Alternatives to Chlorinated Solvents in the Solid Rocket Motor Industry

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Introduction

In response to impending environmental regulations on ozone-depleting chlorinated solvents and hazardous air pollutant emissions from other solvents, the propulsion industry is faced with the challenge to implement new environmentally acceptable solvents for use in manufacturing, maintenance, and other processing operations. Hardware cleaning and coating operations in particular, such as motor case and component degreasing, need to be addressed. while all industries face similar challenges, unique concerns such as case-insulation-propellant bonding characteristics in solid rocket motors and explosives safety issues regarding solvent/energetic material compatibility make transition to alternative processes more problematic in many respects for the propulsion industry. Considerable effort is being devoted to solving these problems. In keeping with the mission of CPIA to compile, analyze, and disseminate information pertinent to propulsion technology, this paper will review work in the area and highlight lessons learned concerning the implementation of environmentally acceptable alternative cleaning processes for solid rocket propulsion systems.

Alternate Cleaning Agents

1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113) and 1,1,1-Trichloroethane (TCA) have been used as universal cleaning agents in the manufacture, refurbishment, processing, and maintenance of solid and liquid rocket propulsion systems. In the solid rocket industry, these materials are primarily used to clean motor case and case insulation surfaces that form critical bonded interfaces of the rocket motor structure.

Several types of alternative cleaning agents are being investigated for potential use in these and other applications. These include non-chlorinated organic solvents; aqueous-based solutions employing alkaline (caustic) ingredients, common detergents, or surfactants; semi-aqueous cleaners, such as a high flash point hydrocarbon solvent/surfactant mixture, which are used in a two step cleaning process in conjunction with a water rinse; and emulsion cleaners, such as a terpene/surfactant mixture. Organic solvents generally are effective in removing heavy oils and greases; however, they may pose flammability concerns, occupational health hazards, environmental 2
concerns, and/or hazardous waste disposal complications and thus be subject to additional regulation under the Resource Conservation and Recovery Act (RCRA), or the Volatile

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Organic Carbon (VOC) or Hazardous Air Pollutant (HAP) sections of the 1990 Clean Air Act Amendments (CAAA). Aqueous cleaners often require the use of some form of mechanical agitation such as sonication or spray operation for efficient removal of oils and greases.[1] They are generally safer to handle; however, flash rusting of metal surfaces might be a concern unless corrosion inhibitors are included in the formulation.[1] Semi-aqueous cleaners might offer the advantage (over aqueous cleaners) in some cases of reduced waste water treatment burden associated with the spent solvents.[2] A large variety of these alternative cleaning agents are available commercially. Another approach might be to mix homemade solutions on-site using only active ingredients present in promising commercial cleaners (not including colorants, fragrants, etc.) in order to avoid future supply problems for a qualified material.[3]

Cleaning Agent Selection Methodology and Results

The most important property of a potential alternative cleaning agent is of course cleaning effectiveness for the particular application. Many secondary factors important to the practical use of a cleaner must also be considered, however. Although a particular material may be a very effective cleaner, full scale implementation may be impractical due to environmental or occupational safety regulatory considerations, availability, or cost.

Once a list of replacement candidates has been developed from a survey of technical and vendor literature, a preliminary assessment to estimate cleaner effectiveness, identify additional environmental and occupational safety regulatory concerns, and identify commercial cleaners having similar formulations of active ingredients can be made in order to reduce the list of potential candidates to a reasonable number for actual testing. This can be accomplished almost entirely by reviewing physical/chemical property data and information from the Material Safety Data Sheet. Figure I illustrates a set of solvent parameters and respective selection criteria proposed by Thecal to evaluate replacement cleaners.[2] Data for most of the parameters, with the exception of solvent effectiveness for the particular application of interest and propellant/materials compatibility, should be readily available. Investigators at Arrogate defined a similar pre-screening approach to eliminate candidates with vapor pressures greater than 45 mm Hg at 20 C (the regulatory limit for solvents on the HAP list under the CAAA), highly flammable candidates posing fire hazards similar to acetone or methyl ethyl ketone, and candidates posing extraordinary toxic hazards from vapor inhalation or skin absorption. In addition, Arrogate workers obtained VOC content measurements in order to evaluate candidates with respect to the provisions of the VOC section of the CAAA.[3]

Figure 1. Thecal Solvent Replacement Selection Criteria

SOLVENT CRITERIA

Solvency:	Remove HD-2 Grease 100 gm/gal. at 150°F in less than 30 min
Ozone Depletion:	None
Instability:	Stable at Ambient Conditions
Flash Point:	>150°F Close Cup Method
Vapor Pressure:	20 mm Hg Max at 200°C
PH:	5 to 9
Compatibility:	Causes no Propellant Sensitivity Increase. Causes no Detrimental Effects to Part Being Cleaned
Health Hazard:	<2 NFPA
Flammability:	<2 NFPA
Specific Hazard:	No Other Specific Hazard
Reactivity:	>2
Cost:	Below $20/gal.
Availability:	Large Quantities Available from Several Sources
Recyclable:	Less than a 3 Step Process

Figure 1. Thiokol Solvent Replacement Selection Criteria

The pre-screening process should yield a set of candidates for further experimental evaluation. The experimental evaluation then focuses on the effectiveness of the cleaner for the particular application, and any compatibility or corrosion issues of concern. The most important property of a potential cleaner for solid rocket motor manufacturing operations is the resulting effect on the structural integrity of the motor case-insulation-propellant bond. Bond integrity can be evaluated directly using bond strength tests of sample specimens or indirectly using surface cleanliness measurements of coupon specimens.

Thecal and NASA Marshall Space Flight Center (MSFC) have been working for several years to identify alternate methods to replace TCA vapor degreasing for precision cleaning of the steel motor case during manufacture of the Space Shuttle Redesigned Solid Rocket Motor (RSRM).[1] Preliminary work resulted in the selection of five candidates from each of three solvent classes, organic, semi-aqueous, and aqueous, for formal testing. A comprehensive set of surface cleanliness and bond quality characterization tests were used in a two phase program to evaluate the aqueous candidates. A logic diagram illustrating the aqueous cleaner assessment test protocol for steel and aluminum motor case material substrates with Diala hydrotest oil, magnetic particle inspection solution residue, and HD-2 metal case preservative grease contaminants is shown in Figure 2.1 In 4 phase I of the program, five materials representing the major chemical variations of aqueous cleaners were evaluated as 10% solutions in two cleaning processes, agitated immersion and high pressure spray application (including a rinse/air dry cycle) both at 155 ^{0}F. The cleaners include:

Brulin 815GD - containing ingredients of detergents, nonylphenoxypolyethyleneoxyethanol, and alkaline cleaners;

Metalube 4U - containing nonylphenoxypolyethyleneoxyethanol, aromatic sulfonate detergents, diethyleneglycolmonobutylether solvent, and other additives;

Turco 3878 LFNC - containing a mixture of anionic surfactants;

Remoxide 32-M - a sodium hydroxide solution; and

Detrex EC375d - a potassium hydroxide solution containing surfactants.

Figure 2. Thecal/NASA-MSFC Logic Flow for Cleaner Down-Selection

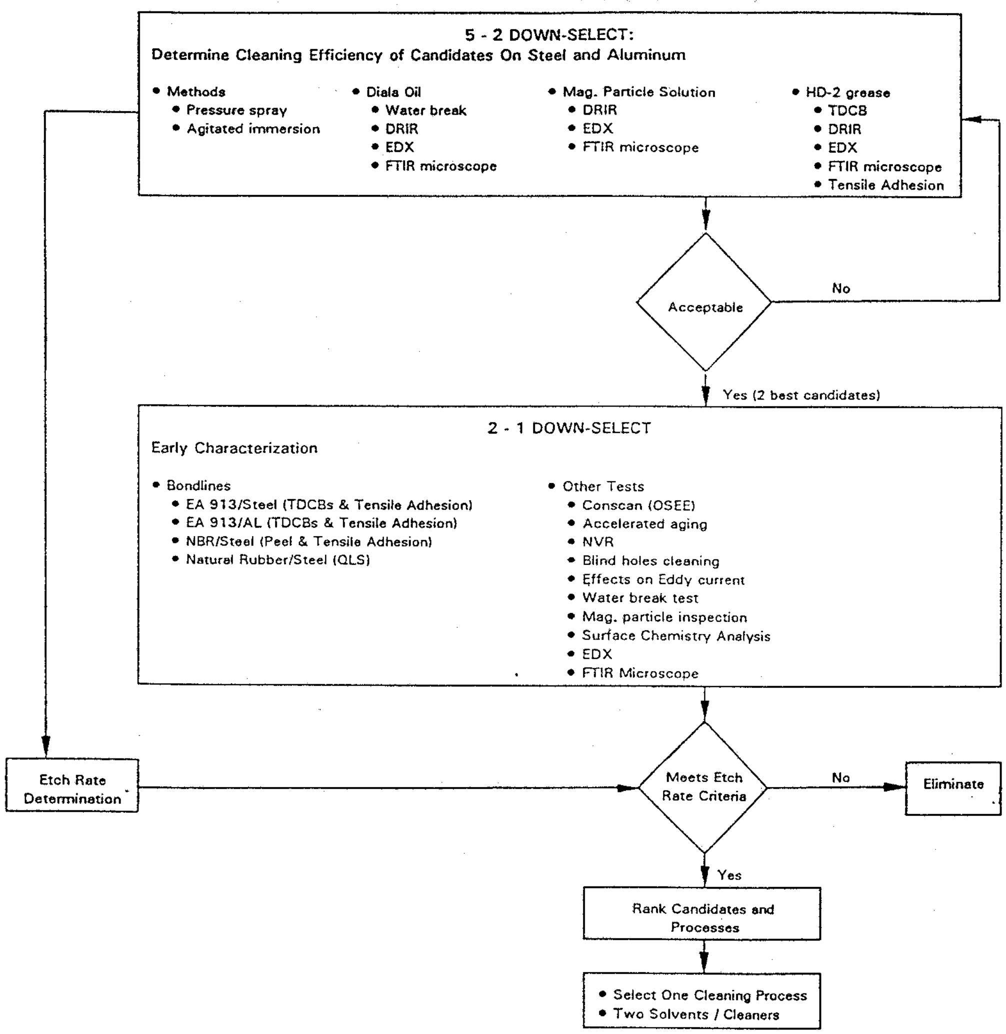

Figure 2. Thiokol/NASA-MSFC Logic Flow for Cleaner Down-Selection

Surface cleanliness of the case material coupons was determined in phase I of the program using diffuse reflectance infrared (DRIR) spectroscopy, Energy Dispersive X-ray Fluorescence (EDX), Fourier Transform Infrared (FTIR) microscopy, and a water break test (in one case). Bonding properties with HD-2 grease contaminant were evaluated using fracture energy (for propagation of a crack through an adhesive bondline) tests with tapered double cantilevered beams (TDCB) according to ASTM D3433-75 and tensile adhesion tests with motor case-epoxy structural adhesive bond specimens. This arrangement did not simulate the actual adhesive-insulation system used in the motor, but was thought to better discriminate surface effects of candidate cleaners. Samples were also analyzed for visible corrosion, and in those instances where corrosion was observed, separate etch-rate immersion tests were performed. Most of the candidates met the performance requirements for tensile adhesion, adhesive bondline crack propagation, and surface cleanliness in both cleaning processes. Three of the materials (Turco, Remoxide, and Dextrex) caused visible aluminum corrosion when used in the immersion process. while this does not directly affect RSRM processing, spray cleaning appeared to be a more forgiving process with respect to corrosion and was selected for further development.

Figure 3 summarizes the results of trials with the five cleaners for removing the metal preservative grease contaminant using a spray process.[1] The results illustrate the value of using an array of structural bond and surface cleanliness tests in determining the best candidate replacements. Based on surface cleanliness measurements (not shown), all of the cleaners appear to perform as well as TCA. From the tensile adhesion data for the steel substrate, it is easy to see that in fact Turco performs much worse than the other candidates. Relying on this data alone, one would conclude that the four remaining candidates provide equivalent bond properties (in fact, they appear to perform as well as TCA). However, the TDCB test results show that Remoxide and Detrex yield much lower fracture energies than the other candidates (and TCA) and that these, as well as Turco, yield very high degrees of adhesive bond failure (indicative of a weak bond). Thus, Brulin and Metalube appear to be the best candidates. Of course, these conclusions require full-scale verification. The results also seem to indicate that some surface cleanliness measurement methods may not adequately gauge real cleanliness and therefore bondability.

Figure 3. Thecal/NASA-MSFC Adhesion Data for Samples Prepared Using Candidate Cleaners to Remove Case Preservative Grease Contamination

Solvent	Criteria	TCA (Typical)	Brulin 815 GD	Turco 3878 LFNC	Metalube 4U	Remoxide 32M	Detrex EC 375 D
Cleaning Process		VD	SIA	SIA	SIA	SIA	SIA
Steel TDCB Tests •Fracture energy (in.lb/in²) •Avg. failure mode (% A/m)	>1.2 - 4.3	11 - 13	13.92(9.2) 1[4]	11.20(11.0) 66[3]	13.61(9.59) 4[4]	5.67(53.0) 94[4]	1.21(39.7) 100[4]
Aluminum TDCB Tests •Fracture energy (in.lb/in²) •Avg. failure mode (% A/m)	>1	3 - 6	15.28(19.5) 3[4]	9.21(59.9) 53[4]	10.36(65.0) 20[4]	NT	NT
Steel Tensile Adhesion Tests •Strength (psi) •Avg. failure mode (% A/m)	>5600 - 6500	7000 - 8000	7666(3.43) 0[7]	2030(6.9) 80[8]	7306(8.78) 4[6]	7156(5.95) 6[7]	7396(5.1) 12[8]
Aluminum Tensile Adhesion Tests* •Strength (psi) •Avg. failure mode (% A/m)	>4000	6000 - 7000	7659(6.93) 3[8]	7270(6.58) 17[6]	7536(4.8) 3[8]	NT	NT

Legend:
Coefficient of variation is identified in parenthesis.
Number of specimens tested is identified in brackets.
NT = reacted violently with cleaner; test deleted.
* Test panels measured 8" x 12" x1/2" (Standard thickness)

SIA = spray-in-air
A/m = Adhesive failure between adhesive and metal
VD = vapor degrease

Figure 3. Thiokol/NASA-MSFC Adhesion Data for Samples Prepared Using Candidate Cleaners to Remove Case Preservative Grease Contamination

In phase II of the program, these two cleaners were further evaluated for performance with all of the representative contaminants in order to down-select the best candidate for process development.[1] Surface cleanliness was evaluated using Optically Stimulated Electron Emission (OSEE), non-volatile residue (NVR), and black light tests. Fracture energy and tensile adhesion data were also confirmed. In general, Brulin performed slightly better than Metalube. Samples were also evaluated using tensile adhesion tests after accelerated aging at 135 ^{0}F and 70% relative humidity for four weeks. Again, Brulin performed slightly better than Metalube although both performed well (only a slight decrease in tensile adhesion strength with aging and still above the performance criteria).

Thus, Brulin was down-selected as the aqueous cleaner of choice. A similar program has been completed to down-select a semi-aqueous cleaner, Jettacin, for comparison with the aqueous cleaner. Further testing proposed in order to select the final candidate includes adhesion tests on specimens simulating the actual motor bondlines both as prepared and after 16 weeks accelerated aging.

In a related program, Thecal/MSFC evaluated two cleaners, Brulin 815GD and Jettacin, as spray cleaners in a three step process involving sequential water blast, spray cleaning, and grit blast unit processes for motor case refurbishment/precision cleaning during processing of the MNASA rocket motor, a quarter scale Space Shuttle test motor used for evaluation of motor materials and components.[4] (Water blasting is being investigated as a refurbishment method for these reusable motor cases while spray clean/grit blasting is being examined to replace the current TCA vapor degrease/grit blast precision cleaning procedure for removing preservative grease from the motor case). A baseline process consisting of a water blast followed by a grit blast was also assessed for comparison. In addition, PF Degreaser, a d-limonene based material, was also evaluated as a hand wipe cleaner for precise cleaning of small areas. OSEE, NVR, and black light surface cleanliness and bond strength measurements were used to evaluate the substitute processes. In this case, however, bond strength was evaluated using tensile adhesion and peel strength tests with specimens representing the actual steel motor case-primer-adhesive-ethylene propylene diene monomer (EPDM) rubber case insulation system. A representative sample configuration used by NASA for these bond tests is shown in Figure 4~S Results of the test program for several different primer-adhesive systems are shown in Figure 5~4 All of the processes examined, including the baseline water blast/grit blast procedure, performed about the same and well above the program performance requirements defined as 100 psi tensile adhesion strength and 12 pli peel strength. In addition, the bond strength tests demonstrated high degrees of cohesive failure in the insulation indicative of structurally sound bonds. Although not further pursued, the water blast/grit blast process is an important example of an apparently effective solvent-free motor case cleaning method.

Figure 4. Steel Case/EPDM Insulation Bond Sample Configuration

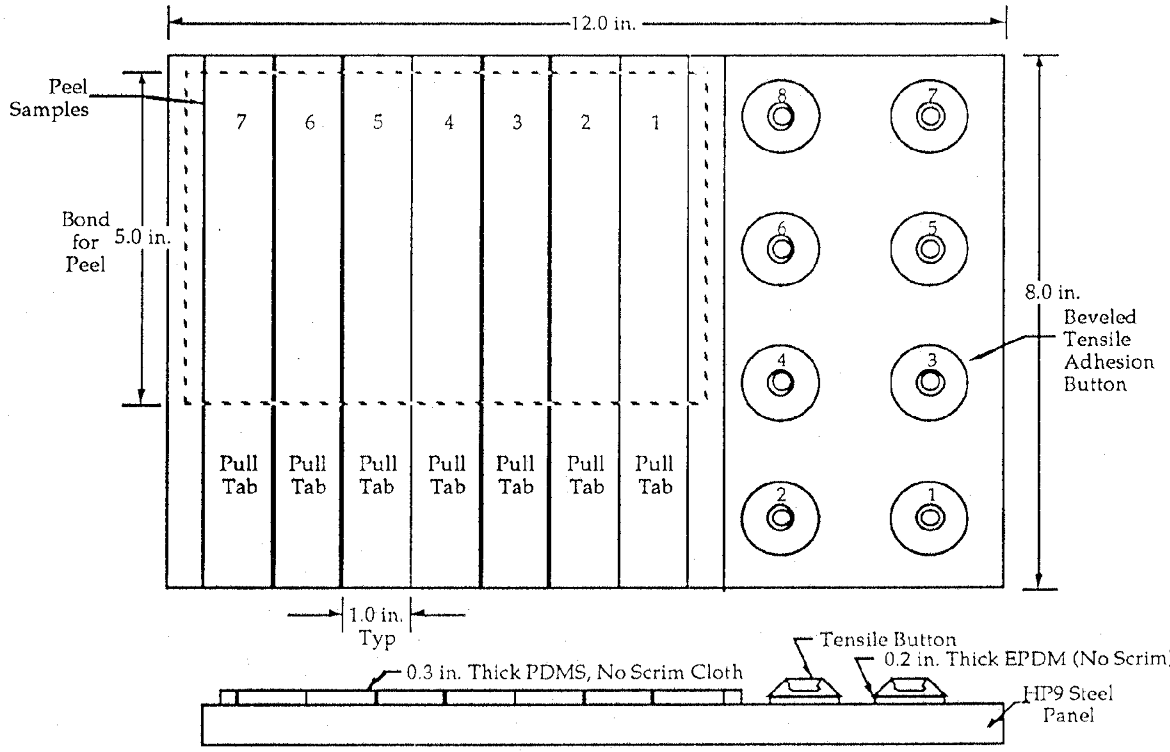

Figure 4. Steel Case/EPDM Insulation Bond Sample Configuration

Figure 5. Thecal Data on Steel Case/EPDM Insulation Bond Strength and Surface Cleanliness Results for Removal of Case Preservative Grease by Candidate Cleaning Processes

Sample Set	1	2	3	4	5	6	7	8	9	10	11	12
Process	WB/SC/GB			WB/SC/GB			HC			WB/GB		
Cleaner	Brulin 815 GD			Jettacin			PF Degreaser			None		
NVR OSEE, cV												
Baseline	596			589			625			680		
Pre NVR	614			605			224			726		
NVR, mg/ft²	1.36			0.33			1.51			1.00		
Primer	CL205	CL205	CL805	CL205	CL205	CL805	CL205	CL205	CL805	CL205	CL205	CL805
Adhesive	CL236 A	CL236 X	CL828	CL236 A	CL236 X	CL828	CL236 A	CL236 X	CL828	CL236 A	CL236 X	CL828
Bond OSEE, cV												
Baseline	699	595	708	731	695	739	658	643	705	631	618	643
Pre bond	546	593	668	657	659	576	243	221	205	608	635	609
Tensile Adhesion Strength, psi	387	393	379	348	397	401	393	384	373	399	376	374
(CoV)	(9.23)	(7.48)	(2.35)	(13.6)	(5.79)	(6.08)	(5.43)	(3.78)	(2.33)	(5.41)	(17.0)	(3.51)
[# tested]	[8]	[8]	[8]	[8]	[8]	[8]	[8]	[8]	[8]	[8]	[8]	[8]
Failure mode (% cohesive)	93	89	96	71	96	98	100	99	95	99	94	99
Average Peel Strength, pli	194	202	192	196	194	189	119	190	190	212	186	204
(CoV)	(2.98)	(5.63)	(7.03)	(5.67)	(7.02)	(7.06)	(10.4)	(5.06)	(6.59)	(2.16)	(6.54)	(4.10)
[# tested]	[7]	[7]	[7]	[7]	[7]	[7]	[7]	[7]	[7]	[7]	[7]	[7]
Failure mode (% cohesive)	99	100	100	99	100	100	100	100	98	100	98	100

Legend:
WB = Water blast
SC = Spray clean
cV = centivolts
GB = Grit blast
HC = Hand clean
NVR = Non-volatile residue
CL = Chemlok
CoV = Coefficient of Variation
OSEE = Optically stimulated electron emission

All panels passed post water blast water break free and black light.
All panels passed post clean black light.

Figure 5. Thiokol Data on Steel Case/EPDM Insulation Bond Strength and Surface Cleanliness Results for Removal of Case Preservative Grease by Candidate Cleaning Processes

The program also included NVR analysis of full scale spray clean/grit blast precision cleaning process demonstration motor cases and tensile adhesion and peel tests on witness specimens processed along with the actual hardware. NVR, tensile adhesion, and peel strength results were comparable (exceeding program requirements) to the previous coupon tests.[4]

Arrogate evaluated a number of candidate methyl ethyl ketone (MEK) replacements for hand wipe cleaning of titanium motor cases before application of the nitrile rubber insulation and TCA replacements for hand wipe cleaning of the inner insulation surface prior to application of the propellant liner and subsequent propellant casting as part of the Minuteman motor remanufacture program.[3] Although this effort was devoted to replacing MEK used to clean the motor case, the lessons learned should be relevant to similar TCA replacement studies. Figure 6 provides a list of candidates selected from the pre-screening assessment including vapor pressure and VOC data.[3] The cleaners were evaluated for effectiveness in removing contaminants representing the effects of handling, such as skin oils and other dirt.

Figure 6. Candidate Replacements Examined by Arrogate

MEK Replacements	Vapor Press. mm Hg	VOC g/l
MEK (Control)	71	806
Deionized Water	15	0
Abrade and Dry Wipe	0	0
10% 2-butoxyethanol	15*	39
MIL-C-43616 Alkaline	Unknown	138
MIL-C-87936 Alkaline	15.0	141
MIAK	4.5	888
MIBK	15.0	798
Diisobutyl Ketone	1.7	805
Dipentene	1.0	838
TCA Replacements		
TCA (Control)	100	1349
Deionized Water	15	0
Dry Wipe	0	0
10% 2-butoxyethanol	15*	39
Axarel tw™	<0.1	804
N-butanol	4.4	810
2-butanol	16.0	808
Amyl Alcohol	2.0	814
Hurrisafe™ (8:1 dil.)	14.2	23
Octowet 75™	15*	--
M-Pyrol™	0.29	1027
0.1% Tergitol™	15*	100
PF Solvent™	<1.0	760

*Estimated as approximately equal to water.

Figure 6. Candidate Replacements Examined by Aerojet

Standard ASTM lap shear test specimens were used to evaluate candidate motor case cleaning materials. Adherends made of the titanium motor case material were coated with representative contaminants, cleaned with the candidate material, and then bonded together using the adhesive-nitrile rubber insulation system used in the rocket motor. The test specimen is shown in Figure 7~3 All of the MEK replacement candidates provided motor case-insulation bond strengths exceeding the design requirement of 118 psi as determined in the ASTM lap shear test and at least two performed as well as the MEK baseline. Two candidates, MIL-C-87936 Alkaline Cleaner and a dry abrade and wipe process, were down-selected for additional testing. The abrade and wipe method involves lightly scouring the surface with grit paper followed by wiping with clean dry cheese cloth. This dry process could be very advantageous in general; however, it may not be effective for heavily contaminated areas and may be undesirable to use with materials that have a special surface treatment.

Figure 7. MEK Replacement Lap Shear Test Configuration

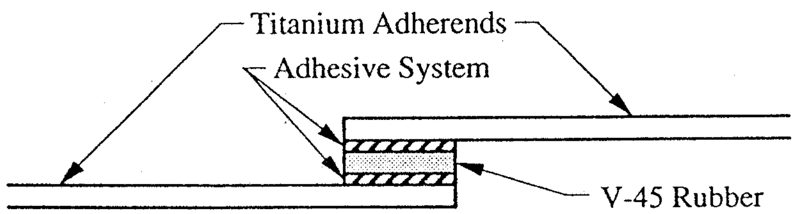

Figure 7. MEK Replacement Lap Shear Test Configuration

Double plate tensile and peel tests were used to evaluate replacements employed for cleaning of the inner surface of the case insulation. Likewise, two candidates were down-selected for further evaluation as TCA replacements to clean the insulation surface. Again, a simple procedure such as wiping with cheese cloth soaked in deionized water followed by a dry cheese cloth wipe was determined to be as effective (exceeding the design requirements of 50 psi and 10 pli, respectively) as TCA in double plate tensile and peel tests of propellant-insulation bond specimens. Another candidate was a 10% solution of 2-butoxyethanol, the active ingredient in many common commercial cleaners. This also performed as well as the TCA control. Impact, friction, and differential thermal analysis (DTA) tests on Minuteman (ammonium perchlorate (AP)/inert binder/aluminum) composite propellant contaminated with each TCA replacement cleaner were also performed to evaluate compatibility for situations such as field maintenance activities where accidental contact may occur. Small decreases in DTA exotherms were observed indicating that some solvent was absorbed into the propellant; however, no significant safety concerns due to incompatibility between the cleaners and AP were evident. D-limonene based solvents such as PF Solvent have also been determined to be compatible with high energy HMX and RDX ingredients used in some solid propellants.[6] Further testing to qualify a selected cleaner (or cleaners) including static firing of a full-scale motor and aging/surveillance studies of full-scale motors was proposed.

Thecal/Huntsville and the U. S. Army Missile Command (MICOM) conducted a program to evaluate a variety of cleaning materials for removing representative contaminants found in a tactical motor production environment from both steel and graphite composite motor case materials and polyisoprene internal insulation.[7] AS part of the program, several surface cleanliness measurement techniques were compared using silicone oil contaminant. OSEE was found to provide the lowest detection limit, 0.5 mg/sq ft, but was only applicable at very low levels of contamination and could not be used with the non-metal graphite composite motor case substrate. Black light analysis provided a detection limit of about 30 mg/sq ft.

Visible light examination was also found to provide similar (to black light) detection levels. IR microscopy provided a detection limit of 16 mg/sq ft. This method was subsequently determined to provide adequate sensitivity to quantify all organic contaminants of interest. EDX was also evaluated to measure inorganic contaminants such as dust and dirt.

Coupons were contaminated with different materials, cleaned by immersion in an ultrasonic bath, and inspected for surface cleanliness using IR microscopy, visible/black light analysis, and EDX. The results for the case materials are shown in Figure 8.~ All of the candidates appeared to be effective in removing lard (analog of skin oil), hydroxy-terminated polybutadiene (HTPB) prepolymer, and dioctyl adipate (DOA) plasticizer. None of the candidates were universally effective in removing the greases, uncured polysulfide liner, or inorganic dirt; however, if the work environment can be controlled to limit the variety of contaminants present, then some of the cleaners may be useable. Another concern, however, is that some of the more effective solvents such as toluene and MEK are subject to additional HAP regulations under the CAAA. Finally, recalling the potential deficiencies of surface cleanliness measurements alone, based on previously discussed NASA work, conclusions regarding actual cleaning effectiveness should be considered preliminary without complimentary bond test data.

Figure 8. Thecal/MICOM Data on Solvent Effectiveness for Representative Contaminants on Steel and Graphite Epoxy Substrates

	Silicone Grease	Lard	R45M	DOA	Polysulfide	Vaseline
Xylene	NE	E	E	E	PE	NE
Methylene Chloride	PE	E	E	E	E	E
PF Degreaser	NE	E	E	E	PE	NE
Mineral Spirits	NE	E	E	E	PE	PE
Methyl Ethyl Ketone	NE	E	E	E	E	PE
d-Limonene	NE	E	E	E	PE	PE
Toluene/Ethyl Acetate	NE	E	E	E	PE	PE
Toluene	NE	E	E	E	PE	PE
M1045	PE	E	E	E	PE	E
KNI-2000*	NE	E	E	E	PE	NE

E = Effective
NE = Not Effective
PE = Partially Effective
*Evaluated on Graphite Epoxy

Figure 8. Thiokol/MICOM Data on Solvent Effectiveness for Representative Contaminants on Steel and Graphite Epoxy Substrates

Other Findings

Additional work on aqueous cleaning has been performed by investigators from Science Applications International Corporation (SAIC) and NASA/MSFC in support of the Space Shuttle Advanced Solid Rocket Motor (ASRM) program.[5] Sample ASRM steel motor case coupons contaminated with metal case preservative grease were successfully cleaned (compared to TCA) using a spray process with either Brulin or Turco solutions, as determined by tensile adhesion and peel tests with specimens representing the steel motor case-Kevlar/EPDM internal insulation system (applied using the same primer-adhesive compounds as the above studies with the RSRM). Tensile adhesion and peel tests were also performed after accelerated aging for up to Six months under conditions of 120 ^{0}F and 80% relative humidity. Essentially no bondline degradation was observed for substrates cleaned with either material.

Arrogate has also been fairly successful in identifying alternate motor ease cleaning methods for its tactical motor operations. A process facility consisting of emulsion solvent cleaning, deionized water rinse, and hot air dry unit operations is currently being qualified.[8]

General Conclusions

Results seem to indicate that adequate substitutes for chlorinated solvents exist for cleaning applications involving solid rocket propulsion systems. The challenge in identifying alternatives appears to be determining the best cleaner for a particular application with minimal resource expenditures. No single replacement appears to be as universally effective, safe, and easy to handle as chlorinated solvents currently in use.

There are a large number of potential candidate cleaners to choose from. Both commercially available solvents as well as homemade solutions might be useful. Some evidence indicates that simple methods such as dry abrasion, water blasting, or wiping with water wet rags may be sufficient in many instances. These techniques would be most desirable, eliminating potential regulatory concerns associated with occupational safety, environmental protection, and solvent waste disposal.

It is apparent that alternate cleaner selection methodologies used by various organizations are somewhat diverse. Some evidence indicates that a very simplistic approach based on surface cleanliness measurements alone may not be adequate to evaluate new cleaning methods. A more desirable situation might be to develop and use a standard assessment protocol to permit easy comparison and utilization of results.

Finally, full scale qualification of new cleaning processes for flight hardware will undoubtedly require significant additional work.

Future Directions

In addition to overcoming the technological issues of identifying alternate cleaning methods, the solid rocket industry faces another challenge of implementing new guidelines and policies to replace government specifications and standards, standard operating procedures, and other instructions that specify the use of ozone depleting chlorinated solvents. Department of Defense 13 (DoD) policy with respect to specifications and standards has recently been overhauled to direct DoD to use performance-based specifications if practicable? Implementation of this new philosophy at the operational level in the solid rocket industry might possibly translate into cleaning guidelines that specify, for example, that "the motor case should be cleaned such that the case to insulation bond strength is greater than (some criteria) in the (selected) test." This approach might be more meaningful than a guideline that specifies the use of a particular material such as a chlorinated solvent without any explanation.

Data which could support this kind of approach to cleaning requirements has been generated in some programs. Figure 9 from work by SAIC and NASA/MSFC shows tensile adhesion and peel strength data as a function of substrate contamination (case preservative grease) level for specimens representing a steel motor case-EPDM insulation bond using a standard primer-adhesive System.[5] For this system, no significant degradation in bond strength was observed up to 500 mg/sq ft grease contamination, although the bondline failure mode changed from cohesive to adhesive in the range of 300-500 mg/sq ft. In addition, no significant degradation in bond strength due to 50 mg/sq ft contamination levels was observed for samples aged at 120 ^{0}F and 80% relative humidity for up to six months. Similar tensile adhesion and peel strength data was obtained by Thecal and MICOM for steel case-HTPB liner bond specimens with DOA plasticizer, polysulfide liner, and silicone grease contaminants.[7] Tensile adhesion results showed no degradation of the bond for contaminant levels up to 80 mg/sq ft with DOA and polysulfide, while minimal contamination with silicone grease caused a significant loss in strength. Some reduction in strength was observed in peel data shown in Figure 10.- It is obvious that little silicone grease contamination can be tolerated. For some contaminants, such as the DOA and polysulfide in this study and the case preservative grease in the NASA study, however, the data might be valuable in assessing cleaning requirements with respect to appropriate performance criteria. This type of information may also allow the relaxation of stringent cleanliness requirements thus enabling the elimination of precision cleaning, or more likely, the use of less aggressive cleaning processes using cleaners such as distilled water or other more benign materials.

Figure 9: SAIC/NASA-MSFC Data on Sensitivity of Steel Case/EPDM Insulation Bond Strength to Case Preservative Grease Contamination

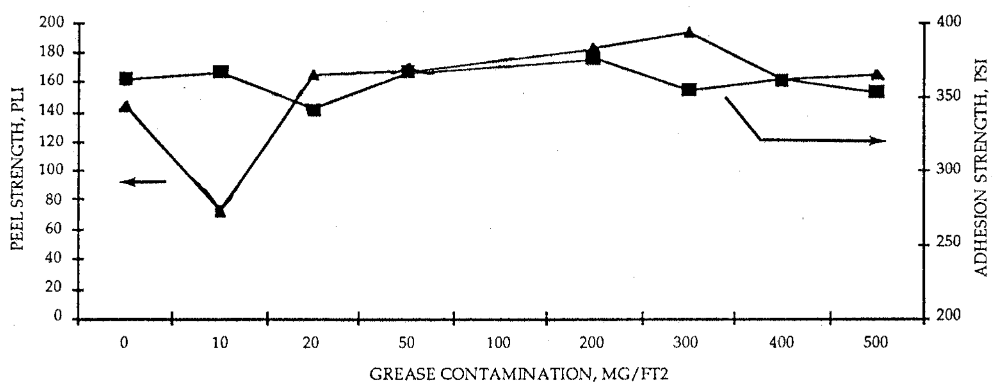

Figure 9: SAIC/NASA-MSFC Data on Sensitivity of Steel Case/EPDM Insulation Bond Strength to Case Preservative Grease Contamination

Figure 10:
Thecal/MICOM Data on Sensitivity of Steel Case/HTPB Liner Bond Strength to Various Contaminants

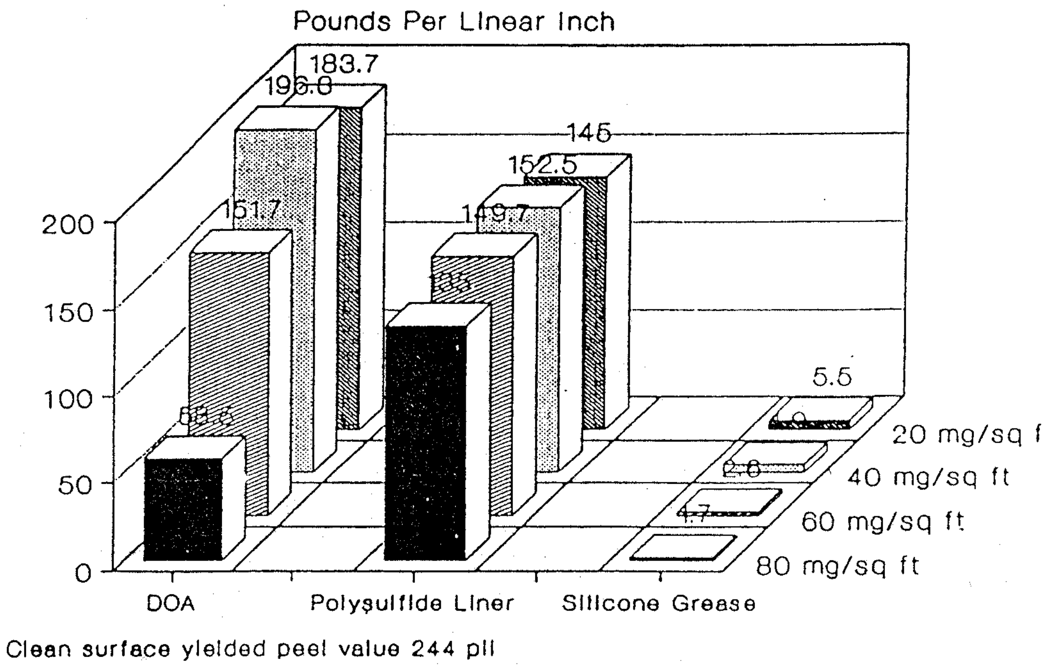

Figure 10: Thiokol/MICOM Data on Sensitivity of Steel Case/HTPB Liner Bond Strength to Various Contaminants

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