NASA Technology Evaluation for Environmental Risk Mitigation
Kennedy Space Center, FL 32899

Test Protocol for Validation of Citric Acid as an Alternative to Nitric Acid for Passivation of Stainless Steels at Kennedy Space Center

Final—Amended

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Test Protocol

For

Validation of Citric Acid as an Alternative to Nitric Acid for Passivation of Stainless Steels at Kennedy Space Center

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Submitted to
NASA Technology Evaluation for Environmental Risk Mitigation Principal Center

AMENDMENTS

Page 4: **Table 1**—Removed all mention of ASTM G 50 under ***Test Methodology References*** for Parameter Optimization Testing, Salt Fog Testing, and Atmospheric Exposure Testing

Page 8: Test Description—Removed the following sentence 'At such times, the specimens shall be removed, photograhed, weighed, and evaluated in accordance with ASTM G 50 (*Standard Practice for Conducting Atmospheric Corrosion Test on Metals*).'

Page 9: **Table 3**—Removed ASTM G 50 from ***Parameters***

Page 9: Data Analysis and Reporting—Replaced the bullet 'Mass loss per ASTM G 50 every 168 hours until failure.' With 'Evaluate per ASTM D 610 every 168 hours until failure.'

Page 11: Test Description—Removed the following sentence 'At such time, the passivated specimens shall be removed, photographed, weighed, and evaluated in accordance with ASTM G 50 (*Standard Practice for Conducting Atmospheric Corrosion Test on Metals*).

Page 11: **Table 5**—Removed ASTM G 50 from ***Parameters***

Page 11: Data Analysis and Reporting—Changed bullet from 'Report coating condition and corrosion data for each coupon.' to 'Evaluate coating condition per ASTM D 610.'

Page 12: Test Description—Changed the following sentence 'Passivated only specimens shall be evaluated per the Rust Grade rating as described by Table 1 of ASTM D 610 (*Standard Practice for Evaluating Degree of Rusting on Painted Steel Surfaces*) and photographed on a monthly basis.' by adding 'for a total of six (6) months.' to the end.

Page 12: Test Description—Deleted the following sentence 'After six (6) months, the test panels shall be removed, photographed, and evaluated per ASTM D 610 (*Standard Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces*).'

Page 12: **Table 6**—Removed ASTM G 50 from ***Parameters***

Page 13: Data Analysis and Reporting—Removed the following sentence 'At failure, report mass loss per ASTM G 50.'

Page 23: **Table 12**—Deleted the row for ASTM G 50

PREFACE

This report was prepared by International Trade Bridge, Inc. (ITB) through the National Aeronautics and Space Administration (NASA) Technology Evaluation for Environmental Risk Mitigation Principal Center (TEERM). The structure, format, and depth of technical content of the report were determined by NASA TEERM, Government contractors, and other Government technical representatives in response to the specific needs of this project.

We wish to acknowledge the invaluable contributions provided by all the organizations involved in the creation of this document.

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LIST OF ACRONYMS

°F	Degrees Fahrenheit
AMS	Aerospace Material Specifications
APL	Approved Products List
ASTM	American Society for Testing and Materials
ft-lbs	foot-pounds
GMW	General Motors Worldwide Engineering Standards
ITB	International Trade Bridge, Inc.
J	Joules
KSC	John F. Kennedy Space Center
ksi	kilo-pound per square inch
LOX	liquid oxygen
MSFC	George C. Marshall Space Flight Center
NASA	National Aeronautics and Space Administration
NOx	Nitrogen oxide emissions
NSF	Notched Fracture Strength
SAE	Society of Automotive Engineers
SLT	Sustained Load Testing
TEERM	Technology Evaluation for Environmental Risk Mitigation Principal Center
USA	United Space Alliance

1. INTRODUCTION

Headquarters National Aeronautics and Space Administration (NASA) chartered the Technology Evaluation for Environmental Risk Mitigation Principal Center (TEERM) to coordinate agency activities affecting pollution prevention issues identified during system and component acquisition and sustainment processes. The primary objectives of NASA TEERM are to:

- Reduce or eliminate the use of hazardous materials or hazardous processes at manufacturing, remanufacturing, and sustainment locations.
- Avoid duplication of effort in actions required to reduce or eliminate hazardous materials through joint center cooperation and technology sharing.

John F. Kennedy Space Center (KSC) has equipment and processes which require the passivation of stainless steel. The standard practice for passivation uses nitric acid. While nitric acid exhibits excellent performance, there are a number of environmental and safety issues:

- Nitrogen oxide (NOx) emissions are considered greenhouse gases
- NOx are volatile organic compounds that contribute to smog
- NOx increase nitrogen loading (oxygen depletion) in bodies of water
- Nitric acid can remove beneficial heavy metals (nickel, chromium, etc) from surface
- Worker safety issues

The longtime military specification for passivation of stainless steel was Federal Specification QQ-P-35 (*Passivation Treatments for Corrosion-resistant Steel)*, but that has been cancelled in favor of American Society for Testing and Materials (ASTM) A 967 (*Standard Specification for Chemical Passivation Treatments for Stainless Steel Parts)* and Aerospace Material Specifications (AMS) 2700 (*Passivation Treatments for Corrosion-resistant Steel)*. Both of the new specifications allow for the use of citric acid in place of nitric acid.

In addition, KSC also uses NASA-STD-5005 (*Standard for the Design and Fabrication of Ground Support Equipment*) and ASTM A 380 (*Standard Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems*).

The primary objective of this effort is to demonstrate and validate citric acid as an alternative to nitric acid for the passivation of stainless steels. Citric acid offers a variety of benefits:

- It is naturally occurring and biodegradable
- Upon disposal, it is rarely classified as a hazardous waste
- It does not remove beneficial heavy metals from surface
- There are no toxic fumes created during the passivation process

This project is follow-on to United Space Alliance (USA) work to optimize the parameters for the use of citric acid and verify effectiveness. USA evaluated the following parameters during their effort: concentration, temperature, and dwell time. This project will use the results from that testing to determine what process parameters will be used in the preparation of test coupons.

This Test Protocol contains the critical requirements and tests necessary to qualify citric acid as an alternative to nitric acid for use at KSC. These tests were derived from engineering, performance, and operational impact (supportability) requirements defined by KSC participants.

A Test Report will document the results of the testing as well as any test modifications made during the execution of the testing. The Test Report will be made available as a reference for future pollution prevention endeavors by other NASA centers, the Department of Defense, and commercial users to minimize duplication of effort. Users of this Test Protocol should check the project's Test Report for additional test details or minor modifications that may have been necessary in the execution of the testing. The technical stakeholders will have agreed upon test procedures modifications documented in the Test Report.

2. ENGINEERING, PERFORMANCE, AND TESTING REQUIREMENTS

A joint group led by NASA TEERM and consisting of technical representatives from KSC reached technical consensus on engineering, performance, and testing requirements for citric acid as an alternative to nitric acid for passivation of stainless steel substrates. The joint group defined critical tests with procedures, methodologies, and acceptance criteria to qualify alternatives against these technical requirements.

Once the test criterion is approved, testing will be performed in a manner that will optimize the use of each test panel. For example, where practical, more than one type of test will be performed on the coated test panels. The number and types of tests performed on a given panel will be determined by the destructive nature of the tests in question.

> **Note:** Tests specified in this Test Protocol may involve the use of hazardous materials, operations, and equipment. This Test Protocol does not address all safety issues associated with its implementation. It is the responsibility of each user of this Test Protocol to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to its use.

This project will compare the performance of citric acid to nitric acid for passivation of stainless steel alloys for use at KSC. The tests described in this Test Protocol are divided into two phases which are summarized in Tables 1 and 2. The tables include acceptance criteria and the reference specifications, if any, used to conduct the tests.

Table 1 Phase 1 Engineering, Performance, and Testing Requirements				
Test	***Section***	***Test Specimen***	***Acceptance Criteria***	***Test Methodology References***
Parameter Optimization Testing	4.1.1	Coupon	Best performing parameters	ASTM B 117 ASTM D 610
Tensile (Pull-off) Adhesion	4.1.2	Coupon	Alternative performs as well or better than control process	ASTM D 4541 NASA-STD-5008B
Salt Fog Testing	4.1.3	Coupon		ASTM B 117 ASTM D 610
Atmospheric Exposure Testing	4.1.4	Coupon		ASTM D 610 ASTM D 714 NASA-STD-5008B
Stress Corrosion Cracking	4.1.5	Coupon		ASTM B 117 ASTM E 4 ASTM E 8 ASTM G 38 ASTM G 39 ASTM G 44 MSFC-STD-3029
Liquid Oxygen (LOX) Compatibility	4.1.6	Sample	Twenty samples must not react when impacted at 72 foot-pounds (ft-lbs) or 98 Joules (J). If one sample out of 20 reacts, 40 additional samples must be tested without any reactions.	ASTM D 2512 NASA-STD-6001

Table 2 Phase 2 Engineering, Performance, and Testing Requirements				
Test	***Section***	***Test Specimen***	***Acceptance Criteria***	***Test Methodology References***
X-Cut Adhesion by Wet Tape	4.2.1	Coupon	Alternative performs as well or better than control process	ASTM D 3359 NASA-STD-5008B
Cyclic Corrosion Resistance	4.2.2	Coupon		GMW 14872
Hydrogen Embrittlement	4.2.3	Coupon		ASTM E 4 ASTM F 519

3. TEST PREPARATION

The following substrate types have been agreed upon by the stakeholders and are listed in prioritized order:

1. Super Austenitic: AL-6XN (UNS N08367)
2. 200 Series Austenitic: A286 (UNS S66286)
3. 300 Series Austenitic: 304 (UNS S30400)
4. Precipitation-Hardened Martensitic: 17-4PH (UNS17400)
5. 400 Series Martensitic: 410 (UNS41000)
6. 300 Series Austenitic: 316 (UNS S31600)
7. Precipitation-Hardened Martensitic: 15-5PH (UNS S15500)
8. Precipitation-Hardened Martensitic: 17-7PH (UNS S17700)

The preparation of coupons will be documented by the technician preparing test coupons. For each test, a minimum of five (5) coupons shall be prepared for each substrate (unless otherwise noted); those with the best coating as determined by the technician shall be used in accordance with the number of coupons required as specified in the *Test Methodology*. Unless otherwise required by a specific test, all coupons will be prepared as follows:

1. Initial degrease to remove machining contamination
2. Test coupons not used for adhesion testing shall be grit blast with carbon steel to eliminate manufacturer's passivation layer and introduce free iron
3. Test coupons to be used for adhesion tests shall be prepared in such a way as to remove the manufacturer's passivation layer without creating a surface profile. Possible procedures include "pickling" or milling or planning the surface. The actual procedure used shall be agreed upon by all stakeholders and documented in the Test Report.
4. Prepare test coupons with nitric acid passivation per the designated process in Appendix A
5. Prepare test coupons with citric acid passivation per the designated process in Appendix B
6. Rinse coupons with deionized water to neutral pH
7. Blow dry with compressed air
8. Store in clean, soft, low lint cloth in controlled environment until needed
9. Apply coating if required for testing

Based on the USA optimization testing, the following information should be used to determine passivation parameters for the identified substrates. For those substrates which are not indicated below, optimization testing of parameters (section 4.1.1) may be performed prior to testing. Passivation parameters may also be determined based on an alloy's similarity to one of the alloys identified below if all stakeholders agree.

- For 304 (UNS30400), temperatures of 100-140 degrees Fahrenheit (°F) and submersion times of 120 minutes provided the best corrosion protection.
- For 410 (UNS41000), temperatures of 140-180 °F and submersion times of 30-75 minutes provided the best corrosion protection.
- For 17-4PH (UNS17400), no increase in corrosion protection was observed for temperatures above 100 °F and submersion times greater than 30 minutes

For paint adhesion testing, test coupons shall be allowed 24 hours of unaided drying time prior to dry film thickness measurements. If liquid coatings are being tested for a destructive test, coupons shall be allowed to cure for an additional 14 days before they undergo any destructive testing to ensure full polymerization of the coating. Coating process parameters, including application method and cure schedule, shall be documented by the facility that prepares the test coupons.

4. TEST DESCRIPTIONS

Test requirements identified in Tables 1 and 2 are further defined in this section to include the test description, rationale, and test methodology. The *Test Methodology* lists the major parameters, test coupon descriptions, number of test coupons, number of coupons per coating system, number of control coupons and acceptance (pass/fail) criteria. Any U*nique Equipment or Instrumentation* requirements and *Data Analysis and Reporting Criteria* are also included. The latest revision of each specification or standard shall be used unless otherwise stated.

4.1 Phase 1 Tests

4.1.1 Parameter Optimization Testing

Test Description

Previous testing by USA determined that the parameters for citric acid passivation that resulted in optimum performance varied by substrate. The data obtained during the original USA study shall be used for the testing of those identified substrates in this test plan. Parameter optimization testing, however, is necessary to determine the best parameters for the substrates included in this test plan that were not part of the USA study.

The USA study determined that a citric acid concentration of 4% provided optimum performance for all substrates and therefore a 4% concentration shall be used for the optimization testing in this test plan. The varying parameters shall be bath temperature and submersion time.

The USA study also found that treatments with elevated temperature and longer submersion times (temperature > 100 °F and time > 30 minutes) provide significantly better corrosion protection than treatments at ambient temperature or shorter submersion times (T ≈ 100 °F and t ≈ 30 minutes).

Based on the available information the following parameters shall be used:

- Time: 60 minutes, 90 minutes, and 120 minutes
- Temperature: 100 °F, 140 °F, and 180 °F

Tests shall be conducted on all coupons in accordance with ASTM B 117 [*Standard Practice for Operating Salt Spray (Fog) Apparatus*]. The test panels shall be evaluated every 168 hours.

The test panels shall be removed from testing once the panels have reached failure. Failure is defined as a Rust Grade rating of “8” as described by Table 1 of ASTM D 610 (*Standard Practice for Evaluating Degree of Rusting on Painted Steel Surfaces*).

The Parameters resulting in the best corrosion resistance shall be used for preparation of that substrate's test panels for the remainder of the testing.

Rationale

The stakeholders agreed that optimization testing was required for those substrates not included in the original USA study.

Test Methodology

Table 3 Test Methodology for Parameter Optimization Test	
Parameters	Per ASTM B 117 and ASTM D 610 Time: 60, 90, and 120 minutes Temperature: 100, 140, and 180°F
Coupons Per Substrate/Coating System	Three (3)
Trials Per Coupon	One (1)
Acceptance Criteria	Parameters resulting in the best corrosion resistance shall be used for preparation of that substrate's test panels for the remainder of the testing

Unique Equipment and Instrumentation

- Salt Fog test chamber

Data Analysis and Reporting

- Evaluate per ASTM D 610 every 168 hours until failure.
- One (1) color photograph of a coupon for each substrate and coating shall be taken before the test. One (1) color photograph of each tested coupon shall be taken at each evaluation and the end of the test.

4.1.2 Tensile (Pull-Off) Adhesion

Test Description

This test evaluates the pull-off strength (commonly referred to as adhesion) of a coating. The test determines either the greatest perpendicular force (in tension) that a surface area can bear before a plug of material is detached, or whether the surface of the

material remains intact at a prescribed force. This test shall be performed in accordance with ASTM D 4541 (*Standard Test Method for Pull-off Strength of Coatings Using Portable Adhesion Testers*).

This test method uses a class of apparatus known as portable pull-off adhesion testers. They are capable of applying a concentric load and counter load to a single surface so that coatings can be tested even though only one side is accessible. Measurements are limited by the strength of adhesion bonds between the loading fixture and the specimen surface or the cohesive strengths of the adhesive, coating layers, and substrate.

The group decided to include coatings that are from the NASA-STD-5008B (*Protective Coating of Carbon Steel, Stainless Steel, and Aluminum on Launch Structures, Facilities, and Ground Support Equipment*) Approved Products List (APL).

> **Note: Specific coatings used during testing will be identified in the test report.**

Rationale

Participants agreed that adhesion is a critical performance requirement and want to compare how citric acid may affect coating adhesion.

Test Methodology

Table 4 Test Methodology for Pull-Off Adhesion Test	
Parameters	Per ASTM D 4541
Coupons Per Substrate/Coating System	Three (3)
Trials Per Coupon	Three (3)
Acceptance Criteria	Alternative performs as well or better than control process

Unique Equipment and Instrumentation

- Patti adhesion tester

Data Analysis and Reporting

- Record the strength at which adhesion fails; there should be three (3) data points for each coupon

- One (1) color photograph of a coupon for each substrate and coating shall be taken before the test. One (1) color photograph of each tested coupon and the dolly shall be taken after the test.

4.1.3 Salt Fog Testing

Test Description

This test evaluates the ability of the passivation process to prevent corrosion when exposed to a controlled corrosive environment. Tests shall be conducted on all test coupons in accordance with ASTM B 117 [*Standard Practice for Operating Salt Spray (Fog) Apparatus*].

The samples shall be evaluated every 168 hours. The panels shall be removed from testing once the panels have reached failure. Failure is defined as a rating of Rust Grade "8" as described by Table 1 of ASTM D 610 (*Standard Practice for Evaluating Degree of Rusting on Painted Steel Surfaces*).

Rationale

The stakeholders agreed that the ASTM B 117 Test provides an acceptable means to compare the corrosion protection of citric acid versus nitric acid passivation.

Test Methodology

Table 5 Test Methodology for Salt Fog Testing	
Parameters	Per ASTM B 117 and ASTM D 610
Coupons Per Substrate	Three (3)
Trials Per Coupon	One (1)
Acceptance Criteria	Alternative performs as well or better than control process

Unique Equipment or Instrumentation

- Programmable salt spray (fog) chamber

Data Analysis and Reporting

- Evaluate coating condition per ASTM D 610.
- Photograph a selected test panel for each substrate prior to test initiation to use as a reference photo, group photographs in the test chamber at the end of each

seven (7)-cycle period, and selected photographs at the terminus of the test to capture the results of the test.

4.1.4 Atmospheric Exposure Testing

Test Description

This test evaluates the performance of the test and control process after outdoor exposure in a marine environment. All panels shall be passivated by either the control or the alternative. Panels will also be prepared and coated with coatings identified for the adhesion tests (sections 4.1.2 and 4.2.1).

Install the test panels at the KSC outdoor exposure rack approximately 150 feet from the ocean high tide line. Follow all KSC test rack procedures for fasteners, exposure angle, and inspection interval as stated in NASA-STD-5008B (*Protective Coating of Carbon Steel, Stainless Steel, and Aluminum on Launch Structures, Facilities, and Ground Support Equipment*).

Passivated only specimens shall be evaluated per the Rust Grade rating as described by Table 1 of ASTM D 610 (*Standard Practice for Evaluating Degree of Rusting on Painted Steel Surfaces*) and photographed on a monthly basis for a total of six (6) months.

Coated test coupons shall be photographed every six (6) months for a total of 18 months. At the conclusion of the 18 months, each coated test coupon condition shall be rated per ASTM D 610 (*Standard Practice for Evaluating Degree of Rusting on Painted Steel Surfaces*). Use the numerical grade scale in ASTM D 610, Table 1, *Scale and Description of Rust Grades*, where 0 indicates 100% surface rusting and 10 indicating less than 0.01% surface rusting. Test coupon condition shall also be rated per ASTM D 714 (*Standard Test Method for Evaluating Degree of Blistering of Paints*); using the reference standards in section 3.

Rationale

This test documents the corrosion protection of the passivation alternative with actual exposure to UV radiation, as well as different cycles of salt spray exposure.

Test Methodology

Table 6 Test Methodology for Atmospheric Exposure Testing	
Parameters	Per NASA-STD-5008B, ASTM D 610, and ASTM D 714
Coupons Per System	Three (3)
Trials Per Coupon	One (1)

Table 6 Test Methodology for Atmospheric Exposure Testing	
Acceptance Criteria	Alternative performs as well or better than control process

Unique Equipment or Instrumentation

- KSC outdoor test rack located 150 feet from ocean high tide line

Data Analysis and Reporting

- For passivated only test panels, evaluate per ASTM D 610 on a monthly basis until failure.
- One (1) color photograph of each passivated only test panel shall be taken before the test. One (1) color photograph of each passivated only test panel shall be taken on a monthly basis. One (1) color photograph shall be taken of the failed coupon at the end of six (6) months.
- For coated test panels, report corrosion rating per ASTM D 610, Table 1, and blister rating per ASTM D 714, Section 3, after 18 months.
- One (1) color photograph of each coated test panel shall be taken before the test. One (1) color photograph of each coated test panel shall be taken at each six (6) month interval and at 18 months.

4.1.5 Stress Corrosion Cracking

Test Description

This test evaluates whether the passivation process will affect the material's resistance to stress corrosion cracking. George C. Marshall Space Flight Center (MSFC)-STD-3029 (*Guideline for the Selection of Metallic Materials for Stress Corrosion Cracking Resistance in Sodium Chloride Environments*) lists the following alloys as Table I materials.

Table I materials experienced no failures of specimen stressed to 75% of the yield strength for 30 days of exposure per ASTM G 44 (*Standard Practice for Preparation of Stress-Corrosion Test Specimens for Weldments*) or ASTM B 117 [*Standard Practice for Operating Salt Spray (Fog) Apparatus*].

Test coupons shall be prepared in accordance with ASTM G *38* (*Standard Practice for Making and Using C-Ring Stress-Corrosion Test Specimens*) or ASTM G 39 (*Standard Practice for Preparation and Use of Bent-Beam Stress-Corrosion Test Specimens*). Three specimens shall represent each alloy considered.

Three unexposed specimens shall be tested per ASTM E 8 (*Standard Test Methods for Tension Testing of Metallic Materials*) to determine the material properties of each alloy.

Three specimens for each alloy are loaded in a test frame to 75% of the material's yield strength per ASTM G 38 or ASTM G 39. The stressed specimen shall be exposed, along with three more un-stressed specimens to 5% salt spray per ASTM B 117 for 30 days.

Rationale

Participants agreed that determining whether citric acid passivation lowers a material's resistance to stress corrosion cracking is vital for approval of its use.

Test Methodology

Table 7 Test Methodology for Stress Corrosion Cracking Test	
Parameters	Specimen shall be prepared per ASTM G 38 or ASTM G 39. The test shall be conducted in 5% salt spray per ASTM B 117 for 30 days.
Coupons Per Substrate	Three (3)
Trials Per Coupon	One (1)
Acceptance Criteria	All of the specimens survive 30 days without fracture.

Unique Equipment or Instrumentation

- Salt fog chamber per ASTM B 117
- Universal test machine per ASTM E 4 (*Standard Practices for Force Verification of Testing Machines*)

Data Analysis and Reporting

- Photograph all test specimens before and after exposure.
- Stress vs. strain curves for all specimens at the completion of the test.

4.1.6 LOX Compatibility

NOTE: After LOX Compatibility Testing was identified by stakeholders, it was found that NASA Marshall Space Flight Center had previously conducted LOX Compatibility testing on citric acid solution. All samples passed showing no reaction. The stakeholders agreed that no additional testing is required.

Test Description

The purpose of this test is to determine if materials in oxygen environments react when mechanically impacted. A reaction from mechanical impact can be determined by an audible report, an electronically or visually detected flash, or obvious charring of the sample, sample cup, or striker pin.

Perform this test in accordance with NASA-STD-6001 (*Flammability, Odor, Offgassing, and Compatibility Requirements and Test Procedures for Materials in Environments that Support Combustion,* issued 1998). Specifically reference Test Method 13A, *Mechanical Impact for Materials in Ambient Pressure LOX*. The test system should be identical to that described in ASTM D 2512 [*Compatibility of Materials with Liquid Oxygen (Impact Sensitivity Threshold* and *Pass-Fail Techniques*), approved 1982].

Rationale

This test is specified in NASA-STD-6001 and was identified as a testing requirement. Materials intended for use in space vehicles, specified test facilities, and specified ground support equipment must meet the requirements of this document.

Test Methodology

Table 8 Test Methodology for LOX Compatibility Test

Parameters	Per NASA-STD-6001; The thickness of the sample must be the worst-case thickness. Test conditions (pressure and temperature) are the ambient pressure of the test facility and the boiling point of LOX at that pressure.
Samples	Twenty (20)*
Trials Per Sample	One (1)
Control Samples Required For Testing	None
Acceptance Criteria	Twenty samples must not react when impacted at 72 ft-lbs (98 J). If one sample out of 20 reacts, 40 additional samples must be tested without any reactions.

*Minimum required

Unique Equipment and Instrumentation

- ABMA-Type Impact Tester

Data Analysis and Reporting

- The test report must include sample identification, configuration, test conditions, number of reactions, and observations from the test. Proper reporting of the test observations, especially of unusual behavior, is critical.

4.2 Phase 2 Tests

4.2.1 X-Cut Adhesion by Wet Tape

Test Description

This test method establishes the adequacy of intercoat and surface adhesion of an organic coating immersed in water by applying pressure sensitive tape over a scribed area of the coating. This test shall be performed in accordance with ASTM D 3359 (*Standard Test Methods for Measuring Adhesion by Tape Test*), Test Method A.

Inspect the X-cut and parallel lines-cut for removal of the coating from the substrate or previous coatings and rate the adhesion in accordance with the 0-5 scale outlined in ASTM D 3359, Method A, Paragraph 7, *Procedure*, with the *0-A* rating being *coating removal beyond the scribed area* and the *5-A* rating being *no peeling or removal*.

The group decided to include coatings that are from the NASA-STD-5008 (*Protective Coating of Carbon Steel, Stainless Steel, and Aluminum on Launch Structures, Facilities, and Ground Support Equipment*) APL coatings.

> **Note: Specific coatings used during testing will be identified in the test report.**

Rationale

The X-cut with parallel lines scribe procedure increases the severity of this test over a dry tape adhesion test using a single "X" scribe and provides quantitative data for the adhesion of a coating system to the underlying metal substrate. All participants have agreed that adhesion testing is a performance requirement.

Test Methodology

Table 9 Test Methodology for X-Cut Adhesion by Tape Test

Parameters	Per ASTM D 3359
Coupons Per Substrate/Coating System	Three (3)
Trials Per Coupon	One (1)
Acceptance Criteria	Alternative performs as well or better than control process

Unique Equipment or Instrumentation

- One (1)-inch wide semitransparent pressure-sensitive tape 3M Code 250 or equivalent
- Four and one half (4.5)-pound rubber-covered roller, approximately three and one-half (3.5)-inches diameter by one (1)-inch wide.
- Cutting tool
- Cutting guide

Data Analysis and Reporting

- Results of the test using the classification guide in ASTM D 3359, Test Method A, paragraph 7.7
- One (1) color photograph of a coupon for each substrate and coating shall be taken before the test. One (1) color photograph of each tested coupon shall be taken after the test.

4.2.2 Cyclic Corrosion Resistance

Test Description

This test evaluates the ability of the passivation process to prevent corrosion when exposed to a simulated neutral pH corrosive environment. Tests shall be conducted on all coupons in accordance with General Motors Worldwide Engineering Standards (GMW) 14872 (*Cyclic Corrosion Laboratory Test*).

Coupons will be exposed for a total of 80 cycles and pictures will be taken every seven (7) cycles and at the end of the exposure period. One test cycle shall equal 24 hours. The following information shall be recorded:

- Frequency of Salt Solution Changes
- Method of Salt Application
- pH of Solution
- Salinity or Conductivity of Solution
- Solution Constituents

Rationale

The stakeholders agreed that the GMW Cyclic Corrosion Laboratory Test provides an acceptable correlation between accelerated laboratory corrosion tests and actual corrosion experienced in the field.

Test Methodology

Table 10 Test Methodology for Cyclic Corrosion Resistance Test	
Parameters	Per GMW 14872 Electrolyte solution: • 0.9% sodium chloride • 0.1% calcium chloride • 0.075% sodium bicarbonate
Coupons Per Substrate	Three (3)
Trials Per Coupon	One (1)
Acceptance Criteria	Alternative performs as well or better than control process

Unique Equipment or Instrumentation

- Programmable salt spray (fog) chamber

Data Analysis and Reporting

- Report coating condition and corrosion data for each coupon.
- Photograph a selected test panel for each substrate prior to test initiation to use as a reference photo, group photographs in the test chamber at the end of each seven (7)-cycle period, and selected photographs at the terminus of the test to capture the results of the test.

4.2.3 Hydrogen Embrittlement

Test Description

This test evaluates whether exposure to chemicals encountered in service environments, such as the passivation process, can cause hydrogen embrittlement in steels.

Tests shall be conducted in accordance with ASTM F 519 (*Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments*). This test method uses specimens made from Society of Automotive Engineers (SAE) 4340 steel (UNS G434006) and heat treated to 260-280 kilo-pound per square inch (ksi).

Prepare/obtain four (4) specimens with ASTM F 519 Type 1d, notched C-ring. This specimen type was selected because it was considered to be the most severe.

Test passivated specimens by the Sustained Load Testing (SLT) to 75% of the manufacturer-reported average Notched Fracture Strength (NSF) for 200 hours in air at room temperature. If all four (4) of the specimens sustain 200 hours SLT without fracture, the process is acceptable.

Rationale

Participants agreed that determining whether citric acid caused hydrogen re-embrittlement is a critical performance requirement.

Test Methodology

Table 11 Test Methodology for Hydrogen Embrittlement Test	
Parameters	Specimen shall be Type 1d, notched C-ring per ASTM F 519. The test shall be conducted in air at room temperature.
Coupons	Four (4) SAE 4340 steel (UNS G434006) heat treated to 260-280 ksi.
Trials Per Coupon	One (1)
Acceptance Criteria	All of the specimens sustain 200 hours SLT without fracture

Unique Equipment or Instrumentation

- Universal testing machine per ASTM E 4 (*Standard Practices for Force Verification of Testing Machines*)
- Device for measuring deflection during stressing

Data Analysis and Reporting

- Record the time under load and sustained or threshold load, or percent of notched fracture strength or notch bend strength of unplated specimens, or displacement as appropriate for the type of specimen tested.
- Photograph a selected test panel for each substrate prior to test initiation to use as a reference photo and photos of post test specimens.

5. REFERENCE DOCUMENTS

The documents in Table 12 were referenced in the development of this Test Protocol.

Table 12 Summarized Test and Evaluation Reference Listing

Reference Document	***Title***	***Test***	***Section***
AMS 2700	*Passivation Treatments for Corrosion-resistant Steel*	N/A	1
ASTM A 380	*Standard Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems*	N/A	1
ASTM A 967	*Standard Specification for Chemical Passivation Treatments for Stainless Steel Parts*	N/A	1
ASTM B 117	*Standard Practice for Operating Salt Spray (Fog) Apparatus*	Parameter Optimization Testing Salt Fog Testing Stress Corrosion Cracking	4.1.1 4.1.3 4.1.5
ASTM D 610	*Standard Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces*	Parameter Optimization Testing Salt Fog Testing Atmospheric Exposure Testing	4.1.1 4.1.3 4.1.4
ASTM D 714	*Standard Test Method for Evaluating Degree of Blistering of Paints*	Atmospheric Exposure Testing	4.1.4
ASTM D 2512	*Compatibility of Materials with Liquid Oxygen (Impact Sensitivity Threshold* and *Pass-Fail Techniques*)	LOX Compatibility	4.1.6

Table 12 Summarized Test and Evaluation Reference Listing			
Reference Document	***Title***	***Test***	***Section***
ASTM D 3359	*Standard Test Methods for Measuring Adhesion by Tape Test*	X-Cut Adhesion	4.2.1
ASTM D 4541	*Standard Test Method for Pull-off Strength of Coatings Using Portable Adhesion Testers*	Tensile (Pull-off) Adhesion	4.1.2
ASTM E 4	*Standard Practices for Force Verification of Testing Machines*	Stress Corrosion Cracking Hydrogen Embrittlement	4.1.5 4.2.3
ASTM E 8	*Standard Test Methods for Tension Testing of Metallic Materials*	Stress Corrosion Cracking	4.1.5
ASTM F 519	*Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments*	Hydrogen Embrittlement	4.2.3
ASTM G 38	*Standard Practice for Making and Using C-Ring Stress-Corrosion Test Specimens*	Stress Corrosion Cracking	4.1.5
ASTM G 39	*Standard Practice for Preparation and Use of Bent-Beam Stress-Corrosion Test Specimens*	Stress Corrosion Cracking	4.1.5
ASTM G 44	*Standard Practice for Preparation of Stress-Corrosion Test Specimens for Weldments*	Stress Corrosion Cracking	4.1.5

Table 12 Summarized Test and Evaluation Reference Listing			
Reference Document	***Title***	***Test***	***Section***
General Motors Worldwide Engineering Standards (GMW) 14872	*Cyclic Corrosion Laboratory Test*	Cyclic Corrosion Resistance	4.2.2
MSFC-STD-3029	*Guideline for the Selection of Metallic Materials for Stress Corrosion Cracking Resistance in Sodium Chloride Environments*	Stress Corrosion Cracking	4.1.5
NASA-STD-5005	*Standard For The Design And Fabrication Of Ground Support Equipment*	N/A	1
NASA-STD-5008B	*Protective Coating of Carbon Steel, Stainless Steel, and Aluminum on Launch Structures, Facilities, and Ground Support Equipment*	X-Cut Adhesion by Wet Tape Tensile (Pull-Off) Adhesion Atmospheric Exposure Testing	4.1.2 4.1.4 4.2.1
NASA-STD-6001	*Flammability, Odor, Offgassing, and Compatibility Requirements and Test Procedures for Materials in Environments that Support Combustion*	LOX Compatibility	4.1.6
QQ-P-35	*Passivation Treatments for Corrosion-resistant Steel*	N/A	1

Appendix A

Excerpt from Nitric Acid Passivation Process

**PASSIVATION AND PROCESSING OF
SMALL/LARGE PARTS, FITTINGS, AND PIPING
PER FED. SPEC. QQ-P-35**

NOTICE: NEITHER WILTECH CORPORATION NOR ANY PERSON ACTING ON BEHALF OF WILTECH ASSUMES ANY LIABILITY RESULTING FROM THE USE OF INFORMATION CONTAINED IN THIS DOCUMENT.

PASSIVATION AND PROCESSING OF SMALL/LARGE PARTS, FITTINGS, AND PIPING PER FED. SPEC. QQ-P-35

Signature on File	**08/05/05**
PREPARED BY	DATE
Signature on File	**08/09/05**
VALIDATED BY	DATE
Signature on File	**08/10/05**
QA/SAFETY	DATE
Signature on File	**08/10/05**
APPROVAL	DATE

NOTICE: NEITHER WILTECH CORPORATION NOR ANY PERSON ACTING ON BEHALF OF WILTECH ASSUMES ANY LIABILITY RESULTING FROM THE USE OF INFORMATION CONTAINED IN THIS DOCUMENT.

COMPONENT PROCESSING OUTLINE FOR:

**PASSIVATION AND PROCESSING OF
SMALL/LARGE PARTS, FITTINGS, AND PIPING
PER FED. SPEC. QQ-P-35**

ITEM	TASK	DOCUMENT	SECT/APPENDIX	PARAGRAPH
1	DECONTAMINATION (If Applicable)	4-0-385/OP-52	8.1	PER CDR
2	PROOF TESTING (If Applicable)	4-0-385	8.13	8.13.3
3	DISASSEMBLY	NOT APPLICABLE OR PER CDR'S SPECIAL INSTRUCTIONS		
4	PAINT REMOVAL (If Applicable)	4-0-385/OP-52	6.3 8.2	6.3.4 8.2.3
5	PRECLEANING	4-0-385/OP-52	8.3	8.3.11
6	FINAL CLEANING (If Applicable) Small Parts Large Parts Tubing Back-To-Back	4-0-385/OP-52	 8.11 8.11 8.11 8.11	 8.11.12 8.11.3.7 8.11.5.1.2 8.11.5.1.4
7	LUBRICATION AND ASSEMBLY	NOT APPLICABLE OR PER CDR'S SPECIAL INSTRUCTIONS		
8	FUNCTIONAL TEST (If Applicable) Media: GN_2 (or GHe Per CDR)	4-0-385/OP-52	8.18	8.18.3
9	SAFETY WIRING (If Applicable)	4-0-385	8.14	8.14.7
10	PACKAGING Material: Poly (Cleaning NOT Required) Aclar For Oxidizer, LO_2, GO_2, and B. Air Nylon For All Others Media	4-0-385/OP-52	8.12	8.12.3 8.12.4.1 8.12.4.2

IDMM NUMBER K-L24A-6S085 REVISION B

TITLE: PASSIVATION AND PROCESSING OF SMALL/LARGE PARTS, FITTINGS, AND PIPING PER FED. SPEC. QQ-P-35

REVISION	ENGINEER'S NAME	DATE COMPLETED	REASON FOR CHANGE
BASIC	Tom Collins	09/06/83	NEW
REV A	Thuy Vanto	08/12/02	Updated
REV B	Don Kahn	08/05/05	Incorp Pink Sheets/Update to new template
REV C			
REV D			
REV E			
REV F			
REV G			
REV H			
REV I			
REV J			
REV K			
REV L			
REV M			
REV N			
REV O			
REV P			
REV Q			
REV R			
REV S			

CHANGE LOG FOR IDMM# 6S085 **SPC BOC CAPPS**

REV - NO.	CHANGE
Revision B	Removed common language and replaced with links.

1. IDMM CHANGE NO. FORMAT: IDMM REVISION - CHANGE NUMBER 1 THROUGH 9. (EXAMPLE: BASIC-1, REV A-2, REV B-5)
2. WHEN THE IDMM IS REVISED, ALL STANDING IDMM CHANGES WILL BE INCORPORATED.
3. MAXIMUM ALLOWABLE IDMM CHANGES PER REVISION LEVEL IS 9.

ROCKWELL SMALL PARTS PASSIVATION (INCLUDING TUBING AND BACK-TO-BACKS NOT REQUIRING ELECTROPOLISHING) STAINLESS STEEL (300 / 400 SERIES) CLEANING AND PASSIVATION

NOTICE: NEITHER WILTECH CORPORATION NOR ANY PERSON ACTING ON BEHALF OF WILTECH ASSUMES ANY LIABILITY RESULTING FROM THE USE OF INFORMATION CONTAINED IN THIS DOCUMENT.

**ROCKWELL SMALL PARTS PASSIVATION
(INCLUDING TUBING AND BACK-TO-BACKS
NOT REQUIRING ELECTROPOLISHING)
STAINLESS STEEL (300 / 400 SERIES)
CLEANING AND PASSIVATION #**

Signature on File	**07/12/05**
PREPARED BY	DATE
Signature on File	**07/13/05**
VALIDATED BY	DATE
Signature on File	**07/15/05**
QA/SAFETY	DATE
Signature on File	**07/15/05**
APPROVAL	DATE

NOTICE: NEITHER WILTECH CORPORATION NOR ANY PERSON ACTING ON BEHALF OF WILTECH ASSUMES ANY LIABILITY RESULTING FROM THE USE OF INFORMATION CONTAINED IN THIS DOCUMENT.

COMPONENT PROCESSING OUTLINE FOR:

**ROCKWELL SMALL PARTS PASSIVATION
(INCLUDING TUBING AND BACK-TO-BACKS
NOT REQUIRING ELECTROPOLISHING)
STAINLESS STEEL (300 / 400 SERIES)
CLEANING AND PASSIVATION**

ITEM	TASK	DOCUMENT	SECT/APPENDIX	PARAGRAPH
1	DECONTAMINATION (If Applicable) Oxidizer Fuel	4-0-385/OP-52	8.1	 8.1.3.14.2 8.1.3.14.3
2	INSPECTION	Visually examine item(s) for any anomalies. Record as required.		
3	PROOF TESTING (If required)	4-0-385/OP-52	8.13	8.13.3
4	PRECLEANING (MAO110-302)	4-0-385/OP-52	8.3	8.3.11
5	CLEANLINESS VERIFICATION General Requirements Fittings and Piece Parts Tubing Back-to-Backs	OP-52	 8.11 8.11 8.11 8.11	 8.11.3 8.11.12 8.11.5.1.2 8.11.5.1.4
6	FUNCTIONAL TEST (If Required) Media: GN_2	4-0-385	8.18	8.18.3
7	PACKAGING Service Media Oxidizer, LO_2, GO_2, Air: All Others:	4-0-385/OP-52 Material Aclar Nylon	8.12	8.12.4.1 8.12.4.2

IDMM NUMBER K-L24A-6S099 REVISION B

TITLE: ROCKWELL SMALL PARTS PASSIVATION (INCLUDING TUBING AND BACK-TO-BACKS NOT REQUIRING ELECTROPOLISHING) STAINLESS STEEL (300 / 400 SERIES) CLEANING AND PASSIVATION

REVISION	ENGINEER'S NAME	DATE COMPLETED	REASON FOR CHANGE
BASIC	Charlie Mitchell	04/21/89	NEW
REV A	Unknown	Unknown	Unknown
REV B	David Poole	07/12/05	Incorp Pink Sheets/Update to new template
REV C			
REV D			
REV E			
REV F			
REV G			
REV H			
REV I			
REV J			
REV K			
REV L			
REV M			
REV N			
REV O			
REV P			
REV Q			
REV R			
REV S			

CHANGE LOG FOR IDMM# 6S099 SPC BOC CAPPS

REV - NO.	CHANGE
Revision A	Add # to precleaning column.
Rev A-1	Process outline page, Item #4, under the heading TASK, is Precleaning (MAO110-302).
Revision B	Added links.

1. IDMM CHANGE NO. FORMAT: IDMM REVISION - CHANGE NUMBER 1 THROUGH 9. (EXAMPLE: BASIC-1, REV A-2, REV B-5)
2. WHEN THE IDMM IS REVISED, ALL STANDING IDMM CHANGES WILL BE INCORPORATED.
3. MAXIMUM ALLOWABLE IDMM CHANGES PER REVISION LEVEL IS 9.

Appendix B

Citric Acid Passivation Process

CITRIC ACID

PROCEDURE FOR PASSIVATION

1.0 Citric Acid Passivation

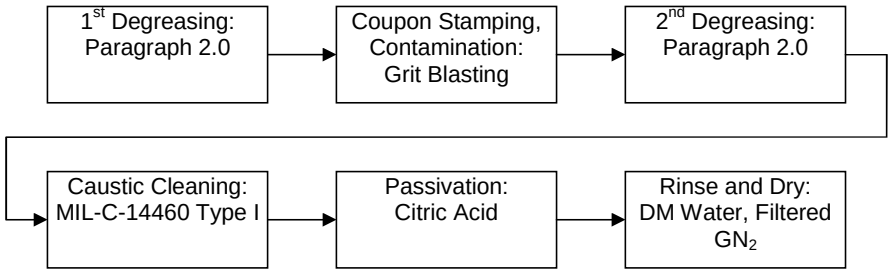

FIGURE 1-1 PROCESS OVERVIEW FLOW CHART

CAUTION

Items with exposed critical surfaces are susceptible to damage during processing. When processing such items, an increased level of care and awareness is mandatory. Do not stack items.

WARNING

Wear face shield, body protection and impermeable rubber gloves when performing hazardous steps.

1.1. Degrease items per paragraph 2.0 of this procedure.

1.2. Metal stamp each coupon in accordance with run table. Number each coupon sequentially, ensuring that the numbers correspond with the coupon materials listed as in Tables 1-1 and 1-2. Center the number on one side only of the 6 inch side approximately ¼ inch from the edge.

NOTE

The following step will be performed at Kennedy Space Center's Corrosion Control Facility by EG&G.

1.3. Grit blast coupons with plain carbon steel shot, both sides, using cross-hatch technique, covering each surface once with horizontal passes and once with vertical passes. Use blast tape to preserve coupon number.

1.4. Degrease items per paragraph 2.0 of this procedure a second time.

1.5. Rack or position parts in a processing basket so that full contact with process solutions and accessibility with rinse waters will be achieved.

1.6. Completely immerse work in Solution E (see Table 3-2) for 15 to 30 minutes.

CAUTION

Ensure a catch basin or drip pan is in place when mechanically applying process solutions to work and that all waste is disposed of properly.

1.7. Flush with demineralized (DM) water for at least 30 seconds. Continue rinsing until wet surface pH is between 6.0 and 8.0.

1.8. Check the wet surfaces for water-breaks. Surfaces shall exhibit continuity of water film when wet with DM water. Formation of droplets or discontinuity of the water film indicates the presence of oily or greasy residue. If a water-break free surface has not been achieved, reclean per paragraphs 1.6 and 1.7 until surface is acceptable. Continue to the next step once an acceptable surface has been achieved.

1.9. Ensure that two bath thermocouples are in place in the Lindberg/Blue M, Model number WB1110A. Reference Figure 1-2 for thermocouple placement. Start temperature data-logger.

1.10. Passivate parts according to the run table. For Citric acid passivation, use the “Constant Temperature Bath” manufactured by Lindberg/Blue M, Model number WB1110A. Position coupons in the bath in accordance with Figures 1-2 and 1-3.

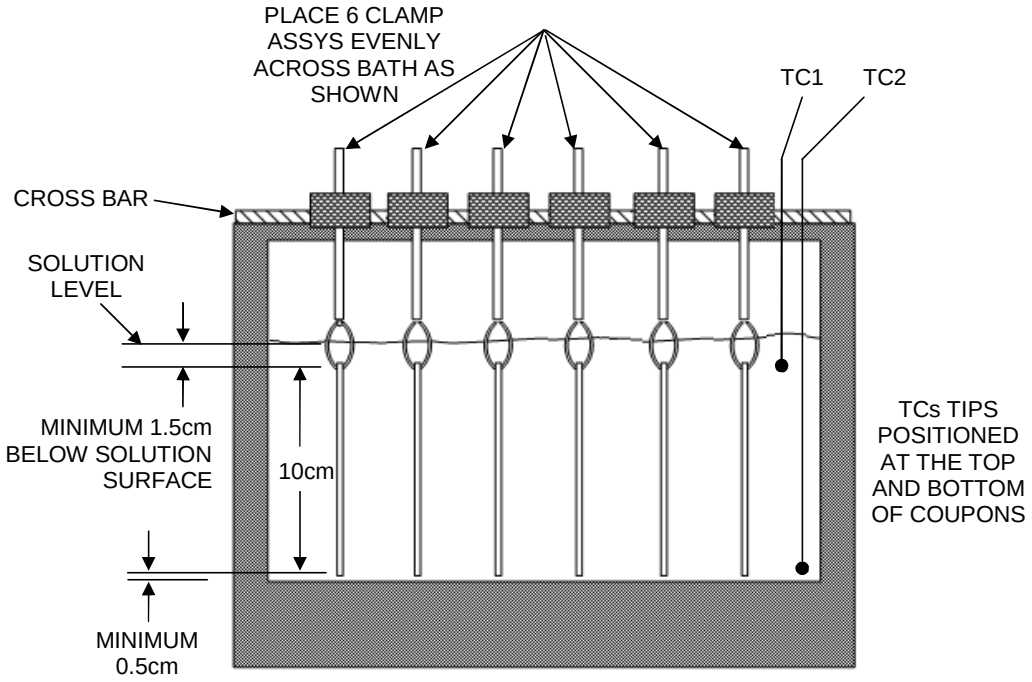

FIGURE 1-2 LINDBERG/BLUE M WATERBATH
MODEL # WB11OA
SECTION VIEW

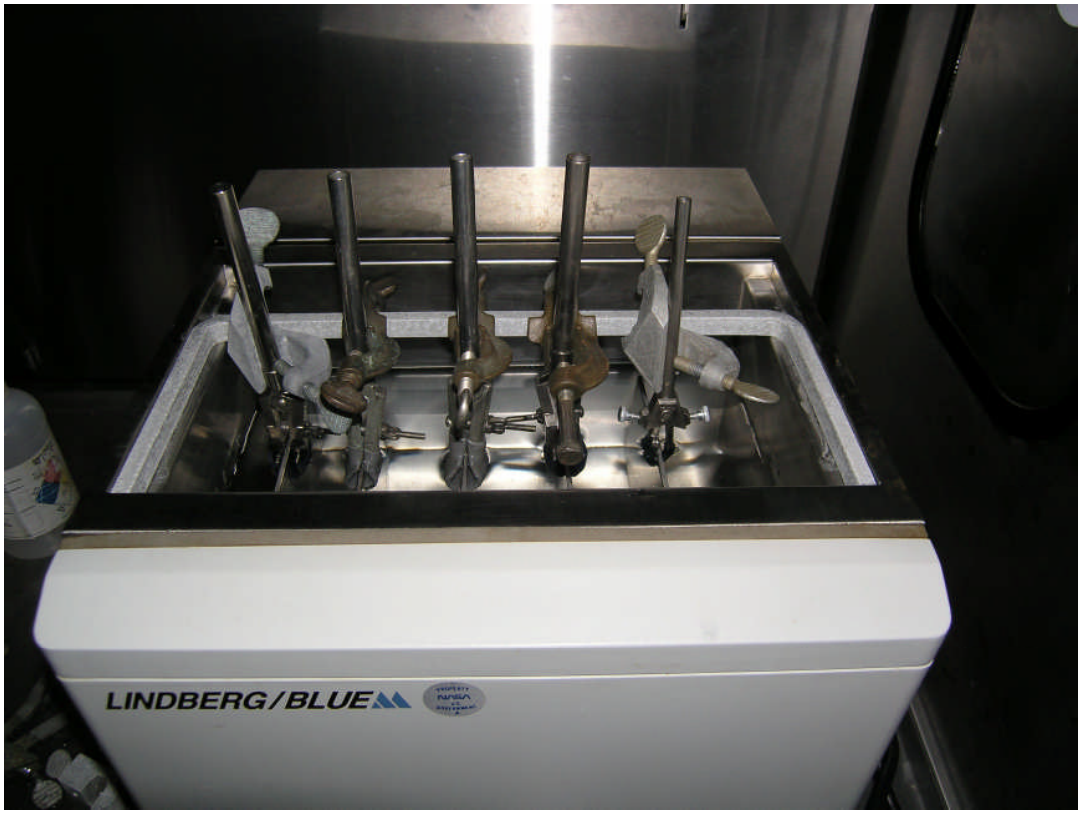

FIGURE 1-3 LINDBER/BLUE M, MODEL # WB1110A SOLUTION BATH

NOTE

After processing, handle parts in a manner to prevent recontamination by dirt, grease, fingerprints, moisture, and any other soil. Personnel handling parts shall wear clean, low-lint gloves. Prepared parts that will require short term storage shall be protected by wrapping with polyethylene material or placing parts in polyethylene bags.

NOTE

When interim packaging is used during processing, it shall be adequate to protect details from contamination, corrosion or damage during handling and storage.

1.11. After passivation, remove coupons from bath and flush with DM water for at least 30 seconds. Continue rinsing until wet surface pH shows no noticeable color change on litmus paper.

1.12. Blow dry the work with filtered GN_2 until visibly dry.

1.13. End temperature data logging session.

1.14. Visually examine parts for evidence of contamination. Reprocess contaminated parts per paragraphs 1.5 through 1.6.

1.15. Package in a single poly zip-top bag. No certification is required.

2.0 Aqueous Degreasing

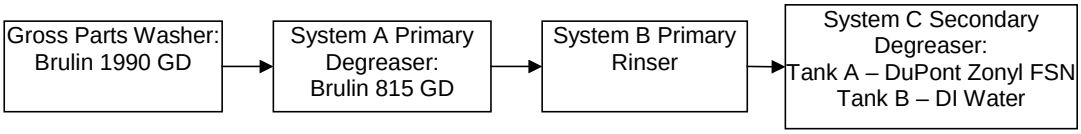

FIGURE 2-1 PROCESS OVERVIEW FLOW CHART

2.1. The aqueous degreasing processing flow is illustrated above.

WARNING

Wear face shield, body protection and impermeable rubber gloves when performing hazardous steps.

CAUTION

Items with exposed critical surfaces are susceptible to damage during processing. When processing such items, an increased level of care and awareness is mandatory.

2.2. Solvent wipe, as required, per paragraph 3.0 to remove labels, tape residues, grease pencil markings, ink, and layout dye.

NOTE

The Hydro P4000 is a suitable substitute for the “Gross Parts Washer” in the removal of gross contamination in subsequent steps. If gross contamination is not evident then the subsequent “Gross Parts Washer” steps may be skipped.

CAUTION

Do not stack parts on top of one another in the rotating basket. Parts with critical surfaces must be positioned so that contact with one another is avoided.

2.3. Rack or position parts in the rotating basket of the "Gross Parts Washer" so that full contact with [Solution OO (see Table 3-2)] spray patterns will be achieved. Use properly sized restraining fixtures, as required, to test coupons if necessary.

2.4. Set the operational controls of the "Gross Parts Washer" per Table 2-1 for this system ("Gross Parts Washer").

2.5. Close the lid and start the unit. Allow the full cycle to terminate before opening the lid.

2.6. After the process cycle is complete, allow the excess fluids to drain/return to sump before opening the lid.

2.7. Visually examine the parts for evidence of gross contamination. Remove the contaminant with the unit's spray wand or reprocess the contaminated parts per paragraphs 2.3 through 2.6.

CAUTION

Do not stack parts on top of one another in the processing basket. Parts with critical surfaces must be positioned so that contact with one another is avoided.

2.8. Remove the parts from the rotating basket of the gross parts washer and carefully place them into proper sized metal processing basket, ensuring they do not touch each other.

2.9. Set the operational controls of System "A" per Table 2-1 for this system.

2.10. Position the work in System "A" so that full contact with the processing solution [Solution MM (see Table 3-2)] is achieved.

CAUTION

Do not touch degreased parts with bare human hands after processing in System “A”. Natural human oils will transfer to clean surfaces of parts.

2.11. When parts are positioned properly within the tank of the Primary Degreaser (System “A”), actuate the start switch and wait for completion of process cycle.

NOTE

To prevent surfactant solutions from drying on hot metal substrates, immediate rinsing of fresh hot DM water (120-150°F) to remove solution residues is required.

CAUTION

Parts removed from System “A” must be moved to System “B” for immediate rinsing.

2.12. Set the operational controls of System “B” per Table 2-1 for this system.

2.13. Turn on the water deluge spray in System “B” and quickly remove degreased parts from System “A” and suspend the parts and basket within the hot demineralized water (DM) water spray, working them in an upward and downward motion for approximately 15 +5 seconds.

2.14. Remove parts and basket from water deluge spray bath and place them into main DM water rinse tank of System “B”.

2.15. When parts are positioned properly within the tank of the Primary Rinser (System “B”), actuate the start switch and wait for completion of process cycle.

2.16. After completion of the rinsing cycle check wet surfaces for water-breaks. Surfaces shall exhibit continuity of water film when wet with DM water. Formation of droplets or discontinuity of water film indicates the presence of oily or greasy residue.

2.17. If a water-break free surface has not been achieved contact engineering for further instructions.

NOTE

Evidence shows that minor contamination is deposited on parts when leaving the chemical processing baths. To assist the cleanliness validation phase of precision cleaning, a secondary degreasing process is required.

CAUTION

Ensure the component parts are placed on a single plane of work, on bottom of basket and not touching one another.

2.18. Rack or position parts in the proper sized metal processing basket, ensuring they do not touch each other.

CAUTION

Verify the surfaces of the component parts are of a neutral pH, 6.0 to 8.0, to prevent acid or alkaline contamination of Solution QQ (see Table 3-2).

2.19. Set the operational controls of System “C” per Table 2-1 for this system.

2.20. Position the work in Tank “A” of System “C” so that full contact with the processing solution [Solution QQ (see Table 3-2)] is achieved.

2.21. When the parts are positioned properly within Tank “A” of the Secondary Degreaser (System “C”), actuate the start switch and wait for completion of process cycle.

NOTE

To prevent surfactant drag-out, pre-rinse the parts back into surfactant bath with the hand wand before placing in main rinse tank “B”.

2.22. Lift parts from Tank “A” and pre-rinse them with the spray wand back into Tank “A”. This will help reduce the effects of surfactant drag-out.

2.23. Position the work in Tank “B” of System “C” so that full contact with the processing solution [Solution JJ (see Table 3-2)] is achieved.

2.24. When the parts are positioned properly within Tank “B” of the Secondary Degreaser (System “C”), press the start switch and wait for completion of the rinsing process.

NOTE

Handle parts, after processing, in a manner to prevent recontamination by dirt, grease, fingerprints, moisture, and any other soil. Personnel handling parts shall wear clean, low-lint gloves. Prepared parts that will require short-term storage shall be protected by wrapping with polyethylene material or placing parts in polyethylene bags.

CAUTION

Icing may occur during vacuum drying - Verify there are no cold spots on parts after drying.

2.25. Dry the work using either the Thermal Vacuum Dryer, Forced Air Over HEPA Dryer or Blow dry the work with filtered GN_2 until visibly dry. Temperature settings for the dryers shall be 150-160°F.

2.26. Visually examine parts for evidence of contamination. Notify engineering for further instructions if contamination is noted.

NOTE

When interim packaging is used during processing, it shall be adequate to protect details from contamination, corrosion or damage during handling and storage.

2.27. Return to passivation processing per paragraph 1.0. If this was the first degreasing, return at paragraph 1.2. If this was the second degreasing, return at paragraph 1.5.

GROSS PARTS WASHER		**SYSTEM A (27KHz)**		**SYSTEM B (40KHz)**		**SYSTEM C (40KHz)**	**A**	**B**
Wash (Minutes)	**10**	Process (Minutes)	**3**	Process (Minutes)	**3**	Process (Minutes)	**3**	**1**
Rinse (Minutes)	**1**	Agitation (Secs. On/Off)	**3**	Agitation (Secs. On/Off)	**3**	Agitation (Minutes)	**3**	
		Ultrasonics Zones 1-4	**OFF**	Ultrasonics Zones 1-4	**AUTO**	Ultrasonics Zones 1-4	**AUTO**	**AUTO**

** Shaded areas represent equipment not used for that Material Class.*

1 =Set ultrasonics **OFF** for Flight & Type I GSE hardware; Set to **AUTO** for other hardware.

TABLE 2-1 EQUIPMENT/LOAD SETTINGS

3.0 Solvent Wipe

CAUTION

Items with exposed critical surfaces are susceptible to damage during processing. When processing such items, an increased level of care and awareness is mandatory.

CAUTION

Do not stack items with exposed critical surfaces.

3.1. Determine type of contaminants, type of materials to be cleaned, and select proper solvent for the cleaning operation from Table 3-1.

Contaminants	Solvent	Flammable (See Caution)	Type of Material (See Note)
Oil, grease, and shop soils, mill markings and tape or decal residue	Solution Q Solution WW	No	All metals and their alloys Painted surfaces and all non-metals except elastomeric (rubber) materials
Identification inks & Most Other Contaminants	Solution S	Yes	Elastomerics (rubber) materials. All metals and their alloys except magnesium and it's alloys
All contaminants	Solution XX Solution NN	No	All metals and their alloys, Painted surfaces and all non-metals, Elastomerics (rubber) materials
Grease pencil markings, layout dye, some mill markings and tape residue	Solution PP Solution DD	Yes	All metals and their alloys except magnesium and it's alloys

TABLE 3-1 SOLVENT USAGE

WARNING

Use non-flammable solvent whenever possible. Use Solutions S, WW, DD, and PP only in well vented areas approved by Industrial Safety. Whenever a solvent splash is possible, Industrial Safety approved eye protection shall be worn.

3.2. Remove loose particulate matter from parts by blowing-off with filtered GN_2.

WARNING

Solvent resistant gloves, eye protection, and body protection shall be worn to prevent skin contact with solvents.

3.3. Dispense solvent onto a clean wiper. Do not dip wiper into solvent.

3.4. Rub or scrub to dislodge the contamination. To spot clean, work around the spot toward its' center to avoid spreading contaminants onto the cleaned surface.

CAUTION

Precautions shall be taken to avoid entrapment of solvents in seams, crevices, between fraying surfaces, etc. If such entrapment does occur remove solvent by blowing with air or GN_2.

3.5. Continue the wiping operation, as necessary, using additional clean wipers and solvent until all contamination is completely removed as indicated by visual examination.

3.6. Wipe dry all accessible surfaces with a clean, dry wiper.

NOTE

Do not return used solvent to the original container. Used wipers shall be placed in Industrial Safety approved containers.

WARNING

Wear eye protection approved by Industrial Safety.

3.7. Dry entrapment areas with filtered GN_2. As an alternate, allow entrapment areas to dry at ambient temperature for 30 minutes minimum.

3.8. Visually examine parts for evidence of oil, grease, layout dye, grease pencil markings, shop soils, or any similar residues. Parts that evidence visual contaminants shall be re-cleaned.

NOTE

Solvent cleaned surfaces shall exhibit no evidence of attack such as etching or pitting of metallic surfaces, softening, smearing, swelling or crazing of non-metallic surfaces, or loss of adhesion at joined material interfaces.

NOTE

Solvent cleaned surfaces shall be visibly dry and free of solvent.

NOTE

Handle parts, after processing, in a manner to prevent recontamination by dirt, grease, fingerprints, moisture, and any other soil. Personnel handling parts shall wear clean, low-lint gloves. Prepared parts that will require short-term storage shall be protected by wrapping with polyethylene material or placing parts in polyethylene bags.

NOTE

When interim packaging is used during processing, it shall be adequate to protect details from contamination, corrosion or damage during handling and storage.

3.9. Return to degreasing at paragraph 2.3.

TABLE 3-2 CLEANING MATERIALS AND SOLUTIONS

SOLUTION	MATERIAL/SOLUTION	APPLICATION/USE LIMITS	MANUFACTURER/ SPECIFICATION	TEMP.	CONC. BY WT/GAL	ANALYTICAL CONTROLS
E	CAUSTIC SCALE REMOVER	SAFE ON STAINLESS STEEL & CARBON STEEL	MIL-C-14460 TYPE I	190° ± 20° F	4.0 TO 6.0 LB/GAL (EQUALS 2-3 LBS NaOH)	TITRATE WEEKLY
S	ISOPROPYL ALCOHOL	DEGREASING, FLUSHING AND SAMPLING	FED-SPEC TT-I-735	AMBIENT	AS PROCURED	PER SE-S-0073, TABLE 6.3-25, WEEKLY
Q	TRICHLOROTRIFLUOROETHANE	VAPOR DEGREASING, ULTRASONICS AND VALIDATION	MIL-C-81302 MSC-SN-C-0037	110° ± 5° AND AMBIENT	AS PROCURED	**ULTRASONICS AND VALIDATION** - H_2O: 60PPM MAX ALCOHOL: 0.3% MAX CHLORIDES: 0.3PPM MAX TWICE WEEKLY **VAPOR DEGREASER** - CHLORIDES: 0.3PPM MAX WEEKLY
DD	ACETONE	PASSIVATION TEST MIL-STD-753 METHOD 102	FED-SPEC O-A-51	AMBIENT	AS PROCURED	N/A
JJ	HIGH PURITY WATER - DM (DEMINERALIZED) WATER, DI (DEIONIZED) WATER, DISTILLED WATER	RINSE, SOLUTION ADDITIVE, VALIDATION FLUID	SE-S-0073 GRADE A	VARIOUS	AS PROCURED	N/A
MM	SURFACTANT DEGREASER	DEGREASING SOLUTION, ULTRASONICS	BRULIN 815 GD	140°-180° F	3% - 10% BY VOL.	TEST DAILY > 8 pH
NN	BIOACT PCG	SPECIALIZED DEGREASING, GLUE AND DECAL REMOVAL	PETROFERM	AMBIENT	> 85%	GAS CHROMATOGRAPH FOR PURITY
PP	MEK	SPECIALIZED PAINT, GLUE AND DECAL REMOVAL	ASTM D-740	AMBIENT	AS PROCURED	N/A
OO	ANTI-FOAM SURFACTANT DEGREASER	DEGREASING SOLUTION, HIGH PRESSURE SPRAY	BRULIN 1990 GD	130°-150° F	1% - 5% BY VOL.	> 8 pH
QQ	NON-IONIC SURFACTANT	SURFACTANT, DEGREASING SOLUTION, VALIDATION SOLUTION	DUPONT ZONYL FSN	AMBIENT TO 140° F	25 PPM IN DM WATER	TEST DAILY OR CONCURRENTLY WITH USE, SURFACE TENSION 28-42 DYNES/CM; NEUTRALITY 6.0-8.0 pH
WW	VERTREL MCA	SOLVENT DEGREASING SOLUTION	DUPONT SE-S-0073 TABLE 6.3-XX	AMBIENT	AS PROCURED	TWICE WEEKLY PER SE-S-0073 TABLE 6.3-XX
XX	HFE-7100	VERIFICATION FLUID / FLUORINATED LUBRICANTS AND LIGHT HYDROCARBONS	3 M SE-S-0073 TABLE 6.3-XX	AMBIENT	AS PROCURED	TWICE WEEKLY PER SE-S-0073 TABLE 6.3-XX