	11	0.0	135								
7	3	0.0	135								
8	7	0.0	135								
9	7	7.0	78								
10	3	7.0	106								
11	11	7.0	135								
12	3	7.0	135								
13	11	14.0	78								
14	3	14.0	78								
15	7	14.0	78								
16	11	14.0	106								
17	3	14.0	106								
18	3	14.0	135								
19	11	14.0	135								
20	7	14.0	135								

FIGURE 10-D

The Catalyst software that was used exclusively did select the number of test runs automatically, it looked for a number of runs that we could afford, and then it allowed us to minimize the number of runs that would give us the optimum number of data points for analysis. This process study (design) developed a mathematically optimized strategy for getting the information we need from an economical set of test runs. Defining the best process design was dependent on the model developed and how the responses interacted with the factors. The software represented this model as a mathematical equation for each response. When two-factor interactions or quadratic effects were significant to the process, we specified these in the program. The software added the necessary terms to its internal model, then generated a design containing the runs we needed to fit all the model terms.

Catalyst automatically determined the number of runs we needed to generate a suitable process design. However, the program did have an option that we chose more than once, and that was to specify the number of runs we could afford. This happens by directing the system to minimize the number of runs required.

After Catalyst created the process design, it generated instructions that identified what factor settings to use for each test run and what responses to measure. To minimize the chance of error, we printed a detailed check list, with each run on a separate page. All data responses were printed on these forms and later entered into the program.

After the test runs were complete, the software automatically fits the model to the data. It displays the results as a matrix of plots with a row for each response and a column for each factor. This display was a powerful indicator of process behavior. It did show us, at a glance, which factors affect key responses most strongly (these are depicted graphically as plots with steep slopes), and which don't affect the process much (these are depicted as flat lines). The plots developed also depicted the directions of the effects (displayed by the sign of the slope), which factors interact (these plots have two lines each), and which ones show quadratic effects (by curvature in the lines).

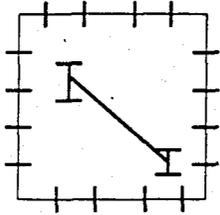
#### Experiment Analysis using Statistical Methods

The purpose of analysis is to find a mathematical model that adequately describes the relationship between the cleaning process inputs and outputs. In keeping with the idea of robust process engineering, the mathematical model included information about how the cleaning process inputs influenced both the location and spread of the outputs.

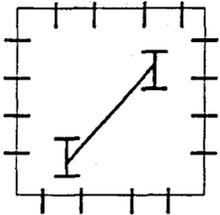
The cleaning process parameter selections as later defined as results, were established at this analytical step. The software presented the data both in numerical and graphical form. Graphically the software displays the results as a matrix of plots (see figure 11). Each plot shows the relationship between a factor and a response. The response forms the Y-axis of each plot, and the factor forms the X-axis of each plot. Numbers along the X-axis of the plots are the limits entered for the factors. The numbers along the Y-axis show the scale of the predicted responses. The bars at the ends of each line show the typical variations in the observed responses.

The Catalyst software automatically calculates a model terms table. This table provides numerical data about the plots. The model terms table displays two numeric values for each factor as it related to each response. The top number or the signal value in the model terms table corresponds to that factor's contribution to the slope of the line in the plot on that response. For linear terms, the signal value is the effect of a change in the factor from its mid to high setting to the response while other factors remain fixed at their mid settings. The bottom number is the noise (variability) of the number above it.

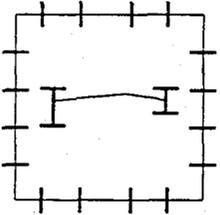
**Plot interpretation** The Model Graph shows how factors affect responses. If the line in the plot is steep, that factor has a strong effect on the response.



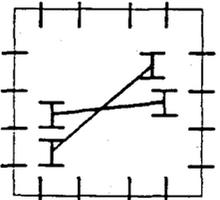
If the line slopes downward, the factor has a negative effect on the response. Changing the factor from its low setting to its high setting decreases the response value.



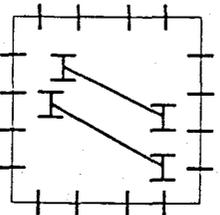
If the line slopes upward, the factor has a positive effect on the response. Changing the factor from its low setting to its high setting increases the response value.



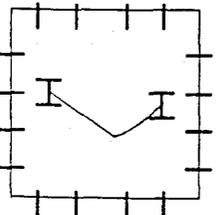
If the line in the plot is flat or nearly flat, that factor has little effect on the response. That is, the response is insensitive to changes in that factor's setting.



The plot displays two lines when that factor interacts with another factor (or factors). One line shows the most positive effect that factor can have on the response. The other line shows the most negative effect that factor can have on the response.



The interaction is strong when the slope of the two lines differs greatly. When the lines are nearly parallel, the interaction is weak.



Plots with a curved line show a quadratic (nonlinear) effects. The sharper the curve in the line, the greater the quadratic effect.

FIGURE 11

The methods used in analyzing the data developed via our test plan were:

- Analysis of mean (ANOM),
- Analysis of second order effects (two factor interactions and/or quadratic effects),
- Analysis of variance (ANOVA),
- Signal-to-noise ratios (SNR),and
- RMS error value optimized

The **Analysis of Mean (ANOM)** determined the effect, which depicted the average performance obtained at each cleaning equipment control factor level.

The **Analysis of second order effects (two factor interactive effect)** defined the interaction between two or more cleaning factors by producing a performance value that is a delta (higher or lower) from the individual contribution factor. In more serious interactions best engineering practices were applied in order to obtain useful results and a successful evaluation.

The **Analysis of second order effects (Quadratic effects)** defined the terms of the signal value (top number in the model terms table) as a change in slope as the factor setting changes from its mid setting to its high setting.

The **Analysis of Variance (ANOVA)** was used to determine the amount of variation from the mean which each factor contributes.

The **Signal-to-Noise Ratios (SNR)** was used as the objective function for evaluating the robustness of the cleaning process parameters. We used the SNR's to compare the influence that each critical control factor level had on the mean performance value to that of the actual variation incurred.

The **RMS Error Value Optimized** was the method we used to move the cleaning responses, as a group, close to their respective goals. The program suggested factor settings for the cleaning process to meet these goals. Catalyst displayed the results of the optimization calculation as a matrix of plots similar to the model graphs that were previously developed. This optimized program allowed us to vary any factor setting from its low to high value, and to see the change in the response values. This graphical data was also used to experiment with making changes to the cleaning process to determine the effect of each component on the responses.

#### Variability Reduction and Process Optimization

The Catalyst software allowed us to see how the cleaning process would respond to new operating conditions with the depiction of a graphical simulation tool. Like the Analysis graph the interpretation graph contained a matrix of plots. This graph showed specific settings for each cleaning factor and predicted values for each response. The plot developed for each factor response pair showed us the result by changing one factor while holding all others at their indicated settings.

Initially, the interpretation graph displayed factor settings selected by the system to optimize process behavior. To establish these settings the system used an iterative numerical optimization algorithm to maximize the total process utility function. This technology matched targets and optimum factor settings that minimize response variability.

In developing total flexibility for evaluating future cleaning process strategies, we now can vary with the data inputs from the system, for any (or all) factor settings to see what happens to the predicted responses. The system re-plots all responses for each new setting, so we can see at a glance how the modified cleaning process will perform. This interactive graphical process simulation capability allowed us to explore various interactions to obtain the best results.

This method of variability reduction was used to increase the cleaning process capability index and reduce the cost of quality. To obtain variability reduction and keep the cleaning process's mean equal to the target we installed control charts to monitor process output. The two types of variable control charts that were used were to cover the average of the process characteristic in the X-bar charts and the variability of the cleaning process characteristic in the R chart.

Monitoring the cleaning process with newly established "optimized" settings was considered to be in control when the process generated a small number of defects (less than 100 PPM). The total number of rejects generated were small and therefore each defect was analyzed to determine its cause. In many cases the tools of continuous process improvements (CIP) were used (brainstorming, cause and effect diagrams, pareto analysis, an data sampling) to determine the most probable cause for each defect. If a deviation of the process was found, the process control factors would be enhanced to allow for settings that were not as variability sensitive.

#### Process Capability and Statistical Analysis

We have determined that there are two ways to increase the quality level of the cleaning process. One, increase the specification limits of the process and allow manufacturing variability to remain the same yet produce fewer defects. Two, reduce manufacturing variability. The ratio of the interaction of these two sources of rejects is called the process capability index, Cp:

$$C_p = \frac{\text{specification width}}{\text{process capability}}$$

$$C_p = \frac{UES - LES}{6 \text{ sigma (total range from -3 sigma to +3 sigma)}}$$

where

UES = upper process specification  
LES = lower process specification  
sigma = manufacturing process standard deviation

The process capability index focused on communication between the robust design, process development, and the product being cleaned. By managing the relationship between design of experiments to manufacturing tolerances, it established a goal for achieving a particular index level that will satisfy both engineering and manufacturing throughput.

The cleaning process variability was approximated by a normal probability distribution, with a mean and a standard deviation. The process capability was again defined as the full range of our normal cleaning process variation measured for a chosen characteristic. We assumed normal distribution, 99.74% of the cleaning process output which is between -3 sigma and +3 sigma.

The Cp value was used to predict the reject rate of new products by using normal probability distribution curves. A high Cp index indicates that the process is capable of replicating faithfully the process characteristics, and therefore will produce cleaning results of the highest quality.

The Cp index depicts a balance of quality responsibility between the robust design and manufacturability. The quality level is set by the ratio of the effects of both. The manufacturing reject rate for the cleaning process was determined by the Cp index. We stated the design tolerance specifications in terms of process capability, it was possible to determine manufacturing reject rates from the normal probability distribution curves.

#### Qualification and Confirmation

Each piece part tested will go through the following tests:

- 100% visual inspection
- Ionograph
- Scanning Auger Microanalysis (SAM)
- Wire bondability
- Solderability

These tests were run on all piece parts tested during Phase I of the overall program. The results will be monitored on their own merits and carefully analyzed against the data developed during Phase I.

The most accurate way of determining if the proposed settings are the best settings is to analyze confirmation runs in comparison to the model's prediction. We used the Interpret Graph calculated by the Catalyst software to set the factor settings to the same levels used during the experiment. Then compare the model predictions to the data collected for that particular run. The raw data must be within the root mean square error (RMSE). We repeated this sampling on runs of special interest and/or until a good confidence level was established. This did build confidence in that the model is accurately predicting the data it has been given.

The Interpret Graphs during confirmation runs predicted the best factor settings to meet the established goals. We utilized the suggested settings and other settings based on knowledge gained from the study.

#### Test results

The test results obtained throughout this test program reflected specific settings for each piece of cleaning equipment, utilizing the earlier specified cleaning solvents on identified product from each M/A-COM product line. It also identified problems that were in the way of accomplishing this goal. These problems are characteristic of reducing the number of variables in the system by establishing fixed equipment parameters.

The results of this analysis will be used by each division to establish process settings, introduce on-line statistical process control, document cleaning procedures, define training and develop piece part specific cleaning fixtures.

The following development tasks were accomplished to reduce the variables of the cleaning systems:

**1. DI water regeneration (closed loop rinsewater systems)**

The DI water regeneration system that was chosen for this evaluation from the Phase I activity was the Kyzen/Zenon KZ100. The system produces DI water from the regeneration of spent rinsewater. This DI water is then processed through a bank of polishing filters to obtain water cleanliness in the area of 2+ megohms.

The problem exists with this system as well as all other like systems on the market that a finite control of the final DI water output to the cleaning systems is not attainable. There is a very broad range of water cleanliness being sent to the rinse cycles of our cleaning equipment. The range is between 50 Kohm and 18 megohm, this extreme variance has not yet been factored into our testing.

Metallurgically a high DI water resistivity could cause pitting in some metals (i.e.; aluminum). A determination as to what range of DI water that will be acceptable for rinsing needs to be accomplished using the same design of experiments used to define the settings of equipment. Early indications show best results when the DI water is between 500 Kohm and 3 Megohm.

**2. Cleaning Fixtures**

One of the most important factors of performing these optimization tests was the ability to place the piece parts under test in the correct cleaning angle. The cleaning equipment only supplies the mechanism to clean, it does not focus on the variability's of product size and configuration. Fixturing becomes critical in achieving repeatability and optimum cleaning. The piece parts under test must be placed in such a fashion as to allow the cleaning solvents and rinse water to attack the contamination and allow for draining of the residues. The degree of cleaning and the ability to remove the spent solvents with DI water is directly proportional to the piece part placement within the cleaning systems.

The material of the fixtures will also play a role in achieving static sensitivity and long life of the fixtures to assure cost affectivity.

**3. Heating of the Solvents**

The Kyzen semi-aqueous cleaning solvents have a much higher flash point (none to boiling) than earlier solvents such as terpenes and petroleum based products. The cleaning systems chosen as a result of Phase I were originally designed around the use of these low flash point solvents. Matter of fact, they were tested prior to shipping using these materials and a residue of left over solvents was present at the start of our testing. This fact resulted in an initial degradation of the membrane filters used for the KZ-100 water regeneration systems.

The cleaning systems using the low flash point solvents ran between room temperature and 105 degrees F., therefore the heaters for the solvents in the accel cleaning system are much too small to bring the Kyzen Ionox solvent up to temperature (140 degrees F.) in a reasonable time. The system solvent heating takes approximately 2 hours to come up to temperature and stabilize. This factor should be realized at the equipment purchasing stage and larger system heaters specified.

The ECD equipment did not offer wash tank heaters at the time of our purchase. However we developed a mechanism that allowed us to compensate for this disadvantage. We installed a precision barrel heater for approximately \$400., to the 5 gallon wash solvent tank supplied and have been able to hold the temperature of the Ionox solvent to within 2 degrees F. The ECD equipment group has since developed a wash tank heater that is available on new systems.

#### 4. White particulate

This is not an uncommon occurrence in alternative cleaning programs. This white powder appears for the most part around solder connections on printed circuit boards. It has not been a problem with the Ionox-HC for all hybrid subassemblies however, for surface mount printed circuit boards we have seen the problem. We have sent samples of this by-product to an outside test lab for evaluation with the following results:

- a) The powder is residue flux in an emulsification state, that is, the flux has turned to soap and has been trapped on the board due to insufficient cleaning and or rinsing.
- b) There is a small component of tin oxide within this soap. It has not been determined if this is all part of the flux cleaning action that takes place in normal soldering operations or if it is a result of the high alkalinity of the Kyzen cleaning solvents. We will continue to evaluate this condition.

#### Final acceptance for implementation

The cleaning systems prior to release for production must be compared to before and after process capabilities (Cp). This comparison will determine if the objectives have been met. If the project's objectives have not been met, a decision must be made as to whether something could be done differently such as considering other factors or responses, or controlling noise factors.

When the system has met all requirements it shall be implemented by manufacturing to the production floor with approved documentation, SPC controls for critical cleaning settings, training plans, correct cleaning fixtures and an approved facility plan for installation.

We still have a significant amount of work ahead of us in qualifying each of the remaining divisions, ordering the equipment, developing the appropriate number and configurations of cleaning fixtures and installing all equipment at all of our M/A-COM divisions.