# EVALUATION OF PROPYLENE CARBONATE IN AIR LOGISTICS CENTER (ALC) DEPAINTING OPERATIONS

by

Foster Wheeler Enviresponse, Inc. Edison, NJ 08837

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# **FOREWORD**

Today's rapidly developing and changing technologies and industrial products and practices frequently carry with them the increased generation of materials that, if improperly dealt with, can threaten both public health and the environment. The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the nation's land, air, and water resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. These laws direct the EPA to perform research to define our environmental problems, measure the impacts, and search for solutions.

The Risk Reduction Engineering Laboratory (RREL) is responsible for planning, implementing, and managing research, development, and demonstration programs to provide an authoritative, defensible engineering basis in support of the policies, programs, and regulations of the EPA with respect to drinking water, wastewater, pesticides, toxic substances, solid and hazardous wastes, and Superfund-related activities. For this project, the EPA applied the resources of its Waste Reduction Evaluations at Federal Sites (WREAFS) Program, with the support of the Strategic Environmental Research and Development Program (SERDP) to provide assistance to Tinker Air Force Base, Oklahoma.

The Pollution Prevention Research Branch of the Risk Reduction Engineering Laboratory has instituted the WREAFS Program to identify, evaluate, and demonstrate waste minimization opportunities in industrial and military operations. This report examines propylene carbonate solvent blends as a substitute for methyl ethyl ketone in ALC depainting operations.

E. Timothy Oppelt, Director Risk Reduction Engineering Laboratory

# **ABSTRACT**

This report summarizes a two-phase, laboratory-scale screening study that evaluated solvent blends containing propylene carbonate (PC) as a potential replacement for methyl ethyl ketone (MEK) in aircraft radome depainting operations. The study was conducted at Oklahoma City Air Logistics Center (OC-ALC) at Tinker Air Force Base (TAFB). TAFB currently uses MEK to depaint B-52 and KC-135 aircraft radomes in a ventilated booth. Because MEK is highly volatile, many gallons vaporize into the atmosphere during each depainting session. Therefore, the U.S. Environmental Protection Agency (EPA) is supporting studies to identify effective, nonvolatile, less toxic substitutes for MEK.

The first phase of this study screened the performance of three solvent blends provided by Texaco Chemical Company. These blends contained varying concentrations of PC, n-methyl pyrrolidone (NMP), dibasic ester (DBE), and other organic solvents. The performance of each blend was compared with that of MEK—both by the paint removal time and by a visual estimate of the amount of paint removed without any visible substrate damage (removal efficiency). The best performer was PC Blend 2, which contained 25 percent PC, 50 percent NMP, and 25 percent DBE. This solvent blend was then compared with MEK during the second phase of this study. The Phase 2 tests measured paint removal time and efficiency, paint adhesion, flexural properties, weight change of the substrate after paint removal and hardness of unpainted substrate test panels.

Phase 2 test results revealed that PC Blend 2 performed favorably in comparison with MEK in removing paint from the fiberglass/epoxy (F/E) test panels and in subsequent paint adhesion tests, despite an indication of possible substrate damage. A preliminary economic analysis performed on PC Blend 2 estimated TAFB would save over \$30,000 the first year of operation by replacing MEK with PC Blend 2. PC Blend 2 should continue to be evaluated as a substitute in the TAFB radome depainting operation. Additional qualification testing, required by the Air Force, and a full-scale demonstration project are recommended before implementation.

This report is submitted in fulfillment of EPA Contract 68-C9-0033 under the sponsorship of the U.S. Environmental Protection Agency Risk Reduction Engineering Laboratory (RREL). This report covers a period from February to September 1993 and work was completed as of September 30, 1993.

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# ABBREVIATIONS AND SYMBOLS

ALC Air Logistics Center

ASTM American Society for Testing and Materials

DBE dibasic ester

EPA U.S. Environmental Protection Agency

°F Degrees Fahrenheit F/E fiberglass/epoxy

FWDC Foster Wheeler Development Corporation

FWEI Foster Wheeler Enviresponse, Inc.

H<sub>2</sub>O water

HP horsepower in. inches L liter

ib, pound force

MEK methyl ethyl ketone

mil unit of thickness; 1 mil = 0.001 inches

mm/mm millimeter/millimeter
MSDS material safety data sheet
NMP n-methyl pyrrolidone

OC-ALC Oklahoma City Air Logistics Center

PC propylene carbonate psi pounds per square inch

PPRB Pollution Prevention Research Branch

QAPP Quality Assurance Project Plan

RREL Risk Reduction Engineering Laboratory
RSD percent relative standard deviation
SEM scanning electron microscope

SERDP Strategic Environmental Research and Development Program

SS stainless steel

TAFB Tinker Air Force Base

TCLP Toxicity Characteristics Leaching Procedure

UCT universal coordinated time

WREAFS Waste Reduction Evaluations at Federal Sites

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# INTRODUCTION

This report summarizes a two-phase, laboratory-scale screening and measurement study to evaluate the potential of substituting solvent blends containing propylene carbonate (PC) for methyl ethyl ketone (MEK) in radome depainting operations at Tinker Air Force Base (TAFB). By eliminating the use of MEK, TAFB will reduce its use of this hazardous substance and the resulting problems associated with its disposal and air emissions.

This project is supported by the U.S. Environmental Protection Agency's (EPA) Risk Reduction Engineering Laboratory (RREL). In keeping with the Agency's responsibility to advise and cooperate with other federal departments on environmental risk reduction, the Pollution Prevention Research Branch (PPRB) has managed a technical support effort known at the Waste Reduction Evaluation at Federal Sites (WREAFS) Program. WREAFS was established to conduct research, develop, and demonstrate opportunities to reduce the generation of waste from federal activities. Through WREAFS, the EPA provides support to federal facilities in researching, developing, and demonstrating pollution prevention technologies and transferring lessons learned from the federal community.

Since 1988, WREAFS has conducted research and development efforts under funding from both EPA and other federal agencies via interagency agreements. In 1990, Congress established the Strategic Environmental Research and Development Program (SERDP) as a multiagency effort to support environmental research and development programs. One of the objectives of SERDP that is addressed by this project is the identification of technologies for national defense purposes that assist government and private sector organizations in addressing environmental concerns. This report is a deliverable of the WREAFS/SERDP programs.

This project also focuses on EPA's 33/50 Voluntary Reduction Program, whose goals are to reduce generation of 17 hazardous substances by 50 percent by the end of 1995 based on the 1992 baseline. One of those 17 chemicals is MEK, a solvent used in aircraft maintenance operations such as cold cleaning applications for removing resins, coatings, and adhesives.

From previous research and background documents (2), RREL has identified PC as a possible alternative to MEK. To date, PC has not been performance-tested as a substitute for MEK in radome depainting operations. The primary objective of this project was a proof-of-concept study to evaluate the performance of solvent blends containing PC as a substitute for MEK.

The program was conducted in two phases. The first phase of the study screened three solvent blends containing PC in varying concentrations; all three blends were provided by Texaco Chemical Company (Texaco). Texaco chose a blend rather than pure PC due to the fact PC's properties would be enhanced by the addition of other solvents. The performance of each blend was compared to that of MEK as measured by the paint removal efficiency and the amount of visible substrate damage from radome test panel specimens provided by Oklahoma City Air Logistics Center (OC-ALC).

Since the PC solvent blends showed adequate paint removal performance from the radome test panel specimens, the most effective solvent blend was chosen for additional testing and comparison to MEK. The second phase included radome test specimen depainting using a simulated depainting spray procedure to determine the paint removal time and efficiency as compared to the current procedure at OC-ALC. The tests performed on the specimens determined the effect of the PC solvent blend and MEK on the composite substrate and included flexural properties, hardness, weight change, and paint adhesion. Microphotographs of the surface and interface of the failed flexural test panels provided visual evidence of possible damage or lack of damage between the radome fiber and epoxy matrix.

The sections that follow provide summarized conclusions of the study and recommendations for additional studies to confirm the potential use of a PC solvent blend as a viable substitute for MEK and provide steps for its introduction into the existing TAFB depainting operation. The report discusses the background and methodology of the overall test program. Test results, cost and quality assurance analyses, and references are provided. Material Safety Data Sheets (MSDS) for the solvents, specific test methodologies and protocols, and definitive laboratory test results are provided in the appendices.

## CONCLUSIONS

The evaluation results indicate that PC Blend 2 is a potentially viable solvent for replacing MEK; further evaluation will be required for proper qualification of its use in TAFB's radome depainting operations.

During screening, the three PC blends provided by Texaco removed paint from condemned radome specimens and appeared to perform better than the pure MEK. PC Blend 2 performed the best and was, therefore, selected for further evaluation tests. These tests included depainting simulation, hardness, flexural properties, paint adhesion, and weight change.

Depainting simulation results were invalid because six of the eight test panels contained a different paint system from the one TAFB currently uses. Although the paint system is no longer used, PC Blend 2 removed the paint faster and more completely than the MEK. The two panels painted with the current radome paint system were tested with PC Blend 2 and were depainted in less than an hour. Because of the inconsistencies in the test panels, results of this test were disregarded and not used in subsequent tests.

To obtain removal time and efficiency for depainting, panels painted with the current paint system were depainted in the simulation unit and then repainted for the paint adhesion test. PC Blend 2 removed the paint in slightly more time than MEK and required a little more scraping for total removal. Both solvents removed 100 percent of the paint. After depainting, the panels were observed indicating possible removal of the top layer of resin by PC Blend 2. The impact of this observation requires further study.

Results from the hardness test indicates that neither PC Blend 2 or MEK embrittle the fiberglass/epoxy (F/E) substrate. The solvents did not affect the flexural properties. Examination of the failed test panels with a scanning electron microscope (SEM) indicated no significant damage to the fibers or the fiber matrix interface. The weight change test panels exhibited weight loss for both solvents after immersion for four hours, although the amount was negligible. The MEK-immersed panels indicated lower weight loss. The paint adhesion rating for both MEK and PC Blend 2 represented complete paint adhesion after a depainting/painting cycle.

Due to limitations of the test procedure, test panels used in the hardness, flexural properties, and weight change testing were cut from prepreg sheets; test panels for screening, simulated depainting, and paint adhesion testing were cut from condemned F/E radomes. However, since the F/E substrate is the same for all tests and there is direct comparison between MEK and PC Blend 2 for each test, it is assumed that conclusions for all tests are valid.

# RECOMMENDATIONS

Based on the tests performed for this evaluation, PC Blend 2 is a potential substitute for MEK in TAFB's radome depainting operation. Since the evaluation tests indicate that PC Blend 2 may damage the substrate, further study is required before full-scale implementation of this solvent can occur. Possible alternative future courses of action are:

- Further evaluation of the potential adverse effects of the solvent on the substrate, and either
- Reformulation of the solvent blend to eliminate or reduce any identified damage, or
- Full-scale demonstration project of PC Blend 2 in TAFB's radome depainting operation if no damage is identified.

The first alternative entails evaluation of whether the extent of resin substrate removal would preclude the use of the PC Blend 2. This evaluation requires thickness measurements of the resin substrate. The depainting operation should be performed over several depainting/painting cycles; thickness of the resin coat should be determined and compared with the minimum allowable thickness to determine suitability of the PC Blend 2. These tests should be repeated with MEK from the sump to permit valid comparison.

Texaco suggested the second alternative, reformulation of the blend to prevent or lessen the substrate damage. This requires tests to determine which blend component damages the substrate, followed by testing of a reformulated blend. The component that causes damage may be isolated by formulating three solutions: PC/NMP, PC/DBE, and NMP/DBE, each with the same concentration as in PC Blend 2. Radome panels would be tested with these three solutions to determine paint removal efficiency and substrate damage. PC Blend 2 could then be reformulated and tested with less of the substrate damaging component.

In a full-scale demonstration project, several areas should be addressed. One area is the disposition of the spent solvent. The EPA does not identify the three components of PC Blend 2 (PC, NMP, or DBE) as hazardous in 40 CFR, Part 261. According to MSDS, the three components are all biodegradable and can be sent to an industrial treatment plant, if local regulations allow. However, this should be tested on a small scale before actual discharge of spent solvent to TAFB's treatment plant. E.I. duPont de Nemours (DuPont) recommends DBE, as a pure component, be recycled in a vacuum distillation unit which indicates that PC Blend 2 can also possibly be distilled and reused. Also, to ensure that the spent PC Blend 2 is disposed properly, testing procedures, such as Toxicity Characteristics Leaching Procedure (TCLP), should be performed on the spent solvent to confirm the nonhazardous designation.

Also to consider is the logistics of retrofitting the existing MEK operation. The three components are not a hazard concern, as is MEK. However, these solvents have some equipment limitations, especially with pump seals. Incompatible materials are Buna-N, Viton®, and Hypalon. Recommended materials are Teflon™, polyethylene, and ethylene propylene rubber. In addition, PC Blend 2 is not volatile and will remain in the

sump for reuse until operators determine it is ineffective. A filtration device may have to be installed in the sump to prevent clogging the pump and piping by paint chips. Depainting booth operators should continue to wear personal protective equipment including respirator, chemical resistant clothing, eye protection, and gloves.

In addition to equipment, the operation must be slightly altered. The time required to remove paint with PC Blend 2 is relatively comparable to MEK. As with the current MEK operation, manual scraping will probably be necessary when using PC Blend 2. However, in the current operation, the MEK evaporates quickly from the radome surface, avoiding any need for a drying step. PC Blend 2 has extremely low volatility and will not quickly evaporate from the surface. An additional step to remove solvent from the surface will have to be added to the depainting operation. The radomes could be heated to evaporate the solvent from the radome surface, by either using infrared panels, moving them to a heated booth, or by spraying them with a heated air curtain. Texaco has also suggested using a volatile solvent, such as dipropylene glycol monomethyl ether, which will dissolve the PC Blend 2 and evaporate. As of this writing, dipropylene glycol monomethyl ether is not characterized as an ozone depleter or hazardous air pollutant under the Clean Air Act. Use of this chemical requires personal protective equipment as in the depainting operation. It is important that any solvent used for this purpose should, as a minimum, be nonhazardous as a solid waste and air pollutant. Part of a qualification program for PC Blend 2 may include testing a solvent for drying.

# **BACKGROUND**

# **EXISTING DEPAINTING PROCEDURE**

Aircraft radomes are painted for appearance and protection from the environment. These protective coatings applied to the radome must not adversely affect the operation of the enclosed equipment, such as radar. Military aircraft radomes are typically fabricated using fiberglass and epoxy or polyester composite material. Large radomes usually consist of a honeycomb structure with a thin coating. The protective coating on F/E radomes consists of a primer and polyurethane rain-erosion coating, followed by a polyurethane antistatic topcoat. Removal of these protective coatings, without damage to the F/E substrate, presents many challenges.

Currently, the radome depainting task at TAFB is performed using MEK. Radomes handled by TAFB include those from KC-135, EC-135, B-52, B-1, and E-3A aircraft. A radome piece can either be a nose radome, "top hat," or side radome. The nose radome, located on the tip of the aircraft, is an oblong dome shape. The "top hat" is the piece located above the nose radome on the aircraft; side radomes are located on the sides of the nose radome. Paint systems vary for different radomes.

Radomes requiring repair are removed from the aircraft and sent to the Composite Repair Facility (TAFB Building 2211), which houses the radome depainting operation. Figure 1 depicts TAFB's radome depainting operation. In this operation, paint is removed in a large ventilated booth by subjecting it to a MEK shower to loosen the paint. The MEK attacks the primer via scribed breaks in the topcoat. According to operators, the paint starts to bubble after about 30 minutes of continuous showering. As the primer dissolves, the topcoat is flushed away from the radome by the MEK shower.

Topcoat residue is filtered from the MEK and flows to a sump for recycling back to the spray header. The operation typically takes 1½ to 3 hours. According to TAFB, a large percentage of the MEK is lost to the atmosphere through the booth exhaust system. After the MEK depainting process, the remaining paint residues are removed by hand sanding. Topcoat chips are captured in a sump and disposed as hazardous waste. In 1991, 719 pounds of topcoat chips were disposed, and an estimated 8,250 gallons of MEK evaporated to the atmosphere.

Because of its properties, MEK is an ideal solvent for the radome depainting operation. MEK lowers the viscosity in paints and adhesives and is relatively inexpensive. However, MEK exhibits the following chemical and physical properties which contribute to its classification as a hazardous material:

- Flammable
- 1.8% lower explosive limit
- 20°F flash point

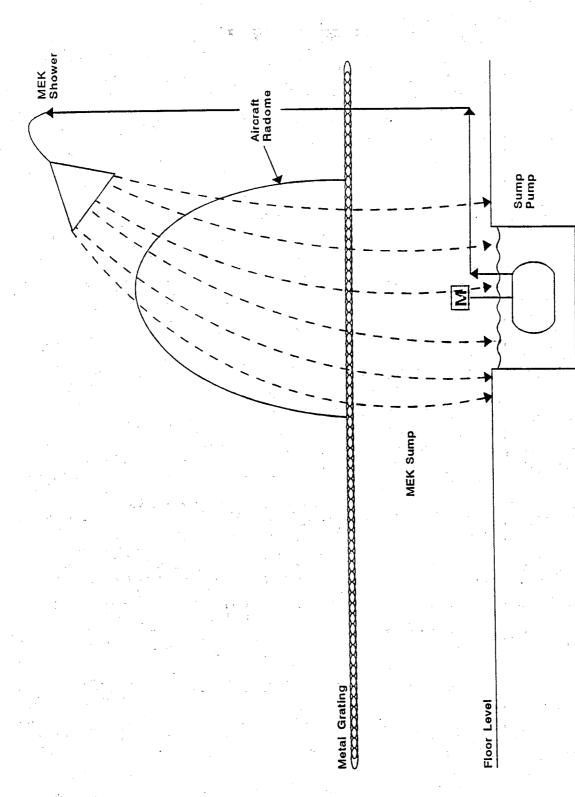


Figure 1. TAFB radome depainting operation.

- High vapor pressure
- High evaporation rate
- · Not miscible with water to any great extent

For these reasons, EPA and TAFB want to eliminate the use of MEK and replace with a nonhazardous or less hazardous alternative.

## PC SOLVENT BLENDS

Texaco provided the solvent blends containing PC. The solution compositions (by weight) are as follows:

#### • PC Blend 1

331/4% propylene carbonate 331/4% n-methylpyrrolidone 331/4% dibasic ester

#### PC Blend 2

25% propylene carbonate 50% n-methyl pyrrolidone 25% dibasic ester

## PC Blend 3

15% propylene carbonate

15% n-methylpyrrolidone

15% methyl isoamyl ketone

40% dibasic ester

15% dipropylene glycol monomethyl ether

Texaco provided the physical properties of these blends, summarized in Table 1, along with properties of MEK. Texaco also provided the MSDS for these blends, included in Appendix A. An MSDS for MEK is also included. Characteristics of the three major components of the solvent blends are discussed below.

TABLE 1. SOLVENT PROPERTIES SUMMARY

Solvent/Blend	Flash Point (°F)	Vapor Pressure (mm Hg @ 68°F)	Specific Gravity (@ 77°F)	Viscosity (cps @ 77°F)
MEK	20	70.21	0.8023	0.41
PC Blend 1	215	0.18	1.0985	2.175
PC Blend 2	210	0.20	1.0797	2.105
PC Blend 3	144	6.51	0.9947	1.919

PC is a clear liquid which has excellent solvent properties of high flash point and low toxicity; a low evaporation rate eliminates the concern of air emissions. PC is typically used as a solvent in such applications as surface coatings, dyes, fibers, and plastics.

NMP is a highly-polar, general-purpose, organic solvent. Like PC, NMP also has a low evaporation rate and high flash point. Typical applications of NMP include paint removal and engine cleaning.

DBE is a product manufactured by DuPont and is a mixture of dimethyl succinate (24 percent by wt.), dimethyl glutarate (60 percent by wt.), and dimethyl adipate (15 percent by wt.) and <1 percent of methanol and water. DBE is a clear, colorless liquid with a mild odor. It is readily soluble with alcohols, ketones, ethers, and most hydrocarbons, and only slightly soluble in water and higher paraffinic hydrocarbons. DBE is stable at normal ambient conditions and, therefore, can be handled and stored without need for precautions to prevent auto-oxidation or hydrolysis. DBE is principally used as an industrial cleaning solvent and paint remover.

#### TEST PROGRAM METHODOLOGY

An EPA Level IV Quality Assurance Project Plan (QAPP) was approved and used as the basis for the propylene carbonate solvent evaluation.

The overall objective of this study was to determine whether a propylene carbonate solvent blend removes paint from a radome without damage to the F/E substrate.

Prior to this program, Texaco laboratory personnel conducted an independent prescreening exercise. FWEI's evaluation consisted of a screening phase and an evaluation phase. The prescreening and two study phases are discussed below.

#### PRESCREENING

For the prescreening, Texaco developed a computer program to predict properties of various solvent blends and to select potential blends, based on criteria entered into the program. Each blend contained PC. A blend was chosen rather than pure PC to enhance paint removal properties of PC. The selection criteria for the PC solvent blends was developed by FWEI and included:

- Nonhazardous mixture
- Low volatility
- · Safe to handle
- Flash point > 140°F
- Biodegradable

Texaco's database produced over 60 solvent blends. The blends were made up and tested on panels cut from a condemned radome by applying solvent to the panel's surface with an eyedropper.

In addition, commercial paint removers Zip Strip and Klean Kutter were selected as standards. According to Texaco, these paint removers softened paint in 1-2 minutes and scraped off fairly easily. The solvent compositions are:

	Zip Strip	K	lean Kutter
79%	methylene chloride	35%	methylene chloride
9%	mineral spirits	20%	toluene
9%	ethanol	20%	acetone
3%	methanol	25%	methanol

PC Blends 1 and 2 removed the paint as well as, or better, than Zip Strip and Klean Kutter. PC Blend 3 and two other formulations attacked the paint less vigorously, requiring slightly more scraping effort to remove it. The other two formulations were:

25% propylene carbonate n-methylpyrrolidone methyl isoamyl ketone
25% propylene carbonate n-methylpyrrolidone n-amyl acetate

The three solvent blends selected for testing were made up in one-gallon batches and sent to the TAFB laboratory.

FWEI conducted Phase 1 screening procedures at TAFB. Phase 2 was the evaluation of substrate damage and paint adhesion of the solvents. The tests are described in the following sections and summarized in Table 2.

#### **SCREENING PROCEDURES**

The purpose of the screening test was to determine if any of the propylene carbonate blends remove paint as effectively as MEK and if so, to select the best of the three blends for more detailed testing.

Screening was performed at TAFB Chemical Laboratory in Building 3001. The screening phase involved qualitative testing of the PC solvent blends to determine their effectiveness in removing paint from radome panels. Pure MEK was evaluated for comparison. The screening test was performed in a laboratory hood.

The three solvent blends and MEK were each poured into two liter beakers with enough volume to ensure total immersion of the test panels. TAFB provided approximately two in. square test panels made of F/E honeycomb from a condemned radome. The panels were unscribed. The radome was a "top hat" section from a B-52 aircraft. The back side and edges of the test panels were masked with aluminum foil tape to prevent damage to the honeycomb structure. The panels were immersed face down in the bath.

After 30 minutes, the panels were removed and visually examined for signs of bubbling of the paint coating. Observations were recorded. The specimens were put back into the bath and examined at 1, 2, 4, 8, and 24 hour intervals after initial immersion. During each examination, the amount of paint removal effectiveness was estimated and recorded. As recommended by the TAFB Materials Group, the panels remained in the bath for 24 hours, even if the paint was removed prior, to provide some visual observation on any possible damage to the substrate.

# **EVALUATION TESTS**

Although the three solvent blends removed paint effectively, PC Blend 2 performed best during the screening procedure and was selected to undergo further testing. The testing included a simulation of the depainting procedure to determine the time and removal efficiency for a spray operation, with a more accurate representation of the depainting operation. TAFB chose other tests to analyze the solvent's effects on the substrate. Tests evaluated hardness, flexural properties, paint adhesion, and weight change. Foster Wheeler Development Corporation (FWDC) personnel performed the tests at FWDC's John Blizard Research Center in Livingston, New Jersey.

TABLE 2. LABORATORY TESTS AND SAMPLE REQUIREMENTS SUMMARY

Test	ASTM Method	Substrate	Paint	Solvent	Number of Panels Tested
Screening	N/A	F/E with honeycomb	Current paint system	PC Blend 1 PC Blend 2 PC Blend 3 MEK MEK/H <sub>2</sub> O	4444
Depainting Simulation	N/A	F/E with honeycomb	Neoprene based paint system	PC Blend 2 MEK	2 4
			Current paint system	PC Blend 2 MEK	0
Hardness	D2583-87	F/E prepreg laminate	Unpainted	PC Blend 2 MEK	4 4
Flexural Properties	D790-92	F/E prepreg laminate	Unpainted	PC Blend 2 MEK	လသ
Paint Adhesion	D3359-92a	F/E with honeycomb	Current paint system	PC Blend 2 MEK	4
Weight Change	N/A	F/E prepreg laminate	Unpainted	PC Blend 2 MEK	4 4
		316 SS (Standard)	N/A	PC Blend 2 MEK	4

# Simulated Depainting Procedure

The simulated depainting procedure was set up for both PC Blend 2 and MEK, in two separate, identical units. The apparatus consisted of a Kleer-flo Cleanmaster parts washer fitted with a ¼ in. diameter spray nozzle. The flow rate of the parts washers was approximately seven liters per minute. Both units had to be modified with a 0.07 HP orbital magnetic drive centrifugal pump because the original pump's seal material was incompatible with both MEK and PC. The nozzle sprayed the two in. square test panels made of F/E honeycomb cut from a condemned B-52 radome. The panels were initially masked with aluminum foil tape on the edges and back to protect the honeycomb structure, but after it was noticed the tape was holding down the edges of the paint and interfering with the paint removal process, the tape was pulled back from the front edges of the panel.

The times required for the solvents to bubble and totally remove the paint were recorded. Along with the time, a qualitative judgment of paint removal was removed was recorded at various time intervals. After bubbling, the paint was removed by hand or a blunt-edged wooden spatula.

# **Hardness**

Hardness tests were performed in accordance with ASTM Test Method D 2583-87. Four test panels were used for each solvent. The panels were two in. squares cut from an unpainted F/E prepreg sheet used for another project at TAFB. A prepreg panel is made of fiberglass fibers that have been soaked or impregnated with a polyester or epoxy resin. The surface of a prepreg panel is identical to an unpainted F/E radome surface. Fabricated prepreg panels are used in tests where an actual radome cannot supply the required surface conditions or dimensions. Panels from the prepreg sheet had to be used for this test because the Barcol Impressor requires a flat surface for measurement. The condemned radome panels available for this experiment had a slight curvature, making them unacceptable for this test.

The hardness test determines indentation hardness of a material using a Barcol Impressor, Model No. GYZJ 934-1. Indentations are made on the specimens and the hardness measured. In accordance with ASTM D 2583, ten measurements were made on each of the four panels, both before and after contact with the solvent. The measurements are no less than ½ in. from the edge or from other measurements. The Barcol Impressor was checked for calibration prior to each set of measurements with standard test disks supplied with the impressor. The panels were sprayed with solvent in the depainting simulation unit for two hours, and gently wiped dry with a paper towel.

# Flexural Properties Test

The flexural properties test was performed in two parts. The first part measured flexural properties of the test panels in accordance with ASTM Test Method D 790-92, Test Method I, Procedure A. Five test panels were used for each of the solvents. The test panels are made from an F/E prepreg sheet used for another project and were provided by TAFB and cut to test specifications. Panels from the prepreg sheet were used instead of condemned radome pieces because flat panels with 1" x 3" dimensions were required for this test. The condemned radome panels available for this experiment had a slight curvature, making them unacceptable for this test.

This ASTM test measured the flexural strength of the panel and allowed inspection to determine if any damage by the solvents occurred to the interface of the F/E and laminate structure. The panel was first subjected to the solvent in the depainting simulation unit for two hours. After drying one hour, the panel was subjected to load in a tensile machine until breakage of outer fibers occurred or when a maximum strain of 0.05 mm/mm was reached.

During the second part, the failed test panels were examined with a scanning electron microscope (SEM). To prepare for SEM examination, the panels were cut into one in. squares with the failure break in the center of the panel. The panels were then mounted onto an aluminum stud using carbon paint, which provides a conductive bond between the stud and the panel. The test panels were then sputtered with gold in a vacuum chamber to make them conductive. After about ten minutes of sputtering at various angles to ensure the gold is applied underneath the fibers, the panel is then placed in another vacuum chamber and viewed by the SEM. The surface was examined at 30X, 300X, and 1200X magnification and the cross-section was examined at 1200X magnification to observe any fiber/matrix interface disbonding. Microphotographs were made at each magnification for representation of the conditions observed. Test panels not exposed to any solvent were also viewed by the SEM to obtain a baseline comparison.

# **Paint Adhesion**

Paint adhesion testing was performed in accordance with ASTM Test Method D 3359-92a, Method A. Four unscribed F/E with honeycomb test panels were used for each solvent. The test panels were painted with TAFB's current paint system.

After paint removal with the solvent in the depainting simulation unit, the test panels were repainted by TAFB personnel with the rain-erosion coating system currently applied to B-52 radomes. The same thickness of coating and painting procedure used for actual radomes was applied to these test panels, approximately a 10 to 12 mil coat. For the tape adhesion tests, two incisions were made in the panels to the substrate layer. A pressure sensitive tape was then applied to the intersection of the cuts for a period of 90 ± 30 seconds. The tape was one in. wide PermaCel 99<sup>m</sup> (manufactured by PermaCel, New Brunswick, NJ). After tape removal, the X-cut area was visually inspected. The adhesion is rated according to a scale of 0A (removal beyond area of X) to 5A (no peeling or removal).

## Weight Change Test

The purpose weight change test is to determine if the substrate has been damaged. TAFB developed the procedure for performing the weight change test (provided in Appendix B).

The test involved weighing a clean, unpainted F/E prepreg test panel before and after immersion in the solvent. A weight loss occurs if the solvent has eaten away the substrate. A weight gain could also occur if the solvent is absorbed into the substructure through microcracks in the substrate. Weight change measurements were recorded in grams. The test panel remained in the solvent for four hours, four times longer than the time required to strip the panel in the screening test. After immersion, the panels were gently wiped dry by hand, and dried in a 150°F oven for one hour. Four weight change tests were performed for each solvent. The balance used in this test was calibrated with Bureau of Standards Class S weights each day of testing. Before each measurement was made, the balance was tared to 0.000 grams.

Panels from the prepreg sheet had to be used for this test because the condemned radome panels may have given false readings. TAFB materials engineers felt that the PC Blend 2 may have absorbed into the honeycomb layers and give a false weight gain. Due to the conical shape of the radome, the honeycomb structure is not in contact with the solvent during the actual depainting operation. Therefore, it was agreed by TAFB and FWEI that panels cut from a prepreg sheet would be more representative of actual conditions.

A parallel experiment was also conducted on a standard to validate the drying procedure. The standard panels are 2½ in. square 316 stainless steel (SS) plates, and were subjected to the same testing conditions as the composite. An inert material, the 316 SS should not absorb or be attacked by the solvent. Any gain in weight indicates solvent residue is present on the surface and did not evaporate.

# **RESULTS AND DISCUSSION**

## SCREENING PROCEDURE

FWEI screened the three propylene carbonate blends and MEK at TAFB Chemical Laboratory in Building 3001 from August 9 through 13, 1993. Four 24-hour runs were performed with each solvent.

In addition, a fifth beaker containing a mixture of 12.5 percent by weight water in MEK was evaluated. This was to determine the potential reuse of an MEK/water mixture which would be recovered from an adsorptive resin system, such as the Purus PADRE™ unit, being evaluated simultaneously at TAFB by FWEI for MEK vapor recovery.

Results of the screening tests are presented in Table 3. As the table indicates, the MEK/water mixture removed the paint in approximately 30 minutes. Although the three PC blends removed the paint, PC Blend 2 removed it most rapidly. For each solvent, paint was removed by hand after the paint had completely bubbled from the surface. The paint bubbled in one piece, which included primer layer, rain-erosion coating, and topcoat. Both MEK and the PC blends attacked the paint in this manner, i.e., by dissolving the bond between the coating and the substrate.

A possible explanation provided by Texaco for why PC Blend 2 was effective at paint removal is due to solubility parameters. This theory is that two substances should be soluble with each other when the solubility parameter of the solvent is equal or nearly equal to the solubility parameter of the solute (in this case, paint) although for this study, the paint formulation was not defined. This theory also explains why the individual components of a blend may not be effective paint removers by themselves, but when combined with other solvents, increase their effectiveness significantly.

The MEK did not perform as well as the MEK/water mixture. After the first run, significant evaporation occurred in both the MEK and MEK/water beakers. To minimize the evaporation, a plastic bag was placed over both beakers after about eight hours into the second run. On the third and fourth runs, a watchglass was placed over the beakers during the entire 24-hour period.

As Table 6-1 indicates, MEK performance became worse in Runs 3 and 4 when the beaker was covered. Given the performance of the MEK/water and the MEK in the first two runs, it appears that the MEK absorbs water from the air, which enhances its paint removal properties. The behavior of the MEK/water mixture versus pure MEK is similar to that of a solvent blend versus a pure solvent, as discussed above. Also, since the volatile MEK is evaporating, the composition of the MEK/water mixture changes with decreasing concentrations of MEK as time increases. Absorption of water by MEK can be checked by doing moisture analyses on several MEK samples obtained from the drainage sump during TAFB depainting operations. The TAFB operators in the radome depainting operation also indicated that MEK sometimes must be used in conjunction with manual scraping techniques to completely remove the paint. The removal time also varies from about 1½ hours to several hours.

TABLE 3. SCREENING PROCEDURE RESULTS

	V-0-2-					
Time	30 Minutes	1 Hour	2 Hours	4 Hours	8 Hours	24 Hours
PC Blend 1 Run 1 Run 2 Run 3 Run 4	50% Bubbling No Change 30% Bubbling No Change	Complete Bubbling 5% Bubbling 90% Bubbling No Change	90% Bubbling Complete Bubbling 20% Bubbling	Complete Bubbling Complete Bubbling		
PC Blend 2 Run 1 Run 2 Run 3 Run 4	25% Bubbling 5% Bubbling 90% Bubbling No Change	Complete Bubbling Complete Bubbling Complete Bubbling 10% Bubbling	Complete Bubbling			-
PC Blend 3 Run 1 Run 2 Run 3 Run 4	No Change Slight Bubbling No Change No Change	Slight Bubbling 60% Bubbling No Change No Change	20% Bubbling Complete Bubbling No Change 10% Bubbling	Complete Bubbling 95% Bubbling Complete Bubbling	Complete Bubbling	·
MEK Run 1 Run 2 Run 3 Run 4	Signs of Flaking No Change No Change No Change	Slight Bubbling No Change No Change No Change	70% Bubbling 10% Bubbling No Change 1% Bubbling	90% Bubbling 75% Bubbling 5% Bubbling 5% Bubbling	Complete Bubbling Complete Bubbling 20% Bubbling 20% Bubbling	20% Bubbling 20% Bubbling
MEK/H <sub>2</sub> O Run 1 Run 2 Run 3 Run 4	Complete Bubbling Complete Bubbling Complete Bubbling 90% Bubbling	Complete Bubbling				

The inconsistency of the paint removal times for the test panels could possibly be explained by the condition of the panels prior to depainting. Some panels had a mottled appearance, suggesting certain areas of the topcoat layer were worn, exposing the white, rain-erosion coating layer. For these panels, paint removal would be easier since the paint thickness was thinner than when originally applied. In Run 4, panels having similar surface conditions were selected. These panels did not have a mottled appearance and, therefore, provided better comparison of the different solvents. As reported in Table 3, complete bubbling took longer in Run 4 than in the previous runs. To satisfy requirements in the QAPP for this project, three additional runs should have been made to confirm results of Run 4. However, because of schedule and limited supply of condemned radome panels with similar surface conditions, this was not possible. The varied surface conditions of the radomes are, however, representative of the radomes encountered in the actual depainting operation.

After the 24-hour period, each of the test panels were evaluated for visible substrate damage. No apparent substrate damage was visible to the naked eye, although white spots indicated some fibers may be exposed. The TAFB materials engineer examined the panels visually and under magnification. He concluded that there could be possible fibers exposed, although all panels, including the MEK, exhibited this characteristic.

TAFB and FWEI conducted a separate experiment in which panels were partially immersed in the solvents (MEK/H<sub>2</sub>O and PC Blend 2 were used since they were removing paint the fastest). When the paint bubbled and peeled away from the panel's surface, the paint was manually removed. After the panels had dried, the paint on the unimmersed portion was partially peeled away by fingernail. This exposed portion of the panel had a darker color of substrate and TAFB concluded it indicated the top layer of resin (referred to as the "gel coat") had been possibly removed by both PC Blend 2 and the MEK/H<sub>2</sub>O. To validate this observation, this experiment should be repeated with pure MEK for comparison and panels should be further evaluated at magnification.

Based on screening results, PC Blend 2 was selected for further testing to evaluate the effect of the solvent on the F/E substrate. MEK was also evaluated in these tests.

## **EVALUATION TESTS**

FWDC performed evaluation tests at their John Blizard Research Center in Livingston, New Jersey. Evaluation tests consisted of simulated depainting, hardness, flexural properties, paint adhesion, and weight change.

## Simulated Depainting Procedure

Results from the depainting simulation are given in Table 4, and includes the time of removal for each test panel and an observation on the degree of paint removal. The PC Blend 2 removed the paint faster and more completely than MEK. It was discovered during this exercise that the paint system on these panels was different from the one used in the screening test. TAFB confirmed that these panels were painted with a neoprene-based coating which consisted of a yellow primer, white primer, and black topcoat. This paint system bonded very well to the substrate, and was extremely difficult to remove. TAFB discontinued using it over six years ago. Apparently, the condemned radome from which these panels were cut still had this paint system on it.

The current paint system consists of a dark red primer, followed by a white, rain-erosion coating and black topcoat. This paint system was on the radome test panels used in the screening tests. Both paint systems appear identical on the test panels. Only when the panels are stripped can they be identified.

TABLE 4. DEPAINTING SIMULATION RESULTS

Sample No.	Solvent	Removal Time (min)	Removal Efficiency
DP-01	PC Blend 2	228	100% removal
DP-02	PC Blend 2	212	100% removal
DP-03	PC Blend 2	44	100% removal
DP-04	PC Blend 2	58	100% removal
DM-01	MEK '	248	100% removal
DM-02	MEK	308	100% removal
DM-03	MEK	307	100% removal
DM-04	MEK	154	100% removal

Runs 3 and 4 for the depainting simulation with PC Blend 2 had the current paint system and, therefore, removed paint in less than one hour and in one piece, as in the screening. In Runs 1 and 2, the PC Blend 2 removed the old paint in about 3½ hours with vigorous scraping. PC Blend 2 appeared to remove the neoprene paint system more completely than the MEK. The four panels tested with MEK, however, had the neoprene system. MEK took an average of 4½ hours to remove this paint, with vigorous scraping.

Due to schedule considerations, this test could not be repeated with panels coated with the current system. However, the panels for the paint adhesion test had the current paint system. An additional step of recording the paint removal time was added to that test.

# Hardness

Hardness results are summarized in Table 5. Hardness measurements were made on unpainted F/E prepreg test panels. Ten measurements were made on each panel prior to exposure of solvent. The panels were then sprayed with solvent in the depainting simulation unit for two hours. After drying, ten additional hardness measurements were made on each panel. Table 5 lists the measurements and the arithmetic average.

The hardness test objective was to achieve a Barcol hardness of 55 or greater. As indicated in Table 5, hardness measurements met this objective and also did not change significantly after being exposed to solvent. Measurements ranged from 75 to 85 Barcol hardness units, with an overall average of 80.4. These results show that both MEK and PC Blend 2 do not chemically embrittle the substrate.

## Flexural Properties Test

Flexural properties test results are listed in Table 6. For each test panel, measurements of length, width, depth, and rate were made. Flexural strength, maximum strain, and modulus of elasticity were calculated by the following formulas:

TABLE 5. HARDNESS TEST RESULTS (Per ASTM D2583-87)

			Barcol h	ardness
Sample No.	Solvent	Measurement	Before	After
HP-01	PC Blend 2	1 2 3 4 5 6 7 8 9	81 80 80 82 79 82 79 79 80 78	82 84 81 80 80 83 84 84 84
	t. Visit	Average	80	82.6
HP-02	PC Blend 2	1 2 3 4 5 6 7 8 9	79 82 81 82 78 79 81 77 80 78	80 79 80 80 79 79 80 81 80 79
		Average	79.7	79.7
HP-03	PC Blend 2	1 2 3 4 5 6 7 8 9 10 Average	81 79 79 80 82 82 82 79 79 81	81 81 81 82 81 80 81 82 79 79
		· · · · · · · · · · · · · · · · · · ·		
HP-04	PC Blend 2	1 2 3 4 5 6 7 8 9	80 82 80 80 79 81 78 82 77 79	79 82 80 76 80 77 80 80 80
		Average	79.8	79.4
		Total average	80 ± 0.3	80.6 ± 1.4

TABLE 5. (Continued)

			Barcol h	ardness
Sample No.	Solvent	Measurement	Before	After
HM-01	MEK	1 2 3 4 5 6 7 8 9	81 82 82 81.5 81 81 82 82 81.5	79 82 82 79 81 82 81 83
		10 Average	80 81.4	82 81.4
HM-02 ;	MEK	1 2 3 4 5 6 7 8	79.5 81 79 78 81 80 81 79 78.5	85 82 82 81 80 83 82 80
,		10 Average	79 79.6	82 81.8
HM-03	MEK	1 2 3 4 5 6 7 8 9	79.8 79 80 80 80 80 83 78 82 79	82 80 79 82 82 82 82 80 77 79
		Average	80	80.5
HM-04	MEK	1 2 3 4 5 6 7 8 9	76 80 79 80 78 79 79 82 76 80	78 79 81 80 81 80 81 75 82
·	; .e. e	Average	78.9	79.6
		Total average	80 ± 1.1	80.8 ± 1.0

TABLE 6. FLEXURAL PROPERTIES RESULTS (Per ASTM D790-92)

Sample No.	Solvent	Lengüi, (in.)	Width, (in.)	Avg. depth, (in.)	Rate, (in./min)	Flexural strength, (psi)	Max. etrain, (in./in.)	Modulus of elasticity, (psi)
0-	None	2.990	1.015	0.0667	0.11	71,750	0.02520	3.64 x 10 <sup>6</sup>
0-05	None	2.985	1.007	0.0667	0.12	70,980	0.02470	3.60 × 10 <sup>6</sup>
60	None	2.993	1.015	0.0663	0.11	026'69	0.02380	3.70 × 10 <sup>6</sup>
90	None	2.987	1.010	0.0665	0.11	52,390*	0.02460	3.30 × 10 <sup>6</sup>
9	None	2.994	1.018	0.0662	0.12	71,280	0.02410	3.60 × 10 <sup>6</sup>
99 O	None	2.990	1.024	0.0664	0.11	72,562	0.02490	3.67 × 10 <sup>6</sup>
					Average	71,300	0.02455	3.59 × 10 <sup>6</sup>
FP-01	PC Blend 2	2.965	1.012	0.0667	0.11	52,240*	0.02710	3.46 × 10 <sup>6</sup>
FP-02	PC Blend 2	2.920	1.021	0.0660	0.11	74,670	0.02430	3.84 x 10 <sup>6</sup>
FP-03	PC Blend 2	2.993	1.018	0.0668	0.11	71,127	0.02505	3.66 x 10 <sup>6</sup>
FP-04	PC Blend 2	2.980	1.015	0.0661	0.12	51,682*	0.02350	3.59 x 10 <sup>6</sup>
FP-05	PC Blend 2	2.989	1.013	0.0667	0.11	71,160	0.02341	3.66 x 10 <sup>6</sup>
					Average	72,319	0.02467	3.64 × 10 <sup>6</sup>
FM-01	MEK	2.995	1.012	0.0660	0.11	72,544	0.02350	3.84 x 10 <sup>6</sup>
FM-02	WEK	2.991	1.010	0.0656	0.11	53,286*	0.02380	3.63 × 10 <sup>6</sup>
FM-03	MEK	2.998	0.995	0.0668	0.11	71,015	0.02485	3.66 × 10 <sup>6</sup>
FM-04	MEK	2.994	1.011	0.0671	0.11	72,694	0.02738	$3.54 \times 10^{6}$
FM-05	MEK	2.991	1.015	0.0666	0.11	71,433	0.02587	3.64 × 10 <sup>6</sup>
					Average	71,922	0.02508	3.66 × 10 <sup>6</sup>
					Average Population A Population B	71,762 ± 1,214 52,400 ± 665	0.02475 ±0.00119	3.62 x 10 <sup>6</sup> ±0.13 x 10 <sup>6</sup>

<sup>\*</sup> Data points determined to be of separate populations, based on bimodal statistical theory (refer to Section 8).

Flexural strength:

S = <u>3PL</u>

where: S = stress in the outer fibers at midspan (psi)

P = load at a given point on the load-deflection curve (lb<sub>t</sub>)

L = support span (in.)

b = width of beam tested (in.) d = depth of beam tested (in.)

Maximum strain:

r = <u>6Dc</u> L²

where: r = maximum strain in the outer fibers (in./in.)

D = maximum deflection of the center of the beam (in.)

L = support span (in.)

d = depth of beam tested (in.).

Modulus of elasticity:

 $E_{B} = \frac{L^{3}m}{4bd^{3}}$ 

where: E<sub>B</sub> = modulus of elasticity in bending (psi)

L = support span (in.)

m = tangent slope to the initial straight-line portion of the load deflection curve (lb<sub>t</sub>/in.)

b = width of beam tested (in.) d = depth of beam tested (in.)

Values required for the calculations were measured by the test.

Load deflection curves plotting load (lbs) versus deflection (mils) were produced for each test panel. These curves are presented in Appendix C as Figures 2 through 17.

The data presented in Table 6 demonstrates the exposure to either PC Blend 2 or MEK did not affect the flexural strength of the panels. Although most panels failed at approximately 72,000 psi loading, test panels O-4, FP-1, FP-4, and FM-2 had lower flexural strength, failing at approximately 52,000 psi loading. These four panels failed with a straight break across the test panel rather than with the zigzag pattern exhibited with the stronger panels. Since this occurred for the unexposed, PC Blend 2 exposed, and MEK exposed panels, the probable explanation is that those four test panels were cut from a weaker section of the prepreg sheet. A statistical analysis revealed that a bimodal distribution existed, proof that the four panels were taken from a different sample population than the stronger specimens. Comparison of individual readings within the respective populations show that flexural strength is unaffected by exposure to either solvent.

The second part of the test required observation of the failed test panels under an SEM. The panels failed in the first part of the test, but did not break in half. The specimens were manually broken in half so

they could be viewed both at the surface and cross-section of the failure by the SEM. After proper mounting and preparation, the panels were viewed at various magnifications to determine the conditions of the fibers and matrix. Microphotographs were taken at 30X, 300X, and 1200X magnification for representative test panels for each solvent. Selected microphotographs are presented as Figures 18 through 22 in Appendix C.

Figures 18 through 20 represent microphotographs made of the panels not exposed to solvent (i.e. Samples O-3 and O-4). Sample O-4 was examined because of its low flexural strength and its atypical failure pattern. As a comparison of Figures 18 and 20 indicates, Sample O-4 at 30X magnification has a much straighter and cleaner break than that of Sample O-3 at the same magnification. This type of break also occurred on Samples FP-01, FP-04, and FM-02. However, the microphotographs show no evidence of fiber damage on Sample O-4.

The remaining microphotographs were made for panels exposed to solvent. Figures 21 represents Sample FP-05, exposed to PC Blend 2. Figure 22 represents Sample FM-05, exposed to MEK. No difference in appearance was evident when comparing the two solvents' microphotographs at the same magnifications. At 300X magnification, Sample FM-05 appears to have fibers protruding in every direction, whereas Sample FP-05 has unidirectional fibers. However, this is a function of the area selected for photographing. As can be seen in the top portion of Figure 21, Sample FP-05 also has fibers in all directions.

The SEM microphotographs indicate no damage by either solvent of the fiber matrix interface or of the fibers themselves. Had damage occurred, the microphotographs would have shown noticeable gaps where the fibers interface the matrix (most obvious at the 300X magnification). Also, individual fibers appear to be intact, indicating the solvent did not attack the resin binding the fibers.

# Paint Adhesion

Table 7 reports the paint adhesion results and also lists the paint removal time for preparation of each test panel.

TABLE 7. PAINT ADHESION TEST RESULTS (Per ASTM D3359-92a)

Sample No.	Solvent	Removal Time	Paint Adhesion Rating
PC-01	PC Blend 2	45 min.	5A
PC-02	PC Blend 2	21 min.	5 <b>A</b>
PC-03	PC Blend 2	25 min.	5 <b>A</b>
PC-04	PC Blend 2	25 min.	5A
PM-01	MEK	30 min.	5A
PM-02	MEK	29 min.	5A
PM-03	MEK	25 min.	5A
PM-04	MEK	25 min.	5A

Removal times for PC Blend 2 averaged about 30 minutes. The MEK samples had paint bubbling much quicker than the PC panels (after about 10 to 15 minutes). After bubbling, paint on the MEK samples

removed easily. PM-2 and PM-4 required scraping to remove paint on edges which were not in the direct spray pattern of the MEK. The PC Blend 2 panels required minimal scraping to remove 100 percent of the paint. Sample PC-01's longer removal time is attributed to less vigorous scraping than the other panels. The behavior of the paint on PC Blend 2 panels was to bubble after about 20 minutes and peel off five to ten minutes later. Scraping was required to remove the loosened paint from the substrate.

Paint removal efficiency of panels for this test is similar to that of Runs 1 and 2 in the screening exercise. (In Runs 1 and 2, the beakers were left uncovered; in Runs 3 and 4, the beakers were covered to prevent evaporation and removal efficiency decreased significantly.) One possible explanation is that the MEK draws moisture from the air to form an MEK/water solvent blend. Water is a polar solvent which increases the polarity of the solvent blend and, therefore, enhances the paint removal capability of the MEK.

The panels were observed after air drying for one hour. The MEK panels appeared darker than the PC panels.

The paint adhesion ratings determined by the paint adhesion test were 5A for each test panel. This rating indicates no peeling or removal of paint by the pressure-sensitive tape occurred, suggesting complete paint adhesion following a depainting/painting cycle.

# Weight Change Test

Results of the weight change test are shown in Table 8. Weight changes are given in grams and as a percentage of the total weight. The test panels exposed to both PC Blend 2 and MEK have weight loss indicating slight substrate damage although considered to be negligible by FWEI and TAFB. Weight measurements for the 316 SS standards are also given in Table 8. Weight changes for these samples are within the accuracy limits of the balance. Therefore, the solvents had completely evaporated from the surface of the test panel.

TABLE 8. WEIGHT CHANGE TEST RESULTS

		Weight	(grams)	Weight (	Change
Sample No.	Solvent	Before	After	Grams	%
WP-01 WP-02 WP-03 WP-04 WM-01 WM-02 WM-03 WM-04	PC Blend 2 PC Blend 2 PC Blend 2 PC Blend 2 MEK MEK MEK MEK MEK MEK	8.124 8.290 8.177 8.065 8.435 8.337 8.300 8.596	8.113 8.281 8.157 8.040 Average 8.427 8.331 8.287 8.587	-0.011 -0.009 -0.020 -0.025 -0.016 -0.008 -0.006 -0.013 -0.009	0.14 0.11 0.25 0.31 0.20 0.09 0.07 0.16 0.10
Standards	,		Average	-0.009	0.11
WP-01 WP-02 WP-03 WP-04 WM-01 WM-02 WM-03 WM-04	PC Blend 2 PC Blend 2 PC Blend 2 PC Blend 2 MEK MEK MEK MEK MEK	118.612 120.937 120.118 120.710 119.581 119.464 118.044 120.700	118.610 120.935 120.119 120.710 119.581 119.465 118.044 120.700	-0.002 -0.002 +0.001 0.000 0.000 +0.001 0.000	

# **COST ANALYSIS**

An economic analysis was performed for replacing MEK with PC Blend 2, based on available information. Results of this analysis are summarized in Table 9. Assumptions used in the analysis are discussed in the following paragraphs.

TABLE 9. ECONOMIC COMPARISON OF PC BLEND 2 AND MEK

	PC Blend 2	MEK
Cost raw material (\$/gal)	\$9.01 <sup>1</sup>	189 drums @ \$284.13/drum <sup>2</sup> 31 drums @ \$215.29/drum <sup>2</sup>
Usage (gal)	3,025 <sup>3</sup>	12,100 <sup>2</sup>
Total raw material cost (\$)	\$27,255.25	\$60,374.56 <sup>2</sup>
Disposal cost (\$/lb)	\$1.92 <sup>2</sup>	\$1.92 <sup>2</sup>
Quantity disposed (lbs)	3,120 <sup>2</sup>	3,120 <sup>2</sup>
Total disposal cost (\$)	\$5,990.40	\$5,990.40
Labor rate (\$/hr)	\$58.35	\$58.35
Amt. of labor required (hr)	312	260
Total labor cost	\$18,205.20	\$15,171.00
TOTAL	\$51,450.85	\$81,535.96

#### Notes:

The estimated cost savings of using PC Blend 2 is \$30,085. This is based on a raw material cost of \$1.00/lb, estimated by Texaco, from the costs of the individual blend components. An actual commercial product of this blend could cost several times more due to packaging, advertising, and marketing costs. The PC is produced by Texaco, but the other two components, NMP and DBE, are produced by others.

Other assumptions made in this analysis are that usage and disposal quantities for MEK are the same as 1992. Since the Desert Storm conflict occurred in late 1991 and early 1992, an increased workload during this time could be giving an overestimate of annual consumption and generation. The usage of MEK

<sup>1</sup> Texaco estimate

<sup>&</sup>lt;sup>2</sup> 1992 data as supplied by TAFB

<sup>3</sup> Estimated usage of PC Blend 2 is assumed to be 1/4 that of MEK. This is a conservative estimate of PC Blend 2 since its vapor pressure is 1/10 that of MEK. Actual PC Blend 2 usage may be a lower percentage.

increased from 150 drums in 1991 to 220 in 1992. However, TAFB agreed to use 1992 figures since it is the last available year.

The estimated usage of PC Blend 2 is assumed to be ½ the usage of MEK; this is a conservative estimate since PC Blend 2's vapor pressure is 1/10 that of MEK. The quantity of paint chips disposed is assumed to be the same for both PC Blend 2 and MEK. Based on discussion in Section 3, spent PC Blend 2 is assumed to be either discharged to an industrial wastewater treatment facility or recycled. If this is not feasible, an additional disposal cost for the spent solvent would be added to the total disposal cost for PC Blend 2. The MEK is assumed to evaporate 100 percent, therefore, the spent MEK quantity is zero.

TAFB provided the standard labor rate for the depainting area and is a 1993 value. The amount of labor was calculated from an average of the results obtained during the testing program and from the actual depainting operation. In preparing for the paint adhesion test, in which test panels painted with TAFB's current paint system were depainted in the simulation unit, both PC Blend 2 and MEK took about 30 minutes to remove paint from a two in. square panel. According to TAFB personnel, paint starts bubbling at 30 minutes and takes between 1½ to 3 hours to completely depaint the radome. Since both MEK and PC Blend 2 removed paint at the same rate, the time estimated for paint removal of a complete radome with both PC Blend 2 and MEK was three hours. An additional two hours were added to PC Blend 2 and MEK to account for set-up and clean-up time, as well as necessary scraping and disposal. An additional hour was added to the PC Blend 2 to represent a step to remove solvent residue. This operation is assumed to occur once a week for an annual total of 312 hours for PC Blend 2 and 260 hours for MEK.

# **QUALITY ASSURANCE ANALYSIS**

The QAPP, "Evaluation of Propylene Carbonate in ALC Depainting Operations," dated August 12, 1993 was used as a basis for this study.

The purpose of the QA/QC program was to fulfill two related purposes:

- To provide an organized framework for the laboratory testing program, and
- To control data quality, within pre-established limits, to ensure that it was adequate to achieve the project objectives.

The overall QA objective is to evaluate the feasibility of substituting a PC solvent formulation for MEK in depainting aircraft radomes. To support this objective, the following goals had to be achieved:

- The PC solvent blend removes paint as effectively as MEK in 24 hours or less,
- Substitution of the PC solvent blend reduces hazardous waste disposal and eliminates discharge of a hazardous air pollutant, and
- The PC solvent blend does not damage the substrate surface or interface and allows subsequent application of paint to adhere to the substrate.

## DATA QUALITY

The quality control program defined in the QAPP was implemented during the testing program. Some measurements were identified as critical for meeting the primary project objectives. Other noncritical measurements were performed to provide supporting information for the primary objectives. Critical measurements were screening, removal rate and efficiency, paint adhesion, and flexural properties. Noncritical measurements were weight change and hardness.

The QAPP contains quantitative specifications for analytical data quality. Adequate data quality is defined by meeting criteria for precision, accuracy, and completeness. A comparison of the data quality requirements, as defined in the QAPP and what was actually achieved, are provided in Table 10. A summary of calibration standards used for this test program is presented in Table 1 of Appendix C.

Based upon the test results, the data quality was adequate to meet the program objectives. Accuracy objectives were met for all critical tests. Although data quality criteria for precision and completeness were not achieved for some of the tests, supplemental tests were added to address those testing areas which

TABLE 10. DATA QUALITY REQUIREMENTS SUMMARY

		Precision (RSD)	n (RSD)	Acci	Accuracy	Completeness	toness
Critical measurement	Reporting units	QAPP	Actual	QAPP	Actual	QAPP	Actual
Removal rate Depaint simulation Paint adhesion	Minutes	£10	19.4 11.6	භ +I	-3.33 -3.33	955	25 87.5
Removal efficiency Depaint simulation Paint achesion	% removal	s20	00	+ 20	± 20 + 20	8	05 06
Flexural properties	Strain, in./in. Flexural strength, psi Modulus of elasticity, psi	(1) (3) (8)	4.8 1.69 3.59	EEE	<b>! ! !</b>	95 95 95	100 100 <sup>(4)</sup> 100
Paint adhesion	Adhesion rating	(2)	0	(2)	100	92	100

No standard, or method of measurement considered to be a standard, has been established for this test.
ASTM D3359-92a classifies surface in broad subjective classifications. Precision and accuracy should not deviate more than 1 classification.
ASTM D790-92 defines RSD for flexural strength and modulus as 7.08 and 10.1% of the mean, respectively.
Statistical analysis proved results were bimodal; therefore, only results from the higher flexural strength were used to determine precision and completeness.

failed to meet the QA objectives. When the test data are considered in their totality, the project met overall program objectives. These factors are discussed below.

## **Precision**

The precision criteria are expressed as the percent relative standard deviation (RSD) of the replicate values from each test. The data met the precision criteria for the removal efficiency, flexural properties, and paint adhesion data. It did not meet the precision criteria for paint removal rate (time).

#### Paint Removal Rate-

The paint removal rate was to be originally determined from the depainting simulation tests. Since the specimens provided had a different paint system, most of the data from the depainting simulation tests were invalid. Time considerations did not allow rerun of the depainting simulation tests so corrective action consisted of obtaining paint removal times from the paint adhesion tests. This data gave a 11.6 percent RSD which slightly failed the precision criteria. This failure should not affect the main conclusion that paint removal times between the base MEK solvent and the proposed PC Blend 2 were comparable for the following reasons:

- The criterion for this test was set too tightly.
  - With the exception of sample PC-01 that was scraped too lightly, both the MEK and PC Blend 2 data had the same variability.
  - The main purpose for paint removal time data was to show that paint removal times were comparable. Average paint removal times were comparable.
  - The paint removal times were obtained from actual specimens that had been exposed to erosive forces that flying as an external part of an aircraft creates. The specimens showed variations in wear. The paint should be easy to remove from specimens having greater wear.
  - There was no previous data to set a reasonable precision criterion for the paint removal times.
- Since the material tested does not provide true replicates, the precision criterion should have been based upon comparing the standard deviations achieved in the base MEK tests and the PC Blend 2 tests.

## Flexural Strength-

The flexural strength tests met the precision criteria. The samples tested failed around two different strengths, 71,762 psi and 52,400 psi. Within each population the RSD for extreme values were within the 10 percent precision criteria. Data concentrated around two sample means indicate a bimodal distribution. This was confirmed by a statistical analysis that showed that the specimens were taken from two distinct sample populations within 95 percent confidence limits (See Table 6).

#### Accuracy

Accuracy for removal time was defined by calibration of the stopwatch against the WWV broadcast of the Universal Coordinated Time (UCT) over a 63-hour time period. The stopwatch was found to be two seconds slower than UCT after the 63 hours. Removal efficiency is a visual determination and, therefore, usual accuracy definitions do not apply. However, accuracy can be estimated, since the human eye can discern ±20 percent. The accuracy for the flexural properties measurements is defined by ASTM Test Method D 790-92. Accuracy objectives were achieved for these measurements. Although accuracy

requirements for the paint adhesion test are given in the QAPP as 1 classification number, further review of ASTM Test Method D-3359-92a indicates bias cannot be established.

## Completeness

Completeness is the percentage of the total measurements judged to be valid. Completeness objectives were not achieved for removal rate (both depainting simulation and paint adhesion). Removal rate (for depainting simulation) values were not complete because six of the eight test panels were painted with an obsolete paint system. For the paint adhesion removal rate, a completeness value of 87.5 percent represents seven out of the eight panels as valid.

#### **DEVIATIONS**

The following deviations from the procedures and test plan defined in the QAPP occurred:

- The amount of panels tested was less than specified in the QAPP, which was a combination of two substrates and two paint systems for each test. TAFB could provide only one substrate with a single paint system per test. Test panels provided were either F/E with honeycomb or unpainted F/E prepreg. As discussed in Section 5, hardness, flexural properties, and weight change tests were not conducive to panels with a honeycomb structure and, therefore, used the unpainted F/E prepreg. Since the base material is the same (F/E), FWEI would not expect a difference in results obtained with either form.
- An additional solvent blend of MEK with 12.5 percent concentration by weight water, was analyzed during the screening test. The purpose of this additional test was to evaluate the potential to reuse MEK with water which could be recovered in the Purus PADRE<sup>TM</sup> unit, which FWEI is studying simultaneously at TAFB.
- During screening, the test panels were immersed face down into the solvent baths rather than suspended using wires clamped to a ringstand. It was discovered the panels floated and it was more practical to immerse the specimens.
- Panels for the screening and evaluation tests were not scribed, as discussed in the QAPP. TAFB
  explained radomes are not always scribed, and they wanted the test panels to represent actual
  radome conditions.
- An additional test was performed to better evaluate possible resin damage after screening. Two
  panels were partially immersed in beakers: one containing MEK/H<sub>2</sub>O and the other PC Blend 2.
  After the paint bubbled, it was removed manually. After sufficiently drying, the unimmersed area was
  then peeled from the immersed area with a fingernail, exposing the substrate under the unimmersed
  area. This was then compared to the immersed area for any visual signs of resin removal.
- After the depainting simulation runs were conducted, it was discovered some of the panels were painted with a neoprene-based coating which was discontinued over six years ago due to its difficulty in paint removal. The surface of these test panels were identical to those with the current paint system. Out of the eight panels tested, six were painted with this system. Because of this, the times recorded for the depainting simulation are not representative since they do not reflect the current radome conditions. Due to limited sample size (specimens containing the same integrity as the specimens used in the screening were not readily available at the testing site), the test was not redone; instead, times were recorded for the specimen preparation step for the paint adhesion test.

- For the hardness test, panels were cut from an unpainted F/E prepreg sheet. The panels were exposed to the solvent in the depainting simulation unit for two hours. This time was chosen arbitrarily since the depainting simulation did not represent the paint system currently used at TAFB. However, the removal time for both solvents was later determined to be about 30 minutes; based on this finding, two hours was a reasonable exposure time.
- The paint adhesion test measured only one "X" cut on the test panel, rather than the three specified in the QAPP. Each "X" cut, as specified in ASTM D 3359, was 1.5 in. long. Test panels were provided by TAFB and as specified by FWEI, were two in. square. Therefore, only one "X" cut could fit on each test panel. Since the results were consistent for both solvents, and four samples were tested, the extra two "X" cuts would probably give similar results.
- Supporting measurements of temperature and relative humidity were not recorded during tests
  conducted early in the test program because a measuring device was not readily available. During
  the flexural properties and paint adhesion tests, this information was measured and recorded, as
  recommended by the respective ASTM test methods. These measurements are reported with the
  test results in Appendix C.
- The weight change test procedure was revised, since issuing the QAPP. The revised procedure is included in Appendix B as Revision 2. Changes include unpainted F/E prepreg panels used as test specimens. Since the panels were unpainted and did not contain the honeycomb structures, no depainting with MEK or masking the test panel was required to prepare for the test. Also, the panels tested with PC Blend 2 did not undergo a solvent rinse after removal from the simulation unit; it was decided not to introduce another variable into the testing process. The panels were wiped gently with a paper towel and dried in an oven for one hour. As the results from the standard panels indicate, the solvent appeared to sufficiently evaporate and did not affect test results.
- After the flexural properties test, the failed test panels were broken in half at the failure site to allow
  examination with the SEM. The panels were then mounted and viewed by the SEM at various
  magnifications. Microphotographs were made at 30X, 300X, and 1200X magnification rather than
  the 40X and 1000X indicated in the QAPP. The latter magnifications seemed to give a representative
  view of the fiber matrix. The 30X magnification provides an overall view of the failure of the panel.
  The 300X magnification views the fiber and matrix interface, while the 1200X magnification gives a
  close-up of the fiber breakage.

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- 4. ASTM D 3359-92a (Vol. 6.01), Tape Adhesion Test.
- 5. ASTM D 2583-87 (Vol. 8.02), Barcol Hardness Test.
- 6. ASTM D 790-92 (Vol. 8.01), Flexural Properties Test.

