

METRIC

MIL-PRF-25567E <u>17 November 1998</u> SUPERSEDING MIL-L-25567D 15 June 1983

PERFORMANCE SPECIFICATION

LEAK DETECTION COMPOUND, OXYGEN SYSTEMS

This specification is approved for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 <u>Scope</u>. This specification covers two types of a gas leak detection compound that is compatible with oxygen. The leak detection compound is intended for use in detecting leaks in both high- and low-pressure oxygen systems in aircraft and other related oxygen systems.

1.2 <u>Classification</u>. The compound consists of the following types, as specified (see 6.2).

Type I - for use in a temperature range of 1 degree C to 70 degrees C. Type II - for use in a temperature range of -54 degrees C to 1 degree C.

2. APPLICABLE DOCUMENTS

2.1 <u>General</u>. The documents listed in this section are specified in sections 3 and 4 of this specification. This section does not include documents cited in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements documents cited in sections 3 and 4 of this specification, whether or not they are listed.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be used in improving this document should be addressed to: Defense Supply Center Richmond (DSCR), ATTN: DSCR-VBD, 8000 Jefferson Davis Highway, Richmond, VA 23297-5610 by using the Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A

FSC 6850

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2.2 Government documents. None

2.3 <u>Non-government publications</u>. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of the documents which are DoD adopted are those listed in the issue of the DoDISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DoDISS are the issues of the documents cited in the solicitation (see 6.2).

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM B152 ASTM B209-96 ASTM D92	Copper Sheet, Strip, Plate, and Rolled Bar (DoD adopted) Aluminum and Aluminum-Alloy Sheet and Plate (DoD adopted) Test for Flash and Fire Points by Cleveland Open Cup (DoD adopted)
ASTM D1173 ASTM D1177 ASTM D1292	Test for Foaming Properties of Surface-Active Agents Test for Freezing Point of Aqueous Engine Anti-freeze Solutions (DoD adopted) Test for Odor in Water

(Application for copies should be addressed to the American Society for Testing and Materials, 100 Barr Harbor Drive, W. Conshohocken, PA 19428-2959.)

SAE INTERNATIONAL (SAE)

SAE-AMS2400	Plating, Cadmium (DoD adopted)
SAE-AMS2471	Anodic Treatment of Aluminum Alloys Sulfuric Acid Process,
	Undyed Coating (DoD adopted)
SAE-AMS5046	Sheet, Strip, and Plate, Carbon Steel (SAE 1020 and 1025)
	Annealed (DoD adopted)

(Application for copies should be addressed to the SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001.)

2.4 <u>Order of precedence</u>. In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 <u>First article</u>. When specified (see 6.2), a sample shall be subjected to first article inspection in accordance with 4.1.1.



3.2 <u>Performance requirements</u>. A compound shall satisfy the following performance requirements.

3.2.1 <u>Composition</u>. The compound shall be liquid and miscible with water in all proportions.

3.2.1.1 <u>Foreign matter</u>. The compound shall be free from any sediment or suspended matter.

3.2.1.2 <u>Ingredients not allowed</u>. The compound shall not contain (a) mineral oil, vegetable oil, animal oil, or fats or (b) ketones, aldehydes, or alcohols as components in the formulation.

3.2.1.3 <u>Igniting material</u>. The compound shall not contain any material that will ignite or explode when in contact with liquid or gaseous oxygen.

3.2.1.4 <u>Skin irritant material</u>. The compound shall not contain any material that will act as primary skin irritants, skin sensitizers, or produce any other dermatosis.

3.2.2 <u>Odor</u>. The compound shall not possess or produce any objectionable odors. The threshold odor number will be a maximum of 1 with a maximum odor intensity index of 0.

3.2.3 <u>pH Value</u>. The compound shall have a pH value between 6.0 and 7.5 at 21 degrees C.

3.2.4 <u>Nonflammability</u>. The compound shall not support combustion below 100 degrees C.

3.2.5 <u>Leak detection</u>. The compound shall be able to detect leaks.

3.2.6 <u>Foaming ability</u>. The compound shall be able to produce an initial foam height of not less than 145 mm and a foam height of 130 mm after standing for 5 minutes.

3.2.7 <u>Freezing point</u>. Type II compound shall have a freezing point of -60 degrees C or below.

3.2.8 <u>Residue</u>. The compound shall not contain more than 0.50 percent total residue.

3.2.9 <u>Corrosiveness</u>. The compound shall not be corrosive.

3.2.10 <u>Compatibility with oxygen</u>. The compound shall be compatible with oxygen.

3.2.11 <u>Mold growth</u>. The solution shall not support mold growth within the container during use or storage.

3.3 <u>Recycled, recovered, or environmentally preferable materials</u>. Recycled, recovered, or environmentally preferable materials should be used to the maximum extent possible provided the material meets or exceeds all specified requirements and promotes economically advantageous life cycle costs.



4. VERIFICATION

4.1 <u>Classification of inspections</u>. The inspection requirements specified herein are classified as first article inspection (see 4.1.1) and conformance inspection (see 4.1.2).

4.1.1 <u>First article inspection</u>. Perform a first article inspection on the initial productionrepresentative unit of an order or when required by the contract. When a first article inspection is required (see 6.2 and 6.3), it includes all verifications listed in table I.

Title	Requirement	Verification
Performance Requirements	3.2	4.2.2
Foreign matter	3.2.1.1	4.2.2.1
Odor	3.2.2	4.2.2.2
pH value	3.2.3	4.2.2.3
Nonflammability	3.2.4	4.2.2.4
Leak detection	3.2.5	4.2.2.5
Foaming ability	3.2.6	4.2.2.6
Freezing point	3.2.7	4.2.2.7
Residue	3.2.8	4.2.2.8
Corrosion	3.2.9	4.2.2.9
Compatibility with oxygen	3.2.10	4.2.2.10
Mold growth	3.2.11	4.2.2.11

TABLE I. Verification methods.

4.1.2 <u>Conformance inspection</u>. Conformance inspection shall be accomplished in accordance with the requirements and verifications in table I unless explicitly waived in the contract in accordance with 6.4.

4.2 <u>Verification methods</u>. Acceptable verification methods included in this section are visual inspection, measurement, sample tests, full-scale demonstration tests, simulation, modeling, engineering evaluation, component properties analysis, and similarity to previously-approved or previously-qualified designs.

4.2.1 <u>Verification alternatives</u>. The manufacturer may propose alternative test methods, techniques, or equipment, including the application of statistical process control, tool control, or cost-effective sampling procedures to verify performance. See the contract for alternatives that replace verifications required by this specification.

4.2.2 <u>Performance requirements verification</u>. Complete each verification in section 4.2.2.

4.2.2.1 <u>Foreign matter inspection</u>. The compound shall be examined visually to determine if it is clear and free from suspended matter and sediment.



4.2.2.2 <u>Odor test</u>. Odor shall be tested in accordance with ASTM D1292.

4.2.2.3 <u>pH value verification</u>. The compound shall be tested with an electric pH meter that has been buffered with two buffer solutions in the desired pH range and is capable of measuring pH to 0.1 point.

4.2.2.4 Nonflammability test. Nonflammability will be tested in accordance with ASTM D92.

4.2.2.5 <u>Leak detection verification</u>. Mount a laboratory screw hose clamp so that when the end of a rubber or plastic tube of 0.3175 cm is inserted, flush with the clamp jaws; that it will point upward at an angle of 45 degrees. Attach the other end of the tube to a source of compressed oxygen gas provided with a suitable gauge. Apply a pressure of 34 KPa and adjust the lead until, on application of the leak test compound, only slight foaming is observed. Immerse the leak in a shallow vessel containing the compound, and collect the escaping gas in an inverted glass tube marked in 0.1 cc divisions filled with the compound. Remove the leak from the bath and apply the leak detection compound. Immediate foaming shall be observed.

Type I shall be tested with the bath at 21 degrees C and 71 degrees C. Type II shall be tested with the bath at -51 degrees C.

4.2.2.6 <u>Foaming ability test.</u> The compound shall be tested in accordance with ASTM Method D1173 with the temperature at 49 degrees C.

4.2.2.7 Freezing point test. Type II only shall be tested in accordance with ASTM D1177.

4.2.2.8 <u>Residue verification</u>. Pipet 10 ml of the compound into a clean, dry 50 ml beaker pre-weighed to the nearest 0.1 mg. Reweigh the beaker plus compound and then heat the solution to approximately 95 degrees C on a hot plate. Allow 8 ml of the solution to evaporate; then place the beaker with the remaining solution in an oven at 110 + 1/2 degrees C and dry to constant weight. The weight of the residue shall not exceed 0.50 percent of the weight of the original 10 ml of compound.

4.2.2.9 Corrosion test. Complete each test in 4.2.2.9.

4.2.2.9.1 <u>Surfaces</u>. Two test panels (2.54 x 15.24 cm each) shall be made from each of the metals specified in table II. Each test panel will be acetone-washed in a beaker and allowed to air dry for two hours in a desiccator. Each previously cleaned test panel shall be coated with a 0.1 ml of compound over 50 percent of the surface area and the panel dried under an infrared reflector drying lamp. The reflector apparatus shall be so adjusted that a 250-watt drying lamp shall be 15 cm above the test panel. After drying, the panel shall be removed and rinsed under a stream of cold deionized water. The panels shall rinse cleanly, leaving a bright shiny surface with no residue or film noticeable by a visual examination.



4.2.2.9.2 <u>Between the faying edges</u>. Two cleaned test panels (2.54 x 15.24 cm each) from each of the metals specified in table II, shall be clamped with a suitable clamp. Place 0.1 ml of the leak detection compound at the faying edge. After one minute, an additional 0.1 ml of the leak detection compound shall be placed at the faying edge. The panels shall then be rinsed with cold deionized water and allowed to air dry for 2 hours at 25 degrees C. Then the panels shall be separated and inspected for any evidence of corrosion at the faying edge. A second set of control panels shall be used in the same manner as the test panels except that no leak test compound will be applied. The test panels shall not have any greater degree of corrosion than the control panels test results.

TABLE II. Metals for corrosion test.

Metals	Surface	
Aluminum Alloy Alclad (2024) conforming to		
ASTM B209-96 using Temper = 0		
Aluminum Alloy (2024) conforming to	Anodized, in accordance with	
ASTM B209-96 using Temper = 0	SAE-AMS2471D	
Steel, conforming to SAE-AMS5046A-90	Cadmium plated in accordance	
(using SAE 1020 and 1025)	with SAE-AMS2400S	
Copper, conforming to ASTM B152	Annealed	

4.2.2.10 <u>Compatibility with oxygen verification</u>. Under ambient conditions, place 100 ml of the compound into two each 200 ml Dewar flasks. Place a thermometer graduated in 0.1 degrees C in each flask and record the temperature. Bubble a stream of oxygen from a glass tube through one of the solutions at 100 ± 10 ml per minute. Bubble a stream of air at the same rate through the other solution. Continue for 30 ± 2 minutes. Observe any temperature rise. The leak detection compound through which the oxygen was bubbled shall not show any steady or sudden rise in temperature and the final temperature shall not exceed by 0.5 degrees C the final temperature of the compound through which the air was bubbled.

4.2.2.11 <u>Mold growth test</u>. Perform the test in Appendix A. There shall not be any mold growth viewed on the test samples with the leak detection compound. If any mold growth is seen, then the lot will be immediately rejected.

5. PACKAGING

5.1 <u>Packaging</u>. For acquisition purposes, the contract or order shall specify packaging requirements (see 6.2). When DoD personnel perform materiel packaging, those personnel need to contact the responsible packaging activity to ascertain requisite packaging requirements. The inventory control point packaging activity within the Military Department of Defense Agency, or within the Military Department's System Command, maintains packaging requirements. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.



6. NOTES

(This section contains general or explanatory information that may be helpful, but is not mandatory.)

6.1 <u>Intended use</u>. The leak detection compound covered by this specification is military unique. This specification is the industry standard for this compound. No commercial equivalent exists. This compound assures the purity of military aviators' breathing systems by prohibiting the introduction of contaminants. This assures the safety, health, and compatibility of oxygen gas with the variety of methods used in military aircraft. It also assures the stability of breathing systems during military operations (bombing, air-to-air combat evasive maneuvering, etc.). These extreme conditions are not found in commercial applications.

6.2 <u>Acquisition requirements</u>. Acquisition documents must specify the following:

- a. Title, number, and date of this specification and any amendments thereto.
- b. Type of compound (see 1.2).
- c. Size container required as applicable.
- d. Issue of DoDISS to be cited in the solicitation, and if required, the specific issue of individual documents referenced (see 2.3).
- e. When first article is required (see 3.1, 4.1.1 and 6.3).
- f. Instructions on submittal of first article samples and test reports will be cited in the contract.
- g. Packaging requirements (see 5.1).

6.3 <u>First article</u>. When requiring a first article inspection, the contracting officer should provide specific guidance to offerors whether the first article is a first article sample, a first production item, or a number of items to be tested. The contracting officer should also include specific instructions in acquisition documents regarding arrangements for examinations, approval of first article test results and disposition of first articles. Pre-solicitation documents should provide the Government with the waiver rights for samples for first article inspection to bidders offering a previously acquired or tested product. Bidders offering such products, who wish to rely on such production or test, must furnish evidence with the bid, that prior government approval is appropriate for the pending contract.

6.4 <u>Conformance inspection</u>. Affordable conformance inspection with confidence varies depending upon a number of procurement risk factors. Some of these factors include contractor past performance, government schedules and budget, product material and design maturity, manufacturing capital equipment and processes applied, the controlled uniformity of those processes, labor skill and training, and the uniformity of measuring processes and techniques. During the solicitation, contracting documents should indicate those tests desired from table I and their designated frequency based on a risk assessment for the procurement.



6.5 <u>Material safety data sheets</u>. Contractor should furnish a material safety data sheet to the procuring activity using FED-STD-313.

6.6 Subject term (key word) listing.

Corrosion Fungi Ingredients Mold growth

6.7 <u>Changes from previous issue</u>. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

Custodians:	Preparing activity:
Air Force - 68	DLA - GS
Army - AV	
Navy - AS	(Project 6850-1201)

Reviewers: Air Force - 11 Army - EA, MI



MOLD GROWTH TEST

A.1 SCOPE

A.1.1 <u>Scope</u>. This appendix details the procedure for conducting a mold growth test. This appendix is a mandatory part of the specification.

A.2 APPLICABLE DOCUMENTS. None.

A.3 MOLD RETARDANT. The formulation of the mold retardant used shall be certified as to its nomenclature, effect in an industrial environment and the non-detrimental effect on the compounds intended use.

A.4 PROCEDURE

A.4.1 <u>Preparation of mineral salts solution</u>. Using a clean apparatus, prepare a mineral salts solution containing the following, then verify the pH of the mineral salts solution is between 6.0 and 6.5.

Potassium dihydrogen orthophosphate (KH ₂ PO ₄)	0.7 g
Potassium monohydrogen orthophosphate (K ₂ HPO ₄)	0.7 g
Magnesium sulfate heptahydrate (MgSO ₄ 7 H ₂ O)	0.7 g
Ammonium nitrate (NH ₄ NO ₃)	1.0 g
Sodium chloride (NaCl)	0.005 g
Ferrous sulfate heptahydrate (FeSO ₄ 7 H ₂ O)	0.002 g
Zinc sulfate heptahydrate (ZnSO ₄ 7 H ₂ O)	0.002 g
Manganous sulfate monohydrate (MnSO ₄ H ₂ O)	0.001 g
Distilled water	1000 ml

A.4.2 <u>Preparation of mixed spore suspension</u>. Prepare a spore suspension using asceptic techniques containing the following test fungi, listed in table A-I.

<u>PRECAUTIONS</u>: Although the fungi specified for this test are not normally considered to present a serious hazard to humans, certain people may develop allergies or other reactions. It is therefore recommended that standard operation procedures (SOPs) for safety be employed. It is also recommended that the tests be conducted by personnel trained in microbiological techniques.



Fungi	Fungus Sources Identification No.	
	USDA ¹	ATCC ²
Aspergillus niger	QM 386	ATCC 9642
Aspergillus flavus	QM 380	ATCC 9643
Aspergillus versicolor	QM 432	ATCC 11730
Penicillium funiculosum	QM 474	ATCC 11797
Chaetomium globosum	QM 459	ATCC 6205

TABLE A-I. Test fungi.

¹ U.S. Department of Agriculture (SEA/FR) Northern Regional Research Center, ARS Culture Collection, 1815 N. University Street, Peoria, IL 60604

² American Type Culture Collection, 12301 Parklawn Drive, Rockville, MD 20852

A.4.2.1 <u>Spore storage</u>. Maintain pure cultures of the test fungi (see table A-I) separately on an appropriate medium such as potato dextrose agar except that chaetomium globosum shall be cultured on strips of filter paper overlayed on the surface of mineral salts agar. Prepare mineral salts agar by dissolving 15.0 g agar in a liter of the mineral salts solution. Do not keep the stock cultures for more than 4 months at 6 degrees +/- 4 degrees C; after that time, prepare sub-cultures and use them for the new stocks. Verify the purity of fungus cultures prior to the mold growth test.

A.4.2.2 <u>Stock cultures preparation</u>. Incubate subcultures used for preparing new stock cultures or the spore suspension at 30 degrees +/- 1.4 degrees C for 14 to 21 days. Prepare a spore suspension of each of the five fungi by pouring into one subculture of each fungus, 10 ml of an aqueous solution containing 0.05 g per liter of a nontoxic wetting agent such as sodium dioctyl sulfosuccinate or sodium lauryl sulfate.

A.4.2.3 <u>Stock cultures separation</u>. Use a rounded glass rod to gently scrape the surface growth from the culture of the test organism. Pour the spore charge into a 125 ml capped Erlenmeyer flask containing 45 ml of water and 50 to 75 solid glass beads, 5 mm diameter. Shake the flask vigorously to liberate the spores from the fruiting bodies and to break the spore clumps. Filter the dispersed fungal spore suspension into a flask through a 6 mm layer of glass wool contained in a glass funnel. This process should remove large mycelial fragments and clumps of agar.

A.4.2.4 <u>Stock cultures solution</u>. Centrifuge the filtered spore suspension and discard the supernatant liquid. Resuspend the residue in 50 ml of water and centrifuge. Wash the spores obtained from each of the fungi in this manner three times. Dilute the final washed residue with mineral-salts solution in such a manner that the resultant spore suspension shall contain 1,000,000 + 200,000 spores per as determined with a counting chamber. Repeat this operation for each organism used in the test.

A.4.2.5 <u>Control item testing</u>. Complete the control tests in A.4.2.5.

A.4.2.5.1 <u>Viability of spore suspension</u>. Prior to preparing the composite spore suspension, inoculate sterile potato agar plates with 0.2 to 0.3 ml of the spore suspension of each of the



individual fungal species using separate potato dextrose agar plates for each species. Distribute the inoculum over the entire surface of the plate. Incubate the inoculated potato dextrose agar plate at 24 degrees to 31 degrees C for 7 days. After the incubation period, check the fungal growth. The absense of copious growth of any of the test organisms over the entire surface in each container will invalidate the results of any tests using these spores.

A.4.2.5.2 <u>Verification of suitability of the chamber environment</u>. Inoculate a known susceptible substrate along with the test sample to insure that proper conditions are present in the incubation chamber to promote fungal growth. The controlled substrate shall consist of sterile, 100 percent cotton, untreated, and bleached white (of flat construction) fabric strips. Prepare a solution containing the following, then dip the cotton strips into the solution.

Prepared Solution:	10.0 g glycerol
	0.1 g potassium dihydrogen orthophosphate (KH ₂ PO ₄)
	0.1 g ammonium nitrate (NH_4NO_3)
	0.025 g magnesium sulfate heptahydrate (MgSO ₄ 7 H ₂ O)
	0.05 g yeast extract
	Distilled water to a total volume of 100 ml
	HCl or NaOH to adjust the final solution pH to 5.3

After dipping, remove the excess liquid from the strips and hang them to dry before placing them in the chamber and inoculating them. Within the chamber, place the strips vertically in close proximity to and bracketing the test item so that the test strips and test items experience the same test environment. The length of the strips shall be at least the height of the test item. These strips verify conditions to promote fungal growth in the chamber.

A.4.2.6 <u>Fungus test suspension</u>. Blend equal volumes of the resultant spore suspension to obtain the final mixed spore suspension. The spore suspension may be prepared fresh. If not freshly prepared, it should be held at 6 ± 4 degrees C for not more than 7 days.

A.4.3 <u>Mold growth test</u>. Complete the mold growth testing in A.4.3.

A.4.3.1 <u>Preparation of incubation</u>. Assure that the condition of the leak detection compound subjected to testing is similar to that as delivered by the manufacturer or customer for use, or as otherwise specified. Place 150 ml of the compound in a 250 ml beaker in the chamber or cabinet on suitable fixtures. Hold the test compound in the operating chamber for at least 4 hours immediately prior to inoculation. Inoculate the test compound and cotton fabric chamber control items with the mixed fungal spore suspension by spraying it on the control and on the test sample in the form of a fine mist from an atomizer or nebulizer. Care should be taken to cover all external and internal surfaces which are exposed during use or maintenance. Replace covers of the test items loosely. Start incubation immediately following the inoculation.

A.4.3.2 <u>Incubation of the test compound</u>. Incubate the test sample under a daily cycle of temperature and humidity conditions consisting of 20 hours of a relative humidity of 95 +/- 5 percent and an air temperature of 30 degrees +/- 1 degree C followed by a 4-hour period.



The 4-hour period consists of 2 hours in which conditions of 95 ± -5 percent relative humidity and temperature of 25 degrees ± -1 degree C are maintained and the other 2 hours of the 4-hour period is used for the transition(s) of temperature and relative humidity. Temperature and humidity conditions during the transition periods must be as follows: temperature = 24 degrees to 21 degrees C and relative humidity = above 90 percent.

A.4.3.3 <u>Test duration</u>. Repeat the 24-hour daily cycle for the test duration. After 7 days, inspect the growth on the control cotton strips to assure that the environmental conditions in the chamber are suitable for growth. For this assurance, at least 90 percent of the part of the surface area of each test strip located at the level of the test item should be covered by fungi when inspected visually. If not, repeat the entire test with the required adjustments of the chamber to produce conditions suitable for growth. Leave the control strips in the chamber for the duration of the test; note their condition at this time and record it. If the cotton strips show satisfactory fungus growth after 7 days, continue the test for 72 hours from the time of inoculation. If there is a decrease in fungal growth on the cotton strips at the end of the test as compared to the 7-day results, the test is invalid.

A.4.4 <u>Inspection</u>. At the end of the incubation period, inspect the test samples immediately. If possible, inspect the samples within the chamber. If the inspection is conducted outside of the chamber and not completed in 8 hours, return the test samples to the test chamber or similar humid environment for a minimum of 12 hours. There shall not be any mold growth viewed on the test samples with the leak detection compound. If any mold growth is seen, then the lot will be immediately rejected.

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