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3 August 1983
SUPERSEDING
MIL-I-13857A(MR)
20 June 1966

MILITARY SPECIFICATION

IMPREGNATION OF METAL CASTINGS

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

1. SCOPE

1.1 Scope. This specification covers the requirements for the impregnation of castings which may contain micropores but not visible blow holes or other major defects.

1.2 Classification. The impregnation processes covered by this specification shall be of the type as specified.

Type I - For use on castings for fire control instruments
Type II - For use on castings for mortar shell

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications, standards, and handbooks. Unless otherwise specified, the following specifications, standards, and handbooks of the issue listed in that issue of the Department of Defense Index of Specifications and Standards (DoDISS) specified in the solicitation form a part of this specification to the extent specified herein.

SPECIFICATIONS

FEDERAL

O-S-809 - Sulfuric Acid, Technical
P-D-680 - Dry Cleaning Solvent
QQ-A-601 - Aluminum Alloy Sand Castings
QQ-W-343 - Wire, Electrical and Nonelectrical, Copper (Uninsulated)

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Director, US Army Materials and Mechanics Research Center, ATTN: DRXMR-SMS, Watertown, MA 02172 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

NO DELIVERABLE DATA REQUIRED BY THIS DOCUMENT

/ AREA MFFP /

MILITARY

MIL-E-463 - Ethyl Alcohol (for Ordnance Use)
MIL-G-5572 - Gasoline, Aviation: Grades, 80/87, 100/130, 115/145
MIL-H-6083 - Hydraulic Fluid, Petroleum Base, for Preservation and Testing
MIL-G-23827 - Grease, Aircraft and Instrument, Gear and Actuator Screw

STANDARDS

FEDERAL

Fed. Test Method Std. No. 141 - Paint, Varnish, Lacquer, and Related Materials, Methods of Inspection, Sampling and Testing

MILITARY

MIL-STD-286 - Propellants, Solid; Sampling, Examination and Testing

(Copies of specifications, standards, handbooks, drawings, and publications required by manufacturers in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting officer.)

2.2 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein, the text of this specification shall take precedence.

3. REQUIREMENTS

3.1 Material. The impregnants used in the process shall produce a thermoset resin when cured and shall cure without the formation of voids (see 6.1.5).

3.2 Shrinkage. The impregnant shall have a maximum shrinkage of ten percent on curing when tested as specified in 4.4.1.

3.3 Inhibitive effect of copper (type I only). The resin for type I impregnant shall cure to a hard firm mass when polymerized in the presence of copper in accordance with 4.4.2. The copper shall exhibit no greenish discoloration.

3.4 Reactivity (type II only). The reactivity of the type II impregnant with either TNT or Composition B explosive shall not exceed 2.00 ml of gas when tested as specified in 4.4.3.

3.5 Diffusion rate after impregnation.

3.5.1 Before exposure. Test disks when prepared and tested in accordance with 4.4.4 shall show a diffusion rate not greater than 10^{-7} cubic feet per square inch per hour.

3.5.2 After exposure. The impregnated disks after exposure as specified in 4.4.5 shall show a diffusion rate not greater than 10^{-7} cubic feet per square inch per hour when tested as outlined in 4.4.4.

3.6 Exudation. There shall be no observable exudation of impregnants from any surface during the curing operation.

3.7 Workmanship. Quality of workmanship shall be such that impregnation will meet the requirements for quality as specified in 3.2 to 3.6, inclusive. The completed impregnation shall meet the inspection and test requirements for quality when tested as in 4.4.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or purchase order, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.2 Sampling.

4.2.1 Sampling and inspection of the impregnant. Sampling and inspection of the impregnant shall be performed in accordance with method 1031 of Fed. Test Method Std. No. 141. Testing shall be performed in accordance with the applicable procedures of 4.4.

4.2.2 Sampling for testing of the impregnating process. The acceptance test sample shall consist of twelve impregnated disks for each lot of castings impregnated (see figure 3). The disks shall possess initial diffusion rates varying between 10^{-2} and 10^{-5} cubic feet per square inch per hour and spaced as specified in 4.4.4.2 and shall be impregnated at the same time under the same conditions as the lot of castings it represents. A lot shall be defined as not more than 12 hours production. For the type II impregnating process a 50 gram sample of impregnated chips is required in addition to the twelve disks. These chips shall be of a size to pass through a No. 10 mesh sieve and be retained on a No. 14 mesh sieve.

4.2.2.1 At the option of the contracting officer, the diffusion rate test may be performed on impregnated castings from the lot in place of the sample disks.

4.3 Classification of tests. Testing under this specification shall be for acceptance of individual lots. Acceptance tests shall normally consist of tests for all requirements specified in section 3.

4.4 Test methods.

4.4.1 Shrinkage test. A 10 ml. graduated cylinder, or a premarked test tube shall be filled to the mark with impregnant and cured under the conditions recommended for the impregnant by the manufacturer. After cooling to room temperature, the cylinder shall be refilled with water containing a suitable wetting agent from a burette or a pipette. The percent of shrinkage shall be calculated from the formula:

$$\text{Percent shrinkage} = 10V$$

where V = volume of water added in ml.

4.4.2 Inhibitive effect of copper (type I only). One gram of the impregnant shall be placed in a small watch glass containing two one-half inch strands of AWG #18 copper wire conforming to QQ-W-343. The impregnant shall be cured under the conditions recommended by the manufacturer. Examination after curing shall be done by pressing the mass with the fingers and noting of the mass is solid and firm. If the impregnant is tacky or compressible, or the copper wire shows a greenish color the impregnant shall be considered as failing the test and the batch shall be rejected.

4.4.3 Reactivity (type II only).

4.4.3.1 Test method. The test for reactivity of the type II impregnant with explosives shall be conducted in accordance with method 403.1.2 of MIL-STD-286. The test shall be conducted at 100°C using a solution of glycerin and water for the constant temperature bath. Specimens having the following compositions shall be tested:

(a) 2.5 grams of type II impregnated metal chips (see 4.2.2).
(b) 2.5 grams of TNT (see 6.2).
(c) 2.5 grams of Composition B explosive (reduced to a practicable fineness under gentle pressure) (see 6.2).
(d) 2.5 grams of type II impregnated metal chips mixed with 2.5 grams of TNT.
(e) 2.5 grams of type II impregnated metal chips mixed with 2.5 grams of Composition B explosive.

4.4.3.2 Calculation. The unit capacity of the capillary and the volume of gas liberated during the test shall be calculated as specified in method 403.1.2 of MIL-STD-286. The reactivity of the impregnant with explosives shall be calculated as follows:

Reactivity, in ml. of gas = X - (Y+Z)

X = ml. of gas produced by the mixture of 2.5/2.5 grams of type II impregnant and explosive
Y = ml. of gas produced by 2.5 grams of impregnant alone
Z = ml. of gas produced by 2.5 grams of explosive alone

4.4.4 Diffusion rate.

4.4.4.1 Test equipment. The diffusion test equipment shall be as shown in figures 1 and 2. All stopcocks shall have leak proof seals. High vacuum rubber tubing shall be used where required and shall be kept to minimum length. Where rubber tubing is used, it shall be butyl rubber. Care shall be taken that it shall be heavy enough to withstand the pressure. All glass to rubber tubing shall be made airtight by using a sealing compound. The system shall be as leak proof as possible. The clamping device for the disk shall be provided with two rubber gaskets to insure leak proofness.

4.4.4.2 Test disks. The disks shall be aluminum castings, class B, of QQ-A-601, with dimensions as shown in figure 3. They shall have an initial thickness of 5/32 inch but the top and bottom face surfaces shall be machined (approximately 1/32 inch). The impregnation shall be performed on nine disks with initial leakage rates between 5×10^{-2} and 5×10^{-3} cubic feet per square inch per hour and three disks having widely spaced initial leakage rates between 10^{-3} and 10^{-5} cubic feet per square inch per hour. With these last three disks the leakage rate ratio between successive disks shall be not less than 6 nor more than 10.

4.4.4.3 Diffusion rate of system. To measure the diffusion rate of the system the following procedure shall be followed: (see figs. 1 and 2).

(a) Fasten the plate firmly between the flanges with the gaskets in place.
(b) Open stopcocks #2 and #10.
(c) Close stopcocks #1, #3, #4, #5, #6, #7, #8, and #9.
(d) Start pump and reduce pressure to about 100 microns.
(e) Carefully open stopcocks #3, #4, #5, #6, #7, and #8. (Stopcock #5 should be opened from plate to pump.) (Close stopcock #10.)
(f) Evacuate the system to 10 microns or less.
(g) Close stopcocks #4, #5, #6, #7 and #8.
(h) Continue evacuating the system to 10 microns or less.
(i) Record pressure reading (zero time reading) using the 0-50 micron scale McLeod gauge and return the gauge to the equalizing position.
(j) Simultaneously close stopcock #2 and start timing clock and take pressure readings

(k) Tilt the 0-50 micron scale McLeod gauge to the reading position at 3, 6, 9, 12 and 15 minutes, read, record and return gauge to equalizing position.
(l) From the above recorded data, the rate of change of pressure (R_1) in microns per minute may be computed by dividing the change in pressure (15 minutes reading minus zero reading) by the time.
(m) The diffusion rate of the system shall not be greater than one micron per minute except in the case of the plate being tested having a high diffusion rate, and in no case shall the diffusion rate of the system exceed the diffusion rate of the plate.

4.4.4.4 Diffusion rate of system and plate. To measure the diffusion rate of the system and plate, the procedure shall be as follows:

(a) After completing the measurement of the diffusion rate of the system (see 4.4.4.3), open stopcock #2 connecting the vacuum pump to the system and readjusting the pressure to 10 microns or less.
(b) Open stopcocks #9 and #5 to the helium side, admitting helium to the high pressure side of the system.
(c) Adjust pressure control valve on the helium tank until the absolute pressure is 30 inches of mercury.
(d) Select proper volume (see 4.4.4.2).
(e) Allow at least 15 minutes for the diffusion rate of the helium through the plate to reach equilibrium, making frequent readings.
(f) When the system has reached equilibrium, record this initial or zero time reading and return gauge to equalizing position.
(g) Simultaneously close stopcock #2 and start timing clock.
(h) Read and record the pressure at the end of 3, 6, 9, 12 and 15 minutes. (In the case of a plate with large diffusion rate, it is sometimes necessary to choose a smaller time interval since the pressure will rise rapidly.)
(i) The diffusion rate of the system and plate (R_2) may be computed from the above recorded data in the manner prescribed by step (l), paragraph 4.4.4.3.

4.4.4.5 Diffusion rate of the plate. The diffusion rate of the plate is computed as the difference between the diffusion rate of the system and plate and the diffusion rate of the system.

Diffusion rate in cubic feet per square inch per hour =

where V = volume of calibrated system in cubic feet as

V_1 = volume of calibrated system when all stopcocks except #3 are closed.

V_2 = volume of calibrated system when all stopcocks except #3 and #7 are closed.

V_3 = volume of calibrated system when all stopcocks except #3, #7 and #8 are closed.

R_1 = rate of change of pressure of system in microns per minute.

R_2 = rate of change of pressure of system and plate in microns per minute.

A = effective area of plate in square inches.

Note. When a number of plates are to be tested, the following procedure shall be used in changing plates at the end of a test:

(a) Close stopcocks #2, #3, #4 and #5. The pump is kept in operation.
(b) Close valve on helium tank.
(c) Change plates.
(d) Open stopcocks #4 and #5 from plate to pump.
(e) Evaluate the system for ten minutes.
(f) Open stopcocks #2 and #3, close #5.
(g) Continue with (h) in paragraph 4.4.4.3.

When it is desired to shut down the apparatus, close stopcocks #2, #3, #4, #5 and #6. Open stopcock #1 to the atmosphere and turn off pump. Shut off supply of helium.

4.4.5 Exposure test. Using eight of the nine impregnated disks referred to in 4.4.4.2 which possess initial diffusion rates of between 5×10^{-2} and 5×10^{-3} cubic feet per square inch per hour, subject these disks to the following exposures, one disk per exposure.

(a) Diester grease. A disk shall be immersed in diester grease, MIL-G-23827, for 48 hours at room temperature.
(b) Hydraulic oil. A disk shall be immersed in hydraulic oil, MIL-H-6083, for 48 hours at room temperature.
(c) Sulfuric acid. A disk shall be immersed in an aqueous solution containing 18 percent by volume of sulfuric acid (O-S-809) at room temperature for two hours.
(d) Solvent. A disk shall be immersed in Stoddard Solvent, P-D-680, for 48 hours at room temperature.
(e) Fuel. A disk shall be immersed in aircraft fuel, MIL-G-5572, for 48 hours at room temperature.
(f) Alcohol. A disk shall be immersed in ethyl alcohol, MIL-E-463, for 48 hours at room temperature.
(g) Heat resistance. A disk shall be exposed to 250°F for one week.
(h) Cyclic exposure. A disk shall be exposed to the following cycle:

(1) Approximately 100 percent relative humidity at 100°F for 24 hours and cool to room temperature.
(2) 160°F for 24 hours and cool to room temperature.
(3) -65°F for 25 hours and warm to room temperature.

(i) After each of the exposures the disks shall be cleaned in a suitable manner and subjected to the diffusion rate test in accordance with 4.4.4.

5. PACKAGING

5.1 Packaging requirements. Not applicable to this specification.

6. NOTES

6.1 It is considered that a suitable impregnation process includes the following steps.

6.1.1 Preparation of castings for impregnation. Castings should be thoroughly cleaned to insure satisfactory adhesion. All machining shall be completed prior to impregnation. Castings should be thoroughly dried.

6.1.2 Welding. Castings should not be treated until all welding operations have been completed.

6.1.3 Heating. If heating of castings prior to impregnation is desirable and practical, dry heat should be used.

6.1.4 Chemical treatment. Unless otherwise specified, chrome pickling, dichromate treatments, and chromic acid anodizing should be accomplished prior to impregnation; however, other treatments should be accomplished after impregnation.

6.1.5 Impregnation. Acceptable impregnation should be accomplished with not more than two cycles of vacuum and pressure, with each cycle being followed by the rinsing and curing phases. Some of the impregnants complying with MIL-I-6869 may be suitable for use in this impregnating process.

6.1.6 Rinsing. Unless otherwise specifically stated in applicable drawings, specifications or directives, the castings should be well drained and the surface rinsed free of impregnating material with a suitable solvent or cleaning agent prior to the curing process.

6.1.7 Curing. The castings, after draining and rinsing, should be heated for a suitable time at the proper temperature to insure complete setting of the impregnating compound. After heating, the castings shall be removed from the oven and allowed to cool in air to room temperature. The manufacturer's recommended curing cycle should be followed. Surfaces of cured, impregnated castings should show no evidence of impregnant.

6.2 TNT and Composition B explosives can be obtained from the following:

duPont de Nemours Co. Inc.
Wilmington, Delaware

Eastman Kodak
Rochester, New York

Custodians:
Army - MR
Navy - SH
Air Force - 11

Preparing activity:
Army - MR

Project No. MFFP-0154

Review Activities:
Army - AR, MI, MD

User activity:
Army - ME

(KBWP# ID-0518A/DISK 0110A. FOR AMMRC USE ONLY)

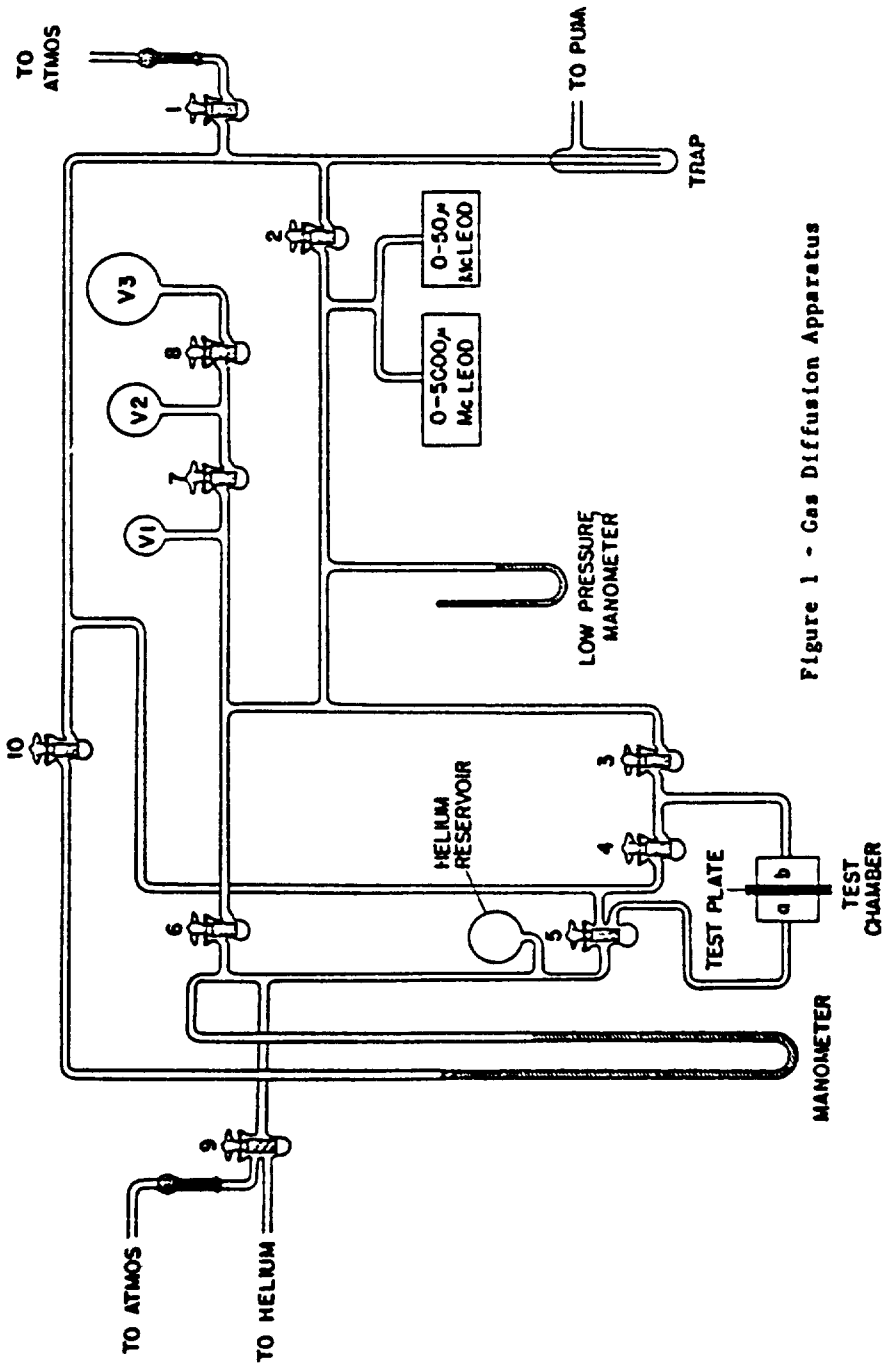

Figure 1 - Gas Diffusion Apparatus

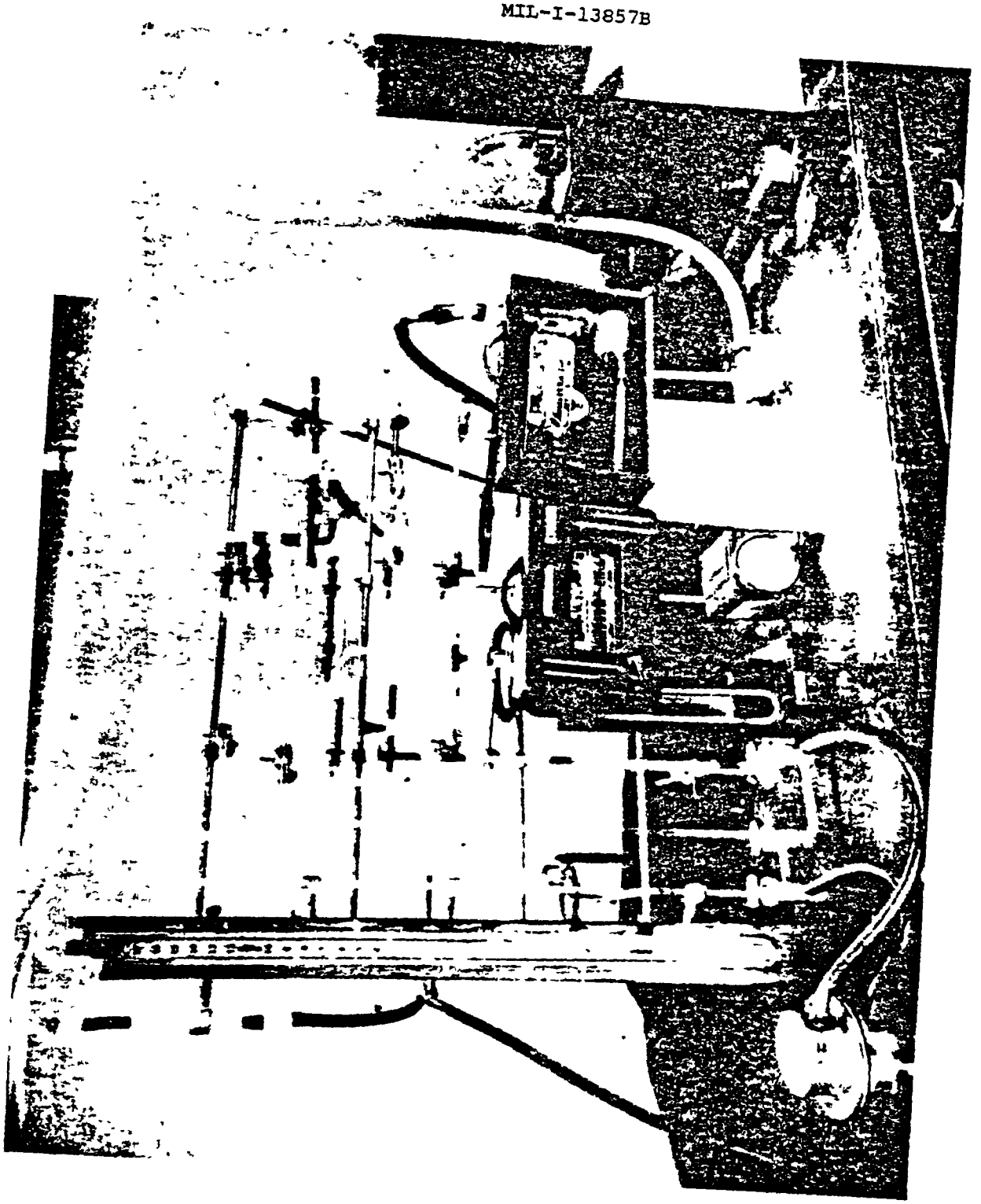

Figure 2 - Gas Diffusion Apparatus

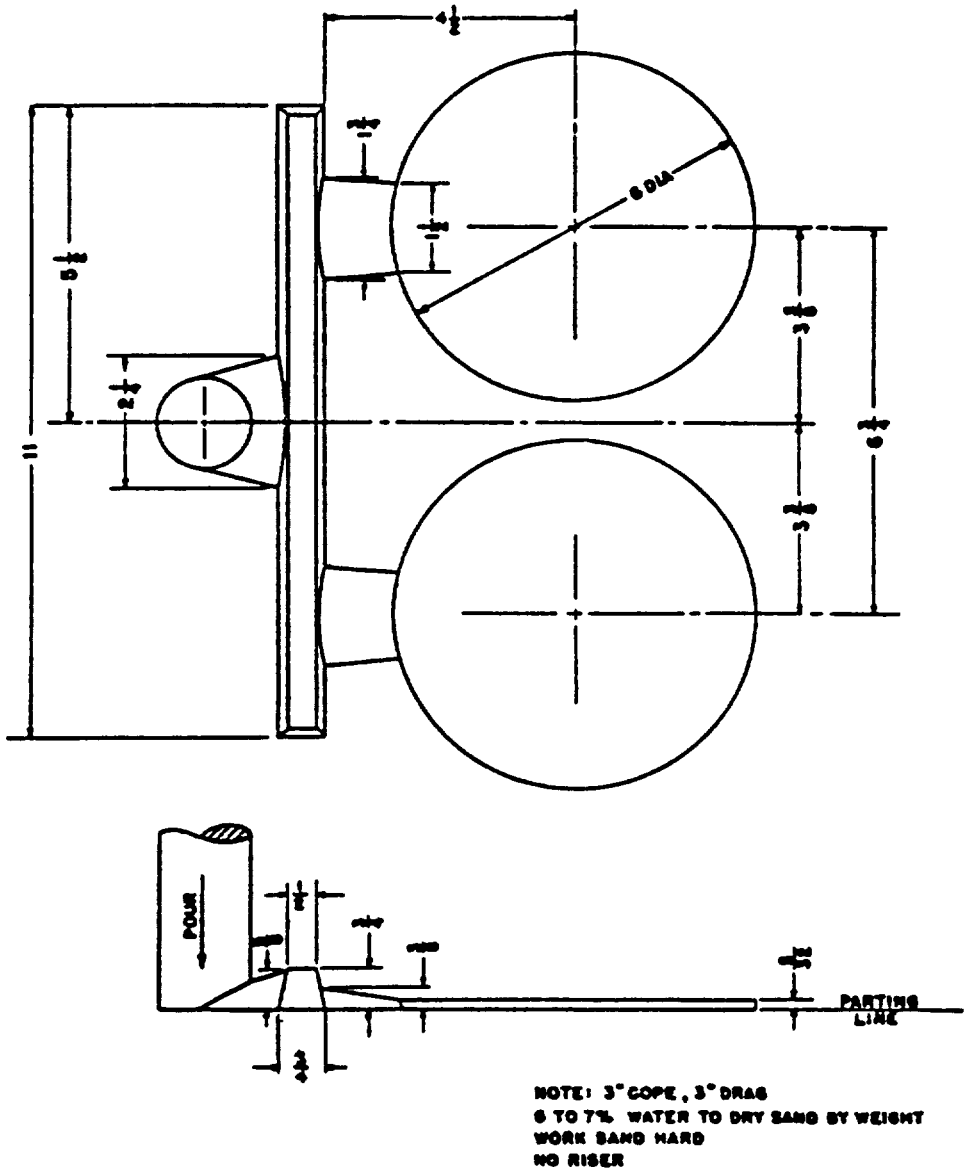

Figure 3 - Pattern Used in Casting Aluminum Discs

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	☐ MANUFACTURER
	☐ OTHER *(Specify)*. _____

5. PROBLEM AREAS

a. Paragraph Number and Wording

b. Recommended Wording

c. Reason/Rationale for Recommendation

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