# US Department of Agriculture Forest Service

# Specification 5100-304d

# Long-Term Retardant, Wildland Firefighting

January 7, 2020 Amended May 6, 2021

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# U.S. DEPARTMENT OF AGRICULTURE FOREST SERVICE

### **SPECIFICATION FOR**

# LONG TERM RETARDANT, WILDLAND FIREFIGHTING

## 1. <u>SCOPE AND CLASSIFICATION</u>.

**1.1.** <u>Scope</u>. The long-term fire retardants described in this specification are for use in wildland fire management. They may be applied from aerial or ground application equipment.

After mixing with water in the prescribed ratio, the mixed retardant is applied to slow the spread and reduce the intensity of the fire. Long-term retardants continue to be effective after the contained water has evaporated. Long-term retardant concentrates may be wet or dry. Products must be one component, i.e., mixed retardants shall be prepared by blending a single concentrate with water.

The mix ratio shall be specified by the manufacturer and confirmed by combustionretarding effectiveness testing. Refer to 3.6 for additional information.

- **1.1.1.** <u>Product Types Covered By This Specification</u>. Long-term retardants for fighting fires in Class A fuels are covered by this specification.
- **1.2.** <u>Classification</u>. The submitter shall specify the classifications of the wildland fire chemical product, according to the following sections, for which qualification is sought.

The evaluation shall be conducted following the test methods and requirements contained in this specification, based on the classifications requested by the submitter.

- **1.2.1.** Form of Concentrate. Each concentrate shall be classified as wet or dry.
- **1.2.1.1.** <u>Wet Concentrate</u>: A single, liquid component which is mixed with water to prepare the mixed product.
- **1.2.1.2.** <u>Dry Concentrate</u>: A single, dry component which is mixed with water to prepare the mixed product.
- **1.2.2.** <u>Color</u>. Long-term retardant may be uncolored or colored. Each mixed product shall be classified as uncolored, non-fugitive (typically iron oxide) colored, or fugitive colored, as described below. All products qualified and approved for aerial application of any type shall be either iron oxide colored or fugitive colored.
- **1.2.2.1.** <u>Uncolored products</u> shall have no ingredients that impart color. The product in the container may have some earth-tone color; however it is not visible when applied to natural fuels.

- 1.2.2.2. <u>Colored products may be:</u>
  - **a.** <u>Fugitive colored</u>; A mixed product that contains one or more ingredients that impart a high degree of visibility from the air when first applied to wildland fuels but will lose visibility gradually over several months. OR
  - **b.** <u>Non-Fugitive, Red Iron Oxide colored</u>; A mixed product that contains at least 12 grams of red iron oxide per mixed gallon to impart red color to provide a high degree of visibility from the air at the time of application to wildland fuels.
  - **c.** <u>Non-Fugitive colored</u>; A mixed product that imparts red color to provide a high degree of visibility and opacity from the air at the time of application to wildland fuels.
- **1.2.3.** <u>Storability</u>.
- **1.2.3.1.** All concentrates shall be evaluated as stored products unless qualified as a pretreatment, non-storable product as specified in 1.2.3.2.
- **1.2.3.2.** <u>Mixed products</u>. Each mixed product shall be classified to indicate the type and length of storage the product is designed for and whether or not recirculation is required or recommended.

<u>Storable</u>. Concentrate is stable for at least 52 weeks. For dried powder concentrates, the mixed product is stable for at least 52 weeks. Products may be recirculated in storage and recirculation may be required to obtain a homogeneous and usable product.

<u>Not Storable</u>. Concentrate is stable for at least 52 weeks. Mixed product is stable for at least 14 days. Products are mixed or blended during transfer to aircraft or other application devices. Minimal additional mixing or recirculation is necessary.

These products are not routinely stored in the mixed form except in application equipment where recirculation is not available.

<u>Pretreatment, Non-storable.</u> Products are not evaluated for storability beyond 14 days. Concentrate is not storable, mixed product is not storable. For immediate use only. Product shall be used for fire prevention pretreatment only. Products are not qualified fo use or storage at permanent airtanker bases. The product not intended for use in direct support of wildfires. For use in helicopter buckets or ground (HB/G) applications only.

**1.2.4.** <u>Application Methods</u>. Each mixed product shall be classified based on the listed application methods.

HF	Helicopters having a fixed tank, either internal or external in direct contact with the helicopter.
FW/Multi-Engine	Fixed-wing (all delivery systems) – land-based, multi engine aircraft having a tank and delivery system for aerial application of wildland fire chemicals.
FW/Single-Engine	Fixed-wing (all delivery systems) - land-based, single-engine (SEAT) aircraft having a tank and delivery system for aerial application of wildland fire chemicals.
HB/G	Helicopters having a bucket suspended below the helicopter such that no chemical is likely to contact the helicopter during normal fire operations; all ground-based application equipment, such as wildland engines, portable pumps, and other such devices.

**1.2.5.** <u>Viscosity Range</u>. Each mixed product shall be classified based on the viscosity of the product. Mixed products must achieve the desired viscosity by hydration of an appropriate amount of guar gum, guar gum derivatives, xanthan, or other thickeners that impart elasticity as well as viscosity.

High Viscosity	Mixed product with a viscosity between 801 and 1500 centipoise (cP).
Medium	Mixed product with a viscosity between 401 and 800 cP.
Low Viscosity	Mixed product with a viscosity between 150 and 400 cP.

**1.2.6.**Base Type. The evaluation shall be conducted following the test methods and<br/>requirements contained in this specification, based on the classifications described.<br/>Qualification for use from specific base types shall be determined by product<br/>performance and storage needs.

Temporary/Mobile Bases	Not storable mixed products are suitable; storable products may be suitable.
	Small volumes of mixed product storage capability and limited auxiliary equipment, including recirculation, are available.
Permanent Bases	Storable mixed products or mixed products made from wet concentrates are suitable.
	Recirculation is possible, large/long-term storage capability, and auxiliary equipment are readily available.

# 2. <u>APPLICABLE DOCUMENTS</u>.

**2.1.** <u>Order of Precedence</u>. In the event of conflict between the text of this document and the references cited herein, the text of this document takes precedence.

Nothing in this document, however, shall supersede applicable laws and regulations unless a specific exemption has been obtained.

- **2.2.** <u>United States Government Documents</u>. The specifications, standards, and handbooks referenced form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents in effect on the date of the invitation for bids or request for proposals shall apply.
- **2.2.1.** <u>Code of Federal Regulations (CFR)</u>. The electronic text of the Codes of Federal Regulation are available at <u>https://gov.ecfr.io/cgi-bin/ECFR</u>
- **2.2.2.** U.S. Department of Agriculture, Forest Service, National Technology and Development Program (NTDP-Missoula).

Manufacturer Submission Procedures for Qualification Testing of Wildland Fire Chemical Products. Available at <u>https://www.fs.fed.us/rm/fire/wfcs/index.htm</u>

Standard Test Methods for the Evaluation of Wildland Fire Chemical Products, version in effect on the date of submission for evaluation. Available at <u>https://www.fs.fed.us/rm/fire/wfcs/index.htm</u>

USDA Forest Service Manual (FSM) 5100, Chapter 5160, Section 5162 – Fire Management Chemicals. Available at <u>https://www.fs.fed.us/im/directives</u>

**2.2.2.1** Paper copies of the submission documents can be obtained from the Program Leader or Project Leader, Wildland Fire Chemical Systems (WFCS), 5785 Highway 10 West, Missoula, MT, 59808, if web access is not available.

2.2.3.	U.S. Department of Agriculture and U.S. Department of Interior.
	Interagency Standards for Fire and Fire Aviation Operations. Department of Agriculture, Forest Service, and Department of the Interior Agencies: Bureau of Land Management, National Park Service, and U.S. Fish and Wildlife Service. Available at <a href="https://www.nifc.gov/policies/pol_ref_redbook.html">https://www.nifc.gov/policies/pol_ref_redbook.html</a>
2.2.4.	U.S. Environmental Protection Agency (EPA), Office of Chemical Safety and Pollution Prevention (OCSPP).
	EPA documents can be obtained from the web site at <u>https://www.epa.gov/aboutepa/about-office-chemical-safety-and-pollution-prevention-ocspp</u>
	By mail from U.S. Environmental Protection Agency, National Service Center for Environmental Publications (NSCEP), P.O. Box 42419, Cincinnati, OH 45242.
2.2.5.	United States Department of Health and Human Services.
	National Toxicology Program: Report on Carcinogens is available at <a href="https://ntp.niehs.nih.gov/">https://ntp.niehs.nih.gov/</a>
2.2.6.	International Agency for Research on Cancer (IARC).
	IARC Monographs of Carcinogens are available at https://monographs.iarc.fr/
2.2.7.	Federal Standards.
	Federal Standards can be obtained from <a href="https://www.gsa.gov/acquisition/purchasing-programs/requisition-programs/gsa-global-supply/supply-standards/index-of-federal-specifications-standards-and-commercial-item-descriptions">https://www.gsa.gov/acquisition/purchasing-programs/gsa-global-supply/supply-standards/index-of-federal-specifications-standards-and-commercial-item-descriptions</a>
2.2.8.	Military Specifications.
2.2.0.	Military Specification can be obtained from <a href="https://www.dsp.dla.mil/Specs-Standards/">https://www.dsp.dla.mil/Specs-Standards/</a>
2.2.9.	Freedom of Information Act (FOIA).
<i>L</i> . <i>L</i> .J.	The Forest Service FOIA information can be found at https://www.fs.fed.us/im/foia/
2.3.	Other Publications. The following publications of the issue in effect on the date of invitation for bids form a part of this specification.
2.3.1.	American Society for Testing and Material (ASTM).
2.01.11	Copies of ASTM publications can be obtained on the web at https://astm.org or
	By mail from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.
2.3.2.	National Association of Corrosion Engineers International (NACE).
	Copies of NACE publications can be obtained at https://www.nace.org or
	By mail from NACE International, 15835 Park Ten Place, Houston, TX, 77084.
2.3.3.	Society of Automotive Engineers, Inc. (SAE).
210101	Copies of SAE publication can be obtained on the web at https://sae.org or
	By mail from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001.
3.	REQUIREMENTS.
3.1.	Wildland Fire Chemical Product Qualification Testing. Qualification testing for wildland

fire chemical products shall be performed prior to use (Forest Service Manual (FSM) 5100, Chapter 5160, Section 5162). Testing shall include a laboratory evaluation and may include a field evaluation during firefighting operations as outlined in section 3.12.

- **3.1.1.** <u>Interim Qualification</u>. Additional requirements are required when seeking interim qualification of new formulations. This testing is optional and the expense of the submitter.
- **3.1.2.** <u>Additional Testing at the Discretion of the Forest Service.</u> Additional tests not specified in this document may be required at the discretion of the Forest Service when information provided in the product information or otherwise known to the Forest Service suggests a need.

The submitter shall be informed, before any additional testing is performed, of the specific tests to be performed and the reason for the tests.

**3.2.** <u>Manufacturer Submission Process</u>. The submitter (manufacturer, distributor, or supplier) shall make a request for evaluation to the USDA Forest Service, Branch Chief for Fire Equipment and Chemicals.

### 3.3. Product Information

**3.3.1.** Formulation Disclosure Sheet (SF 5100-37). The submitter shall submit a Formulation Disclosure Sheet (Table 1 of Manufacturer Submission Procedures) including the required information on all ingredients contained in the formulation.

Full disclosure of the types and amounts of each chemical in the product, the Chemical Abstract Services (CAS) number, quality or grade, and manufacturer or source shall be included for each ingredient.

The manufacturing process, manufacturing site, and other related information about each ingredient should also be provided.

- **3.3.2.** <u>Mix Ratio</u>. The submitter shall specify the mix ratio for which the product is designed and qualification is being sought.
- **3.3.3.** <u>Health and Safety Information.</u> The submitter shall submit the following safety information to the Government for review, prior to shipping the product for testing.
  - SDS for each ingredient of the proposed product from the manufacturer/supplier shown on the formulation disclosure sheet.
  - SDS for the proposed product from the manufacturer/supplier shown on the formulation disclosure sheet.
  - Optional: Summary of any toxicity or related safety test results conducted by or for the manufacturer prior to submission to the Government.
- **3.3.4.** <u>Technical Data Sheets</u>. The submitter shall provide completed Technical Data Sheets (Tables 2 and 3 of Manufacturer Submission Procedures) providing all required information on the physical properties and characteristics of the long-term retardant.

A description of the field mixing and handling requirements shall be included.

- **3.3.5.** <u>Reaction Ingredient Residual.</u> When the submitted product is the result of a chemical reaction, information explaining the reaction process and the residual amounts of the reaction ingredients shall be provided.
- **3.3.6.** <u>Other Technical Information.</u> The submitter shall provide information regarding laboratory mixing, field mixing, handling, loading, and any special cleanup procedures that are required for laboratory or field personnel.
- **3.3.7.** <u>Patents</u>. Copies of patents covering any aspect of the formulation or its application in wildland fire operations should be included in the submission documentation.
- **3.4.** <u>Chemicals of Concern</u>.

- **3.4.1.** <u>Unacceptable Ingredients</u>. The following ingredients shall not be accepted:
  - Sodium ferrocyanide (Yellow Prussiate of Soda or YPS)
  - Dichromates
  - Thiourea
  - Borate or other boron-containing compounds
  - Polychlorinated biphenols (PCB)
  - Polybrominated diphenyl ethers (PBDE)
  - Nonylphenol ethoxylates (NPE)
  - Ammonium Sulfate
  - Per- and polyfluoroalkyl substances (PFAS), including but not limited to:
    - Perfluorooctanoic acid (PFOA)
    - Perfluorooctanesulfonate (PFOS) compounds
- **3.4.2.** <u>Environmental and Health Regulations</u>. A review of environmental regulations as they apply to the formulation and the ingredients of the formulation shall be completed by the Government as a part of the overall review. Specifically, the status of each chemical with regard to the regulatory lists shown below shall be determined.

Additional regulations or restrictions may be added in the future.

- a. National Toxicology Program's Annual Report on Carcinogens.
- **b.** International Agency for Research on Cancer (IARC) Monographs for Potential Carcinogens.
- **c.** 40 Code of Federal Regulations (CFR) 355 Appendix A. Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), List of Extremely Hazardous Substances and their Threshold Planning Quantities.
- **d.** 40 CFR 302.4. CERCLA, List of Hazardous Substances and Reportable Quantities.
- e. 40 CFR 261.33. Resources Conservation and Recovery Act (RCRA), Acutely Hazardous and Toxic Wastes.
- f. 40 CFR 372. Superfund Amendment and Reauthorization Act (SARA) Title III, sec 313, Emergency Planning and Community Right to Know (EPCRA), Toxic Release Inventory (TRI).

### 3.4.3. <u>Chemical Profile and Risk Assessment</u>.

All chemical profiles and risk assessments shall be performed by the Government or an approved third party selected by the Government, using accepted methodology. Based on the information provided in accordance with 3.3, chemical profiles and/or a risk assessment as described in 3.4.3.1 and 3.4.3.2 may be required before lab-work begins.

- **3.4.3.1.** <u>Chemical Profile.</u> When no existing chemical profile exists or the existing profile is out of date, a new profile shall be developed by the Government or qualified third party selected by the Government, at Government expense.
- **3.4.3.2.** <u>Risk Assessment</u>. A risk assessment shall be performed by the Government OR a qualified party selected by the Government, at submitter expense, prior to a product placement on the Qualified Products List (QPL).

If a risk assessment is required before the laboratory evaluation is performed, based on information developed in 3.4, the Government shall notify the submitter of the flagged chemicals found during the review of the regulatory lists as required by 3.4.2.

3.4.3.3. Environmental Consultation with Regulatory Agencies. A new product may be included in an environmental consultation with the Federal regulatory agencies [U.S. Fish and Wildlife Service (FWS) and National Oceanic and Atmospheric Administration - National Marine Fisheries Service (NMFS)]. The extent of the consultation will be based on the similarities and differences to other products from the same submitter.

> The composition of a product, the results of laboratory testing required by this specification, applicable chemical profiles, and product risk assessments will be shared with these regulatory agencies to inform them of the characteristics of the product and assist them in making their determination.

> The consultation will result in a formal statement of findings from each regulatory agency.

#### 3.5. Performance Requirements.

Determination of Laboratory Mixing Procedures. If necessary, a suitable set of 3.5.1. conditions and methods for preparing laboratory samples of the mixed product shall be determined.

> This procedure shall be used to prepare all samples for the laboratory evaluation. If standard mixing operations are not acceptable/sufficient, the submitter shall provide acceptable mixing equipment, capable of accurately measuring to the specified dilution, and associated mixing procedures. Costs of additional equipment and/or personnel time shall be the responsibility of the submitter.

- Health and Safety. 3.5.2.
- Mammalian Toxicity and Irritation Tests. As required by 3.5.2.1.1. and 3.5.2.1.2., the 3.5.2.1. mammalian toxicity and irritation performance of the concentrate and mixed product shall be determined in accordance with 4.1.1.
- Concentrate. The toxicity of the wet or dry concentrate shall meet the requirements in 3.5.2.1.1. Table 1 when tested in accordance with 4.1.1.

Table 1. Toxicity and irritation requirements for wet or dry concentrate.					
<u>Test</u>	<u>Requirement</u>				
Acute oral toxicity	LD <sub>50</sub> > 500 mg/kg.				
Acute dermal toxicity	LD <sub>50</sub> > 2000 mg/kg.				
Primary eye irritation for single and double washed eyes	Mildly irritating or less. If more irritating, recommend protective gear and safe handling procedures.				
Primary dermal irritation	Primary irritation index < 5.0. If more irritating, recommend protective gear and safe handling procedures.				

. . . . .

- 3.5.2.1.2. Mixed Product. The toxicity of the mixed product shall meet the requirements in Table 2 when tested in accordance with 4.1.1.
- 3.5.2.1.3. Report of Mammalian Toxicity and Irritation Test Results. The results of the toxicity testing shall be certified by the testing laboratory and submitted to the Government, in accordance with 4.1.1.1.

Table 2. Toxicity and irritation requirements for mixed product.			
Test	<u>Requirement</u>		
Acute oral toxicity	LD <sub>50</sub> > 5000 mg/kg.		
Acute dermal toxicity	LD <sub>50</sub> > 2000 mg/kg.		
Primary eye irritation for single and double washed eyes	Mildly irritating or less If more irritating, recommend protective gear and safe handling practices.		
Primary dermal irritation	Primary irritation index < 5.0.		

**3.5.2.1.4.** <u>Review of Mammalian Toxicity and Irritation Test Results</u>. When the test results for a concentrate indicate that personal protective equipment / safe handling procedures are needed, the manufacturer shall make recommendations to be added to the product label and the Safety Data Sheet (SDS).

In accordance with 4.1.1.2, the results and related recommendations shall be reviewed by the Government.

- **3.5.2.2.** Aquatic Toxicity. The  $LC_{50}$  for rainbow trout exposed to the concentrate shall be greater than 200 mg/L when tested in accordance with 4.1.2.
- **3.6.** <u>Combustion-Retarding Effectiveness</u>. All mixed retardants shall meet one the following criteria:
  - a) Conformance to 3.6.1, confirmed by testing to 4.3.1
  - b) Conformance to 3.6.2, as tested in 4.2.
- **3.6.1.** <u>Required Retarding Salt Concentration</u>. A product containing one of the following retarding salts or mixtures of salts at or greater than the listed concentrations may not require a burn test.

The salt concentration shall be verified by chemical analysis during the evaluation.

- Diammonium phosphate (DAP), industrial grade or better (NPK# 21-53-0), in the mixed retardant at a concentration of 10.6 percent or greater.
   Fertilizer grade and other lower grades shall be burn tested to establish an acceptable mix ratio.
- b. Monoammonium phosphate (MAP), industrial grade or better (NPK# 12-62-0), in the mixed retardant at a concentration of 9.2 percent or greater.
   Fertilizer grade and other lower grades shall be burn tested to establish an acceptable mix ratio.
- **c.** P2O5 in fertilizer grade ammonium polyphosphates (APP; NPK# 10-34-0 or 11-37-0) in the mixed retardant at a concentration of 8.0 percent or greater total phosphate.
- d. Combinations of DAP and MAP, industrial grades or better, having a total of 10.6 percent DAP (NPK# 21-53-0) equivalents or greater using the conversions described below.

Use the DAP concentration without conversion.

Use the MAP concentration multiplied by 1.15.

e. Fertilizer grade DAP or MAP, alone or in combination shall require a burn test.

3.6.2.	<u>Combustion-Retarding Effectiveness Test</u> . When a mixed retardant does not meet one of the criteria in 3.6.1, the product shall undergo a fire effectiveness test in accordance with 4.2.
	A reduction index greater or equal to the reduction index of the standard chemical, 10.6-percent DAP, shall be acceptable.
3.7.	<u>Physical Properties.</u> In accordance with 4.3, the physical properties of the dry and wet concentrate and all mixed retardants shall be determined as specified in 3.7.1, 3.7.2, and 3.7.3.
	These test results shall define the standard characteristics for the submitted product and be used to address quality issues.
3.7.1.	<u>Physical Properties of the Dry Concentrate</u> . In accordance with 4.3, the retarding salt content of the dry concentrate shall be determined.
	The values determined shall be used as baseline values for stability tests as required in 3.10.
3.7.2.	Physical Properties of the Wet Concentrate. In accordance with 4.3, the retarding salt content, viscosity, density, and pH of the wet concentrate shall be determined.
	The values determined shall be used as baseline values for stability tests as required in 3.10.
3.7.3.	<u>Physical Properties of the Mixed Retardant</u> . In accordance with 4.3, the retarding salt content, the refractometer reading, steady-state viscosity, density, and pH of the mixed retardant shall be determined.
	The values determined shall be used as baseline values for stability tests as required in 3.10.
3.7.3.1.	<u>Retarding Salt Content</u> . When tested in accordance with 4.3.1 the retarding salt content shall meet the requirements of 3.6.1 or 3.6.2.
3.7.3.2.	<u>Steady State Viscosity</u> . When tested in accordance with 4.3.3.1, the steady state viscosity shall meet the requirements of the classification for which the product was submitted.
	When the mixture is prepared in 70°F fresh water, no viscosity less than 150 or greater than 1500 centipoise shall be acceptable.
3.7.3.3.	<u>Maximum Density of the Mixed Retardant for Aerial Applications</u> . A maximum density of the mixed retardant shall be no greater than 9.2 lbs/gallon. If a retardant meets all other criteria but exceeds this maximum density, it will be acceptable for use from ground applications only.
3.7.3.4.	<u>pH.</u> When tested in accordance with 4.3.5, the pH shall be measured. A pH between 4.0 – 10.0 pH units is acceptable.
3.8.	<u>Materials Effects</u> . As required by 3.8.1 through 3.8.4, the effects of the wet concentrate and mixed retardant on metallic and non-metallic materials shall be determined in accordance with 4.4.
3.8.1.	<u>Uniform Corrosion</u> . When tested in accordance with 4.4.1, wet concentrate and freshly prepared mixed retardant shall not have corrosion rates exceeding those shown in Table 3 for the alloys listed.

	2024-T3 Aluminum			4130	Steel		Yellow Brass	Az31B Magnesium					
	То	otal	Par	tial	Т	otal	Par	rtial	Partial	To	otal	Par	tial
Temperature: <sup>°</sup> F	70	120	70	120	70	120	70	120	120	70	120	70	120
							mils-pe	r-year					
Concentrates Wet concentrates	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
for fixed-tank helicopters	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Wet concentrates <sup>2</sup> except fixed-tank helicopters	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0				
Mixed Products													
Fixed-tank helicopters <sup>3</sup>	2.0	2.0	2.0	2.0	5.0	5.0	5.0	5.0	5.0	4.0	4.0	4.0	4.0
Fixed-wing air tankers <sup>4</sup>	2.0	2.0	2.0	2.0	5.0	5.0	5.0	5.0	5.0				
Helicopter bucket and <sup>2</sup> Ground-based application	2.0	2.0	2.0	2.0	5.0	5.0	5.0	5.0	5.0				

### Table 3. Maximum Allowable Corrosion Rates (mils-per-year) for Wildland Fire Chemical Products.<sup>1</sup>

<sup>1</sup>All uniform corrosion rates shall be determined by 90-day weight loss tests. All uniform corrosion rates are the maximum allowable average of all replicates.

<sup>2</sup>Magnesium uniform corrosion tests shall be performed for performance information. Intergranular corrosion tests are not required on aluminum or magnesium.

<sup>3</sup>Intergranular corrosion tests shall be performed on aluminum and magnesium coupons; no intergranular corrosion is allowed.

<sup>4</sup>Intergranular corrosion tests shall be performed on aluminum coupons; no intergranular corrosion is allowed. Magnesium uniform corrosion tests shall be performed for performance information. Intergranular corrosion tests are not required on magnesium.

**3.8.2.** Intergranular Corrosion (IGA). When tested in accordance with 4.4.2, the alloys specified in 3.8.2.1 through 3.8.2.4 shall show no evidence of intergranular corrosion as summarized in Table 4.

Table 4. Intergranular Corrosion				
Application Method	Alloy			
	Aluminum 2024-T3	Magnesium Az-31-B		
Helicopter Fixed-Tank	No detectable IGA	No detectable IGA		
Multi-Engine, Fixed-Wing Airtanker	No detectable IGA	Not Applicable		
Single-Engine, Fixed-Wing Airtanker	No detectable IGA	Not Applicable		
Helicopter Bucket.	Not Applicable	Not Applicable		
Ground Based Equipment	Not Applicable	Not Applicable		

- **3.8.2.1.** <u>Helicopter Fixed Tank</u>. When tested in accordance with 4.4.2, coupons made of alloy 2024-T3 aluminum and Az-31B magnesium shall not exhibit intergranular corrosion following exposure to mixed retardant during the uniform corrosion tests.
- **3.8.2.2.** <u>Multi-Engine, Fixed-Wing Air Tanker</u>. When tested in accordance with 4.4.2, coupons made of alloy 2024-T3 aluminum shall not exhibit intergranular corrosion following exposure to mixed retardant during the uniform corrosion tests.
- **3.8.2.3.** Single-Engine, Fixed-Wing Air Tanker. When tested in accordance with 4.4.2, coupons made of alloy 2024-T3 aluminum shall not exhibit intergranular corrosion following exposure to mixed retardant during the uniform corrosion tests.
- **3.8.2.4.** <u>Helicopter Bucket and Ground Based Application Equipment</u>. There are no intergranular corrosion requirements for helicopter buckets.
- **3.8.3.** Effects of Concentrate and Mixed Product on Non-Metallic Materials. In accordance with 4.4.3, the wet concentrates and all mixed retardants shall be tested to determine their effect on the non-metallic materials listed in Table 5 and their ability to meet the requirements of 3.8.3.1.

<b>Table 5.</b> Materials Tested to Determine the Effect of Exposure to Wet Concentrate and Mixed Retardant on Non-Metallic Materials.			
Material	Material Specification		
Chloroprene Rubber	AMS 3208M		
PVC Plastic, Flexible	MIL A-A-55859A		
Fiberglass/Epoxy Resin	AMS C-9084		
High-Density Polyethylene	ASTM D 4976		
Low-Density Polyethylene	ASTM D 4976		
Flexible Cross-Linked Polyolefin	AMS DTL-23053/5		

**3.8.3.1.** Effect of Exposure to Wet Concentrate and Mixed Product on Non-Metallic Materials. When tested as required in 3.8.3, the changes in hardness and volume of each of the materials listed in Table 5 shall be determined.

Characteristics	Reportable Change
Hardness	≤ 10-percent decrease
Hardness	≤ 20-percent increase
Volume	≤ 0.5 mL volume change

**3.8.4.** <u>Abrasion.</u> When tested in accordance with 4.5, all wet concentrates and mixed retardants prepared from dry concentrates, shall be tested for the abrasiveness of the retardant to aluminum 2024-T3.

Total abrasion of the disc and the wear plate shall not exceed 0.010 inch (0.25 mm), when rotated at 1800 rpm for 50 hours.

**3.9.** <u>Pumpability</u>. When tested in accordance with 4.6 the pumpability of all wet concentrates and mixed retardants prepared from dry concentrates shall be determined.

A minimum flow rate of 18 gallons (68.1 liters) per minute is required.

- **3.10.** <u>Product Stability</u>. When tested in accordance with 4.7, concentrates and mixed retardants shall meet all applicable requirements of 3.10.1 through 3.10.3.
- **3.10.1.** <u>Outdoor Storability</u>. When tested in accordance with 4.7.1, the concentrate and mixed products shall meet all applicable requirements of 3.10.1.1 and 3.10.1.2. Compliance with the criteria in Table 6 shall be determined. Pending the results of this testing, a designation of the stability will be determined as either "Storable" or "Not Storable."
- **3.10.1.1.** <u>Concentrates</u>. All concentrates shall meet the requirements of either 3.10.1.1.1 or 3.10.1.1.2.
- **3.10.1.1.1**. <u>Dry Concentrates</u>. In accordance with 4.7.1.1.1, dry concentrates shall be stored outdoors for 52 weeks, unless product is classified as pretreatment, non-storable as described in 1.2.3.2.

The stored concentrate shall have no visual separation such as discoloration or caking. Lumps shall fit through a 0.25-inch (0.625 cm) sieve-size. The stored concentrate shall be used to prepare mixed retardant as required in 3.10.1.1.3.

**3.10.1.1.2.** <u>Wet Concentrates</u>. In accordance with 4.7.1.1.2, wet concentrates shall be stored outdoors for 52 weeks, unless product is classified as pretreatment, non-storable as described in 1.2.3.2.

There shall be no separation resulting in particles larger than 0.25-inch (0.625 cm) sieve-size.

The stored concentrate shall be tested to determine the following properties:

- a. Viscosity, in accordance with 4.3.3,
- b. Density, in accordance with 4.3.4, and
- c. pH, in accordance with 4.3.5.

The results will be evaluated to the pass/fail criteria of Table 7.

The stored concentrate shall be used to prepare mixed retardant as required in 3.10.1.1.3.

**3.10.1.1.3.** <u>Mixed Retardant from Stored Concentrate</u>. In accordance with 4.7.1.1.3, and as required by 3.10.1.1.1 and 3.10.1.1.2, the mixed retardant shall be prepared from the stored concentrate and tested as required in 3.10.1.1.4 through 3.10.1.1.6.

- **3.10.1.1.4.** <u>Physical Properties of Mixed Retardant from Stored Concentrate</u>. The mixed retardant, prepared as required in 3.10.1.1.3, shall be tested to determine the following properties:
  - a. Steady State Viscosity, in accordance with 4.3.3.1.,
  - b. Density, in accordance with 4.3.4, and
  - c. pH, in accordance with 4.3.5.

These values shall be within the allowable variation, as shown in Table 6. The original values from fresh concentrate (determined in 3.7.3), are used as the baseline for this testing.

<ul> <li><u>Table 6</u>. Allowable Variation of Physical Properties of:</li> <li>Mixed Retardant (stored for 14-days), and</li> <li>Mixed Retardant prepared from concentrate stored for 52 weeks</li> </ul>		
<u>Property</u>		
Steady-State Viscosity	Shall not exceed ± 15 percent change from initial value	
Density	Shall not exceed ± 1 percent change from initial value	
рН	No change criteria listed. Reading shall be between 4.0 and 10.0 pH units	

- **3.10.1.1.5.** <u>Stability of Mixed Retardant from Stored Concentrate</u>. The mixed retardant, prepared as required by 3.10.1.1.3, shall be stored outdoors for 14 days, in accordance with 4.7.1.2.2, for freshly prepared mixed retardant. All products shall meet the acceptance criteria listed in Table 6 for the 14-day stability tests. The stored mixed retardant shall be tested as required in 3.10.1.1.4 and 3.10.1.1.5.
- **3.10.1.1.6.** <u>Corrosivity of Mixed Retardant from Stored Concentrate</u>. The mixed retardant, prepared as required by 3.10.1.1.3, shall be tested to determine uniform and intergranular corrosion and shall meet the uniform and intergranular corrosion requirements of 3.8.1 and 3.8.2.
- **3.10.1.2.** <u>Outdoor Stability of the Fresh Mixed Retardant</u>. In accordance with 4.7.1.2, the fresh mixed retardant shall be stored outdoors for 14 days.

At the end of the storage period, the stored mixture shall be examined visually and shall have no separation resulting in particles larger than 0.25-inch (0.635 cm) sieve size.

The stored mixed retardant shall be tested as required in 3.10.1.2.1 or 3.10.1.2.2.

**3.10.1.2.1.** <u>Stability of the Mixed Retardant: Storable</u>. In accordance with 4.7.1.2.1, dry concentrate products shall be mixed and stored outdoors for 52 weeks.

Following recirculation, there shall be no separation resulting in crystals or other particles larger than 0.25-in (0.635 cm) sieve size.

The mixed retardant shall be tested to determine the following physical properties:

- a. Steady-State Viscosity, in accordance with 4.3.3.1,
- b. Density, in accordance with 4.3.4, and
- c. pH, in accordance with 4.3.5.

These values shall be within the allowable variation from the initial values, determined in 4.3, physical properties, on the fresh retardant, as shown in Table 7. The mixed retardant shall meet the corrosion requirements shown in Table 3 for uniform and intergranular corrosion (Table 4) when tested in accordance with 4.4.1 and 4.4.2.

<ul> <li><u>Table 7</u>. Allowable Variation of Physical Properties of 52-week stored:</li> <li>Mixed Retardant, and</li> <li>Wet Concentrates</li> </ul>			
Property			
Steady-State Viscosity	Shall not exceed $\pm$ 40 percent change from the initial value		
Density	Shall not exceed $\pm$ 1 percent change from the initial value		
рН	No change criteria listed. Reading shall be between 4.0 and 10.0 pH units		

**3.10.1.2.2.** <u>Stability of the Mixed Retardant: Not Storable</u>. In accordance with 4.7.1.2.2, the mixed retardant shall be stored outdoors for 14 days.

Following recirculation, there shall be no separation resulting in crystals or other particles larger than 0.25-in (0.635 cm) sieve size.

The mixed retardant shall be tested to determine the following physical properties:

- a. Steady-State Viscosity, in accordance with 4.3.3.1,
- b. Density, in accordance with 4.3.4, and
- c. pH, in accordance with 4.3.5.

These values shall be within the allowable variation from the initial values, determined physical properties, on the fresh retardant, as shown in Table 6.

- **3.10.1.2.3.** <u>Storable and Not-Storable Products</u>. Figure 2 in Appendix A provides a flow-diagram of this evaluation.
  - All wet concentrates shall be evaluated as "Storable" products.
  - Dry concentrates shall be evaluated for stability in both concentrate and mixed forms.
    - The concentrate shall meet the requirements of Table 6 and,
    - The mixed product shall meet the requirements of Table 7 to be classified "Storable."

**3.10.2.** Optional: Accelerated Stability Testing on Concentrate and Mixed Retardant. In accordance with 4.7.1, and Appendix A the product concentrate and mixed retardant (prepared from dry concentrate) shall be subjected to temperature cycling. This testing shall only be performed when interim qualification is being sought in accordance with Appendix A of this specification.

The mixed retardant shall be tested to determine the following properties:

- a. Steady-State Viscosity, in accordance with 4.3.3.1.,
- b. Density, in accordance with 4.3.4, and
- c. pH in accordance with 4.3.5.

Changes in these properties shall be calculated and compared to the original results (from section 3.7) to meet the acceptance values of Table 7.

The liquid concentrate will be evaluated to the limits of Table 6. All concentrates shall be used to prepare mixed retardant which shall be tested as required in 3.10.2.1.

- **3.10.2.1.** <u>Mixed Retardant Prepared from Accelerated Stability Testing-Cycled Concentrate</u>. As required by 3.10.2, mixed retardant prepared from temperature-cycled concentrate and fresh water shall be tested in accordance with 4.7.1.1.3 to determine the following properties:
  - a. Steady-State Viscosity, in accordance with 4.3.3.1,
  - b. Density, in accordance with 4.3.4, and
  - c. pH, in accordance with 4.3.5.

Changes in these properties shall be calculated and compared to the original results (from section 3.7) to meet the acceptance values of Table 6.

- **3.10.3.** Resistance of Wet Concentrates and Mixed Retardant to Microbial Growth. After 14 days in storage in accordance with 4.7.2, wet concentrates and mixed retardant shall show no visible sign of microbial contamination, including growths on the surface or within the fluid, significant discoloration, or other change in appearance.
- **3.11.** <u>Color Properties</u>. All mixed retardants shall be evaluated in accordance with 4.8.1.1 through 4.8.1.4 and meet the requirements of 3.11.1 through 3.11.3 as applicable for the color system used.
- **3.11.1.** <u>Acceptable Colors</u>. Color of the long-term retardants shall be easily identifiable as different from that of colored water enhancers.

In accordance with 4.9.1.1., Long-term retardant shall be uncolored or a shade of red in the color spectrum greater than 590 nm. Pink hues that don't fall within the conventional color spectrum of visible light may also be permitted at the discretion of the Government.

- **3.11.1.1** Opacity of Mixed Retardant. When tested in accordance with 4.8.1.3, mixed retardant shall be tested to determine their opacity on a black-white block opacity chart.
- **3.11.2.** <u>Color of Iron-Oxide Colored Retardant</u>. The iron-oxide colored mixed retardant shall contain a minimum of at least 12 grams of iron oxide per gallon to impart red color to the mixed retardant.
- **3.11.3.** <u>Laboratory Evaluation of Fugitive-Colored Mixed Retardant</u>. As required by 3.11.3.1 and 3.11.3.2, fugitive-colored mixed retardant shall be tested to determine the opacity and fading of films applied in accordance with 4.8.1.1 through 4.8.1.4.

- **3.11.3.1.** Fading of Fugitive-Colored Mixed Retardant. In accordance with 4.8.1.4, at the end of the exposure period in accordance with 4.8.1.2, the mixed retardant with fugitive colorant shall be no more colored than a sample of the uncolored mixed product, applied and treated in the same manner as the mixed retardant.
- **3.12.** <u>Operational Field Evaluation (OFE)</u>. In accordance with 4.10, after meeting requirements of 4.4 through 4.9, an analysis shall be undertaken to determine the need for an operational field evaluation. A copy of the analysis shall be provided to the submitter.

The analysis will document the rationale for no field test or provide a summary of the issues and performance to be addressed during the field evaluation.

Product for the operational field evaluation shall be purchased by the Government according to the classification established during qualification testing. All other costs associated with the operational field evaluation shall be the responsibility of the submitter.

The product shall perform satisfactorily as determined by the Government under operational conditions during a fire season. An acceptable test should include firefighting operations on a variety of fuel types, slopes, aspects, and exposures.

Formulations shall be listed on the QPL as either fully qualified or interim qualified prior to an OFE.

- 4. <u>**TEST PROCEDURES.</u>** Detailed test methods are described in Standard Test Procedures for the Evaluation of Wildland Fire Chemical Products (STP).</u>
- 4.1. <u>Health and Safety</u>
- **4.1.1.** <u>Mammalian Toxicity and Irritation Tests (STP-1.3)</u>. As required by 3.5.2.1, mammalian toxicity and irritation test on wet and dry concentrates and the mixed retardant, shall be conducted by an independent biological testing laboratory approved by the Government.

All testing shall be conducted in compliance with 40 CFR 160 and 792 Good Laboratory Practice Standards, in accordance with EPA/OCSPP Health Effects Test Guidelines, series 870 and shall include:

- a. OCSPP 870.1100, Acute Oral Toxicity
- b. OCSPP 870.1200, Acute Dermal Toxicity
- c. OCSPP 870.2400; Primary Eye Irritation
- d. OCSPP 870.2500; Primary Dermal Irritation
- **4.1.1.1.** <u>Report of Test Results</u>. The results of the mammalian toxicity and irritation testing shall be certified by the testing laboratory and submitted directly to the Government as required by 3.5.2.1.3.
- **4.1.1.2.** <u>Review of Mammalian Toxicity and Irritation Test Results</u>. When required in accordance with 3.5.2.1.4, the Government shall review the results of the testing and the submitter's recommended protective gear and safe handling procedures to ensure adequate protection for firefighters and the general public who may come into contact with the product.
- **4.1.2.** <u>Fish Toxicity (STP-1.5)</u>. As required by 3.5.2.2, the toxicity of the concentrate to rainbow trout (Oncorhynchus mykiss) shall be determined in accordance with OCSPP 850.1075, Ecological Effects Test Guidelines, Fish Acute Toxicity Test, Freshwater and Marine.

Static test (non-renewal) conditions in ASTM soft water (described in ASTM E729) at  $12 \pm 2$  °C shall be maintained throughout the 96-hour test period.

All fish shall be  $60 \pm 15$  days post hatch.

**4.2.** <u>Combustion Retarding Effectiveness Test (STP-2.1)</u>. As required by 3.6.2, when the retardant does not meet the salt content requirements of 3.6.1., the combustion retarding effectiveness of the mixed retardant shall be determined.

Fuel beds of aspen excelsior or Ponderosa pine needles shall be prepared and treated with mixed retardant or 10.6-percent diammonium phosphate (control) and then dried at standard temperature and humidity to remove the water contained in the retardant or control.

The mixed retardant-treated fuel beds shall be tested and the effect of the mixed retardant on the rate of flame spread and rate of fuel weight loss determined. The reduction index shall be calculated by comparing the rate of flame spread and rate of weight loss of the retardant-treated and control-treated beds to the untreated beds made from the same fuels as the treated beds.

- **4.3.** <u>Physical Properties</u>. As required by 3.7, the concentrate and the mixed retardant shall be tested to determine the retarding salt content, refractometer reading, viscosity, steady state viscosity, density, and pH.
- **4.3.1.** <u>Retarding Salt Content Test (STP-4.1)</u>. As required by 3.7.1. through 3.7.3, the mixed retardant shall be tested using recognized analytical methods to determine the retarding salt content.

Ortho and total phosphate shall be determined in accordance with AOAC accepted test methods.

- **4.3.2.** <u>Refractometer Reading (STP-4.2)</u>. As required by 3.7.3, the refractometer reading of a properly mixed retardant shall be determined using a hand-held refractometer that incorporates the arbitrary scale found in industrial fluid testers or the Brix scale when needed.
- 4.3.3. <u>Viscosity Test (STP-4.5)</u>. As required by 3.7.2 and 3.7.3, the viscosity of all wet concentrates and mixed retardants at 70 °F shall be measured using a Brookfield Viscometer, model LVF, or equal, set at 60 rpm with the appropriate spindle. The same spindle shall be used for the initial and final viscosity measurements to determine stability performance.
- 4.3.3.1. Steady State Viscosity. As required by 3.7.3.2, the viscosity of the mixed retardant at 10 minutes, 1 hour, 4 hours, 8 hours, 1 day, and daily for 8 days after mixing shall be determined as described in 4.3.3. Viscosity values shall be graphed against time. The viscosity value corresponding to the plateau of the viscosity curve, typically 24 hours, shall be determined. This shall be referred to as the steady state viscosity.
- **4.3.4.** <u>Density Test (STP-4.3)</u>. As required by 3.7.2 and 3.7.3, the density of the wet concentrate and mixed retardant shall be determined to the nearest 0.001 g/mL by fluid displacement or electronic density meter.
- **4.3.5.** <u>pH Value Test (STP-4.4)</u>. As required by 3.7.2 and 3.7.3.4, the pH of wet concentrates and mixed retardant shall be determined using a full range pH meter, capable of being read to 0.1 pH.
- **4.4.** <u>Materials Effects Tests</u>. As required by 3.8, wet concentrates and mixed retardant shall be tested to determine uniform and intergranular corrosion of selected alloys and the effects to non-metallic materials.

**4.4.1.** <u>Uniform Corrosion (STP-5.1)</u>. As required by 3.8.1 and 3.10.1.1.6., the wet concentrate and mixed retardant, prepared with fresh water, of all concentrates shall be tested for uniform corrosion as summarized below.

Test coupons of 2024-T3 aluminum, 4130 steel, UNS C26000 yellow brass, and Az31B magnesium shall be engraved with a unique identification number, measured, cleaned, dried, and weighed.

Each coupon shall be immersed in the test solution and allowed to remain undisturbed at the required conditions for 90 days.

At the end of the test duration, each coupon shall be cleaned, dried, and weighed, and the corrosion rate calculated.

All corrosion rates for the same product, alloy, immersion condition and temperature shall be averaged.

**4.4.2.** <u>Intergranular Corrosion (STP-5.2)</u>. As required by 3.8.2., products shall be tested for intergranular corrosion as summarized below.

At least one coupon for each exposure and temperature from the uniform corrosion tests on the specified alloys shall be sliced as shown in Figure 1.

The coupon shall be mounted, polished to 0.3 micron alumna finish, and etched using Keller's reagent for aluminum coupons and Nital reagent for magnesium coupons.

The etched coupons shall be examined microscopically with a magnification of 500X.

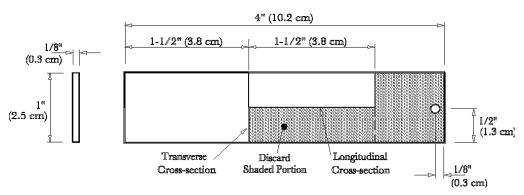


Figure 1. Diagram for cutting and examining coupons for intergranular corrosion.

**4.4.3.** Effect of Wet Concentrate and Mixed Retardant on Non-Metallic Materials (STP-5.3). As required by 3.8.3, the wet concentrate and all mixed retardants shall be tested to determine their effect on non-metallic materials, as summarized below.

Prior to exposure of the non-metallic materials, the hardness and volume of each non-metallic sample shall be determined. A hand-held durometer shall be used to measure the hardness and either fluid displacement or dimensional analysis shall be used to determine the volume.

The test pieces of each non-metallic material shall be exposed for 20 cycles. Each cycle shall consist of the material being immersed in the fluid at night and on weekends and in the air during the work day.

At the end of the test period, each test piece shall be rinsed, allowed to air dry, and the hardness and volume of each piece determined on the same day as the exposure ends.

The change in hardness and volume from the initial value of each shall be calculated.

If the result of either exceeds the allowable maximum, the measurements shall be repeated the next day and the change calculated. No additional measurements shall be allowed.

The results of the last set of measurements taken shall be used to determine performance.

**4.5.** <u>Abrasion Test (STP-5.4)</u>. As required by 3.8.4, the abrasiveness of the wet concentrate or mixed retardant from dry concentrate to aluminum 2024-T3 shall be determined as summarized below.

A disc and a wear plate made of aluminum 2024-T3 shall be installed on the apparatus, parallel to each other with a 0.020-inch (0.5-mm) gap between them, and submerged in retardant.

The top plate shall be rotated at 1800 rpm for 50 hours.

The plate and disc shall be measured to the nearest 0.001 inch (0.025 mm) before and after the test.

The maximum wear on the disc and the wear plate shall be added to determine the total abrasion.

**4.6.** <u>Pumpability Test (STP-7)</u>. As required by 3.9, the pumpability of the wet concentrate or mixed retardant from dry concentrate shall be determined as summarized below.

The test apparatus shall consist of a storage tank, a pump and a scale-mounted weighing tank.

The time required for the retardant to be transferred from the storage tank to the weighing tank shall be determined.

The change in weight over time shall be used to calculate the flow rate of the product.

- **4.7.** <u>Product Stability Test</u>. As required by 3.10, all concentrates and mixed retardant shall be tested for product stability as summarized in 4.7.1 and 4.7.2.
- **4.7.1.** <u>Outdoor Storage Test (STP-6.1)</u>. As required by 3.10.1, concentrates and mixed retardant shall be tested to determine the effects of storage in outdoor weather conditions.
- **4.7.1.1.** <u>Concentrates</u>. Each retardant concentrate shall be evaluated to determine outdoor stability in accordance with 4.7.1.1.1 or 4.7.1.1.2.

**4.7.1.1.1** <u>Dry Concentrates</u>. As required by 3.10.1.1.1, each dry concentrate shall be evaluated for outdoor stability.

To document the initial condition of the product, the fresh concentrate shall be examined visually to determine the general condition of the concentrate, including the fluidity, presence or absence of lumps, the ease of crumbling the lumps, or visually separate layers.

The fresh concentrate shall then be stored, in large sample containers outdoors at NTDP-Missoula and NTDP-San Dimas for 52 weeks.

At the end of the 52 week storage period, the samples shall be examined visually to determine that changes in the general condition, such as fluidity and/or presence of hard lumps, have not occurred.

As required by 3.10.1.1.1, the stored concentrate shall be used to prepare mixed retardant in accordance with 4.7.1.1.3.

**4.7.1.1.2.** <u>Wet Concentrates</u>. As required by 3.10.1.1.2, each wet concentrate shall be evaluated for outdoor stability.

The initial condition of the fresh concentrate shall be documented including the presence or absence of crystals or other solids greater than 0.25 inch (0.635 cm).

A 4130 grade steel coupon is suspended in the fresh concentrate, and the container sealed. The fresh concentrate shall then be stored, in large sample containers outdoors at NTDP-Missoula and NTDP-San Dimas for 52 weeks.

At the end of the 52 week storage period, the steel coupon shall be removed and samples shall be inspected to determine that changes in the general condition of the concentrate have not occurred and tested as required in 3.10.1.1.2.

As required by 3.10.1.1.3, the stored concentrate shall be circulated and used to prepare mixed retardant in accordance with 4.7.1.1.3.

**4.7.1.1.3.** <u>Mixed Retardants from Stored Concentrate</u>. As required by 3.10.1.1.3, the mixed product shall be prepared from stored concentrate.

As required by 3.10.1.1.3 and 3.10.1.1.4, mixed product shall be prepared from stored concentrate and fresh water and tested to determine the density, pH, and steady-state viscosity.

As required by 3.10.1.1.5 and 3.10.1.1.6, mixed product shall be prepared from stored concentrate and fresh water and tested to determine the outdoor stability and corrosivity of the mixed retardant from stored concentrate.

- **4.7.1.2.** <u>Mixed Retardant</u>. Each mixed retardant shall be evaluated to determine outdoor stability in accordance with 4.7.1.2.1 or 4.7.1.2.2.
- **4.7.1.2.1.** <u>Storable</u>. As required by 3.10.1.2.1, the mixed retardant shall be stored in large sample containers, each containing a large, 4130 steel stability coupon, outdoors at NTDP-Missoula and NTDP-San Dimas for 52 weeks.

At the end of the 52-week storage period, the sample shall be mixed for one minute with low shear (1800 rpm with 2-bladed propeller-type stirrer).

The recirculated sample shall then be tested in accordance with 4.3 to determine steady-state viscosity, density, refractometer, pH value, and 4.4.1 and 4.4.2 to determine uniform corrosion and intergranular corrosion.

**4.7.1.2.2.** <u>Not Storable</u>. As required by 3.10.1.2.2, the mixed retardant shall be stored in large sample containers, each containing a large, 2024-T3 aluminum stability coupon, outdoors at NTDP-Missoula and NTDP-San Dimas for 14 days.

At the end of the 14-day storage period, the sample container shall be opened. The stored product shall be mixed for one minute with low shear (1800 rpm with 2-bladed propeller-type stirrer).

The recirculated sample shall be tested in accordance with 4.3 to determine, steadystate viscosity, density, refractometer, and pH value.

**4.7.2.** <u>Resistance to Microbial Growth Test.</u> As required by 3.10.3, the mixed retardant shall be observed, and assessed for microbial contamination.

The physical appearance, including growths on the surface or within the fluid, significant discoloration, or other changes shall be described and recorded at the initiation of the test and after the 14 days have elapsed.

- **4.8.** <u>Color Properties.</u> As required by 3.11, the iron oxide-colored, fugitive-colored, and uncolored mixed retardant shall be evaluated to determine the color characteristics of the mixed products.
- **4.8.1.** <u>Laboratory Visibility Test of Fugitive-Colored Retardant</u>. As required by 3.11.3, the mixed retardant shall be tested to determine the opacity and fading characteristics of the fugitive-colored retardant.
- **4.8.1.1.** <u>Preparation of the Test Panels</u>. The fugitive-colored product and the product without color, as a control, shall be used to prepare the test panels.

Four test panels of plate glass shall be treated by applying a 0.064 inch (4 GPC) thick layer of the test product with a Gardner knife or equivalent.

Four control panels shall be treated in the same manner with the uncolored product.

**4.8.1.2.** <u>Light Exposure of the Mixed Retardant</u>. The test and control panels shall be exposed outdoors to natural light at a test facility acceptable to the Government.

All exposures shall be performed in accordance with ASTM G-24 (Standard Recommended Practice for conducting Natural Light Exposures) until 50,000 Langleys are accumulated.

Visual observations and photographic records shall be made after each 10,000 Langleys of exposure.

At the end of the exposure period, the test panels shall be returned to the laboratory for final assessment in accordance with 4.8.1.4.

- **4.8.1.3.** Opacity of the Mixed Retardant. As required by 3.11.1.1, the opacity of the mixed retardant film on black-white block opacity chart shall be determined immediately after application and again after 24 hours.
- **4.8.1.4.** <u>Assessment of Fading</u>. As required by 3.11.3.1, the acceptability of fading of the test panels shall be assessed.

The outer edges of the film shall not be considered during the assessment. This area, the outer edge of the film, approximately 1 in (2.5 cm), shall be removed or masked.

Each panel shall be examined and the appearance of the film shall be compared with the appearance of the control panels. The appearance of the panels with the test material shall be neutral in color and not significantly different from the appearance of the control material.

**4.9.** <u>Spectral Evaluation.</u> As required by 3.11, the mixed retardant shall be tested to determine the spectral color wavelength.

- **4.9.1.** <u>Laboratory Spectral Color Evaluation of Mixed Retardant</u>. As required by 3.11.1, the mixed retardant shall be tested to determine the spectral color value in accordance with 4.9.1.1.
- **4.9.1.1.** <u>Spectral Color Analysis</u>. As required by 3.11.1., the mixed retardant shall be tested to determine the acceptability to the designated wavelength range. The testing instrument shall be a HunterLab Spectrophotometer (or equivalent) capable of determining L, a, b, color values or other acceptable color-space values.
- **4.10.** <u>Operational Field Evaluation (STP-12)</u>. As required by 3.12, the Government shall determine the need for an operational field evaluation, based on the nature of the formulations and/or results of the laboratory evaluation.

All laboratory testing shall be completed prior to conducting an operational field evaluation. An OFE may be initiated, if all preliminary criteria are met in accordance with Appendix A for Interim Qualification. When an operational field evaluation is required, a test plan will be developed by the Government.

The evaluation will be conducted in accordance with the developed test plan. Detailed test methods are described in Standard Test Procedures for the Evaluation of Wildland Fire Chemical Products.

# 5. <u>PREPARATION FOR DELIVERY</u>.

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- **5.1.** Submission of Samples for Laboratory Evaluation. When requested, and at no cost to the Government, the submitter shall provide the required amount of concentrate for use in the laboratory evaluation tests.
- **5.2.** Packaging and Labeling. Upon completion of documentation submission as required by 3.3.1. through 3.3.7., the product shall be supplied to the laboratory for testing.
- **5.2.1.** <u>Packaging</u>. The packaging of all wildland fire chemicals submitted for evaluation shall conform to regulations governing ground and air transport of materials.

....

The concentrates, in the quantities shown, shall be packaged as specified in Table 8.

Table 8. Test sample quantity and packaging.				
Product Type	Packaging	<u>Quantity</u>		
Dry concentrate	5-gallon (18.9 liter) Plastic Pails with Removable Lids	20 Pails – Each containing the amount of concentrate to be added to 25 gallons (95 liters) of water		
Wet concentrate	5-gallon (18.9 liter) Plastic Pails with Removable Lids	225 gallons (852 liters) in pails weighing $\leq$ 50 lbs (22.7 kg) each		
Note: Based on specific product information, the Project Leader may specify a different amount of product than shown here.				

**5.2.2.** <u>Labeling.</u> Individual containers of products submitted for evaluation shall be legibly marked in accordance with Federal Standard 123.

Labeling shall comply with Department of Transportation, Occupational Safety and Health Administration, and applicable State and Local requirements. Labeling shall be legible, permanent and clear in addition it shall include the following:

- **a.** Manufacturers or submitter's name or trademark
- **b.** Product identification including formulation identification codes and production information codes.
- c. Volume of concentrate (weight in the case of a dry concentrate) per container.
- **d.** Month and year of submission.
- 5.3. <u>Safety Data Sheets (SDS).</u>
- **5.3.1.** <u>Product SDS.</u> An SDS for the product from the submitter of the product, typically the manufacturer, shall accompany the application for qualification and each shipment of product.
- **5.3.2.** <u>Ingredient SDS.</u> All individual ingredients. The SDS from the manufacturer or the supplier of the chemical ingredient, as indicated on the Formulation Disclosure Sheet (FS 5100-37) shall be included for every ingredient, as a part of the submission paperwork.
- **5.3.3.** <u>Updated/Modified SDS.</u> It is the responsibility of the submitter to send copies of the SDSs when updates become available.
- **5.4.** <u>Additional Quantities.</u> When additional supplies are required, each container shall be marked with the original formulation identification and a secondary name or formulation identification number clearly identifying this as a remake of the original formulation.

This remake shall contain all of the original ingredients from the original sources and no other ingredients.

- **5.4.1.** <u>Labeling Additional Quantities.</u> All containers of the remake shall be clearly labeled and identified as a new or additional production using the original formula or recipe. Example: Product XYZ, original product name and product ID; unique remake code/identifier 123 for the remake this can be the production code or other identification.
- **5.5.** <u>Shipping Submission Sample to Wildland Fire Chemical Systems (WFCS).</u> The laboratory test sample shall be shipped at the submitter's expense to WFCS at the National Technology and Development Program (NTDP) in Missoula, Montana.

The complete address shall be provided as part of the shipping instructions when the product is requested.

An SDS for the product shall accompany the shipment.

If the product is imported, the submitter / supplier shall be responsible for the entire process necessary to deliver the product to the WFCS laboratory.

**5.5.1.** <u>Excess Submission Sample.</u> After the evaluation has been completed, any remaining unused and excess product shall be returned to the submitter at the submitter's expense.

## 6. <u>GENERAL INFORMATION.</u>

**6.1.** <u>Intended Use.</u> The products covered by this specification are intended for use in wildland firefighting.

The prescribed tests were selected to assess the effectiveness of the products to meet this goal.

The toxicity and environmental tests were selected to minimize hazards to firefighters, members of the general public, and the environment.

- 6.2. <u>Qualification.</u> Products that have been submitted for evaluation as described in this specification and submission procedures and have successfully met the requirements stated herein shall be added to the Forest Service Qualified Products List (QPL). Specific use designations, based on the requirements for each use or application, will be included.
- **6.3.** <u>Collection Agreement and Test Fees.</u> A Collection Agreement between the submitter and Wildland Fire Chemical Systems (WFCS) National Technology and Development Program (NTDP); USDA Forest Service shall be prepared.

This document describes the roles and responsibilities of the Government, WFCS laboratory personnel, and the submitter.

Specific information in the agreement includes a list of authorized contacts for the Government and for the submitter, as well as an estimate of the cost and time required for the evaluation.

- 6.4. <u>Submission of Manufacturer Documentation and Information.</u> All product information described shall be provided to the Government and reviewed by the designated agency representative, as summarized in 3.3 and described in "Manufacturer Submission Procedures for Qualification Testing" prior to acceptance of samples for testing.
- 6.4.1. <u>Paper copies available</u>. Paper copies of the required forms and documents can be obtained from the Program Leader or Project Leader, Wildland Fire Chemical Systems (WFCS), 5785 Highway 10 West, Missoula, MT, 5980, if web access is not available.
- **6.5.** <u>Mixing and Loading Equipment.</u> Suppliers are responsible for providing information relative to the equipment needs associated with the mixing and loading of their product at portable or permanent sites. They are responsible for demonstrating acceptable performance of their systems with their product.
- 6.6 <u>Definitions.</u> The definitions below may be specific to the fire chemical products although most of the terms are in general use. A few of the definitions may not appear in this specification but may be found in related documents such as the "Manufacturers' Submission Procedures." They are provided here as a convenience to the reader.

**ASTM Soft & Hard Water** – defined in ASTM E729 1996 (2007), Standard Guide for Conducting Acute Toxicity Tests on Test materials with Fishes, Macroinvertabates and Amphibians.

ASTM E729 1996 (2007) Table 1 - Add to high quality water conductivity less than 1 micro-ohm/cm and either total organic carbon (TOC) less than 2 mg/L, OR chemical oxygen demand (COD) less than 5 mg/L.

Acceptable water can usually be prepared using properly operated deionization, distillation, or reverse osmosis units.

See text of standard for additional info and details. Numbers listed are in mg/L.

Salts, mg/L	Very Soft	<u>Soft</u>	<u>Hard</u>	Very Hard
NaHCO <sub>3</sub>	12	48	192	384
CaSO <sub>4</sub> ·2H <sub>2</sub> O	7.5	30.0	120.0	240.0
MgSO <sub>4</sub>	7.5	30.0	120.0	240.0
KCI	0.5	2.0	8.0	16.0

**Class A Fuels.** Materials such as vegetation, wood, cloth, paper, rubber, and some plastics in which combustion can occur at or below the surface of the material.

**Component.** Each combination of ingredients, packaged together by the manufacturer for use in preparation of the mixed product by the user.

**Coupon, Corrosion.** A metal test specimen, approximately 1 in x 4 in x 1/8 in (2.5cm x 10.2 cm x 0.3 cm), made of 2024-T3 aluminum, AISI 4130 steel, C26000 yellow brass, or Az31B magnesium for use in uniform corrosion testing.

**Coupon, Large Stability.** A metal sample, approximately 2 in x 12 in x 1/8 in (5 cm x 30 cm x 0.3 cm), made of mild steel or 2024-T3 aluminum for use in outdoor stability testing.

**Density.** The mass in grams of 1 milliliter (mL) of product.

**Dry Concentrate.** A dry, single component which is mixed with water to prepare the mixed product.

**Fire Retardant, Long-Term.** A product containing one or more inorganic salts to reduce the intensity of a fire. It contains water which serves to aid in uniform distribution of the retardant salts over the target fuel.

The product continues to be an effective fire retardant after the water it originally contained has completely evaporated.

**Fire Suppressant.** Any agent used to extinguish the flaming and glowing phase of combustion by direct application to the burning fuel.

**Forest Service.** The term Forest Service as used throughout this document refers to the U.S. Department of Agriculture, Forest Service.

**Fugitive Color.** A coloring agent which imparts a high degree of visibility to the mixed product when first applied to wildland fuels but will gradually disappear over several months.

**GPC. (Gallons per 100 square feet).** A measure of the application of mixed product onto forest fuels to prevent or slow the spread of wildland fire. Gallons of mixed product per 100 square feet.

**Hydration.** The action of a combination of concentrate with water required to produce a thickened product.

**Ingredient.** Each single chemical used by the manufacturer in the formulation of the product. The supplier and quantity of each ingredient shall be specified in the submission paperwork.

Intergranular Corrosion. A corrosive attack on metal at the grain boundary.

**LC**<sub>50</sub>. The concentration of product in water that results in the death of 50 percent of the aquatic test specimens within a specified time frame.  $LC_{50}$ , In this document, expressed as milligrams of product in a liter of solution.

**LD**<sub>50</sub>. The dosage of a product at which 50 percent of the test animals die within a specified time frame.  $LD_{50}$ , in this document, expressed as milligrams of the product per kilogram of body weight of the test animal.

Manufacturer. The company or other entity who makes a product.

**Mixed Product.** The combination of a wet or dry concentrate and water at the qualified mix ratio for use in fire management activities.

Mix Ratio. The proportion of concentrate and water in the mixed product.

The mix ratio can be expressed in several ways:

- · Pounds of dry concentrate added to a gallon of water
- Gallons of wet concentrate to be added to a gallon of water
- Volume percentage of concentrate and water typical for foams and wet concentrate water enhancers

**pH.** A measure of the acidity or alkalinity of a solution, represented on a numeric scale with 7 representing neutral solutions. Higher numbers represent alkaline solutions and lower numbers represent acidic solutions.

**Pre-treatment Retardant.** A product designed specifically for ignition prevention, it is applied prior to an incident in areas at increased risk of wildfire starts, such as roadsides or power line rights-of-way. Products are generally durable, and may remain effective for an extended period of time, however, durability is not evaluated as part of this Forest Service Specification.

**Reduction Index.** A measure of the reduction in fire intensity (flame spread and weight loss) during the combustion retarding effectiveness test.

**Retarding Salt.** A single salt or combination of salts that impart combustion retarding effectiveness.

**Sample Container, large.** A 5.5-gallon (20 liter), low-density polyethylene carboy without spigot. Carboy shall be closed with a size 13.5 rubber stopper secured by a polypropylene screw cap.

**Standard Chemical.** Technical grade diammonium phosphate (DAP) mixed with water to produce a 10.6-percent (weight/weight) solution. This solution is used as a reference for the combustion-retarding effectiveness test.

**Steady State Viscosity.** The viscosity after hydration is complete and viscosity is stable.

**Temperature.** Each temperature included in the specification consists of a Fahrenheit temperature and allowable variation from that temperature and the Celsius equivalents for the temperature and range.

Commonly used temperatures and variations are shown in the first section below and included in the specification requirements and test descriptions by listing a simple Fahrenheit temperature.

<u>Fahrenheit</u>	<u>Celsius</u>	
5 °F± 5 °F	-15 °C $\pm$ 2.8 °C	
15 °F± 5 °F	-9.4 °C ± 2.8 °C	
35 °F± 2 °F	1.7 °C ± 1.1 °C	
40 °F± 5 °F	4.4 °C $\pm$ 2.8 °C	
70 °F $\pm$ 5 °F	21.1 °C ± 2.8 °C	
100 °F± 5 °F	37.8 °C ± 2.8 °C	
120 °F± 5 °F	48.9 °C ± 2.8 °C	

Uniform Corrosion. Removal of metal by chemical means over the entire surface.

Viscosity. A measure of the resistance of a liquid to flow, reported in centipoise (cP).

**Water, Deionized.** Water treated by distillation, ion exchange, reverse osmosis, or a combination of these methods to remove most salts in conformance to ASTM D-1193 Type IV reagent water.

Water, Fresh. Tap water with a hardness of 120 to 180 ppm of calcium carbonate.

**Wet Concentrate.** A liquid, single component which is added to water to prepare the mixed product.

# INTERIM QUALIFICATION AND ACCELERATED STABILITY TESTING

# 1. <u>SCOPE AND CLASSIFICATION</u>

- 1.1. <u>Scope</u>. Wildland fire chemicals undergo extensive testing that can take up to 24 months to complete. By incorporating initial screening tests, this appendix offers an alternate path resulting in interim qualification. This protocol does not change any of the test requirements but instead offers additional accelerated testing, at the cost of the submitter, reducing the 24 month testing timeline. All requirements of the base specification must still be met for full qualification and listing on the Qualified Products List (QPL).
- 1.2. <u>Interim Qualification</u>. The interim qualification process for a new wildland fire chemical can be requested by any company seeking qualification of a new product. The interim qualification must meet all criteria of Section 3 of this Appendix before being listed on the QPL under an interim designation. Upon successful completion of the requirements listed below, a product may be added to the QPL on an interim basis and proceed with an Operational Field Evaluation (OFE).

Interim qualification shall remain in effect for the OFE only. The interim qualification status shall not extend past the field season in which the OFE is conducted. Upon completion of all laboratory tests in accordance with FS 5100-304d, the QPL will be updated to reflect "Fully Qualified" if all requirements are met or it will be removed from the QPL if it fails to meet any requirement. A letter will be sent to the company indicating this change of status.

## 2. <u>APPLICABLE DOCUMENTS</u>

- 2.1. <u>Order of Precedence</u>. In the event of conflict between the text of this document and the references cited herein, the text of the specification (5100-304d) takes precedence. Nothing in this document, however, shall supersede applicable laws and regulations unless a specific exemption has been obtained.
- 2.2. <u>Specification</u>. The base specification (5100-304d) used for qualification is referenced throughout this document. This document also contains all testing procedures and detailed requirements.
- 2.3. <u>Terms and Conditions.</u> The Company shall provide a signed letter accepting the terms, conditions and requirements of the interim qualification.

# 3. <u>REQUIREMENTS</u>

- 3.1. <u>Wildland Fire Chemical Product Qualification Testing</u>. Laboratory testing to this appendix is supplementary to that of 5100-304d. New wildland fire chemicals may first be deemed acceptable for interim use per this Appendix. Products will continue full product testing and be noted as "Pending full qualification in accordance with 5100-304d" with a footnote on the QPL. A flow-diagram for this testing is outlined in Figure 1.
- 3.2. <u>Product Information</u>. As required by 5100-304d, all collection agreements, submission processes, and document review must first be completed before accepting any products for evaluation.
- 3.3. <u>Chemical Profile and Risk Assessment</u>. As required by 5100-304d section 3.4.3., chemical profiles shall be prepared and a risk assessment shall be performed on all products prior to being placed on

the Qualified Products List (QPL) for interim use. This assessment requires the results of toxicity testing prior to Risk Assessment and Chemical Profile initiation.

3.4. <u>Environmental Consultation with Regulatory Agencies</u>. As required by 5100-304d section 3.4.3.3, a new product may be included in an environmental consultation with the regulatory agencies.

The consultation will result in a formal statement of findings from each regulatory agency.

3.5. <u>Accelerated Stability Testing</u>. Outdoor stability testing may be simulated in an environmental chamber to reproduce the 1-year stability testing requirement. Following the accelerated stability testing, the samples will be required to meet the same test requirements of 5100-304d section 3.10 and 4.7. This test option cannot be pursued without also being subject to 1-year outdoor stability testing in accordance with section 3.10 of FS 5100-304d.

**NOTE**: Accelerated stability testing can provide false positives, resulting in a more difficult test protocol to pass. If a failure is encountered during this accelerated cycle, no interim qualification will be granted. The product shall complete the 52-week outdoor stability test to validate its use as is standard practice for 5100-304d. If a product passes the accelerated cycle, but fails the outdoor 52-week stability test, the product would fail to meet the terms of this specification. Failure of both stability (accelerated and outdoor) tests will deem the product unsuitable for use and exclude it from QPL listing.

# Figure A1. Stability Testing Scenarios<sup>1</sup>

Accelerated Stability passes; Interim Qualified on QPLAccelerated S passes; Interin Qualified on QPL1-Year Outdoor Stability passes; Fully Qualified on QPL1-Year Outdoo Stability fails; removed from Network	n Fails; No Interim Status. PL Continue testing on outdoor testing or 1-Year Outdoor Stability	Accelerated Stability Fails; No Interim Status. Continue testing on outdoor testing 1-Year Outdoor Stability fails; <i>Fails</i>
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<sup>1</sup>This figure provides a description of Stability test scenarios, and does not capture all associated testing that may change a product's status. If a product fails to meet any terms of this specification and Appendix, it will be removed from evaluation.

- 3.5.1. <u>Low-Temperature Accelerated Stability Testing</u>. A testing protocol that simulates the low temperature climate of Missoula, MT will be condensed to a less than a 60-day cycle. This will include temperature cycling in conformance with MIL-STD-810G. The annual regional averages are used to establish accelerated temperature conditions, per ASTM F1908, and a minimum of 5 freeze-thaw cycles to subject the product to chemical fatigue that would normally be endured over a 1-year outdoor test.
- 3.5.2. <u>High-Temperature Accelerated Stability Testing</u>. A testing protocol that simulates the high temperature climate of San Dimas, CA will be condensed to less than a 60-day cycle. This will include temperature cycling in conformance with MIL-STD-810G. The annual regional averages temperatures are used to establish accelerated temperature conditions, per ASTM F1908, to subject the product to chemical fatigue that would normally be endured over a 1-year outdoor test.
- 3.6. <u>Initial 90-Day Corrosion Testing</u>. The product is required to meet the initial corrosion requirements of 5100-304d Table 3, to be acceptable.
- 3.6.1. <u>Intergranular Corrosion</u>. Following the 90-day uniform corrosion test, samples will be selected and analyzed for intergranular corrosion analysis in accordance with 5100-304d, section 3.8.3.2.

- 3.7. <u>Toxicity Testing</u>. Toxicity testing is required on both mammalian and aquatic organisms in accordance with 5100-304d, section 3.5.2.
- 3.7.1. <u>Mammalian Toxicity</u>. The mammalian toxicity results for the product concentrate and the mixed product must both meet acceptance in accordance with 5100-304d, section 3.5.2.1.
- 3.7.2. <u>Aquatic Toxicity</u>. The aquatic toxicity results for the product concentrate must meet acceptance in accordance with 5100-304d, section 3.5.2.2.
- 3.8. <u>Effectiveness Testing</u>. The combustion retarding effectiveness (burn) test is the standard for this effectiveness testing. Other test methods may be used to validate initial effectiveness to that of the 10.6% DAP standard (i.e. ASTM E84, Cone calorimeter testing, etc.) but a burn test may still be required for products that do not meet section 3.6 of 5100-304d.

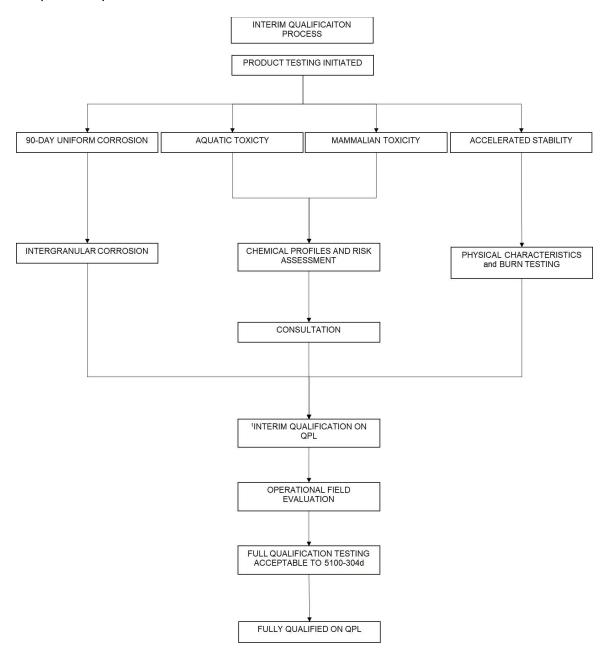


Figure A2-Interim Qualification Process with Accelerated Stability Testing. This outline summarizes the testing, more specific testing information is found in the following sections as a supplement to 5100-304d.

<sup>1</sup>Interim Qualification; Use of Fire Chemical is deemed acceptable for use in the Operational Field Evaluation stage for 1 fire season. Fire chemical product must meet full acceptance in accordance with 5100-304d by the following fire season.

- 4. **<u>TEST PROCEDURES</u>**. All test procedures are outlined in 5100-304d and its associated references.
- 5. <u>PREPARATION FOR DELIVERY</u>. See 5100-304d and the "Manufacturer Submission Procedures" documents for this direction.
- 6. <u>GENERAL INFORMATION</u>. This accelerated testing may be revised or updated to include any other screening tests necessary to better serve the field users.
- 6.1. If a product fails any required test in 5100-304d, the interim status of the product shall be revoked and the product shall be removed from the QPL.