

AEROSPACE MATERIAL SPECIFICATION

Submitted for recognition as an American National Standard

Molybdenum Disulfide, Technical, Lubrication Grade

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1. SCOPE:

1.1 Scope:

This specification covers the requirements for procurement of one grade of powdered molybdenum disulfide to be used in lubricants and greases for surfaces where boundary conditions exist.

2. APPLICABLE DOCUMENTS:

The following publications, of the issues in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

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2.1 U.S. Government Publications:

Available from DODSSP Subscription Services Desk, Building 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094.

MIL-R-3043 Resin Coating, Unpigmented, For Engine Components And Metal Parts
MIL-S-7952 Steel, Sheet and Strip, Uncoated, CARBON (1020 and 1025) (Aircraft Quality) (Asg)
QQ-C-576 Copper Flat Products With Slit, Slit And Edge-Rolled, Sheared, Sawed or Machined Edges (Plate, Bar, Sheet and Strip)
TT-T-291 Thinner, Paint, Volatile Spirits (Petroleum-Spirits)

MIL-STD-105 Sampling Procedures For Inspection By Attributes
MIL-STD-290 Packaging, Packing and Marking of Petroleum And Related Products
FED-STD-313 Material Safety Data Sheets, Preparation And Submission Of
FED-STD-791 Lubricants, Liquid Fuels and Related Products; Methods of Testing

3. REQUIREMENTS:**3.1 First article inspection:**

The molybdenum disulfide furnished under this specification shall be a product which has been inspected and has passed the first article inspection specified herein. (See 4.2.1).

3.2 Materials:

The molybdenum disulfide shall be a powder of a purity and grade suitable for general lubricating use.

3.3 Chemical and physical characteristics:

The chemical and physical characteristics of the molybdenum disulfide shall be as specified in Table I.

TABLE I Chemical and Physical Characteristics

Characteristic	Limit	Applicable Paragraph
Moisture (loss of weight), percent, max.	0.7	4.4.2
Water solubles, percent, max.	0.5	4.4.3
Oil content (acetone extractable), percent, max.	0.5	4.4.4
Total insolubles, percent, max.	1.0	4.4.5
Molybdenum disulfide, percent, min.	98.0	4.4.5
Corrosion resistance 1/	Pass	4.4.6
Steel and copper corrosion 2/	Pass	4.4.7
Fineness, microns (average particle size)	4-10	4.4.8

1/ The molybdenum disulfide, when in the form of a bonded solid film, shall show or cause no discoloration, pitting, formation of white deposits, or other evidence of corrosion.

2/ The molybdenum disulfide shall cause no pitting, etching, or heavy staining on polished copper or sandblasted steel surfaces. A slight stain or discoloration shall not be cause for rejection.

3.4 Toxicity:

The molybdenum disulfide shall have no adverse effect on the health of personnel when used for its intended purpose. The powder shall contain no components which produce noxious vapors in such concentrations as to be an annoyance to personnel during formulation or use under conditions of adequate ventilation while exercising caution to avoid prolonged contact with the skin. The properties of the powder shall be such that its use shall not require personnel protective equipment, special control procedures, or special handling equipment. Questions pertaining to the toxic effects shall be referred by the procuring activity to the appropriate departmental-medical service who will act as an advisor to the procuring activity. A Material Safety Data Sheet (MSDS) shall be prepared in accordance with FED-STD-313 and be submitted either prior to or with the First Article Samples (3.1).

3.5 Workmanship:

The molybdenum disulfide, when subjected to visual examination, shall appear homogeneous.

4. QUALITY ASSURANCE PROVISIONS:

4.1 Responsibility for inspection:

Unless otherwise specified in contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any commercial laboratory acceptable to the Government. The Government reserves the right to perform any of the examinations and tests set forth in the specification where such are deemed necessary to assure supplies and services conform to prescribed requirements.

4.2 Classification of inspection:

The inspection and testing of the molybdenum disulfide shall be classified as follows:

- (a) First article inspection (4.2.1)
- (b) Quality conformance inspection (4.2.2)

4.2.1 First article inspection: The first article inspection shall consist of testing the first article sample (4.3.1) for conformance with the corrosion resistance requirement (3.3). If the results of the first article inspection are in accordance with the requirements of this specification, the consistency of production quality will thereby be verified. Failure of the molybdenum disulfide to pass a first article inspection shall require that the acceptance of a further shipment of molybdenum disulfide to be withheld until the contractor has corrected the conditions which led to the failure. Material Safety Data Sheets shall be prepared in accordance with FED-STD-313 and submitted to the cognizant laboratory (4.3.1).

4.2.2 Quality conformance (lot-by-lot) inspection: The quality conformance inspection shall include examining and testing the quality conformance samples (4.3.2.2) for conformance to all the Section 3 requirements, except corrosion resistance (3.3), and an examination of the samples of filled containers (4.3.2.3) for conformance to the Section 5 requirements.

4.3 Sampling:

4.3.1 First article sample: First article sample shall consist of 5 pounds of molybdenum disulfide selected at random from the first lot of molybdenum disulfide processed under a contract or order. First processed inspection samples shall be forwarded to the Naval Air Development Center, Code 60612, Warminster, PA 18974. The samples shall be plainly identified by securely attached durable tags or labels marked with the following information:

Sample for first article inspection
MOLYBDENUM DISULFIDE, TECHNICAL, LUBRICATION GRADE
Specification MIL-M-7866C
Name of manufacturer
Product code number
Date of manufacture
Contract or order number
Batch number

4.3.2 Quality conformance samples: The quality conformance sample shall consist of a sample for tests (4.3.2.2) and a sample for examination of filled containers (4.3.2.3). Samples shall be labeled completely with information identifying the purpose of the sample, name of product, specification number, lot and and batch number, date of sampling, and contract number. Individual samples shall not be mixed, shall be placed in separate air-tight and water-tight containers, which shall be nearly filled, covered, and sealed to prevent atmospheric effects.

4.3.2.1 Inspection lot: A lot shall consist of the material produced by one manufacturer under essentially the same manufacturing conditions. Each batch shall constitute a lot.

4.3.2.2 Sample for tests: The sample for tests shall consist of one can of molybdenum disulfide taken at random from each lot of molybdenum disulfide to be offered or processed for delivery under a contract or order. The lot shall be unacceptable if a sample fails to meet any one of the test requirements specified.

4.3.2.3 Sample for examination of filled containers: A random sample of filled containers and a sample of shipping containers fully prepared for delivery shall be selected from each lot of molybdenum disulfide in accordance with MIL-STD-105 at inspection level I and acceptable quality level (AQL)=2.5 percent defective.

4.4 Inspection methods:

4.4.1 Unless otherwise specified, the molybdenum disulfide shall be tested as taken from the container without treatment or preconditioning of any kind.

4.4.2 Moisture:

4.4.2.1 Samples: The moisture test shall be made on the first portion withdrawn from the sample container.

- 4.4.2.2 Method: A 10 gm sample shall be weighed into a previously dried and weighed flat form weighing bottle (30 by 70 mm). The bottle shall be transferred to an oven set at $107\pm 3^{\circ}\text{C}$ ($225\pm 4^{\circ}\text{F}$) to be dried for at least 16 hours. The bottle shall be removed to a desiccator to cool for one half hour, then reweighed. This shall be repeated until constant weight is attained.

$$\text{Percent moisture} = \frac{\text{loss in weight}}{\text{sample weight}} \times 100$$

- 4.4.3 Water solubles: A 10 gm sample of the dried molybdenum disulfide shall be weighed into an 80 by 33 mm single thickness thimble. The thimble shall be placed in the extraction tube of a size B Soxhlet extraction apparatus. One hundred and fifty ml of water shall be added to a previously dried and weighed 250 ml flat bottom extraction flask, and the complete extraction apparatus assembled on an electric hotplate. A blank determination shall be run simultaneously. The extraction of the powder and the blank shall be allowed to continue for 4 hours. The tared flasks shall be placed on a steam bath and evaporated to dryness. The flasks containing the residues shall be removed to an oven maintained at $107 \pm 3^{\circ}\text{C}$ ($225 \pm 5^{\circ}\text{F}$) for 1 hour, then cooled in a desiccator, and weighed.

$$\text{Percent water soluble water} = \frac{A - B}{W} \times 100$$

Where: A = Increase in weight of sample flask
B = Increase in weight of blank flask
W = Weight of dried sample

- 4.4.4 Oil content: The thimble containing the water insolubles (4.4.3.2) shall be removed to an oven maintained at $107\pm 3^{\circ}\text{C}$ ($225\pm 5^{\circ}\text{F}$) and allowed to dry overnight. A 6 gm moisture free sample shall be weighed into a tared fritted glass crucible (30 ml capacity, fine porosity). Prior to use the crucible shall be cleaned with hot chromic acid-sulfuric acid solution, washed with water, rinsed with acetone, and dried in an oven maintained at $107\pm 3^{\circ}\text{C}$ ($225\pm 5^{\circ}\text{F}$) for 1 hour. The sample shall be leached with a total of 100 ml of acetone, using 10 ml portions to cover the powder for several minutes before drawing the acetone through the filter with suction. The crucible and residue shall be dried in an oven at $107\pm 3^{\circ}\text{C}$ ($225\pm 5^{\circ}\text{F}$) for 1 hour, then removed to a desiccator for 0.5 to 1 hour before reweighing. The drying shall be repeated to constant weight.

$$\text{Percent oil content} = \frac{A}{W} \times 100$$

Where: A = loss in weight of crucible (in grams)
W = weight of moisture free sample (in grams)

- 4.4.5 Insolubles and molybdenum disulfide: The insolubles and molybdenum disulfide content shall be determined in accordance with Method 3710 of FED-STD-791.

4.4.6 Corrosion resistance of molybdenum disulfide:

4.4.6.1 Solid film formulation: The powder is incorporated into a solid film formulation by thoroughly mixing 1 part by weight of molybdenum disulfide with two parts phenolic resin conforming to MIL-R-3043 with the further addition of a mixture consisting of 50 percent c.p. methylethyl ketone, 25 percent c.p. xylene, and 25 percent c.p. N Butyl Alcohol until spraying consistency is reached.

4.4.6.2 The corrosion resistance of the molybdenum disulfide shall be determined in accordance with Method 3814 of MIL-STD-791. A spray technique with continuous agitation shall be used in film deposition. A temperature of 149°C (300°F) for 60 minutes is required for curing.

4.4.7 Steel and copper corrosion:

4.4.7.1 Panels: The steel panels shall be sandblasted SAE 1020 cold rolled steel, 2 by 4 by 1/8 inch, conforming to MIL-S-7952. The copper panels shall be 2 by 4 by 1/8 inch conforming to QQ-C-576 and shall be polished to a roughness of 3 microinches rms.

4.4.7.2 Reagent: The mineral spirits shall conform to TT-T-291. After filtering this solvent through absorbent cotton, the nonvolatile content shall be checked by evaporating a 100 ml volume to dryness in white dish over a steam bath. No residue shall remain.

4.4.7.3 Method: A slurry shall be prepared by mixing 10 grams of molybdenum disulfide with 5 ml of mineral spirits. The slurry shall be brushed onto the copper and steel panels, suspended vertically until drainage ceases, and then transferred to an oven at 102±2°C (216±4°F) for 24 hours. The panels shall be removed and immersed in boiling mineral spirits to eliminate the film of molybdenum disulfide. For complete removal of the powder, a small wad of absorbent cotton soaked with the solvent shall be lightly rubbed over the panel surface. The panels shall be cooled and dried, then examined microscopically at 15X magnification for evidence of pitting, etching, brown or black stains. Slight stains shall be considered unobjectionable. Blank panels of copper and steel shall be carried through simultaneously for comparison.

4.4.8 Fineness:

4.4.8.1 Test apparatus: The average particle size of the molybdenum disulfide shall be determined using a Fisher Sub-Sieve Sizer or equivalent. This instrument is composed of a regulating device, a precision bore sample tube, a standardized double range air flowmeter, a calculator chart, and a calibrator.