

**Joint Test Protocol  
J-01-WL-001-P6**

**Validation of Corrosion Resistance Properties of  
Lubricants for Military Weapons**

**Revised Draft**

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## TABLE OF CONTENTS

	Page
<b>ACRONYM LIST .....</b>	<b>iv</b>
<b>PREFACE.....</b>	<b>v</b>
<b>JTP REVISION HISTORY .....</b>	<b>vi</b>
<b>1.0 INTRODUCTION.....</b>	<b>1</b>
1.1 Scope.....	1
1.2 Execution .....	1
1.3 Document Maintenance .....	3
<b>2.0 JTP DOCUMENT GUIDE.....</b>	<b>4</b>
<b>3.0 APPLICATIONS .....</b>	<b>6</b>
3.1 Guidelines .....	6
3.2 Methodology .....	7
<b>4.0 TESTING REQUIREMENTS.....</b>	<b>8</b>
4.1 Performance Testing Requirements.....	10
4.1.1 Fluid Lubricants Testing Requirements.....	10
4.1.2 Semi-Fluid Lubricants Testing Requirements .....	11
4.2 Special Testing Requirements.....	12
4.2.1 Fluid Lubricants Testing Requirements.....	12
4.2.2 Semi-Lubricants Testing Requirements.....	12
4.3 Test Descriptions .....	13
4.3.1 Corrosion Protection (Humidity Cabinet).....	13
4.3.1.1 Scope.....	13
4.3.1.2 Equipment .....	13
4.3.1.3 Reagents .....	13
4.3.1.4 Procedure .....	13
4.3.1.5 Acceptance Criteria.....	14
4.3.2 Oxidation Stability .....	15
4.3.2.1 Scope.....	15
4.3.2.2 Equipment .....	15
4.3.2.3 Reagents .....	15
4.3.2.4 Procedure .....	15
4.3.2.5 Acceptance Criteria.....	16
4.3.3 Rubber Compatibility.....	17
4.3.3.1 Scope.....	17
4.3.3.2 Equipment .....	17
4.3.3.3 Reagents .....	17
4.3.3.4 Procedure .....	17
4.3.3.5 Acceptance Criteria.....	18
4.3.4 Rust Inhibition .....	18
4.3.4.1 Scope.....	18
4.3.4.2 Equipment .....	18
4.3.4.3 Reagents .....	18
4.3.4.4 Procedure .....	18
4.3.4.5 Acceptance Criteria.....	19

## TABLE OF CONTENTS (continued)

	<b>Page</b>
4.3.5 Rust Prevention .....	19
4.3.5.1 Scope .....	19
4.3.5.2 Equipment .....	19
4.3.5.3 Reagents .....	20
4.3.5.4 Procedure .....	20
4.3.5.5 Acceptance Criteria .....	20
4.3.6 Copper Corrosion – Rifles .....	20
4.3.6.1 Scope .....	20
4.3.6.2 Equipment .....	20
4.3.6.3 Reagents .....	21
4.3.6.4 Test Procedure .....	21
4.3.6.5 Acceptance Criteria .....	21
4.3.7. Copper Corrosion – Automatic Weapons .....	22
4.3.7.1 Scope .....	22
4.3.7.2 Equipment .....	22
4.3.7.3 Reagents .....	22
4.3.7.4 Test Procedure .....	22
4.3.7.5 Acceptance Criteria .....	22
4.3.8. Rubber Compatibility .....	23
4.3.8.1 Scope .....	23
4.3.8.2 Equipment .....	23
4.3.8.3 Reagents .....	23
4.3.8.4 Procedure .....	23
4.3.8.5 Acceptance Criteria .....	24
4.3.9 Salt Spray (Fog) Resistance .....	25
4.3.9.1 Scope .....	25
4.3.9.2 Equipment .....	25
4.3.9.3 Reagents .....	25
4.3.9.4 Procedure .....	25
4.3.9.5 Acceptance Criteria .....	26
4.3.10 Rust Inhibition .....	26
4.3.10.1 Scope .....	26
4.3.10.2 Equipment .....	26
4.3.10.3 Reagents .....	26
4.3.10.4 Procedure .....	26
4.3.10.5 Acceptance Criteria .....	27
4.3.11 Corrosion Protection from Propellant Reaction .....	27
4.3.11.1 Scope .....	27
4.3.11.2 Equipment .....	27
4.3.11.3 Reagents .....	28

## TABLE OF CONTENTS (continued)

	<b>Page</b>
4.3.11.4 Procedure .....	28
4.3.11.5 Acceptance Criteria.....	28
<b>5.0 FAILURE ANALYSIS .....</b>	<b>29</b>
<b>6.0 REFERENCE DOCUMENTS .....</b>	<b>30</b>

## LIST OF TABLES

Table 1. Test Method Summary for Fluid and Semi-Fluid Lubricants.....	6
Table 2. Fluid Lubricants Performance Testing Requirements .....	10
Table 3. Semi-Fluid Lubricants Performance Testing Requirements.....	11
Table 4. Fluid Lubricants Special Testing Requirements .....	12
Table 5. Reference Documents .....	30

## LIST OF FIGURES

Figure 1. Test Flow Diagram .....	5
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## LIST OF APPENDICES

APPENDIX: List of Technical Stakeholders.....	A-1
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## ACRONYM LIST

ACO	Army Corrosion Office
ARDEC	U.S. Army Research, Development and Engineering Center
ARL	U.S. Army Research Laboratory
ASTM	American Society for Testing and Materials
A2LA	American Association for Laboratory Accreditation
CTC	Concurrent Technologies Corporation
DoD	Department of Defense
JTP	Joint Test Protocol
JTR	Joint Test Report
mg	Milligram
ml	Milliliter
mm	Millimeter
MP	Minimum Performance
NVLAP	National Voluntary Laboratory Accreditation Program
PFL	Product Failure Laboratory
RH	Relative Humidity
SS	Salt Spray
TDPMD	Technology Demonstration for Prevention of Material Degradation
U.S.	United States
VOC	Volatile Organic Compound

## **PREFACE**

This report was prepared by Concurrent Technologies Corporation (*CTC*) under Contract Number Contract No. N65236-02-D-3826. This document was prepared on behalf of, and under guidance provided by the Technology Demonstration for Prevention of Material Degradation (TDPMD) Program. The TDPMD Program is dedicated to reducing the material degradation burden for the Department of Defense (DoD). The objective of the program is to identify, investigate, and validate technologies, procedures, and methodologies that will improve material degradation on Army vehicles and aircraft, thereby reducing life cycle operational costs and maximizing equipment sustainability for the war fighter. The structure, format, and depth of technical content of the report were determined by the TDPMD technical associates, pertinent United States (U.S.) Army personnel, government contractors, and other government technical representatives (hereafter referred to as “stakeholders”) in response to the specific needs of this project.

The *CTC* Joint Test Protocol (JTP) team wishes to thank the participants involved in the creation of this document for their invaluable contributions:

U.S. Army Corrosion Office

U.S. Army Armament Research, Development and Engineering Center (ARDEC)

U.S. Army Research Laboratory (ARL)

### JTP REVISION HISTORY

This section will serve as a means to document revisions and discussions regarding this JTP only. It is intended to help the reader identify updated versions of the JTP, and to organize periodic updates of the JTP as new materials and techniques become available. If the latest entry on the JTP Revisions History is more than two (2) years old, the entry “No revisions have been made for the year 20xy” should be entered where appropriate.

[illegible]

## 1.0 INTRODUCTION

This Joint Test Protocol (JTP) contains the critical test requirements to validate the corrosion resistance properties of lubricants for United States (U.S.) military weapons. This document is a protocol for testing and assessing the performance of any fluid lubricant or semi-fluid lubricant that will hereafter be referred to simply as the “candidate.”

### 1.1 Scope

This JTP presents a plan for the assessment of a candidate to be used in military weapons. This document covers both fluid and semi-fluid lubricants. Solid film lubricants are not covered in this JTP since they are more appropriately categorized as coatings; therefore, they have significantly different properties than fluids and semi-fluids lubricants, and consequently require different tests.

This JTP establishes the minimum corrosion resistance performance requirements that must be met for a candidate to be considered for possible use on military weapons. The candidate must then undergo the remaining physical property tests in the military specifications for further consideration on a specific military weapon. The candidate can then be tested on the specific military weapon. These additional evaluations and tests are specific to the application and are outside the scope of this document.

**It must be emphasized that this JTP document is not a process, material, or product specification, nor is it intended to address ongoing quality issues.** The testing outline in this document confirms the technical capabilities of the candidate for the particular application with respect to corrosion resistance, and qualifies the candidate for consideration for military use by the relevant armed services Corrosion Office invoking the JTP (e.g., the Army Corrosion Manager) or the relevant Program Manager (hereafter referred to as “the invoking authority.” It should also be emphasized that successful completion of the procedures outlined in this JTP does not obligate the U.S. Army or any other Department of Defense (DoD) organization to procure or use the candidate.

### 1.2 Execution

This document is organized in such a manner as to aid the user during the corrosion study planning stage, throughout the testing activity and during the reporting and interpretation phases. This section describes the use of this document by outlining the steps that will guide the user through the process of extracting and utilizing corrosion data. Section 2.0 describes a logical flow to the process of evaluating the results of the corrosion tests. Section 2.0 also provides a test flow diagram. Section 3.0 outlines a requirements summary and its applications. Section 4.0 establishes test requirements (acceptance criteria), test descriptions, and procedures, and Section 5.0 discusses failure analysis. Finally, Section 6.0 provides a list of reference documents that were utilized in the preparation of this JTP.



The performance of candidates under this JTP will be determined through a series of tests. These tests have been derived from engineering, performance, and operational impact (supportability) requirements defined by a consensus of government and industry participants. Many of the tests in this document are based upon recognized commercial and military test standards that are currently in use by relevant test facilities. **In instances where the JTP test method conflicts with the reference standard on which it is based, the JTP test method will take precedence.** The candidate must demonstrate Minimum Performance (MP) under a series of Performance Tests to be considered as an alternative under the authority of this JTP.

Prior to conducting the required Performance and Special Tests, a candidate must undergo a preliminary Feasibility Study, in which the following considerations shall be addressed:

- The candidate must conform to current military environmental regulations and concerns such as atmospheric and groundwater impact, volatile organic compounds (VOCs), disposal, etc., with respect to the use of the candidate.
- Procurement of the candidate must be compatible with standard military business procedures. Considerations include, but are not limited to: distribution status (domestic/offshore), product cost analysis, and vendor capability, reputation, and reliability.

**The Feasibility Study will be conducted prior to the execution of the test program contained in this JTP.** The business issues assessment should be repeated again at the completion of the JTP testing if business issues have changed as a result of product and/or financial changes. The actual implementation of the Feasibility Study shall be conducted under the direction of the invoking authority, and is outside the scope of this JTP.

The testing requirements outlined in this JTP are organized into two categories, Fluid Lubricants and Semi-Fluid Lubricants. Under the Fluid Lubricants category, the requirements are subdivided into Performance and Special Testing requirements. Performance Testing requirements are those tests required for validating any candidate on weapons; the candidate must pass the Performance Tests under these requirements in order to be considered for military use. Special Testing requirements are those requirements identified by some (but not all) technical stakeholders for validating candidates in certain applications, such as exposure to particularly unusual environments. The fluid candidate must meet both Performance and Special Testing requirements to be considered for Special applications. Under the Semi-Fluid Lubricants category, there are only Performance Testing requirements.

Fluid lubricants are considered to be liquid in form, with low viscosity and flow properties approaching those of water. They are commonly called oils. Semi-fluid lubricants have flow properties that are intermediate between solids and liquids and are highly viscous. They are commonly called greases.

A Joint Test Report (JTR), consisting of the results and analysis of actual performance tests and failure analysis tests, will document the testing conducted on each candidate in accordance with this JTP. The JTR will provide a record of experimental specifics, equipment designations and calibration, and laboratory environmental conditions, as well as the results of the testing. If planned execution of the tests varies from that described in this JTP, test procedure modifications must be approved by the technical stakeholders and/or the invoking authority in advance and documented in the JTR. The JTR will be made available as a reference for future endeavors by other DoD and commercial users to minimize duplication of effort.

### **1.3 Document Maintenance**

Annual updates and general maintenance of this document will be the responsibility of a committee chaired by the Army Corrosion Manager or designee. This document should be reviewed and updated on an annual basis with changes being noted on the “JTP Revisions History” page. If no changes have been made, the entry “no revisions have been made for the year 20xy” should be entered where appropriate. This document is considered to be obsolete if the latest entry on the JTP Revisions History is more than two years old. In this case, contact the Army Corrosion Office (ACO) or designee for the most recent revisions before conducting testing in accordance with this JTP.

## 2.0 JTP DOCUMENT GUIDE

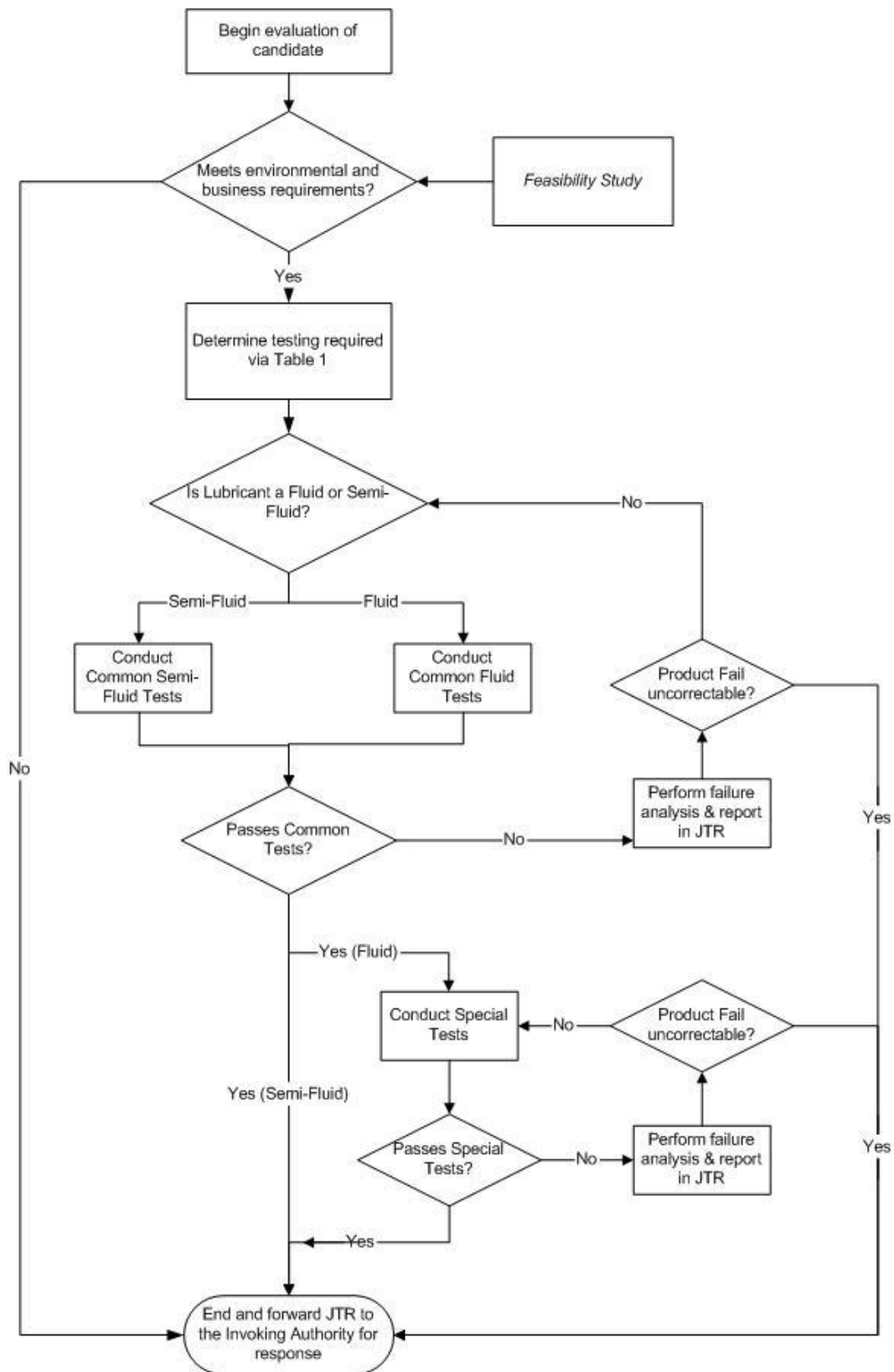
This section of the JTP provides a logical implementation flow process diagram for evaluation of a weapons lubricant candidate. Use of this document for military consideration of a candidate utilizing the Performance and Special Testing sections is described and demonstrated.

Any candidate that is to be considered technically acceptable must meet the criteria for each Performance Test, as established in Section 4.0, Engineering Requirements, Test Descriptions, and Procedures.

A failure analysis can be performed on any candidate that fails either Performance or Special Testing to determine the cause of failure (see Section 5.0) at the vendor's request, if the vendor feels that a failure analysis may be used to show that some processing procedure or material characteristic that caused the failure can be corrected, thus allowing successful retesting. Failure in any test does not necessarily disqualify a candidate for use in all possible applications; however, use of a candidate that has failed either Performance or Special Tests is at the discretion of the invoking authority, and is outside the scope of this document.

Figure 1 illustrates the process flow for conducting Performance and Special Tests, as well as the analysis of candidates that have failed one of the aforementioned tests. The process flow will be similar for both fluid and semi-fluid lubricants. The evaluation process begins with the Feasibility Study. If the candidate conforms to current military environmental regulations, and procurement of the candidate is compatible with standard military business procedures, "Fluid" or "Semi-Fluid" candidates the required testing for either form is determined via Table 1.

Note: In Figure 1 below, there are two potential "infinite loops" that might occur due to testing failures. To avoid this, a provision has been inserted that if failure occurs for any of the Performance Tests after the third cycle, this process is to end and be documented in the JTR, which is forwarded to the vendor for transmittal to the Invoking Authority's response. This provision is likewise applicable for the Fluid Lubricants' Special Tests.



**Figure 1. Test Flow Diagram**

### 3.0 APPLICATIONS

#### 3.1 Guidelines

This section establishes the guidelines for testing a potential candidate for use on military weapons.

Table 1 lists the Performance and Special Tests to be applied for a potential candidate in the Fluid Lubricants and Semi-Fluid Lubricants categories. The table also provides the location of the test procedure within the JTP document along with its applications.

**Table 1. Test Method Summary for Fluid and Semi-Fluid Lubricants**

Fluid Lubricants			Semi-Fluid Lubricants		
JTP Section	Test	Applications	JTP Section	Test	Applications
4.1	<b>Performance Tests</b> Corrosion Protection (Humidity Cabinet)	Low-temperature small arms weapons	4.1	<b>Performance Tests</b>	
4.3.1			4.3.4	Rust Inhibition	Automatic weapons
		Small & large caliber weapons	4.3.5	Rust Prevention	Automatic weapons
	Oxidation Stability	Low-temperature small arms weapons	4.3.6	Copper Corrosion	Rifles
4.3.2			4.3.7	Copper Corrosion	Automatic weapons
4.3.3	Rubber Compatibility	Low-temperature small arms weapons	4.3.8	Rubber Compatibility	Aircraft machine guns
4.2	<b>Special Tests</b>	All tests in this section apply to small & large caliber weapons	4.2	<b>Special Tests</b>	
4.3.9	Salt Spray (Fog) Resistance			Not Applicable	
4.3.10	Rust Inhibition				
4.3.11	Corrosion Protection from Propellant Reaction				

## **3.2 Methodology**

The guidelines for testing candidates under this JTP would be as follows:

1. Determine what tests are required.
2. Perform appropriate testing and obtain test results.
3. Document the test results in a JTR.
4. Submit the JTR to the invoking authority for review.

Performance and Special Tests will be conducted in accordance with the test descriptions and procedures outlined in Section 4.0 of this JTP. The Performance and Special Tests and minimum acceptance criteria are listed in Tables 2 through 4 of this JTP.

## 4.0 TESTING REQUIREMENTS

The technical requirements listed herein establish the acceptable characteristics of candidates for use on military weapons. These requirements have been used to derive tests, with procedures, methodologies, and acceptance criteria, to qualify the candidates. In this section, the requirements are organized into two categories: Fluid Lubricants, which is further subdivided into Performance and Special Testing requirements, and Semi-Fluid Lubricants, with only Performance Testing requirements.

Performance Test methods are identified along with acceptance criteria in Section 4.1. Special Test methods are identified along with acceptance criteria in Section 4.2. Special Tests are program-specific requirements identified by at least one of the stakeholders. Special Tests are performed on actual materials utilizing the candidate. Detailed test descriptions, test procedures, and acceptance criteria are further provided in Sections 4.3. **In instances where the JTP test method conflicts with the reference standard on which it is based, the JTP test method will take precedence.**

All testing shall be performed by a government or independent testing laboratory, which shall be agreed upon by the stakeholders and either be accredited by a recognized governing body (such as the American Association for Laboratory Accreditation (A2LA) or the National Voluntary Laboratory Accreditation Program (NVLAP)) or be an ISO-certified company having its own testing laboratory. **Vendor-supplied testimonials shall be used for informational purposes only, and are not to be used in lieu of tests required under this JTP.** Incorporation of previous studies performed on the candidate by an outside laboratory, at the request of the vendor, is at the discretion of the invoking authority. All tests shall be conducted in a manner that will eliminate duplication and maximize the use of each test specimen; where possible, more than one test should be performed on each candidate specimen. The number and types of tests that can be run on any candidate specimen will be dependant upon the destructiveness of the tests.

The tests described in this JTP may involve the use of hazardous materials, operations, and/or equipment. This JTP does not address all safety issues associated with their use. It is the responsibility of each user of this JTP to establish appropriate safety and health practices, and to determine the applicability of regulatory limitations, prior to the use of such materials, operations, and/or equipment.

The following conditions will apply to all Performance Testing (Fluid and Semi-Fluid) and Special Testing, unless otherwise specified in an individual test description:

- It is preferred that all test coupons be produced from the same material lot.
- Unless otherwise specified, all coupons will be cleaned prior to pretreatment to ensure surfaces are free of water breaks in accordance with the latest version of American Society for Testing and Materials (ASTM) G1, "Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens." Surface cleanliness will be verified by testing in accordance with the latest version of

ASTM F22, “Standard Test Method for Hydrophobic Surface Films by the Water-Break Test.”

- Pretreatment of the test coupons will be dependent upon the lubricant under scrutiny, and will be specified in the JTR.

It is recommended that users of this JTP obtain copies of previous JTRs, if available, from the invoking authority for additional test details or minor modifications that were necessary in the execution of previous testing. Any test procedure modifications described in the JTR will have been agreed upon by the technical stakeholders or the invoking authority before any testing is conducted.



## 4.1 Performance Testing Requirements

### 4.1.1 Fluid Lubricants Testing Requirements

Table 2 lists Performance Testing requirements identified by stakeholders for validating fluid lubricant candidates for use on military weapons.

**Table 2. Fluid Lubricants Performance Testing Requirements**

JTP Section	Test	Applications	Minimum Acceptance Criteria	Test Method References
4.3.1	Corrosion Protection (Humidity Cabinet)	Low-temperature, -59°C to 18°C (-70°F to 0°F) small arms weapons	After 400 hours exposures, test panel shall not have more than three corrosion spots, none of which exceeds one millimeter (mm) in length, width or diameter. One large corrosion spot exceeding one mm in size will be cause for rejection.	FED-STD-791C Method 5329.2 & ASTM D1748
		Small & large caliber weapons, -54°C to 66°C (-65°F to 150°F)	After 900 hours exposure, test panel shall not have more than three corrosion spots, none of which exceeds one mm in length, width or diameter. One large corrosion spot exceeding one mm in size will be cause for rejection.	
4.3.2	Oxidation Stability	For low-temperature small arms weapons	<ul style="list-style-type: none"><li>Change in weight of copper and steel specimens shall be less than 0.2 mg/cm<sup>2</sup>.</li><li>Oil viscosity at -54 °C (-65°F) shall not exceed 1200 centistokes.</li><li>Neutralization number shall not increase more than 0.5 mg potassium hydroxide (KOH).</li><li>Neutralization number of any formed volatile compounds shall not increase more than 0.5 mg KOH.</li><li>No visible evidence of separation of insoluble materials or gumming of oil.</li></ul>	MIL-L-14107C Para. 4.6.4, and 3.7 & ASTM D974
4.3.3	Rubber Compatibility	For low-temperature small arms weapons	<ul style="list-style-type: none"><li>Swelling of standard synthetic rubber Type L by the lubricant shall not exceed 15%.</li></ul>	FED-STD-791C Method 3603.5

Unless otherwise specified, Performance Tests shall be performed on material utilizing the candidate. Results of the Performance Tests are reported in the JTR and submitted to the vendor for transmittal to the invoking authority.

The Fluids Performance Tests (identified in Table 2) and Semi-Fluids Performance Tests (identified in Table 4) are further defined in Section 4.3 with test descriptions, scope, and methodology. Also included are any major or unique equipment and instrumentation requirements, reagents, test procedures, and acceptance criteria. The test procedure identifies the test specimen preparation, test procedure, and method for collecting and reporting test results.

#### 4.1.2 Semi-Fluid Lubricants Testing Requirements

Table 3 lists Performance Testing requirements identified by stakeholders for validating semi-fluid lubricant candidates for use on military weapons.

**Table 3. Semi-Fluid Lubricants Performance Testing Requirements**

JTP Section	Test	Applications	Minimum Acceptance Criteria	Test Method References
4.3.4	Rust Inhibition	Automatic weapons (operating temperature range of -54 °C to 127 °C (-65 °F to 260 °F))	Not more than one of four test specimens that fails by either one rust spot larger than one millimeter (mm) in length, width or diameter; or three or more rust spots smaller than one mm in length, width or diameter, but that increase in size over a 24-hour period.	MIL-L-46000C Para. 4-10 & ASTM D1748
4.3.5	Rust Prevention	Automatic weapons	At least three of four exposed test panels shall be free of any visible rust.	MIL-PRF-85336B Para. 4.5.5
4.3.6	Copper Corrosion	Rifles	Copper strip shall show no evidence of green color, pitting or etching, nor shall any brown or black stain remain on the strip after washing with normal hexane.	FED-STD-791C Method 5309.5
4.3.7	Copper Corrosion	Automatic weapons	Copper strips shall show no brown or black stains. Slight darkening or slight green stain in the lubricant is acceptable.	Fed-STD-791C Method 5304.5
4.3.8	Rubber Compatibility	Aircraft machine guns	Change in weight in MIL-P-5516 O-rings shall be 3% maximum between the candidate lubricant and MIL-L-19701B, Table IV lubricant formulation results.	MIL-L-19701B Tables I, II & IV & ASTM D471 Sections 7, 8, 10 & 17

Performance Testing identified in Table 3 is further defined in this section to include test descriptions, scope, and methodology. Also included, as needed, are any major or unique equipment and instrumentation requirements, and data analysis procedures. The test procedure includes the definition of test parameters and conditions, test specimens and substrates, number of trials per specimen, any baseline (experimental control) specimens required, and acceptance criteria.

## 4.2 Special Testing Requirements

### 4.2.1 Fluid Lubricants Special Testing Requirements

Table 4 lists Special Testing requirements identified and required by some (but not all) technical stakeholders for validating fluid lubricant candidates for use on military weapons for special applications.

**Table 4. Fluid Lubricants Special Testing Requirements**

JTP Section	Test	Applications	Minimum Acceptance Criteria	Test Method References
4.3.9	Salt Spray (Fog) Resistance	For small and large caliber weapons	Not more than three small corrosion spots per test panel, none exceeding one mm in length, width or diameter on any of the test panels. One large corrosion spot exceeding one mm in size on any of three panels will be cause for rejection.	FED-STD-791C Method 4001.3 & Method 5329.2 Para. 6.1 through 6.3b
4.3.10	Rust Inhibition	For small and large caliber weapons	Lubricant shall not produce visual evidence of pitting, etching, dark discoloration or mass change for the following metals in excess of: Zinc 1.5 mg/cm <sup>2</sup> Aluminum 0.2 " Brass 1.0 " Steel 0.2 " Copper 1.5 " Magnesium 0.5 " Cadmium 1.5 "	MIL-L-63460D Amendment 6 Para. 4.10
4. 3.11	Corrosion Protection from Propellant Reaction	For small and large caliber weapons	No rust spots 2 mm or larger involving visible pitting or etching of the metal shall be evident on any of the three humidity cabinet test panels.	MIL-L-63460D Amendment 6 Para. 4.13 & ASTM D1748 Appendix I

Tests identified in Table 4 are further defined in this section to include test descriptions, scope, and methodology. Also included, as needed, are any major or unique equipment and instrumentation requirements. The test procedure includes the definition of test parameters and conditions, test specimens and substrates, number of trials per specimen, any baseline (experimental control) specimens required, and acceptance criteria.

### 4.2.2 Semi-Fluid Lubricants Special Testing Requirements

None.

## 4.3 Test Descriptions

### 4.3.1 Corrosion Protection (Humidity Cabinet)

#### 4.3.1.1 Scope

This method determines the effectiveness of corrosion-inhibiting lubricants under conditions of high humidity. The test consists of dipping steel panels in the lubricant and then placing them in a humidity cabinet. A 400-hour exposure time is specified for low-temperature small arms weapons (–70°F to 0°F) and a 900-hour exposure time for small and large caliber weapons (–65°F to 150°F).

#### 4.3.1.2 Equipment

Test sample size: Approximately 500 milliliter (ml) of the lubricant to be tested.

Humidity cabinet: Conforming to Appendix I of ASTM D1748.

Desiccators: Containing an indicating desiccant.

Panel hook: Stainless steel (for handling panels during processing).

Hooks: Monel or stainless steel (for supporting steel panels).

Sandblast cabinet: A suitable apparatus for sandblasting test panels.

Beakers: Tall-form, 500 ml.

Shaded fluorescent light: A 15-watt balance illuminator type that will permit the panel to be viewed from all angles at a distance of 7.6 cm (3").

Surgical gauze.

Silica sand: White, dry, sharp. The size must meet the following sieve requirements of RR-S-366: 100% must pass through a No. 10 sieve; a minimum of 90% must pass through a No. 20 sieve; and a maximum of 10% shall be permitted to pass through a No. 50 sieve.

#### 4.3.1.3 Reagents

Naphtha: TT-N-95.

Methanol: O-M-232.

#### 4.3.1.4 Procedure

FS 1009 steel panels (3): Fabricated from open-hearth, low carbon, No. 4 soft temper, cold-rolled sheet or strip conforming to QQ-S-698. Panel dimensions must conform to Appendix I of ASTM D1748. Cold drawn bar stock is not satisfactory.

Preparation: Emboss or scribe an identification number on the backside of each steel panel. Round the edges and ream out the suspension holes in accordance with Appendix I of ASTM D1748. Wipe the surfaces of the panels with solvent-soaked rags and scrub with surgical swabs in a beaker of hot naphtha. Rinse in a beaker of hot methanol. Cool the panels in desiccators until further processing. Test panels must be handled with a panel hook at all times, avoiding contact with any type of contaminated surface.

Maintain the solvents at a temperature high enough to keep the temperature of the panels above the dew point during the cleaning operation.

Caution: Naphtha is flammable. Use only in a well-ventilated area. Methanol is both toxic and flammable. Do not allow it to come into contact with the skin or breathe its fumes. Keep all flames away from naphtha and methanol.

Sandblast the edges of the panels and lightly sandblast the back of the panels with silica sand. Sandblast the unnumbered side, or test surface, of the panels to a fresh, uniformly abraded surface. Immediately after sandblasting, place the panels in a beaker of anhydrous methanol. Heat the anhydrous methanol so that the solvent will evaporate from the panels immediately after withdrawal from the solvent. Remove the remaining residue by holding the panels in a rack at approximately 20 degrees from the vertical and spraying downward with the naphtha. Spray the test surface, then the back of the panel and the test surface again. Rinse the panels in hot naphtha and hot methanol and store in a desiccator until cool. Panels are to be used the same day as prepared.

Test procedure: Dip the test panels in the test sample at a temperature of  $24^{\circ}\text{C} \pm 3^{\circ}\text{C}$  ( $75^{\circ}\text{F} \pm 5^{\circ}\text{F}$ ) and agitate gently for one minute. Suspend the panels by means of monel or stainless steel hooks and drain for two hours in a room held at  $24^{\circ}\text{C} \pm 3^{\circ}\text{C}$  ( $75^{\circ}\text{F} \pm 5^{\circ}\text{F}$ ) and a maximum of 55% relative humidity. Place the panels in the humidity chamber described in Appendix I of ASTM D1748 for the time required by the applicable specification (400-hour exposure for low-temperature small arms weapons and 900-hour exposure for small and large caliber weapons). Dummy panels, fabricated from monel or stainless steel, are used to fill all spaces not occupied by test panels. They are also placed under the three arm supports of the rotating stage. Open the cabinet twice each day, except Saturday and Sunday, once for 15 minutes and again for five minutes (approximately five hours between openings). Check the rate of air to the cabinet, air temperature, pH and water level and regulate. Check remaining standards weekly. Maintain test room atmosphere at a temperature of  $24^{\circ}\text{C} \pm 3^{\circ}\text{C}$  ( $75^{\circ}\text{F} \pm 5^{\circ}\text{F}$ ) and a maximum relative humidity of 55%. At the end of the exposure period, remove the panels and rinse in methanol. Follow with a rinse in naphtha and methanol and examine the significant area of the test surface as defined in Appendix I of ASTM D1748, using a 15-watt, shaded fluorescent light.

Report: Record the number of corrosion spots that are 1 mm in diameter or less and the number of corrosion spots that are greater than 1 mm in diameter.

#### **4.3.1.5 Acceptance Criteria**

One large corrosion spot exceeding one mm in size on any of the three panels will be cause for rejection. If more than three small corrosion spots, none of which exceeds one mm in length, width or diameter, are evident on each test panel this shall be cause for rejection. The total number of small corrosion spots on all three test panels shall not exceed three per panel. Corrosion on the outer 6 mm ( $\frac{1}{4}$ " ) of the panels shall not be cause for rejection. These acceptance criteria are applicable for low-temperature small arms weapons with a 400-hour exposure, and small and large caliber weapons with a 900-hour exposure.

## 4.3.2 Oxidation Stability

### 4.3.2.1 Scope

This test determines the oxidation stability of corrosion-inhibiting lubricants. The test consists of passing air through a test tube containing the lubricant with copper and steel specimens immersed in the lubricant while the tube is held in a water bath maintained at  $100^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  ( $212^{\circ}\text{F} \pm 1^{\circ}\text{F}$ ) for a period of 168 hours.

### 4.3.2.2 Equipment

Pyrex test tube: 50 cm (20") long by 5 cm (2") in diameter fitted with an air inlet tube and a reflux condenser.

Test sample size: Approximately 500 ml of the lubricant to be tested.

300-ml Erlenmeyer flask.

Trap: Containing  $50 \pm 1$  ml of 0.1 normal potassium hydroxide (KOH) diluted with 150 ml of distilled water or water of equal purity.

Two towers: One containing soda lime and the second containing glass wool. Size of the towers shall be sufficient to handle the airflow rate.

Balance: Sensitivity 1 milligram (mg).

Horizontal metal polishing wheel.

Phenolphthalein indicator.

240-grit silicon carbide or aluminum oxide abrasive.

Viscosity measuring equipment (suitable for the test lubricant's viscosity range).

### 4.3.2.3 Reagents

Dry cleaning solvent: P-D-680.

Naphtha: TT-N-95.

Methanol: O-M-232.

Benzene.

Acetone.

Ethanol: Neutralized grade.

Sulfuric acid.

Potassium Hydroxide (KOH).

### 4.3.2.4 Procedure

Two metal specimens: Each approximately 4.45 cm (1.75") by 0.95 cm (0.375") by 0.06 cm (0.025"). One specimen made from copper with slit conforming to QQ-C-576. One specimen made from low carbon steel with slit conforming to FS 1009, QQ-S-698.

Preparation of test specimens: Polish the specimens to remove all pits, burrs and irregularities from the faces and edges. Initial polishing may be done with a slow speed horizontal metallurgical polishing wheel. Final polishing may be done with a 240-grit silicon or aluminum oxide abrasive having cloth or paper backing moistened with solvent. After polishing, spray the specimens with naphtha followed by a rinse in hot

naphtha and hot methanol. Do not touch the specimens with the hands after polishing and cleaning.

Caution: Naphtha is flammable. Use only in a well-ventilated area. Methanol is both toxic and flammable. Do not allow it to come into contact with the skin or breathe its fumes. Keep all flames away from naphtha and methanol.

Test procedure: Measure the initial viscosity of the lubricant (for comparison with that of the oxidized lubricant). Weigh the metal specimens. Pour lubricant weighing  $150 \pm 5$  grams into the Pyrex test tube fitted with an air inlet tube and reflux condenser. Connect the copper and steel specimens with a copper wire and support them with a glass holder equipped with glass hooks. Arrange the specimens so they will not touch each other during the test. Immerse the specimens in the lubricant filled test tube so they are completely covered. Place the tube and condenser assembly in a water bath maintained at  $100^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  ( $212^{\circ}\text{F} \pm 1^{\circ}\text{F}$ ). By means of a glass tube, pass air through the two towers, one containing soda lime and the second containing glass wool, and then through the lubricant at a rate of  $5 \pm 0.2$  liters per hour. Pass the gases, that escape through reflux condenser into a trap containing  $50 \pm 1$  ml of 0.1 normal KOH diluted with 150 ml of distilled water or water of equal purity. Conduct the test for a total of  $168 \pm 1$  hours. At the end of the test, remove the test tube and condenser assembly from the bath, disassemble it and remove the metal specimens. Wash the metal specimens with reagent-grade benzene and reagent-grade acetone. Weigh the specimens. Examine the oxidized lubricant for insoluble materials and gumming. Determine the viscosity of the oxidized lubricant at  $-53.9^{\circ}\text{C} \pm 0.1^{\circ}\text{C}$  ( $-65^{\circ}\text{F} \pm 0.2^{\circ}\text{F}$ ). Determine the neutralization number of the lubricant by ASTM D974. If this does not provide a definite end point, the alternate procedure described in the next paragraph may be used. Determine the acid number of the volatile material collected in the trap as specified in the last paragraph of this section.

Alternate procedure for neutralization number: Weigh  $10 \pm 0.5$  grams of lubricant to the nearest 0.1 gram into a 300 ml Erlenmeyer flask and add  $50 \pm 1$  ml of neutralized reagent grade ethanol. Agitate the flask thoroughly, heat it to boiling and cool it to room temperature. Remove the lubricant by decantation. Heat the alcohol phase in the flask to boiling and titrate to the end point using phenolphthalein as the indicator. If the end point is in doubt, add  $50 \pm 2$  ml of boiling neutralized distilled water and observe the solution for the end point color. If necessary, additional titration will produce the desired color that marks the end point.

Acid number of volatile materials: Determine the acid number of the volatile materials formed by the oxidation of the lubricant by back titration of the solution in the KOH trap. Add a known amount of 0.1 normal sulfuric acid, approximately 10% in excess of the KOH contained in the solution. Boil the mixture for  $5 \pm 1$  minutes under a reflux condenser. Remove the reflux condenser and titrate the hot solution with standard 0.1 normal KOH using phenolphthalein indicator. Record all test data.

#### 4.3.2.5 Acceptance Criteria

The change in weight of the copper and steel specimens shall not exceed 0.2 mg per square centimeter of surface. Oxidized oil viscosity shall not exceed 1200 centistokes at  $-54^{\circ}\text{C}$  ( $-65^{\circ}\text{F}$ ). Lubricant's neutralization number shall not have increased by more than 0.5 mg KOH from the lubricant's original neutralization number. The acid number of

any volatile materials formed shall not exceed 0.5 mg KOH. There shall be no visual evidence of separation of insoluble materials or gumming of the lubricant.

### 4.3.3 Rubber Compatibility

#### 4.3.3.1 Scope

This test method is used to determine the effect of the lubricant on synthetic rubber. The test consists of determining the initial and final volumes of three standardized rubber sheets by water displacement, before and after storing them in the lubricant for 168 hours at 70°C (158°F), then determining the average change in volume of the sheets.

#### 4.3.3.2 Equipment

Test tube: 5 cm (2") in diameter and 40 cm (16") long.

Test sample size: Approximately 300 ml of lubricant.

Balance: Sensitivity 1 mg, suitable for weighing in both air and water.

Oven: Gravity-convection, 70°C ± 1°C (158°F ± 2°F).

Filter paper.

Tank for water displacement measurements (for measuring rubber sheet volumes).

Graduated Cylinder

#### 4.3.3.3 Reagents

Distilled water.

Ethanol or methanol: Anhydrous.

#### 4.3.3.4 Procedure

Three test sheets: Rubber standard synthetic Type L, approximately 2.5 cm (1") by 5.1 cm (2") by 0.19 cm (0.76"). Rubber stock shall not be older than six months.

Preparation: Determine the water displacement weight of each test sheet to the nearest 1 mg by weighing each sheet in air, then in distilled water at 24°C ± 3°C (75°F ± 5.4°F). Record the weights and compute the difference between the weight in air and the weight in water for each sheet. Calculation of the percent change in volume as follows:

$$V = \frac{(A - B) \times 100\%}{B}$$

Where: V = Percent change in volume

A = Displacement weight in mg at end of test

B = Displacement weight in mg before immersion in lubricant

Test procedure: Fill the test tube with approximately 20 ml of lubricant for each gram of total weight of the test sheets. Blot dry each test sheet with filter paper, and then immerse sheets completely into the lubricant in the test tube. Store the test tubes in an



oven at  $70^{\circ}\text{C} \pm 1^{\circ}\text{C}$  ( $158^{\circ}\text{F} \pm 2^{\circ}\text{F}$ ) for 168 hours  $\pm$  0.5 hours (approximately one week). Maintain a ratio of 20 ml of lubricant to each gram of test sheet. Makeup lubricant shall be the same temperature as that in the test tube. At the end of the storage period, cool the lubricant to  $24^{\circ}\text{C} \pm 3^{\circ}\text{C}$  ( $75^{\circ}\text{F} \pm 5.4^{\circ}\text{F}$ ). Remove the test sheets and wipe them to remove most of the lubricant. Dip the sheets in alcohol and wipe or blot them dry, using filter paper (or soft cloths) and note the time. Within five minutes of drying the test sheets, re-determine the water displacement of each sheet as specified above. Record the average percent volume change of the three rubber sheets.

#### **4.3.3.5 Acceptance Criteria**

Swelling of standard synthetic rubber Type L caused by the lubricant shall not exceed 15 percent in volume.

#### **4.3.4 Rust Inhibition**

##### **4.3.4.1 Scope**

This test determines the rust inhibiting characteristics of the semi-fluid lubricant in accordance with either a 168-hour cyclic humidity chamber test or a 400-hour saturated humidity chamber test.

##### **4.3.4.2 Equipment**

Humidity chamber: Any humidity chamber that is capable of meeting and maintaining the temperature and humidity requirements specified in the test procedure in Paragraph 4.3.3.4.

Material: 500 ml of the lubricant.

Abrasive paper or cloth: Having No. 600A silicon carbide grit or No. 500 aluminum oxide grit.

Centrifuge. Any centrifuge capable of holding the four 1020 carbon steel rods as specified in test procedure 4.3.4.4.

##### **4.3.4.3 Reagents**

Reagent-grade n-hexane.

Reagent-grade Petroleum ether: Having a boiling point of  $30^{\circ}\text{C}$  to  $65^{\circ}\text{C}$  ( $86^{\circ}\text{F}$  to  $149^{\circ}\text{F}$ ).

##### **4.3.4.4 Procedure**

Four 1020 carbon steels rods: Conforming to ASTM A575 or A663. Dimensions of the specimens are 102 mm (4") long by 9.5 mm (0.375") in diameter with a hole of suitable size drilled approximately 19 mm (0.75") from one end.

Preparation: Polish the cylindrical steel rod specimens with abrasive paper or cloth.

Wash the specimens in reagent-grade n-hexane, wipe them with absorbent cotton and then wash them in the reagent-grade petroleum ether. Immerse the specimens completely

in the lubricant. Insert each specimen into a rubber stopper with a diameter slightly smaller than the centrifuge trunnion cup. Place the four specimens in the centrifuge and rotate them at 1000 rpm for 10 minutes. Remove each specimen from the centrifuge by inserting a glass rod in the hole near the end of the steel rod.

Caution: n-hexane is both toxic and flammable. Use only in a well ventilated area. Do not breathe its fumes nor allow it to come in contact with the skin. Keep all flames away from n-hexane.

Test procedure: Expose the test specimens in either a cyclic humidity chamber for 168 hours or a saturated humidity chamber for 400 hours.

For 168-hour test: Suspend the test specimens in a cyclic humidity chamber and expose them to the following conditions: four hours at a relative humidity (RH) of  $80\% \pm 5\%$  and a temperature of  $43^{\circ}\text{C} \pm 3^{\circ}$  ( $110^{\circ}\text{F} \pm 5^{\circ}\text{F}$ ) followed by four hours at RH of  $95\% \pm 5\%$  and a temperature of  $55^{\circ}\text{C} \pm 3^{\circ}$  ( $131^{\circ}\text{F} \pm 5^{\circ}\text{F}$ ).

For 400-hour test: Suspend and expose the specimens for 400 hours in a saturated humidity chamber in accordance with the specifications and instructions in ASTM D1748 (including Appendix I), except that the specimens shall be cylindrical rods instead of panels.

Report: Examine the specimens for evidence of rust at the end of each 24-hour interval. Express the test results as the average of the times-to-failure of the four specimens. Record the test results.

#### **4.3.4.5 Acceptance Criteria**

Not more than one of the four test specimens shall have failed by either the appearance on any given specimen of one rust spot larger than 1 mm in length, width or diameter, or the appearance on any given specimen of three or more rust spots, all smaller than 1 mm in length, width or diameter, but that increase in size over a 24-hour period.

### **4.3.5 Rust Prevention**

#### **4.3.5.1 Scope**

This test determines the effectiveness of corrosion-inhibiting lubricants on carbon steel. It consists of applying the test lubricant to plain carbon steel panels and dripping a one percent solution of pure sodium chloride onto the lubricant film.

#### **4.3.5.2 Equipment**

Test sample size: Approximately 500 ml of the lubricant.

Glass rod: 6 mm to 8 mm (0.24" to 0.31") in diameter.

Burette tip.

Glass tube: 2 mm to 3 mm (0.08" to 0.12")

320-grit abrasive cloth or paper.

Magnifier: 10X magnification

#### 4.3.5.3 Reagents

Sodium chloride solution: One percent.

Acetone.

#### 4.3.5.4 Procedure

Four steel panels: Fabricated from plain carbon steel 51 mm x 102 mm x 3.2 mm (2" x 4" x 0.125").

Preparation of steel panels: Remove contamination, preservatives and other foreign material. Wash with hot water and detergent. Rinse thoroughly with hot water followed by acetone. Abrade with clean 320-grit abrasive cloth or paper. Rinse with hot tap water followed by distilled water. Allow to drain and dry.

Test procedure: Apply the lubricant immediately (with a glass rod 6 mm to 8 mm (0.24" to 0.31") in diameter) by rubbing across the panel surface several times and remove the excess by passing the rod down the length of the panel. The rod shall be spaced 0.13 mm (0.005") above the surface at each side to leave an even film. The panels shall be placed with one end elevated so that it is inclined 15 degrees from the horizontal. A burette tip or other small (2 mm to 3 mm (0.08" to 0.12")) glass tube shall be mounted vertically above the panel with the end of the tube 50 mm (2") above a point on the panel on its centerline and 25 mm to 30 mm (1" to 1.2") from the upper end. A one percent solution of pure sodium chloride in distilled water shall be allowed to drip from the tube onto the lubricant film at  $2 \pm 0.2$  ml/minute. Constant flow rate can be achieved by siphoning the solution from a constant level reservoir. After 100 ml of the solution passes over each panel, it shall be examined with a 10X magnifier for evidence of rusting in the impingement or drainage areas. Four tests shall be made on the lubricant sample.

Reports: Record any visible rust on each of the panels.

#### 4.3.5.5 Acceptance Criteria

At least three of the four exposed panels shall be free of any visible rust.

### 4.3.6 Copper Corrosion – Rifles

#### 4.3.6.1 Scope

This test determines the corrosive properties of grease at elevated temperatures.

#### 4.3.6.2 Equipment

Oven: 100°C (212°F).

Microscope: 60X magnification.

Test tube: 2.5 cm x 10 cm (1" x 4").

Support beaker: For holding test tube approximately vertical.

Forceps: Stainless steel.

Test sample size: Approximately 20 ml of grease.

Polishing materials: 240-grit silicon carbide or aluminum grit paper.

Fine silicon carbide.

150 mesh silicon carbide grains.

#### 4.3.6.3 Reagents

Kerosene.

n-Hexane: ACS grade non-corrosive (as determined by ASTM D130).

Acetone: ACS grade.

#### 4.3.6.4 Test Procedure

Copper strip: (annealed, approximately 1.3 cm x 7.6 cm x 0.15 cm or 0.3 cm (0.5" x 3" x 0.06" or 0.12")).

Surface preparation: Remove all blemishes from all six sides of the copper strip with silicon carbide or aluminum grit paper. Moisten with kerosene or acetone and rub the strip against the paper with a rotary motion. Avoid contacting the strip with fingers by using ashless filter paper. Finish with 240-grit silicon carbide or aluminum grit paper. Then immerse the strip in acetone and withdraw immediately.

Final polishing: Remove a strip from the acetone. Hold with fingers protected with the ashless filter paper. First, polish the ends and sides with 150-mesh silicon carbide grains picked up from a clean glass plate with a pad of absorbent cotton moistened in acetone. Wipe vigorously with new absorbent cotton pad and handle by stainless steel forceps. Clamp in a vise and polish the main surfaces with silicon carbide grains. Rub the strip in the direction of the long axis, and carry the stroke beyond the strip's end before reversing the direction. Clean metal dust by rubbing with clean pads of absorbent cotton until a fresh pad remains unsoiled. Immerse the strip in the prepared sample when the strip is clean.

Test procedure: Fill test tube with sample to 5 cm (2") depth. Tap the tube on the bench or table to remove air pockets from the grease. Using stainless steel forceps, insert copper strip until it touches the bottom of the tube. Place the test tube in oven for 24 hours. Remove the test tube and cool to room temperature. Remove copper strip by using forceps, and wash in non-corrosive n-hexane.

Caution: n-hexane is both toxic and flammable. Use only in a well ventilated area. Do not breathe its fumes nor allow it to come in contact with the skin. Keep all flames away from n-hexane.

Report: Report whether the color of the strip section immersed in the sample is darker than the exposed section; whether there is any sign of development of green color in the grease; or whether there is any sign of corrosion in the strip by using a microscope of 60X magnification.

#### 4.3.6.5 Acceptance Criteria

Copper strip shall show no evidence of green color, pitting or etching, nor shall any brown or black stain remain after washing the strip with n-hexane.

#### 4.3.7. Copper Corrosion – Automatic Weapons

##### 4.3.7.1 Scope

This test determines the presence of corrosive compounds in grease or other semi-fluid lubricants at room temperature, 25°C (77°F). It consists of covering half of two copper strips with the lubricant under test and examining for corrosion after 24 hours.

##### 4.3.7.2 Equipment

Watch glass: For covering sheets.

Abrasive paper (silicon carbide or alumina), of various grades or grit, including silicon carbide paper, 240-grit.

Silicon carbide: 150-mesh grit.

Cotton: Absorbent.

Test lubricant: Approximately 5 grams.

##### 4.3.7.3 Reagents

n-Hexane: ACS grade.

Acetone: O-A-51, technical grade.

##### 4.3.7.4 Test Procedure

Copper strips (2): QQ-C-576, 2.5 cm x 2.5 cm x 0.3 cm (1" x 1" x 0.125").

Copper strip preparation: Using progressively finer grades of abrasive paper, remove all surface blemishes from one side of the strip. Polish the strips with the 240-grit paper to remove any marks produced by the coarser abrasive. Do not touch the strips with fingers, which could cause corrosion. Handle the strips only with lint-free paper or tongs. Store strips in acetone until ready for final polishing. Remove strips from the acetone and final polish one side to a mirror finish, using the 150-mesh silicon carbide grit (held on an acetone-moistened cotton pad). Clean all metal dust from the strips by rubbing vigorously with clean pads of absorbent cotton until a fresh pad remains unsoiled. Wash strips in fresh warm n-hexane, then in fresh warm acetone and allow to air dry.

Procedure: Place approximately one-half of the lubricant on each strip, taking care to leave approximately half of the polished strip exposed. Cover the strips with inverted watch glasses and let stand at room temperature for 24 hours. Remove the watch glasses and visually examine the lubricant for a green color. Wash the copper strips in non-corrosive n-hexane and inspect them for brown or black stains.

Report: Report the lack or presence of any green color in the lubricant and brown or black stains on the copper strips.

##### 4.3.7.5 Acceptance Criteria

The copper strips shall show no brown or black stains. Slight darkening or slight green stain in the lubricant is acceptable.

#### 4.3.8. Rubber Compatibility

##### 4.3.8.1 Scope

This method describes the procedures to evaluate the ability of rubber to withstand the effects of semi-fluid lubricants at 70°C (158°F) and after 72 hours of immersion time.

##### 4.3.8.2 Equipment

Materials: Semi-fluid lubricant to be tested.

Aging chamber or blocks.

Glass test tube: 38 mm (1.5") outside diameter and an overall length of 300 mm (12 ").

Reflux condenser: For volatile semi-fluid lubricants.

Stoppers: Loose fitted for non-volatiles and tight fitted for volatiles.

Oven: Type IIB as specified ASTM E145 or type IIA for higher temperatures with interior size of 900 mm x 900 mm x 1200 mm (36" x 36" x 48").

Balance: Sensitivity 1 mg.

Recording thermometer.

Thermostatic temperature control: Preferred location is adjacent to the recording thermometer.

Note: Maintain accurate and uniform heating in all parts of the aging chamber.

Filter paper.

##### 4.3.8.3 Reagents

Acetone.

##### 4.3.8.4 Procedure

Test specimens: Rubber O-rings (3), 13 mm (0.5") external diameter and 1.7 mm (0.07 in) cross-sectional diameter and fabricated from rubber meeting the requirements of MIL-P-5516.

Preparation of specimens: Prepare in accordance with ASTM D3182 and ASTM D3183.

Test procedure: Weigh each test specimen and record the weight as W1 to the nearest 1 mg. Immerse the specimens in the test tube, as shown in Figure 1 of ASTM D471, containing 100 cm<sup>3</sup> (6.1 in<sup>3</sup>) of the lubricant to be tested. Separate each test specimen from any adjacent test specimen and the test tube walls by 6 mm (0.25"). For non-volatile lubricants, condition the test tube assembly at 70°C ± 2°C (158°F ± 3.6°F) for 72 hours. For volatile lubricants, fit the test tube with a reflux condenser and condition the test tube assembly at 70°C ± 2°C (158°F ± 3.6°F) for 72 hours. Check the actual temperature of the test tube's lubricant to ensure the desired temperature for the specified period. After the specified length of time in the immersion test, remove the test specimens. Cool the test specimens to room temperature by transferring them to a cool clean portion of the lubricant to be tested for 30 to 60 minutes. Dip the specimens quickly in acetone (at room temperature), blot lightly with filter paper and immediately place them in a tarred, stoppered weighing bottle. Determine the after-test weight, W2,

of each specimen to the nearest 1 mg. Immerse the specimen (after weighing) in the same test lubricant if data is desired on the progressive changes that occur with increasing times of immersion.

Notes: When dealing with volatile lubricants at room temperature, no more than 30 seconds should elapse between removal from the test lubricant and stoppering the weighing bottle. For relatively viscous lubricants, it may be difficult to remove the lubricant by dipping in acetone and wiping the specimens. To allow most of the lubricant to drip off the surface of the test specimens, suspend the specimens for about 30 minutes at room temperature. Then proceed with the acetone dipping and blotting.

Test Results: Calculate the percent change in mass (weight) as follows:

$$\Delta W = \frac{(W2 - W1) \times 100\%}{W1}$$

Where:  $\Delta W$  = change in weight in percent  
 $W1$  = initial specimen weight  
 $W2$  = after immersion specimen weight

Report: Record the test results.

#### 4.3.8.5 Acceptance Criteria

The maximum difference or percent change in weight of the MIL-P-5516 O-rings, in 72 hours at 70°C (158°F), shall not exceed three percent between the tested lubricant and the lubricant formulation results shown in the following table:

Component		Weight Percent
a. Isodecyl pelargonate, lubricant grade	1)	28.6 ± 0.5
b. Bis (2-ethylhexyl) sebacate, lubricant grade	2)	9.0 ± 0.5
c. Dimethyl silicon fluid, 7.5 cs	3)	39.2 ± 1.0
d. Slightly phenylate silicon fluid, 50 cs	4)	14.2 ± 0.5
e. Phenylstearic acid		1.0 ± 0.05
f. Basic barium dinonylnaphthalane sulfonate (50% volatile solvent)	5)	2.0 ± 0.1
g. Oxidation inhibitor	6)	0.4 ± 0.05
h. Lithium stearate	7)	5.6 ± 1.5

- 1) Equivalent to that supplied by Emery Industry as Emolein 2911.
- 2) Equivalent to that supplied by Hatco Chemical Corp. as Hatcol 3110.
- 3) Equivalent to that supplied by Dow Corning Corporation as DC-200 (7.5 cs may be obtained by blending equal portions of 5 cs and 10 cs fluids.
- 4) Equivalent to that supplied by Dow Corning Corporation as DC-510, 50 cs.
- 5) Equivalent to that supplied by R. T. Vanderbilt Co., Inc.
- 6) 4-tert-butyl-2-phenylphenol, phenyl-a-naphthylamine and 2, 6-di-tert-butyl-4-methylphenol are satisfactory.

The use of a very pure grade of lithium stearate is essential to attain the required performance by the finished lubricant. The following procedure may be used to determine the suitability of the lithium stearate for this use. An infusion of lithium stearate shall be prepared by briefly boiling 0.75 g of lithium stearate in 25 ml of distilled water and filtering through fine filter paper. Care shall be taken to exclude detergents and other surface-active materials. The infusion shall not be diluted or allowed to evaporate appreciably. The surface tension of the infusion shall then be measured by the ring method. ASTM D971 shall be followed in regard to the apparatus, the preparation and calibration of the apparatus and the determination of the surface tension of water. That portion of the procedure dealing with oil-water interfacial tension is not used. The surface tension of the infusion should be no less than 380 micro-newtons/meter.

### 4.3.9 Salt Spray (Fog) Resistance

#### 4.3.9.1 Scope

This method determines the corrosion protection provided by the lubricant by exposing the test panels to 5% sodium chloride solution.

#### 4.3.9.2 Equipment

Exposure chamber: This equipment should consist of a fog chamber with racks and suitable heater/controls, a salt solution reservoir, a supply of conditioned (oil- and contaminant-free) compressed air, atomizing nozzles, and a humidifier.

Test sample size: 500 ml of the lubricant to be tested.

Cleaning tissues.

#### 4.3.9.3 Reagents

Sodium chloride: O-C-265; 5% solution.

Hydrochloric acid: O-C-265.

Sodium hydroxide: O-C-265.

Bromthymol blue solution: MIL-B-11845.

Indicator: pH range from 6 to 7.

Distilled water: (ACS grade).

Acetone.

#### 4.3.9.4 Procedure

Three (3) 1010 steel panels: (low carbon, open-hearth, cold-finished) with dimensions of 7.5 cm by 5.0 cm by 0.32 cm (3" by 2" by 1/8"). Prepare the test panels in accordance with FED-STD-791C Method 5329.2, paragraphs 6.1 through 6.3b.

Preparation: Clean uncoated metal panels with acetone at 38°C to 54°C (100°F to 129°F) and cleaning tissues. Avoid handling panels on significant surfaces. Dry all panels thoroughly. Apply the lubricant to be tested to the test panels. Place the test panels approximately 15 degrees from the vertical position and parallel to the principal direction of horizontal flow of fog through the chamber.

Test procedure: The SS (fog) chamber should be operated continuously for a minimum of 100 hours. The chamber shall be operated at 35°C + 1°C, -1.5°C (95°F + 2°F, -3°F) in the exposure zone. In addition, all exposed parts shall be maintained in such a manner that a suitable receptacle placed at any point in the exposure zone will collect from 0.5 to 3 ml of solution per hour for each 80 cm<sup>2</sup> (12.4 in<sup>2</sup>) of horizontal collecting area (10 cm (4") diameter) for an average run of 16 hours. At the conclusion of the 100-hour exposure period, remove the coupons and clean them by gently flushing with running tap water not warmer than 38°C (100°F) and brush lightly.



#### 4.3.9.5 Acceptance Criteria

Not more than three small corrosion spots per test panel, none exceeding 1 mm in length, width or diameter on any of the test panels. One large corrosion spot exceeding one mm in size on any of the three panels will be cause for rejection.

#### 4.3.10 Rust Inhibition

##### 4.3.10.1 Scope

This test determines the rust inhibition requirements for a lubricant used (for short-term preservation not to exceed 30 days) of small and large caliber weapons operating in the temperature range of  $-54^{\circ}\text{C}$  to  $66^{\circ}\text{C}$  ( $-65^{\circ}\text{F}$  to  $150^{\circ}\text{F}$ ).

##### 4.3.10.2 Equipment

Oven: Convection, up to  $100^{\circ}\text{C}$  ( $212^{\circ}\text{F}$ ).

Balance: Sensitivity 1 mg.

Oxide paper: 240-grit aluminum.

Filter paper.

Lubricant to be tested.

##### 4.3.10.3 Reagents

Cleaning solvent: In accordance with MIL-P-D-680.

Naphtha: TT-N-95.

Methanol: Anhydrous, O-M-232, Grade A.

##### 4.3.10.4 Procedure

Test coupons (7): 25 mm x 51 mm x 6.4 mm (1" x 2" x 1/4"), one each made of the following metals: zinc (MIL-A-18001), aluminum (QQ-A-250/4), brass (QQ-B-626, composition 22), steel (ASTM A109), copper (QQ-C-576), magnesium (QQ-M-44, A231B, condition H24 or H26), and cadmium (QQ-A-671).

Preparation: Polish all surfaces of the test coupons using progressively finer abrasive paper, finishing with a 240-grit polishing medium; hold the coupon with tongs or filter paper and avoid touching with fingers. Use a cloth with dry-cleaning solvent after final polish; do not use wet or dry papers. Clean the test coupon with hot naphtha with a final rinse in warm anhydrous methanol (Grade A of O-M-232).

Caution: Naphtha is flammable. Use only in a well-ventilated area. Methanol is both toxic and flammable. Do not allow it to come into contact with the skin or breathe its fumes. Keep all flames away from naphtha and methanol.

Test procedure: Weigh the coupons and place them in a mason jar (88.9 mm (3.5") diameter and 95.25 mm (3.75") deep) with a screw cap. Stand the test coupons on one 6.4 mm (0.25") edge in a circular pattern so that the 25 mm (1") length is parallel to the jar circumference as shown in Figure 1 of MIL-L-63460D. Cover the test coupons with

adequate lubricant containing no solid particles, with the coupon tops at least 6 mm (0.24 in) below the surface of the liquid product. After closing the jar by screwing on the lid, place the jar in a convection oven setting at temperature of  $54.4^{\circ}\text{C} \pm 1^{\circ}\text{C}$  ( $130^{\circ}\text{F} \pm 2^{\circ}\text{F}$ ) for seven days  $\pm 15$  minutes. At the end of the test, remove the lubricant and any loose corrosion products from specimens by swabbing with gauze pads moistened with naphtha; then with gauze pads moistened with methanol and followed by clean solvent rinses. Then weigh the specimens and calculate the weight loss or gain in milligrams per square centimeter.

#### 4.3.10.5 Acceptance Criteria

Lubricant shall not produce visual evidence of pitting, etching, or dark discoloration. Weight changes shall not exceed those for the following metals:

<u>Metal</u>	<u>mg/cm<sup>2</sup></u>
Zinc	1.5
Aluminum	0.2
Brass	1.0
Steel	0.2
Copper	1.5
Magnesium	0.5
Cadmium	1.5

#### 4.3.11 Corrosion Protection from Propellant Reaction

##### 4.3.11.1 Scope

This test determines the lubricant's ability to retain its corrosion protection characteristics after being exposed to the by-products of propellant ignition. (Test to be conducted at Picatinny Arsenal.)

##### 4.3.11.2 Equipment

Environmental chamber: Any environmental chamber that is capable of meeting and maintaining the temperature and humidity requirements specified in the test procedure in Paragraph 5.1.2.3.4. The chamber must also be able to accommodate the test fixture described in the same paragraph. A Tenney Model # TH-10, or equivalent, is an example.

Three test panels: Conforming to ASTM Method D1748, Appendix Section A1-10.

Three porcelain dishes: 120 mm (4.7") in diameter with 195 ml capacity.

Materials: Nine grams of WC-844 propellant powder.

#### 4.3.11.3 Reagents

5% Sodium chloride.

Naphtha: TT-N-95.

Methanol: O-M-232.

#### 4.3.11.4 Procedure

Preparation: Clean the three test panels thoroughly, first in the heated petroleum naphtha and then in the heated methanol. (Heat the solvent sufficiently so it will evaporate from the panels immediately after withdrawal from the solvent baths.) Place the panels in a dessicator.

Test Procedure: After they have cooled, agitate each panel in a beaker of the lubricant for one minute. Suspend the panels from a rack and drain for two hours. After two hours, agitate each panel for one minute in a beaker containing a solution of 5% sodium chloride by weight dissolved in distilled water. Drain the panels again for 15 minutes. While the panels are draining, weigh out  $3 \pm 0.1$  gram of WC-844 propellant powder into each of the three porcelain-evaporating dishes. Concentrate the powder into a strip measuring approximately 25 mm (1") across the bottom of the dish. After completion of the 15-minute drain period, place a panel across the top of a test fixture constructed of four humidity test panels fastened together to form a box-like structure opened at the top and bottom with interior dimensions of 10.2 cm (4") in length, 4.45 cm (1.75") in width and 5.1 cm (2") in height. Place the fixture with the test panel on it across the rim of the evaporating dish centered over and parallel to the strip of propellant. Ignite the propellant so that one face is exposed to the burning propellant. Repeat this operation on the other two panels and then suspend the three panels in an environmental chamber maintained at  $49^{\circ}\text{C} \pm 2^{\circ}\text{C}$  ( $120^{\circ}\text{F} \pm 5^{\circ}\text{F}$ ) and  $95\% \pm 5\%$  relative (condensing) humidity. The panels should be suspended side by side forming one row so that the test surfaces are facing the front of the chamber and the distance from the rear of the chamber to each panel is 350 mm (13.75") and the distance from the chamber floor to bottom of the panels is 430 mm (17"). After a period of 96 hours, remove the test panels and wipe them lightly with a soft cloth or tissue paper soaked with petroleum ether.

Report: Observe for any rusting on the test face of the panels.

#### 4.3.11.5 Acceptance Criteria

No rust spots 2 mm or larger involving visible pitting or etching of the metal shall be evident on any of the three test panels. Rust spots occurring within 3 mm (0.125") from the edges of the test panel shall not be cause for failure.

## **5.0 FAILURE ANALYSIS**

Any candidate that is to be considered technically acceptable for the affected system must pass all performance tests and special tests, if applicable. Any candidate that fails one or more performance tests should be documented with regards to failure mechanism/phenomenon before being discarded. If the candidate fails any specific performance or special test, at the candidate vendor's request and expense, a Failure Analysis procedure can be undertaken. Such failure analysis can be a useful vendor option if it can be used to demonstrate that some promising procedure or material characteristics that caused the observed performance failure can be corrected and then lead to acceptable performance metrics. However, after failing any of the Performance Tests for the third time, further iterations of that Performance Test are not permitted. Instead, the JTP process should be terminated and results noted in that JTR. Likewise, for failure of any specific Special Tests, if failure occurs for the third time, do not repeat the Special Testing. Instead, the JTP process should be terminated and results noted in that JTR. The JTR should then be forwarded to the invoking authority for his response.

Marginal test results must be either overcome by retesting or documented before discarding the candidate. The failure mechanisms of candidates that fail one or more of any performance tests should be documented. In the event of any testing-related dispute between vendor and tester, such as causes of premature failure, a third-party testing laboratory will be mutually agreed upon as a credible testing source by the invoking authority. This Product Failure Laboratory (PFL) must have no pre-existing connections to either the vendor of the candidate or the original laboratory that conducted the testing. The process flow is illustrated in Figure 1, which appears in Section 2.0, JTP Document Guide.

Failure in any test does not necessarily disqualify a candidate lubricant for use in all possible applications. The initial JTR and all related JTRs (specifically failure analyses) must be submitted to the relevant invoking authority or designee.

## 6.0 REFERENCE DOCUMENTS

The documents listed in Table 5 were referenced in the development of this JTP.

**Table 5. Reference Documents**

Reference Document	Title	Applicable Section(s) of Reference Document	JTP Test	JTP Section Cross- Reference	Document Source
MIL-L-14107C	Lubricating Oil, Weapons, Low Temperature	Para. 3.4 & Table II	Corrosion Protection (Humidity Cabinet)	4.3.1	DoD
		Para. 3.7 & 4.6.4	Oxidation Stability	4.3.2	
		Para. 3.6 & Table II	Rubber Compatibility	4.3.3	
MIL-PRF-63460D Amendment 6	Lubricant, Cleaner, and Preservative for Weapons and Weapons Systems	Para. 3.7.1 & 4.11	Corrosion Protection (Humidity Cabinet)	4.3.1	DoD
		Para. 3.7.2 & 4.12	Salt Spray (Fog)	4.3.9	
		Para. 3.6 & 4.10 Table 1 & Figure 1	Rust Inhibition	4.3.4	
		Para.3.7.3 & 4.13	Corrosion Protection from Propellant Reaction	4. 3.11	
MIL-PRF-85336B	Lubricant, All-Weather (Automatic Weapons)	Para. 3.4 & 4.5.5 Table I	Rust Prevention	4.3.5	DoD
		Para. 3.4 Tables I & III	Copper Corrosion – Automatic Weapons	4.3.7	
MIL-L-19701B	Lubricant, All-Weather, Semi-Fluid, for Aircraft Ordnance (Machine Guns)	Para. 3.3 Tables I, II, & IV	Rubber Compatibility	4.3.8	DoD

**Table 5. Reference Documents (Continued)**

Reference Document	Title	Applicable Section(s) of Reference Document	JTP Test	JTP Section Cross- Reference	Document Source
MIL-G-46003A (WC)	Grease, Rifle	Para. 3.8 & 4.6	Copper Corrosion – Rifles	4.3.7	DoD
MIL-L-46000C	Lubricant, Semi-Fluid (Automatic Weapons)	Para. 3.11 & 4.10	Rust Inhibition	4.3.4	DoD
FED-STD-791C Method 5329.2	Corrosion Protection (Humidity Cabinet)	All	Corrosion Protection (Humidity Cabinet)	4.3.1	DoD
FED-STD-791C Method 3603.5	Swelling of Synthetic Rubber	All	Rubber Compatibility	4. 3.8	DoD
FED-STD-791C Method 4001.3	Corrosion Protection by Coating: SS (Fog) Test	All	SS (Fog) Resistance	4.3.9	DoD
FED-STD-791C Method 5309.5	Corrosiveness of Grease (Copper Strip, 100°F)	All	Copper Corrosion – Rifles	4.3.6	DoD
FED-STD-791C Method 5304.5	Corrosiveness of Grease or Semi- Solid Products at 25°C	All	Copper Corrosion – Automatic Weapons	4.3.7	DoD
ASTM D974	Acid and Base Number by Color- Indicator Titration	All	Oxidation Stability	4.3.2	ASTM
ASTM D471	Rubber Property – Effect of Liquids	Sections 7, 8, 10, and 17	Rubber Compatibility	4.3.8	ASTM
ASTM D1748	Rust Protection by Metal Preservatives in the Humidity Chamber	All	Corrosion Protection (Humidity Cabinet)	4.3.1	ASTM
		Annex A1-10	Corrosion Protection from Propellant Reaction	4.3.11	
		All	Rust Inhibition	4.3.4	

## **APPENDIX**

### **List of Technical Stakeholders**

### List of Technical Stakeholders

FIRST NAME	LAST NAME	COMPANY/ ORGANIZATION	ADDRESS	CITY/STATE/ ZIP	PHONE	FAX	E-MAIL
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