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Investigation of Cleanliness Verification Techniques for Rocket Engine Hardware

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Abstract

Oxidizer propellant systems for liquid-fueled rocket engines must meet stringent cleanliness requirements for particulate and nonvolatile residue. These requirements were established to limit residual contaminants which could block small orifices or ignite in the oxidizer system during engine operation. Limiting organic residues in high pressure oxygen systems, such as in the Space Shuttle Main Engine (SSME), is particularly important. The current method of cleanliness verification for the SSME uses an organic solvent flush of the critical hardware surfaces. The solvent is filtered and analyzed for particulate matter followed by gravimetric determination of the nonvolatile residue (NVR) content of the filtered solvent. The organic solvents currently specified for use (1,1,1-trichloroethane and CFC-113) are ozone-depleting chemicals slated for elimination by December 1995.

A test program is in progress to evaluate alternative methods for cleanliness verification that do not require the use of ozone-depleting chemicals and that minimize or eliminate the use of solvents regulated as hazardous air pollutants or smog precursors. Initial results from the laboratory test program to evaluate aqueous-based methods and organic solvent flush methods for NVR verification are provided and compared with results obtained using the current method. Evaluation of the alternative methods was conducted using a range of contaminants encountered in the manufacture of rocket engine hardware.

Introduction

Background

Stringent particulate and nonvolatile residue (NVR) cleanliness requirements for liquid rocket engine hardware, including fuel, oxidizer and pneumatic systems, are imposed by NASA and other customer specifications. The current method for cleanliness verification involves a final flush of the hardware surfaces immediately following the vapor degrease or solvent flush precision cleaning operation. A sample of the final flush solvent is collected, filtered for particulate matter analysis and tested for nonvolatile residue by a gravimetric technique. The specified cleanliness requirement is less than 1 mg of NVR per square foot of significant surface area. Significant surfaces are defined as those hardware surfaces that may contact the propellants or pneumatic gases during engine operation.

A variety of hardware configurations, material substrates and contaminants are encountered during the final cleaning and verification process. The hardware includes simple detail parts such as seals, bearings, nuts, and bolts; complex detail parts such as lines, pump and valve housings, and pump volutes and impellers; moderate size subassemblies such as valves, flex joints, and turbopumps; and large complex subassemblies such as flexible propellant ducts, powerheads, nozzles and main combustion chambers. Material substrates include nickel, iron and cobalt-base superalloys; stainless and low alloy steels; aluminum alloys; copper alloys; nickel, silver, gold and copper plating; polychlorotrifluoroethylene, polytetrafluoroethylene, polyimide and polyethylene terephthalate plastics; various elastomers; carbon; and dry film lubricants. Typical contaminants include machine coolants; machining, cutting and tapping fluids; hydraulic fluids; inks; dye penetrants and other fluids to support NDT operations; hydrocarbon, fluorinated and silicone greases; paraffin waxes; detergent residues; tape adhesive residues; and fingerprints.

The solvent predominantly utilized by Rocketdyne for precision cleaning and cleanliness verification is 1,1,1-trichloroethane (TCA). TCA is an ozone-depleting substance and will be banned from manufacture as of December 1995. Rocketdyne has an environmental task to evaluate and implement alternative methods of cleanliness verification that do not require the use of ozone-depleting chemicals and that minimize or eliminate the use of other regulated solvents. In general, the alternative technique(s) must be capable of detecting a variety of contaminants, be suitable for use on a variety of surface finishes, be capable of sampling internal cavities where the residue is most likely to be entrapped, not recontaminate the hardware, yield quantitative results that can be correlated to the current technique, be compatible with hardware materials, be applicable in a production environment and at the same time meet environmental and safety constraints.

Approach

The overall task proceeds from laboratory evaluation of the candidate techniques to hardware-scale demonstration to final production implementation. The status of the laboratory-scale evaluation of the aqueous technique and the organic solvent investigation will be discussed.

NASA-KSC has developed an aqueous verification technique in which the cleaned part is ultrasonically agitated in heated deionized (DI) water to remove any residual organic contaminants from the surfaces.(1) The contaminant concentration in the water is then determined by total organic carbon (TOC) analysis. Based upon the promising results obtained by NASA-KSC, the technique is under evaluation using the contaminants, substrates and configurations specific to Rocketdyne. However, as the technique is applicable to hardware of a limited size and some items (i.e., dry film lubricated parts and fragile instrumentation) are damaged by the ultrasonics, additional techniques will also be required. Organic solvents, other than the ozone-depleting chemicals, are under evaluation for these applications.

To evaluate the aqueous technique developed by KSC, the effectiveness of ultrasonic agitation for removing contaminants from surfaces was determined and the method of TOC analysis for determining the contaminant concentration in an aqueous medium was investigated. The effectiveness of ultrasonics was initially determined on small, flat coupons. Investigation of the TOC technique included determination of sample preparation techniques, construction of correlation curves and determination of the limits of detection. Finally, the entire verification process, ultrasonic removal and TOC analysis, was performed on coupons with a known level of contamination and the results verified gravimetrically. Additional testing will be conducted using complex test samples and small hardware.

The evaluation of alternative organic solvents will proceed along the same path as the investigation of the aqueous method except greater emphasis will be placed on solubility characteristics rather than on mechanical agitation techniques to remove the contaminant from a surface. The candidate fluids were identified, physical and chemical properties tabulated, and a review performed by Rocketdyne's Health, Safety & Fire and Environmental Protection departments. Downselected candidates were tested for residue and contaminant removal effectiveness. Once the final candidates have been identified, they will be tested for rinsability and material compatiblity. Removal testing on complex coupons and small hardware will then proceed as with the aqueous method.

Procedure

Ultrasonic Contaminant Removal Tests

Initial testing to determine the effectiveness of ultrasonic agitation with heated water to remove contaminants from flat coupons was evaluated. Small (1" x 1") flat Alloy 718 coupons were initially prepared by ultrasonic cleaning in tetrachloroethylene, drying, ultrasonic cleaning in heated DI water and drying. The coupon weights were monitored after each drying step to verify cleanliness. The cleaning cycle was repeated until no weight change was noted. Upon verification of cleanliness, each coupon was contaminated by spreading approximately 2 mg of the contaminant over one surface. The coupons were then heated for 1 hour at 95°C, allowed to cool and reweighed to determine the amount of contaminant remaining. The heating step eliminated any highly volatile species and more closely simulated the nature of a contaminant residue as it is present on actual hardware. Each coupon was then immersed in 100 mL of deionized water heated to 52°C and ultrasonically agitated for 10 minutes. The coupons were dried for 0.5 hour at 95°C, allowed to cool and reweighed to determine the residual contaminant remaining on the coupon. The percentage contaminant removed from each coupon was calculated from the weight data.

Testing was conducted using a 0.75-gallon, 47-kHz ultrasonic tank with a 150-watt power level and a 5-gallon, 25-kHz ultrasonic tank with a 600-watt power level. All tests were performed in triplicate using the contaminants listed in Table 1.

Test Contaminant	Description
Cool Tool, Monroe Fluid Tech.	Hand-applied cutting and tapping fluid containing paraffinic oil
MIL-H-83282 Micronic 882, Bray Oil Co.	Synthetic hydrocarbon hydraulic fluid containing triphenyl phosphate
MIL-H-5606	Hydraulic fluid containing naphthenic distillate with polymer additives and triphenyl phosphate
Rust Foil L-492 Preservative Oil, Franklin Oil Co.	Solvent-dispersed corrosion preventive compound containing aliphatic hydrocarbons and mineral oil
Krytox 240 AC, DuPont	Perfluoroalkylether grease with TFE filler used as a lubricant for oxygen systems and for pressure testing
CIMSTAR 3700, Cincinnati Milacron	Semi-synthetic water soluble metal working fluid containing mineral oil, di- and tri-ethanolamines, aminomethylpropanol and a synthetic lubricant
DTE 24, Mobil Oil	Petroleum distillate oil
Lapping Compound 38-1200, USP	Ultra fine grit lapping compound containing aluminum oxide
CRC 3-36, CRC Industries	Petroleum distillate and paraffinic oil containing lubricant and rust inhibitor
Centerpoint Lube, Chicago Manuf. & Dist.	High viscosity, grease-like extreme pressure machining lubricant containing petroleum oil, wax, and rosin ester
Bio-Pen P6R, Ardrox Inc.	Detergent-based visible, solvent-removable dye penetrant
Bio-Pen P6F-4, Ardrox, Inc.	Fluorescent, water-washable dye penetrant
Bio-Pen NQ-1 Developer, Ardrox Inc.	IPA and silica containing spray developer for penetrant inspection
Turco 3878 LF-NC, Turco Products	Aqueous emulsion cleaner containing sodium tripolyphosphate, glycol ether, and proprietary salts of anionic surfactants
Braycote 236, Castrol Inc.	Petrolatum used to lubricate o-rings during pressure test
Paraffin Wax	Low-melting-point, 107°F, wax used as machining maskant
Vacuum Grease, Dow Corning	Silicone vacuum grease used during pressure test
China Marker 165-T, Empire Berol Corp.	Red water-resistant marking pencil
Hydro Marker 665-T, Empire Berol Corp.	Red water-soluble marking pencil

Table 1. Test Contaminants

TOC / Contaminant Concentration Correlation Curves

To relate the TOC value of a sample to the actual contaminant concentration, a series of correlation curves were generated using prepared standards. Standard solutions of 20.5 ppm, 50.5 ppm and 80.3 ppm of Cool Tool; 19.5 ppm, 50.8 ppm, and 93.3 ppm of MIL-H-5606 hydraulic fluid; 24.0 ppm, 52.3 ppm, and 81.0 ppm of CRC 3-36; and 20.3 ppm, 59.3 ppm and 88.0 ppm of Centerpoint Lube each in 400 mL of DI water were prepared. To emulsify the contaminant, each solution was agitated by manual shaking for 30 seconds and then immersed in a 25-kHz ultrasonic bath at 52°C and agitated for 10 minutes after thermal equilibrium had been obtained. Each solution was then analyzed in triplicate for TOC content using a Rosemount Analytical Model DC-190 High Temperature Combustion TOC Analyzer. A sample of DI water was also analyzed to obtain a blank value.

TOC Analysis of Water from Coupon Tests

A quick assessment of the feasibility of the ultrasonic agitation/TOC analysis for cleanliness verification was performed using the small Alloy 718 coupons. The coupons were cleaned, verified and contaminated as in the ultrasonic removal tests, except varying levels of initial contaminant were applied. The target contamination levels were 1 mg, 2 mg and 5 mg. Two contaminants were tested, Cool Tool and Centerpoint Lube. Each sample was ultrasonically agitated in 100 mL of heated DI water as described previously. The water sample was then analyzed for TOC content. From the correlation curves, the contaminant concentration in the water was calculated and the total amount of contaminant removed from the coupon was calculated using Equation 1.

$$\text{Contaminant Removed (mg)} = \frac{TOC - B}{M} \times V \qquad (1)$$

TOC	=	TOC value in ppm or mg/liter
B	=	the y-intercept of the correlation curve, i.e., the TOC of the DI water blank in ppm or mg/liter
M	=	slope of line of correlation curve in ppm TOC / ppm contaminant concentration or (mg/liter) / (mg/liter)
V	=	volume of water used for ultrasonic extraction in liters

For comparison, the amount of contaminant removed was also calculated by the change in coupon weight after ultrasonic immersion.

Organic Solvent Evaluation

A list of candidate alternate solvents was compiled from a literature search, published data bases and supplier information. The categories of solvents considered included chlorinated solvents, hydrochlorofluorocarbons (HCFCs), perfluorocarbons (PFCs), hydrofluorocarbons (HFCs), fluoroiodocarbons (FICs), alcohols, terpenes, ketones, aliphatic and alicyclic hydrocarbons, ethers, glycol ethers, esters, amines, aromatic hydrocarbons, methyl siloxanes, and parachlorobenzotrifluoride. An abbreviated list of candidates was compiled using the following criteria:

Essential characteristics:

- Moderate to high volatility to promote evaporation from complex hardware and ease of performing a gravimetric NVR
- Comparable solubility to trichloroethane for a range of contaminants used at Rocketdyne
- Available in high purity and leaves little or no residue upon evaporation
- Existing or near-term availability
- Non-ozone depleting

Desirable characteristics:

- Not currently listed nor proposed to be listed on California Proposition 65, AB2588 or classified as a HAP (hazardous air pollutant)
- Does not contribute to global warming
- Not classified as a VOC (volatile organic compound)
- Permissible exposure level greater than 100 ppm
- Nonflammable
- Inoffensive odor

Contaminant removal tests were then performed to assess the effectiveness of the candidate solvents relative to TCA. Approximately 10 to 30 mg of contaminant were applied to the bottom of a clean, tared aluminum weighing pan. The contaminated pan was heated for 1 hour at 95°C, allowed to cool to room temperature and reweighed to determine the amount of contaminant remaining. The dish was then sequentially filled and drained with the filtered test solvent until a total of 100 mL had been used. The pan was dried for 1 hour at 108°C to evaporate any residual solvent, allowed to cool to room temperature and reweighed. The percentage of the initial contaminant removed was calculated using the weight data. A gravimetric NVR was also performed on each solvent sample and a percentage of contaminant removed was calculated. Tests were performed in triplicate with the contaminants listed in Table 1.

Results and Discussion

Ultrasonic Contaminant Removal Tests

The results from the removal tests for the seventeen contaminants tested using both the 47-kHz ultrasonic bath and the 25-kHz bath are shown graphically in Figure 1. In general, the 25-kHz bath was slightly more effective than the 47-kHz bath. The average removal of all of the contaminants was 83% for the 47-kHz bath and 85% for the 25-kHz bath. For the majority of the contaminants, ultrasonic agitation in water was greater than 90% effective in removing the contaminant residues. The Krytox grease, silicone vacuum grease, paraffin wax and China marker were the most difficult contaminants to remove as shown by their 10 to 80% removal. Not surprisingly, these contaminants are also the most difficult to remove using organic solvents.

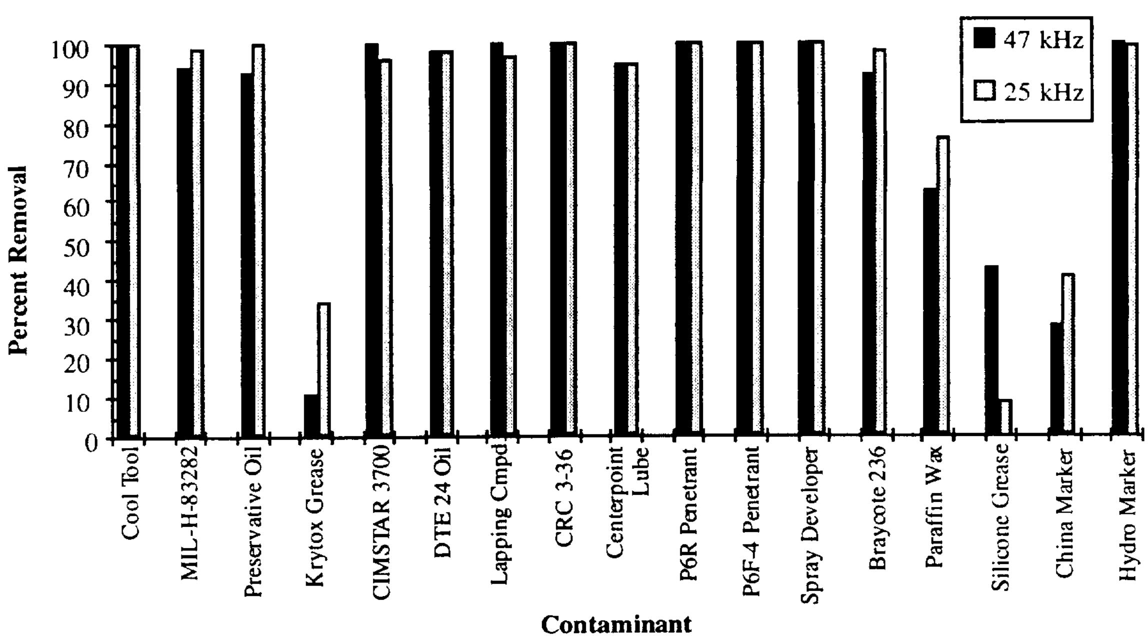

Figure 1. Contaminant Removal Effectiveness by 10-Minute Immersion in 52°C DI Water with Ultrasonic Agitation

Because of the simple configuration of the test coupons used, the removal efficiencies may not translate directly to more complex geometries. The coupons were selected so the contaminant removed could be determined by simple gravimetric means with relatively little error. For example, the accuracy of the percentage removal data is approximately ± 5% with the error derived from the limitations of the balance. To fully evaluate the effectiveness of the ultrasonics, more complex test coupons and hardware will be tested and an organic solvent verification method used to assess the results.

TOC / Contaminant Concentration Correlation Curves

In order to relate the measured TOC value to the actual concentration of the contaminant in water, correlation curves were generated. The curves for four contaminants, Cool Tool, Centerpoint Lube, CRC 3-36 and MIL-H-5606 hydraulic fluid are shown in Figure 2. The average measured TOC value for each of the standard solutions prepared was plotted and a best fit line determined by the method of least squares. As shown, for the concentration ranges tested, the linear fit of the data is excellent. Testing is continuing to generate correlation curves for the other contaminants of interest.

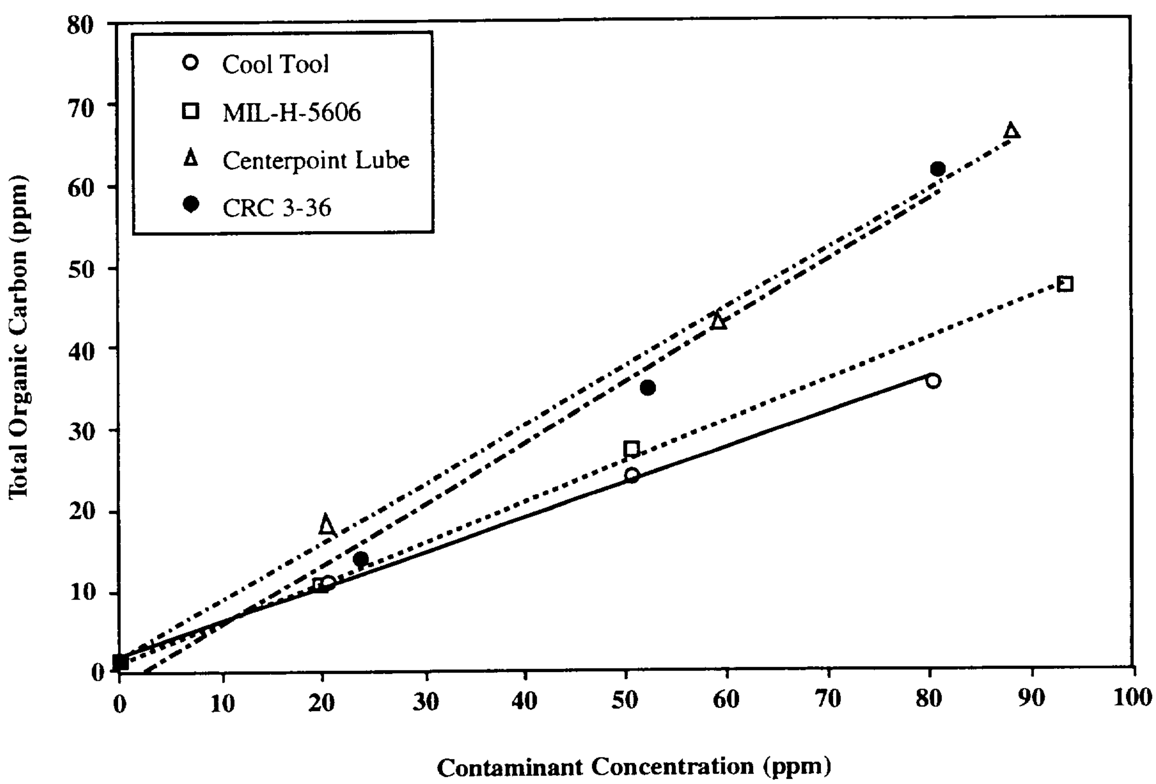

Figure 2. Correlation of Contaminant Concentration with TOC

TOC Analysis of Water from Coupon Tests

Testing was performed using the small coupons to verify the ability of the TOC analysis to accurately measure the amount of contaminant removed after ultrasonic agitation. The contaminant removed was calculated both gravimetrically and by TOC analysis. These coupons were used because cleanliness assessment could be made gravimetrically rather than by the more time consuming solvent verification method. As shown in Figures 3 and 4, contaminant removal results determined by the TOC method compare favorably to those obtained by the gravimetric method. The favorable results indicate that the aqueous verification technique is viable, at least for simple geometries and the contaminants tested to date. Based upon these successful results, testing has been initiated with more complex geometries.

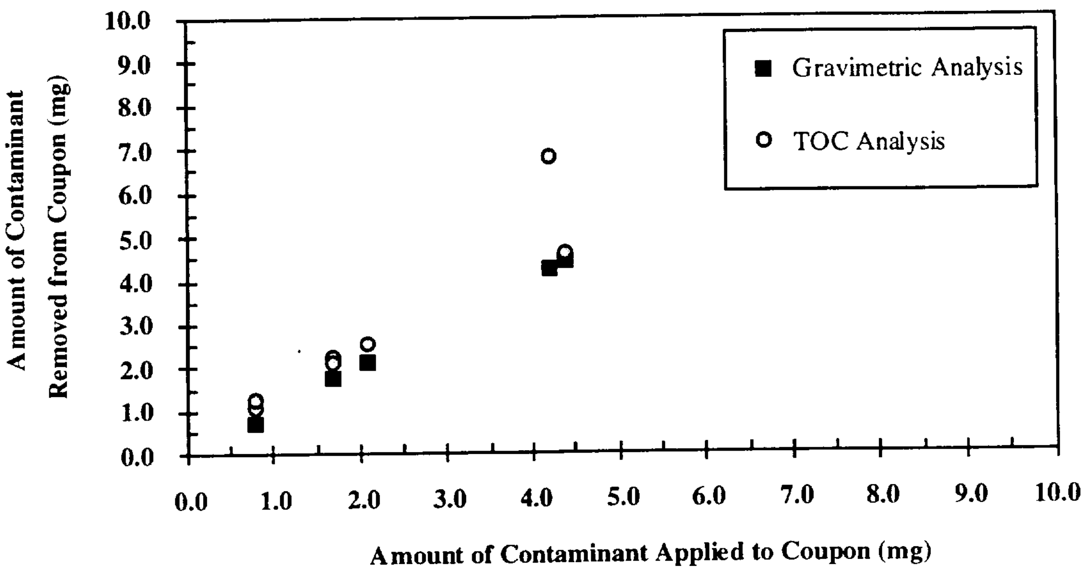

Figure 3. Comparison of Aqueous Verification with Gravimetric Results for Determining Cool Tool Residue on Alloy 718 Coupons

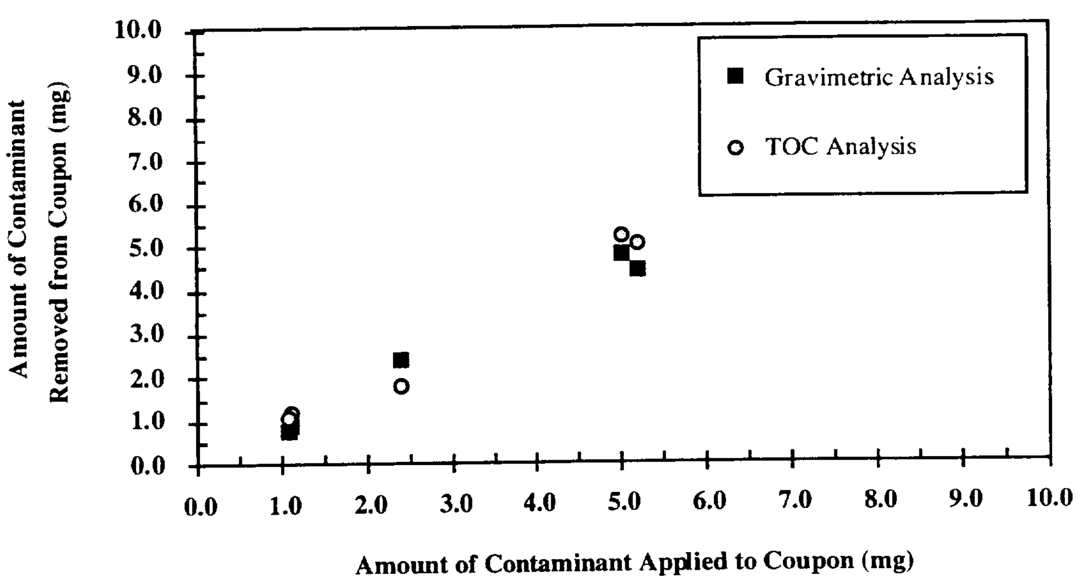

Figure 4. Comparison of Aqueous Verification with Gravimetric Results for Determining Centerpoint Lube Residue on Alloy 718 Coupons

Organic Solvent Alternatives

Candidate organic solvents under initial consideration as alternatives to TCA include isopropyl alcohol (IPA), cyclohexane, ethyl acetate, an IPA/cyclohexane azeotrope, HCFC 225 and acetone. Of these, IPA, cyclohexane, and ethyl acetate have been subjected to solubility testing. These solvents were selected for initial evaluation based upon promising results obtained by other companies that require alternative verification methods. Testing was also conducted using TCA for comparison.

A comparision of the percentage of contaminant removed as determined by coupon weight data and by solvent NVR was conducted to assess the accuracy of the current gravimetric technique. Testing was performed for all of the contaminants using TCA, IPA, cyclohexane and ethyl acetate; however, only the TCA results are shown. From Figure 5, it can be seen that for those contaminants readily removed by the solvent, the NVR results are typically 85 to 100% of the results obtained gravimetrically, except for the MIL-H-5606 and the DTE 24. The difference between the NVR and the gravimetric results is primarily attributed to the volatility of the contaminant, i.e., some of the contaminant is evaporated along with the solvent during the evaporation phase of the NVR procedure. This is particularly apparent with the light hydrocarbon contaminants, such as the MIL-H-5606 hydraulic fluid and the DTE 24. Previous testing in which the contaminant was not initially dried resulted in an even greater difference betweeen the NVR and weight data. Drying the contaminant prior to testing aids in reducing the difference but does not entirely eliminate the effect.

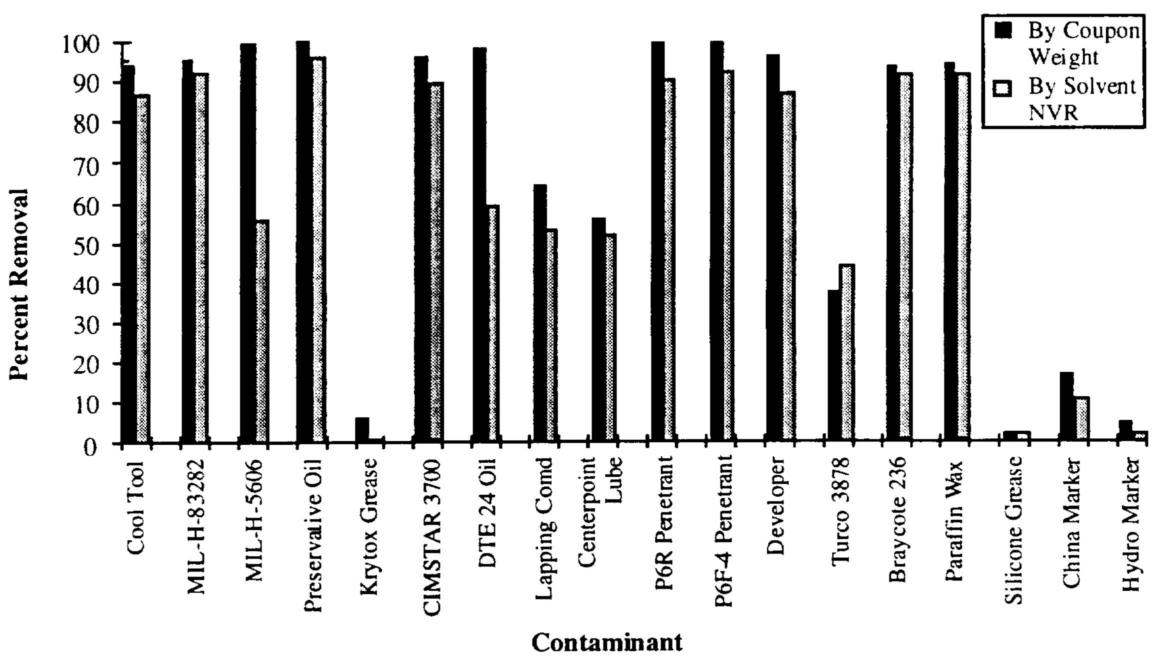

Figure 5. Contaminant Removal Tests with Ambient Temperature TCA

The effectiveness of each solvent for removing the selected contaminants is presented as a percentage removal in Figures 6 and 7. The results are based upon the gravimetric data rather than the NVR values and are shown with the TCA results for comparision. As shown, isopropyl alcohol is considerably less effective than TCA for the contaminants tested, whereas cyclohexane and ethyl acetate are nearly as effective as TCA. As shown, even the TCA only partially removed the lapping compound, the Centerpoint Lube and the Turco 3878. Furthermore, the TCA is relatively ineffective in removing the Krytox grease, the silicone vacuum grease, and the markers. For these contaminants, it was noted that any removal at all was primarily the result of mechanical action rather than dissolution in the solvent. Of the solvents tested to date, cyclohexane showed the most promise as an alternative to TCA. However, testing is still in progress with some of the other candidates.

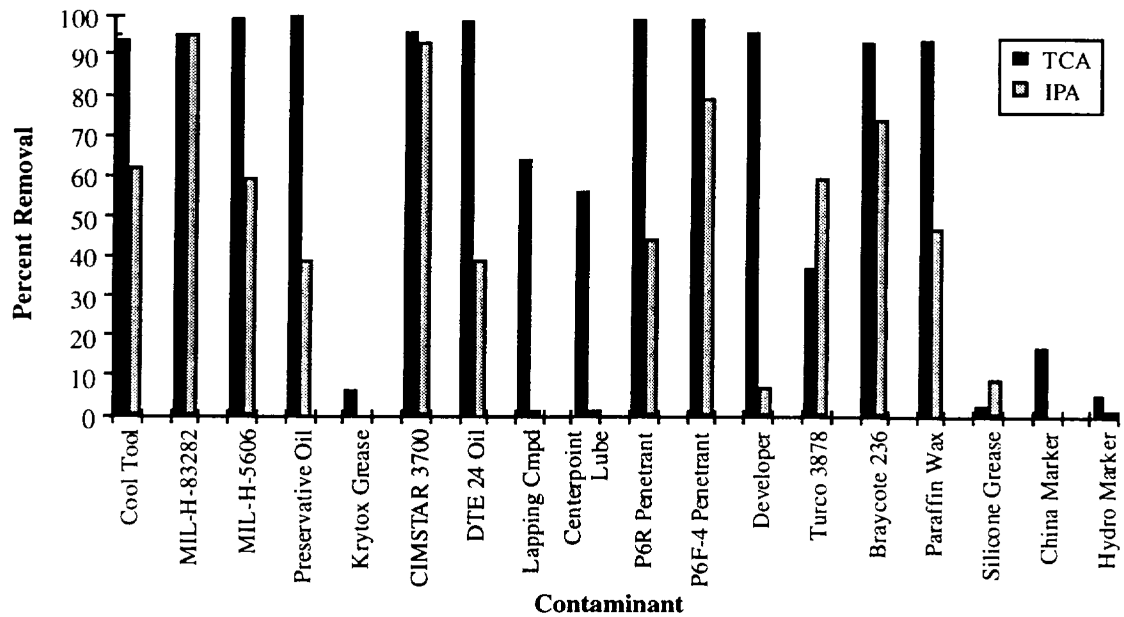

Figure 6. Contaminant Removal Tests with Ambient Temperature 1,1,1 Trichloroethane and Isopropyl Alcohol

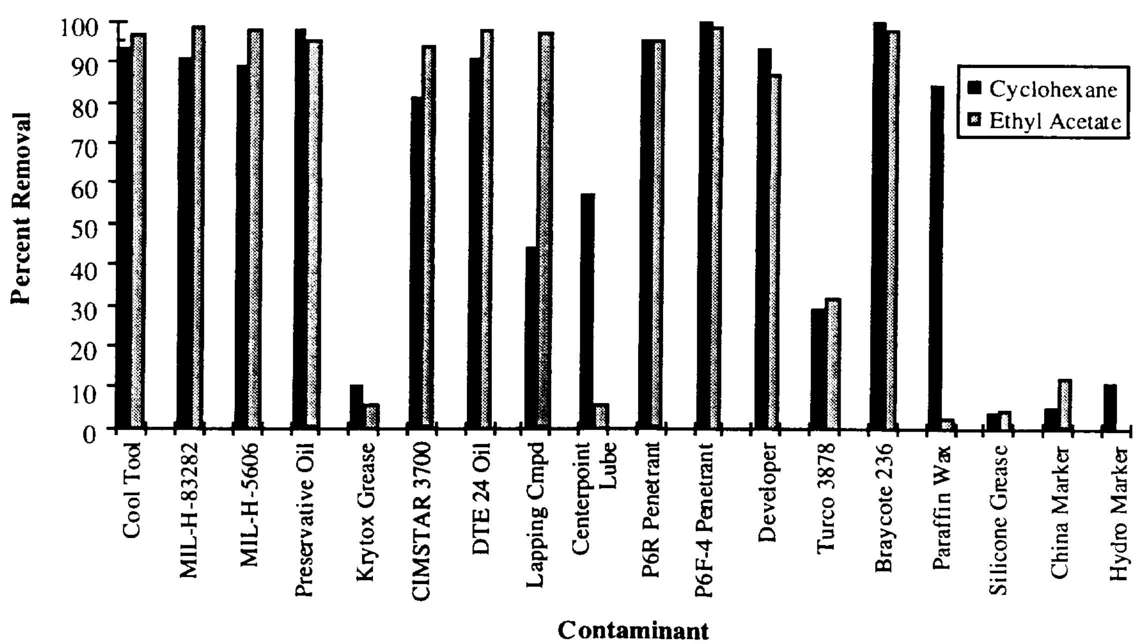

Figure 7. Contaminant Removal Tests with Ambient Temperature Cyclohexane and Ethyl Acetate

Conclusions

Based upon limited laboratory testing, the aqueous verification technique developed by NASA-KSC has been shown to be feasible for some Rocketdyne applications. Use of 25-kHz ultrasonic agitation is slightly more effective than 47-kHz ultrasonic agitation for removing the majority of the test contaminants with heated DI water. Through the use of correlation curves, TOC analysis of the water used during the ultrasonic cleaning is accurate in determining the amount of contaminant removed from the surface. Testing, however, was very limited and must be extended to the more difficult to remove contaminants and more complex geometries.

The majority of the organic solvents that can be considered as alternatives to TCA have disadvantages such as toxicity, flammability, or classification as VOCs (volatile organic compounds) or HAPs (hazardous air pollutants). From the limited testing performed to date, cyclohexane is nearly equivalent to TCA in contaminant removal. However, cyclohexane is a VOC and has a low flash point. Furthermore, as with the majority of the solvents, cyclohexane is not compatible with oxygen and must be completely removed from any oxidizer hardware. Testing will continue to evaluate other alternatives with final technique validation performed on full-scale hardware.

Acknowledgement

The assistance of S. Klee, N. Cimarusti, M. Maseda, and M. Silvia for developing test techniques and performing testing in support of this project is gratefully acknowledged.

Reference

(1) Allen, G. J., Hoppesch, C. W., Johnson, R. S., Buckley, M. D., "Aqueous Nonvolatile Residue Validation of Precision Cleaned Hardware", Alternatives to Chlorofluorocarbon Fluids in the Cleaning of Oxygen and Aerospace Systems and Components, ASTM STP 1181, 1993, pp. 37 - 48.

INVESTIGATION OF CLEANLINESS VERIFICATION TECHNIQUES FOR ROCKET ENGINE HARDWARE

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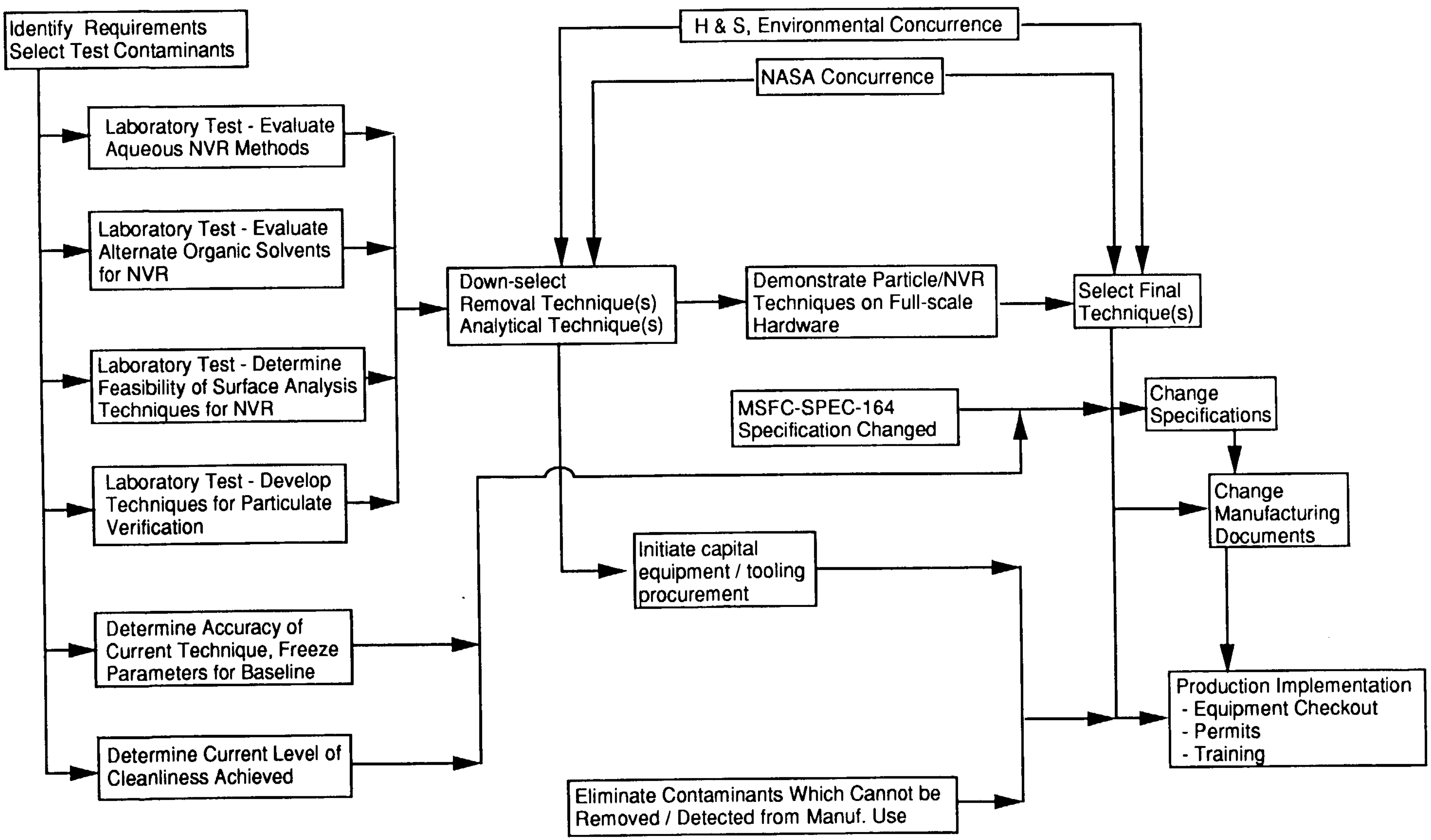
HUNTSVILLE, AL
AUGUST 10-11, 1994

CLEANLINESS VERIFICATION TASK FLOWCHART



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CONTAMINANTS SELECTED FOR TESTING

- Machining / Lapping Compounds
  - CIMSTAR 3700
  - Cool Tool
  - DTE 24 Oil
  - BP200 EDM Oil
  - Lapping Compound
- Preservative Compounds
  - Preservative Oil (Franklin L-492, RB0210-016)
  - Centerpoint Lube
- Hydraulic Fluids
  - MIL-H-83282
  - MIL-H-5606
- Penetrant Compounds
  - 2A Dye Penetrant
  - 4C Dye Penetrant
  - Spray Developer
- Detergent Residues
  - Turco 4215 Additive
  - Turco 3878
  - Turco 4215 NC-LT
- Waxes / Greases
  - Petrolatum (Braycote 236)
  - Paraffin Wax
  - Silicone Vacuum Grease
  - Krytox Grease
  - Grease Pencils
    - China Marker
    - Hydromarker

CONTAMINANT REMOVAL TESTS

- Objective: Determine the effectiveness of sonicated, heated, DI water for removing contaminants from Inco 718 coupons.

- Approach:
  - Clean light (0.5 gram) coupons with perc to constant weight
    - Verify no wt. change occurs in sonicated, heated DI water
    - Dry, allow to cool, weigh each coupon (W1)

  - Apply ~ 2 mg of contaminant evenly over one surface of coupon
    - Weigh (W1.5)
    - Heat coupon for 1 hour at 200°F
    - Allow to cool to RT, weigh (W2)

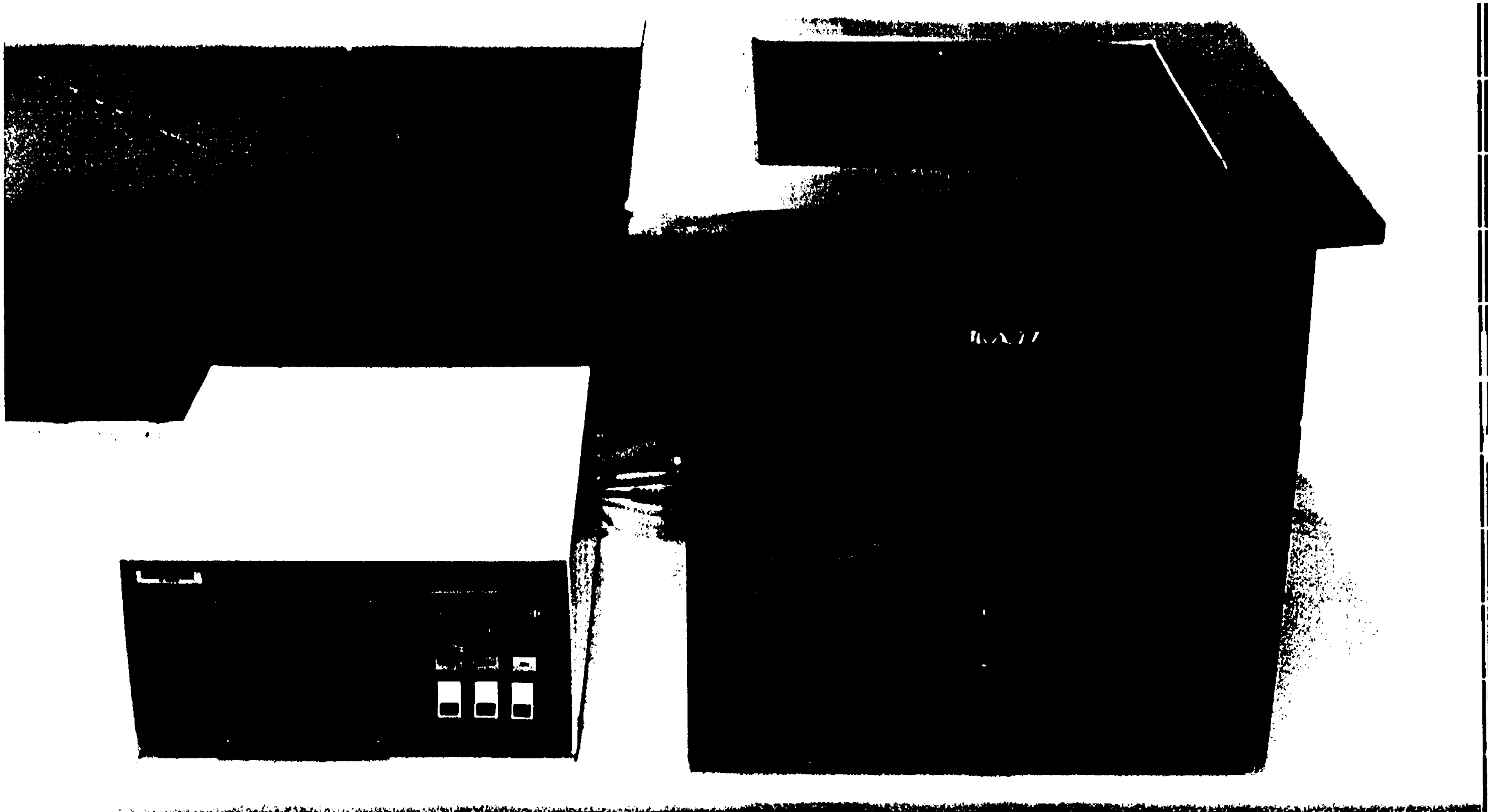
  - Place coupon in 100 ml of DI water at 52°C. Sonicate for 10 min.

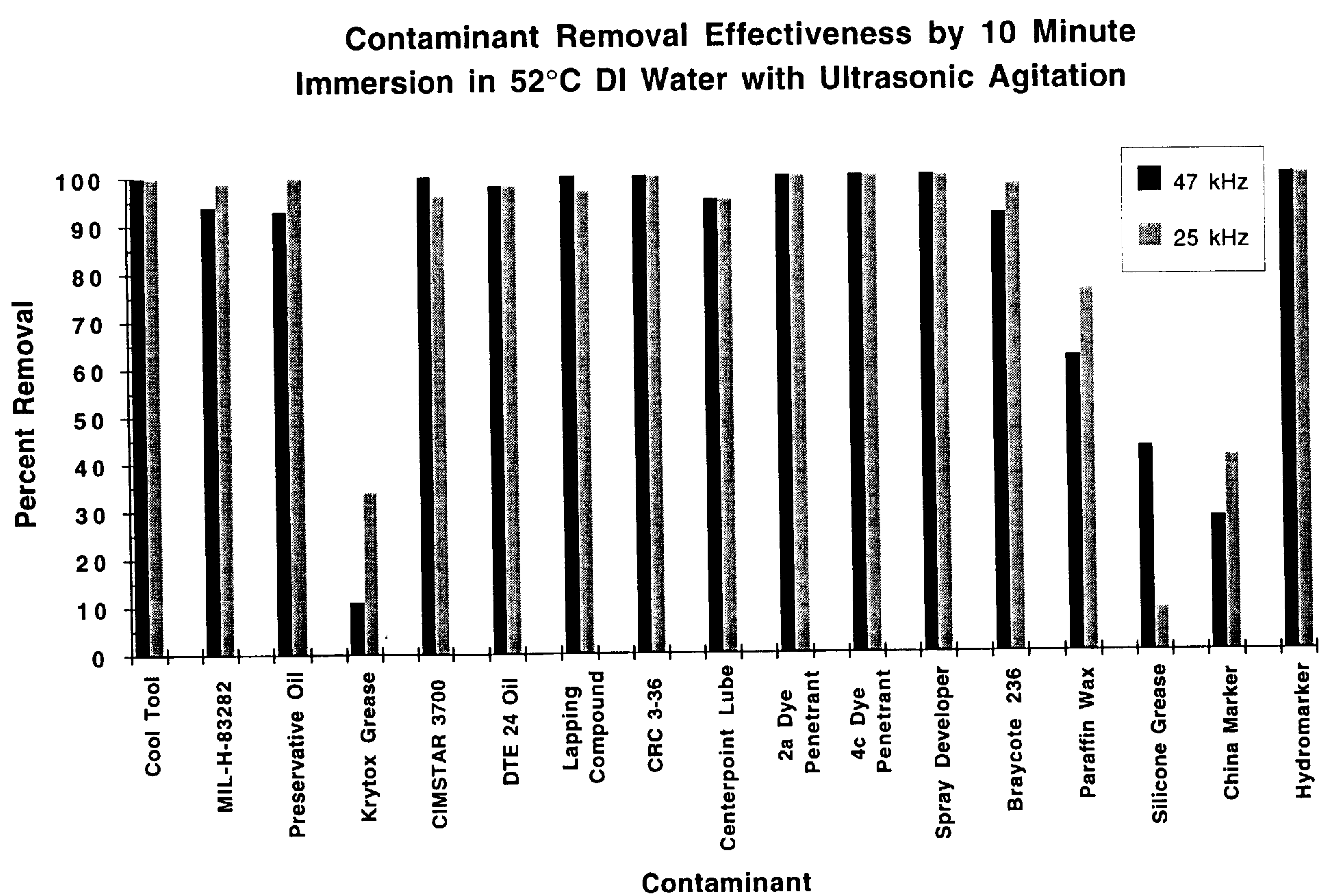
  - Dry coupon at 200°F. Allow to cool to RT. Weigh (W3)

  - Calculate % removal = $\frac{W2 - W3}{W2 - W1} \times 100\%$

  - Clean coupons in perc to constant weight

Blackstone 25-kHz Ultrasonic Bath




Contaminant Removal Effectiveness by 10 Minute Immersion in 52°C DI Water with Ultrasonic Agitation
47 kHz
25 kHz
Percent Removal
100
90
80
70
60
50
40
30
20
10
0
Cool Tool
MIL-H-83282
Preservative Oil
Krytox Grease
CIMSTAR 3700
DTE 24 Oil
Lapping Compound
CRC 3-36
Centerpoint Lube
2a Dye Penetrant
4c Dye Penetrant
Spray Developer
Braycote 236
Paraffin Wax
Silicone Grease
China Marker
Hydromarker
Contaminant

CORRELATION OF CONTAMINANT CONCENTRATION WITH TOC

Objective:

To generate correlation plots for Cool Tool, MIL-H-5606 oil, CRC 3-36, and Centerpoint Lube.

Procedure:

1) Weigh appropriate amount of contaminant and deposit in 500 ml bottle.

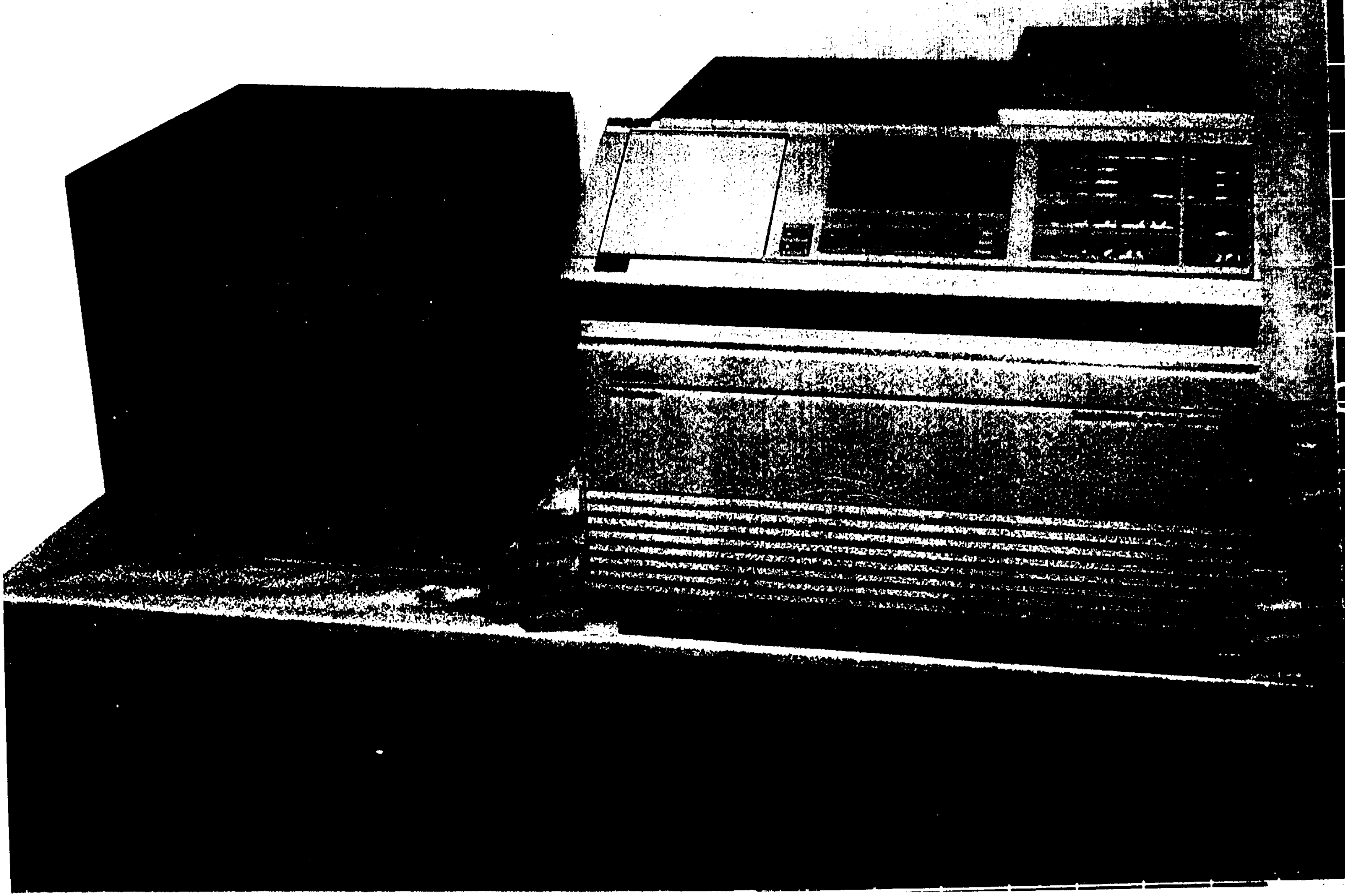
2) Add 400 ml of distilled water and shake bottle manually for 30 sec.

3) Place bottle in 52°C ultrasonic bath, and allow 30 min. for bottle's contents to reach temperature.

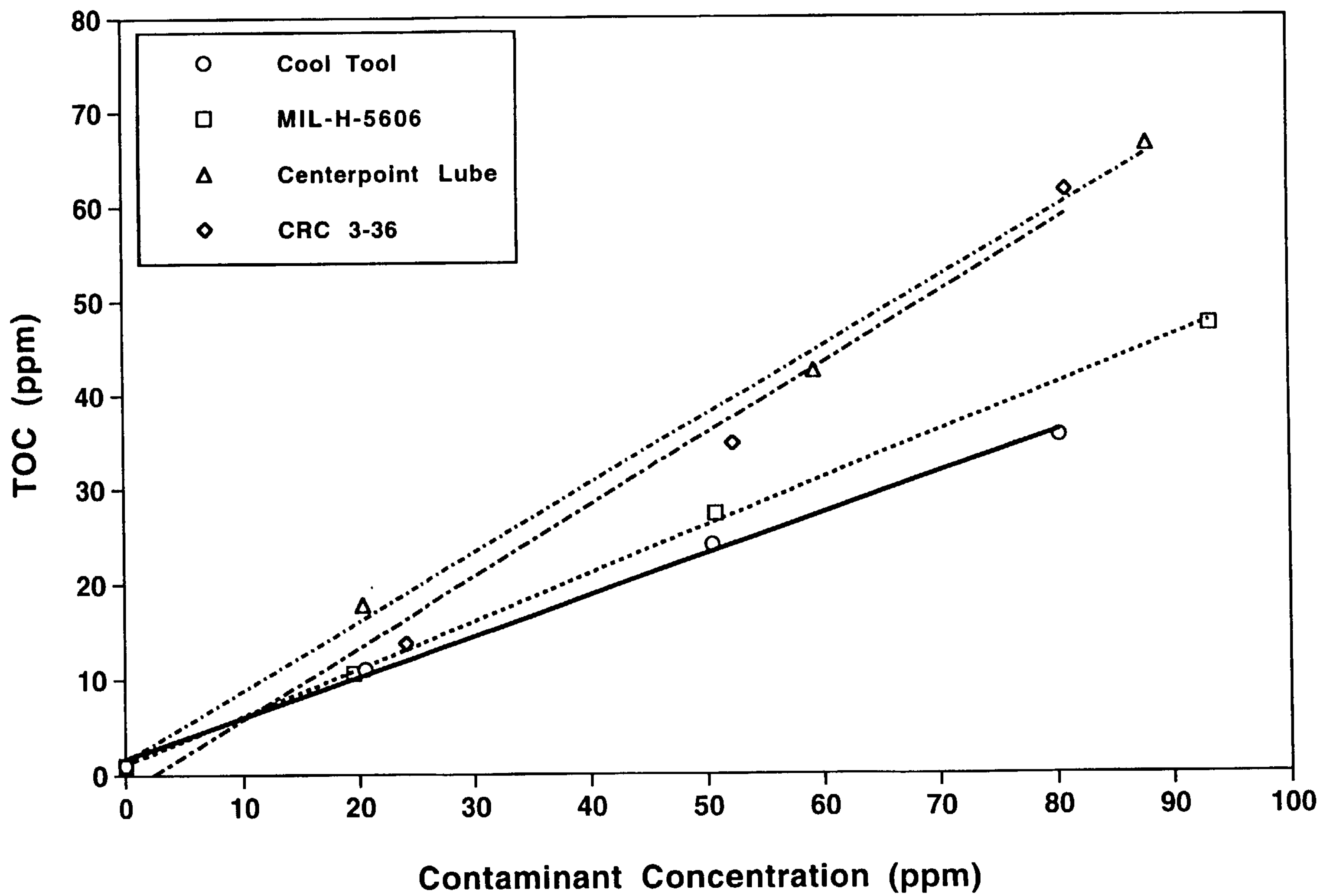
4) Sonicate sample for 10 min.

5) Begin TOC sampling immediately.

Rosemount Total Organic Carbon Analyzer & Balston TOC Gas Generator



CORRELATION OF CONTAMINANT CONCENTRATION WITH TOC



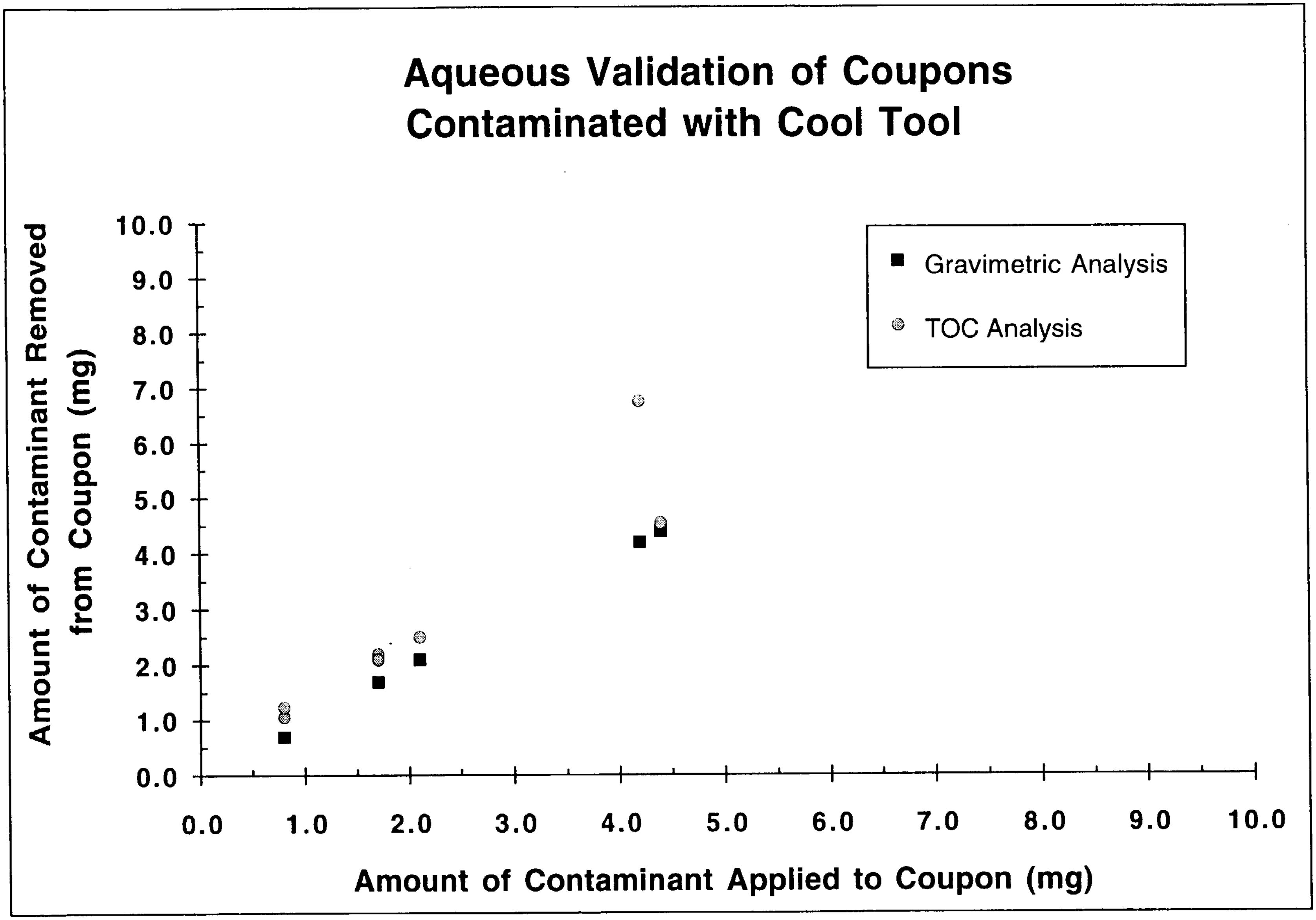
ANALYSIS OF WATER FROM CONTAMINANT REMOVAL TESTS

Objective:

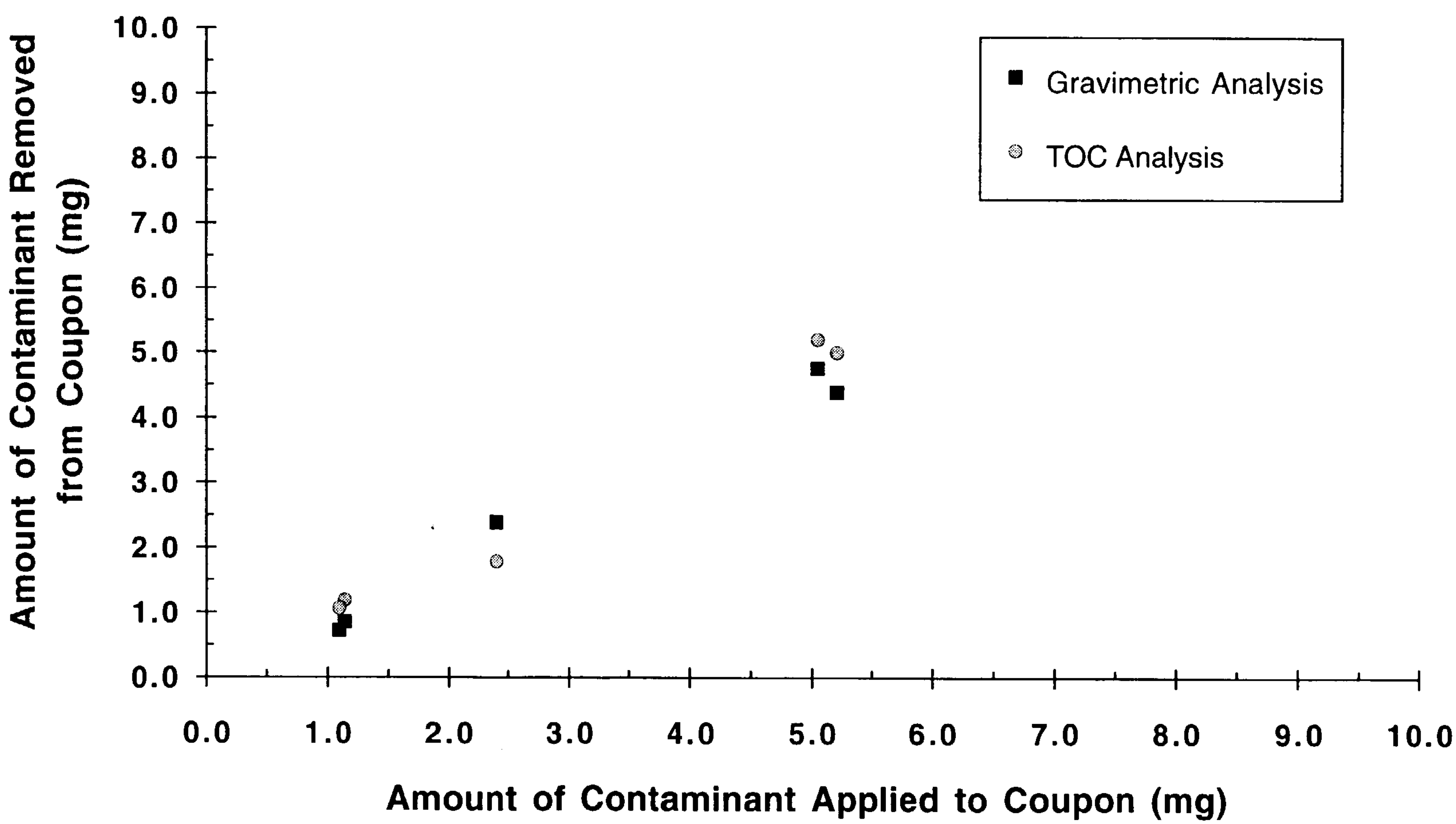
To investigate the feasibility of using TOC analysis to assess contaminant removal from coupons.

Approach:

- Conduct ultrasonic contaminant removal tests with DI water
- Sample water used in the test
- Analyze water for TOC value
- Using correlation curves, determine contaminant concentration from TOC value
- Compare milligrams of contaminant removed as determined by TOC to gravimetric results


Aqueous Validation of Coupons
Contaminated with Cool Tool
Gravimetric Analysis
TOC Analysis
Amount of Contaminant Removed from Coupon (mg)
Amount of Contaminant Applied to Coupon (mg)
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0

**Aqueous Validation of Coupons
Contaminated with Centerpoint Lube**



SOLVENT CONTAMINANT REMOVAL TESTS

- Objective: Screen organic solvents for contaminant removal efficiency as compared to TCA and CFC-113.

- Approach:
  - Apply 10 - 15 mg of contaminant to bottom of tared Al dish
    - Heat coupon for 1 hour at 200°F
    - Allow to cool to RT, weigh

  - Rinse dish with 100 ml of filtered solvent & collect solvent in a second tared Al dish
    - Evaporate collected solvent to 5 ml on a steam bath

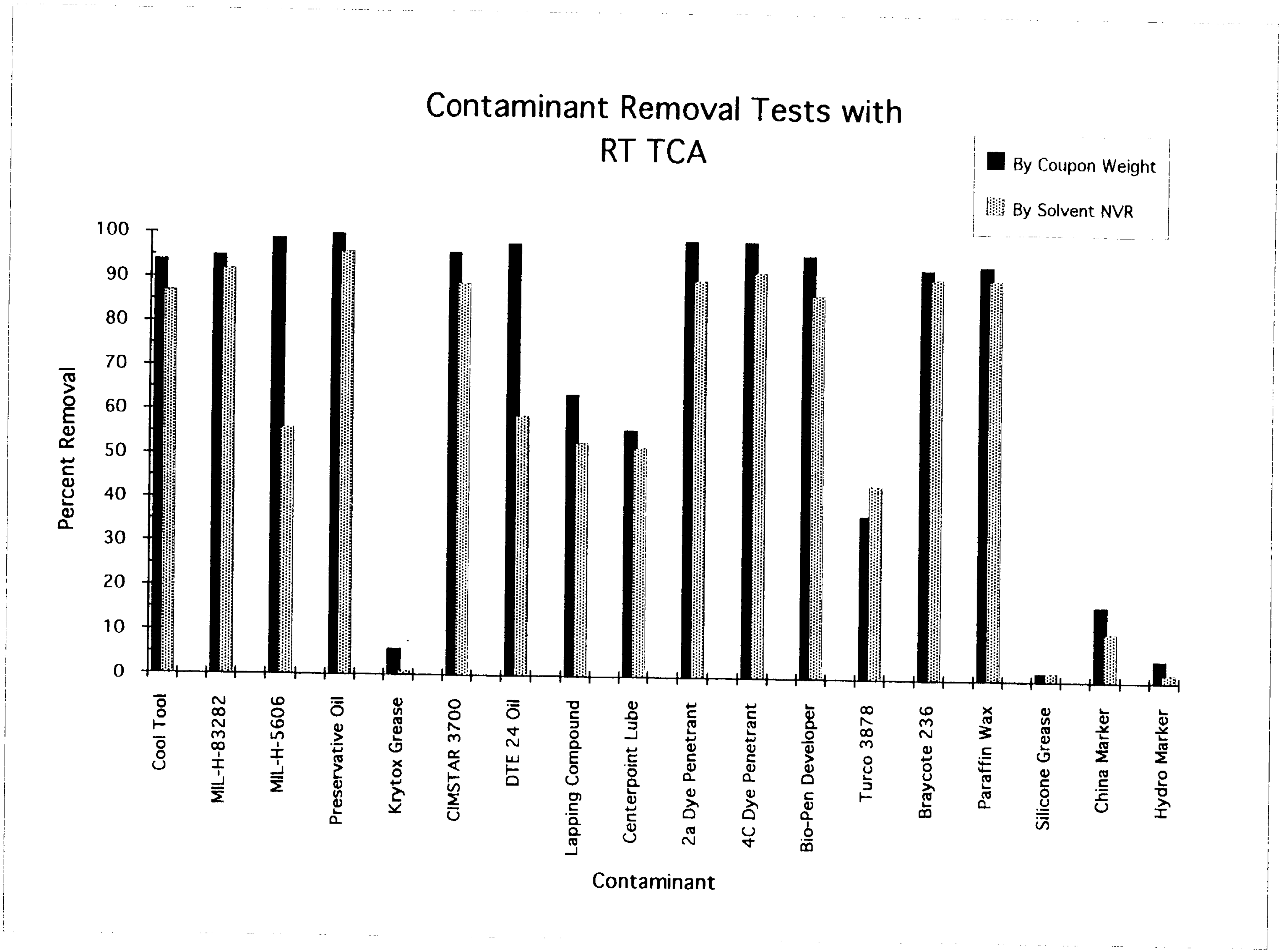
  - Dry both dishes for 1 hour at 228°F

  - Allow to cool to RT and reweigh each dish

  - Adjust weights by results obtained from solvent blank analysis

  - Calculate % removal from residual weight of contaminant in dish

  - Calculate % removal from solvent NVR


Contaminant Removal Tests with RT TCA
By Coupon Weight
By Solvent NVR
Percent Removal
100
90
80
70
60
50
40
30
20
10
0
Cool Tool
MIL-H-83282
MIL-H-5606
Preservative Oil
Krytox Grease
CIMSTAR 3700
DTE 24 Oil
Lapping Compound
Centerpoint Lube
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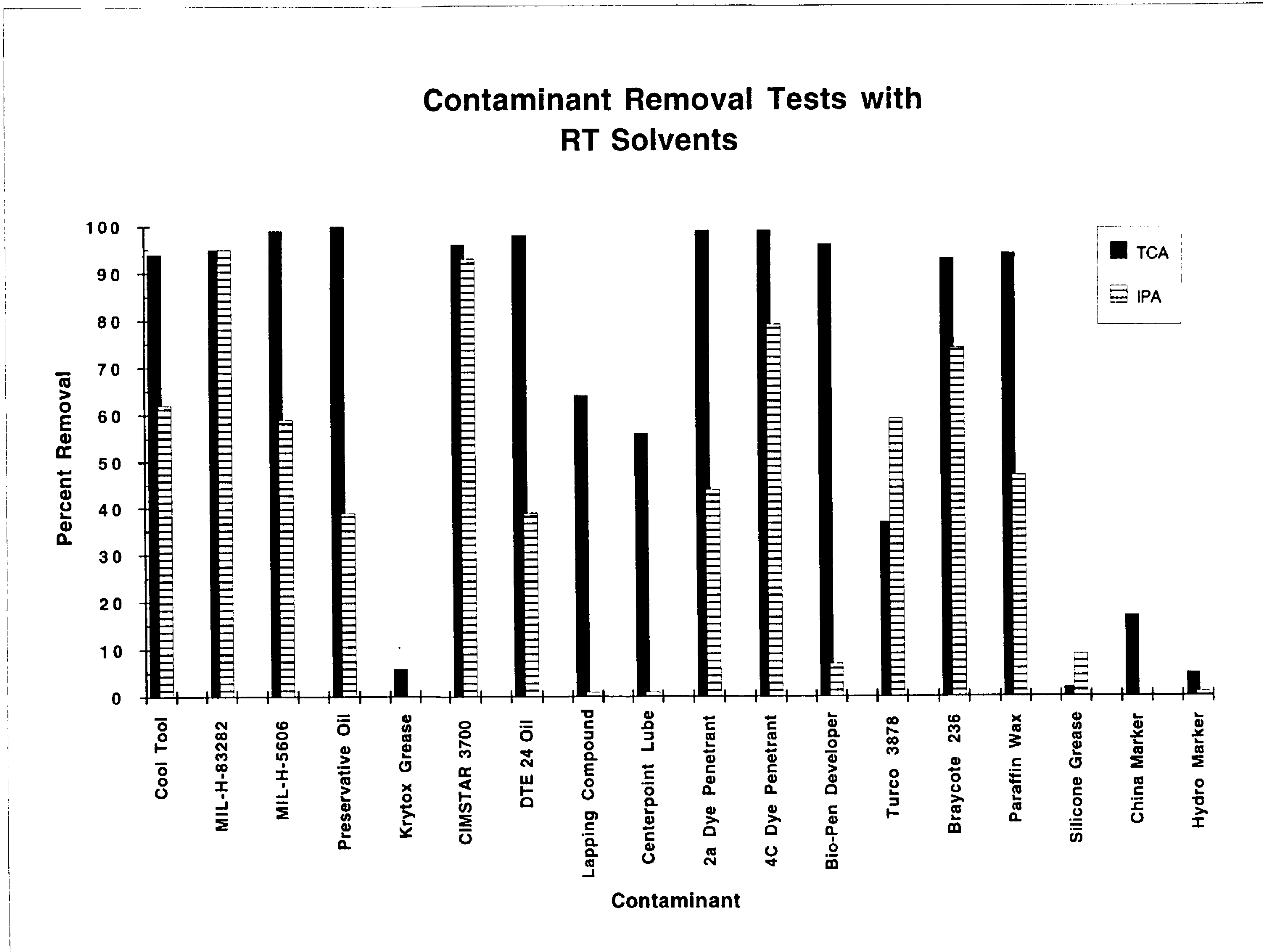
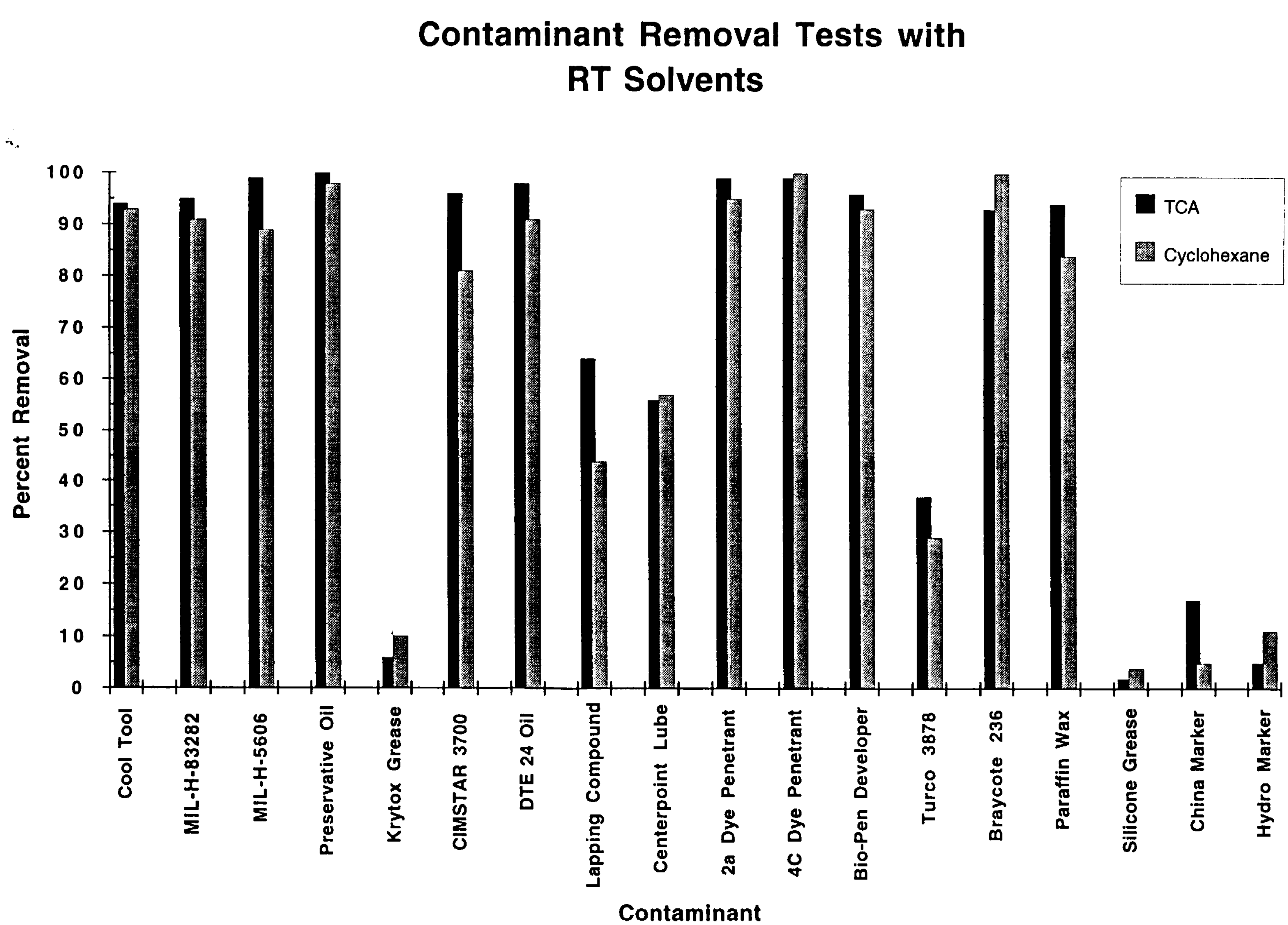
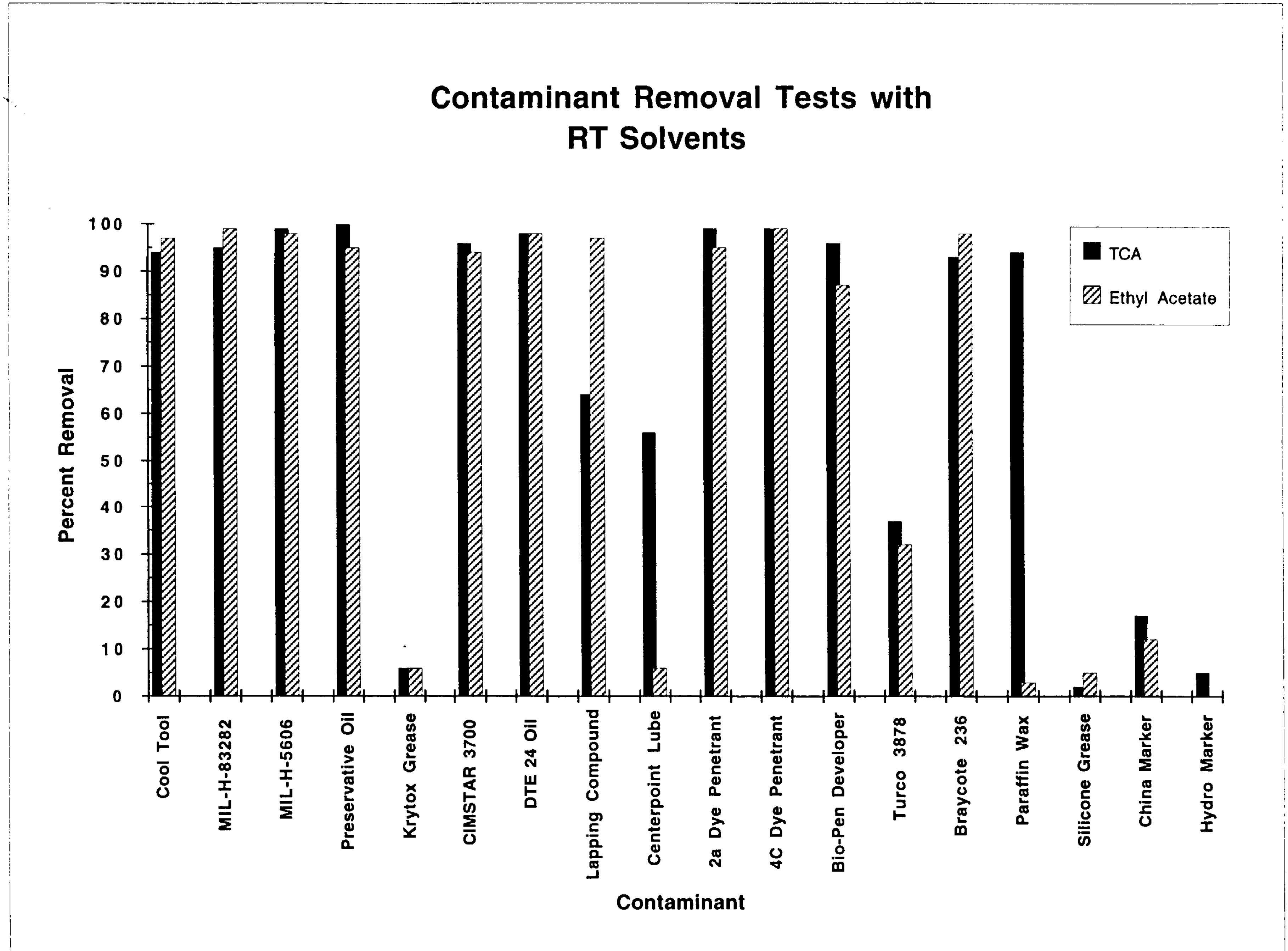
Silicone Grease
China Marker
Hydro Marker
Contaminant



Contaminant Removal Tests with
RT Solvents
Percent Removal
100
90
80
70
60
50
40
30
20
10
0
TCA
IPA
Cool Tool
MIL-H-83282
MIL-H-5606
Preservative Oil
Krytox Grease
CIMSTAR 3700
DTE 24 Oil
Lapping Compound
Centerpoint Lube
2a Dye Penetrant
4C Dye Penetrant
Bio-Pen Developer
Turco 3878
Braycote 236
Paraffin Wax
Silicone Grease
China Marker
Hydro Marker
Contaminant



Contaminant Removal Tests with RT Solvents
Percent Removal
100
90
80
70
60
50
40
30
20
10
0
TCA
Cyclohexane
Cool Tool
MIL-H-83282
MIL-H-5606
Preservative Oil
Krytox Grease
CIMSTAR 3700
DTE 24 Oil
Lapping Compound
Centerpoint Lube
2a Dye Penetrant
4C Dye Penetrant
Bio-Pen Developer
Turco 3878
Braycote 236
Paraffin Wax
Silicone Grease
China Marker
Hydro Marker
Contaminant



Contaminant Removal Tests with
RT Solvents
Percent Removal
100
90
80
70
60
50
40
30
20
10
0
TCA
Ethyl Acetate
Cool Tool
MIL-H-83282
MIL-H-5606
Preservative Oil
Krytox Grease
CIMSTAR 3700
DTE 24 Oil
Lapping Compound
Centerpoint Lube
2a Dye Penetrant
4C Dye Penetrant
Bio-Pen Developer
Turco 3878
Braycote 236
Paraffin Wax
Silicone Grease
China Marker
Hydro Marker
Contaminant

ALTERNATE ORGANIC SOLVENTS

SUMMARY

- Average effectiveness of alternate solvents relative to TCA

Solvent	Overall Avg.	Avg. excluding Krytox, silicone, markers
TCA	1.00	1.00
IPA	0.72	0.59
Cyclohexane	1.06	0.93
Ethyl Acetate	0.92	0.89

CONSIDERATIONS FOR FUTURE VERIFICATION

- **Anticipate More Than One Technique Will Be Used**
- **Technique Selected Will Depend on**
  - **Hardware Configuration**
    - Hardware Which Can Be Aqueously Fine Cleaned
      - Small Parts Which Can Be Immersed in U/S Bath
      - Parts Which Can Not Be Immersed in U/S Bath
    - Hardware Which Can Not Be Aqueously Fine Cleaned
  - **Expected Contaminants**
- **Possibility That Sequential NVR Techniques Will Be Required**
- **Critical to Verify at Detail Level & Eliminate Verification at Assembly Level When Possible**
- **Contaminants Which Can Not Be Detected May Need to be Eliminated From Use in Manufacturing**