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REPORT NO. NADC-85084-60



**MODIFICATION OF FEDERAL TEST METHOD STANDARD
NO. 791C METHOD 5331.1 "CORROSION PROTECTION
OF STEEL AGAINST SULFUROUS ACID-SALT SPRAY
BY SOLID FILM LUBRICANTS"**

AD-A222 410

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FEBRUARY 27, 1985

FINAL REPORT
AIRTASK No. A530-5204/001-2/3201-000-001
TASK AREA No. 201000001A
Work Unit No. AE 140

Approved for Public Release; Distribution Unlimited

Prepared for
NAVAL AIR SYSTEMS COMMAND
Department of the Navy
Washington, DC 20361

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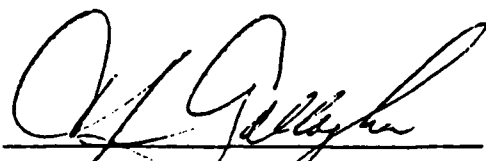
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SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

1a REPORT SECURITY CLASSIFICATION N/A		1b RESTRICTIVE MARKINGS	
2a SECURITY CLASSIFICATION AUTHORITY N/A		3 DISTRIBUTION AVAILABILITY OF REPORT Approved for public release; distribution unlimited.	
2b DECLASSIFICATION/DOWNGRADING SCHEDULE N/A			
4 PERFORMING ORGANIZATION REPORT NUMBER(S) NADC-85084-60		5 MONITORING ORGANIZATION REPORT NUMBER(S) N/A	

6a NAME OF PERFORMING ORGANIZATION NAVAL AIR DEVELOPMENT CENTER	6b OFFICE SYMBOL (if applicable)	7a NAME OF MONITORING ORGANIZATION N/A	
6c ADDRESS (City, State, and ZIP Code) WARMINSTER, PA 18974-5000		7b ADDRESS (City, State, and ZIP Code) N/A	

8a NAME OF FUNDING SPONSORING ORGANIZATION N/A	8b OFFICE SYMBOL (if applicable)	9 PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER N/A	
8c ADDRESS (City, State, and ZIP Code) N/A		10 SOURCE OF FUNDING NUMBERS	
PROGRAM ELEMENT NO	PROJECT NO	TASK NO	WORK UNIT ACCESSION NO
N/A	20100000/A	A530-5204/001-27	AE140

11 TITLE (Include Security Classification)
Modification of Federal Test Method Standard 791C Method 5331.1 "Corrosion Protection of Steel Against Sulfurous Acid-Salt Spray by Solid Film Lubricants."

12 PERSONAL AUTHOR(S)
Alfeo A. Conte

13a TYPE OF REPORT Final	13b TIME COVERED FROM TO N/A TO N/A	14 DATE OF REPORT (Year, Month, Day) February 27, 1985	15 PAGE COUNT 13
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16 SUPPLEMENTARY NOTATION
N/A

17 COSATI CODES			18 SUBJECT TERMS (Continue on reverse if necessary and identify by block number) Corrosion Test, Solid Film Lubricants, MIL-L-8937, MIL-L-23398, Materials, Sulphur Dioxide, Steel, salt spray tests
FIELD	GROUP	SUB-GROUP	
N/A	N/A	N/A	

19 ABSTRACT (Continue on reverse if necessary and identify by block number)

A deficiency was uncovered in Federal Test Standard No. 791C Method 5331.1 "Corrosion Protection of Steel Against Sulfurous Acid-Salt Spray By Solid Film Lubricants". It was determined that the potency of the sulfurous acid-salt spray rapidly diminished after the initial test cycle and thus was not consistent throughout the test procedure. A modification of this test procedure was established in order to insure consistent exposure of solid film lubricant-coated test panels. This involved the use of freshly prepared sulfurous acid-salt spray solution after each test cycle and the redefinition of the test cycle as two hours salt spray exposure and twenty-two hours drying period. Solid film lubricants conforming to MIL-L-8937 and MIL-L-23398 were evaluated using this new test procedure and found to meet the four cycle specification requirement with no evidence of visible corrosion, pitting or staining.

20 DISTRIBUTION AVAILABILITY OF ABSTRACT <input type="checkbox"/> UNCLASSIFIED/UNLIMITED <input checked="" type="checkbox"/> SAME AS RPT <input type="checkbox"/> DTIC USERS		21 ABSTRACT SECURITY CLASSIFICATION	
22a NAME OF RESPONSIBLE INDIVIDUAL Alfeo A. Conte, Jr.		22b TELEPHONE (Include Area Code) 441-2835	22c OFFICE SYMBOL 6061

TABLE OF CONTENTS

	<u>Page No.</u>
INTRODUCTION	1
BACKGROUND	1
PROCEDURE	1
APPROACH AND RESULTS	2
RECOMMENDATION	2
APPENDIX A – PROPOSED TEST METHOD	A-1

LIST OF TABLES

Table No.

1	Stability of Sulfurous Acid	3
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LIST OF FIGURES

Figure No.

1	Corrosion of AISI-1010 Disks Under Various Bath conditions	4
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INTRODUCTION

Federal Test Method Standard No. 791C, Method 5331.1 "Corrosion Protection of Steel Against Sulfurous Acid-Salt Spray by Solid Film Lubricants" is a test method requirement in solid film lubricant specifications MIL-L-8937 and MIL L-23398. Solid film lubricant-coated steel disks are exposed to a sulfurous acid-salt spray in a cyclic fashion, one cycle consisting of two hours exposure to the salt spray and a minimum two hour drying period. In a normal work day, two test cycles can be completed. The coated disks must withstand four cycles of exposure with no evidence of visible corrosion, pitting, or staining, in order to meet specification requirements.

A deficiency in this method was uncovered in that the potency of the sulfurous acid-salt spray solution rapidly diminished after the initial test cycle. This report describes modifications to the test method designed to provide a more consistent and meaningful procedure. This work was performed under AIRTASK NO. A530-5204/001-2/3201-000-001 "Production Engineering Support for Aircraft Materials/Processes".

BACKGROUND

It is common knowledge in the chemical community that sulfurous acid, which is prepared by solvating sulfur dioxide in water, is a relatively unstable material. This is substantiated by the data presented in Table 1. One hundred mL of sulfurous acid was placed in a 400 mL beaker in a laboratory environment. The concentration of sulfur dioxide was determined at various time intervals by titrating 2 mL samples with standardized sodium hydroxide. The results in Table 1 show that for the first three hours of exposure only about 10% of the original SO₂ concentration is lost; however, after 24 hours approximately 85% is dissipated.

PROCEDURE

In this test procedure, the sulfurous acid-salt spray solution is atomized to create a mist which condenses on the test disk. This procedure could accelerate the rate of sulfurous acid decomposition. Because of the low concentration of sulfurous acid present in the salt solution (2 mL of sulfurous acid per 900 mL of synthetic sea water containing 41.95 g of synthetic sea salt per liter of distilled water), detection by normal titration techniques would be impossible. An alternate approach centered on determining the potency of the sulfurous acid-salt spray bath, qualitatively, using uncoated AISI-1010 steel disks. Figure 1 shows the results of the study. Four series of tests were performed under the following spray conditions:

1. Distilled water only.
2. 2 mL of sulfurous acid per 900 mL of distilled water.
3. Salt solution only (41.95 g synthetic sea salt per liter of distilled water).
4. 2 mL sulfurous acid per 900 mL of synthetic sea water containing 41.95 g synthetic sea salt per liter of distilled water.

Differences in the corrosion tendency of each of the four spray mediums are readily apparent in Figure 1. With only distilled water, a few isolated corrosion spots can be observed even after three cycles of exposure. Adding sulfurous acid severely corrodes the disk after only one cycle. Replacing this disk with a fresh one for the second cycle on the bath shows that the potency of the spray is significantly reduced. For the third and fourth cycles which were performed on the following day, the fresh disks exhibited only a few isolated corrosion spots.

With only salt solution, corrosion is observed after the first cycle although it is not as severe as that with sulfurous acid in distilled water. The corrosion can be observed to progress with the increase in number of exposure cycles.

The final series of tests used sulfurous acid in combination with salt solution which represents the currently used spray medium. As the test progresses, the corrosion potency of the bath decreases as shown by inserting fresh disks after each cycle.

These test results clearly show that dissipation of sulfurous acid occurs in the first test cycle and that continuation of the test to the four cycle specification requirement is meaningless.

The need for a more consistent test method is therefore readily apparent. This report summarizes the modifications introduced in order to satisfy this need.

APPROACH AND RESULTS

The approach taken toward modifying the sulfurous acid-salt spray test, in order to provide a consistent test method, included the following:

<u>Modification</u>	<u>Rationale</u>
Redefine test cycle as 2 hours exposure to sulfurous acid-salt spray and a 22 hour drying period	Provide same time interval between successive cycles
Use freshly prepared sulfurous acid-salt spray solution after each cycle	To maintain potency of solution throughout test sequence

Solid film lubricants listed on the Qualified Products List (QPL) MIL-L-8937 and QPL, MIL-L-23398 were evaluated using the procedure found in Appendix A. All of the products tested met the requirement of four cycles exposure with no visible signs of corrosion, pitting, or staining.

RECOMMENDATION

It is recommended that Federal Test Method No. 791C Method 5331.1 "Corrosion Protection of Steel Against Sulfurous Acid-Salt Spray by Solid Film Lubricants" be revised in accordance with the proposed test method found in Appendix A.

TABLE 1
STABILITY OF SULFUROUS ACID

Sample Size: 100 mL

Beaker Size: 400 mL

Temperature: $25 \pm 3^{\circ}\text{C}$

Relative Humidity: $50 \pm 5\%$

Initial SO_2 Concentration: 6.07 wt %

<u>Open Beaker Exposure Time (hrs)</u>	<u>SO_2 Concentration (wt %)</u>
0.0	6.07
0.25	6.07
0.50	6.06
1.0	5.96
2.0	5.75
3.0	5.51
5.0	3.86
24.0	0.97

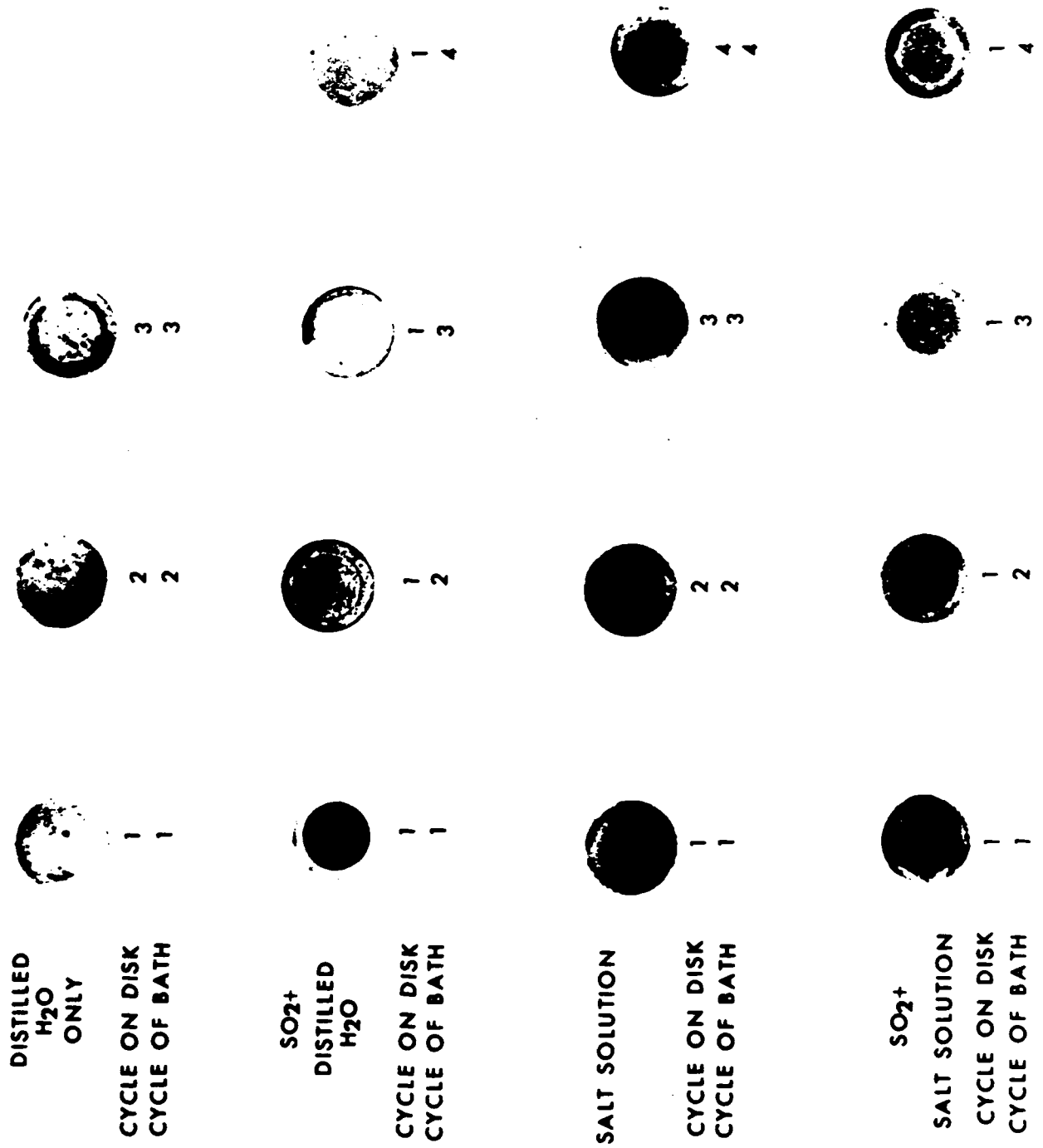


Figure 1. Corrosion of AISI-1010 Disks Under Various Bath Conditions

APPENDIX A

PROPOSED TEST METHOD

CORROSION PROTECTION OF STEEL AGAINST SULFUROUS ACID-

SALT SPRAY BY SOLID FILM LUBRICANTS

1. SCOPE

1.1 This method is used to evaluate the corrosion preventive properties of dry, solid film lubricant under the combined conditions of salt, sulfurous acid and humidity environment.

2. SUMMARY

2.1 A steel disk having solid film lubricant deposited on one surface is subjected to the cycling effect of salt spray and drying. After each cycle of salt spray and drying, the disks are examined for evidence of corrosion.

3. SAMPLE SIZE

3.1 180 mL of solid-film lubricant of a sprayable consistency.

4. REFERENCES, STANDARDS AND APPARATUS

4.1 Air Supply – filtered, 70 to 100 kPa.

4.2 Air flow regulating – capable of regulation air flow at 472 cm³/s.

4.3 Spray Nozzle (Figure A-2)-corrosion resistant with a 2.7 cm diameter acrylic baffle which prevents direct impingement of the spray, but allows a fine mist full contact with the disks.

4.4 Turntable Assembly which consists of:

a. Turntable – corrosion resistant, 26.7 cm diameter with holes for mounting test holders and specimens.

b. Driving Mechanism – capable of mechanically rotating turntable at 1/3 RPM by an electric motor (slow speed), encased within a protective acrylic box and mounted above jar on an arrangement of flexaframe support rods.

4.5 Specimen Holders – holder cap (Figure A-3) and ice jacket (aluminum) that is so placed as to be in direct contact with back of test specimen.

4.6 Jar – 30.5 cm I.D. and 30.5 cm high.

4.7 Support Rods – flexaframe or equivalent.

FED. TEST METHOD STD. NO. 791C

METHOD 5331.1

4.8 Flexible Tygon Tubing – 0.96 cm I.D.

5. MATERIALS

5.1 Steel Specimen – disk with 5.4 cm diameter and thickness of 0.16 cm, made of carbon steel conforming to FS 1010 with a 0.08 cm hole for handling disk with wire.

5.2 Synthetic Sea Water-Sulfurous Acid Test Solution – Synthetic sea water (Formula ASTM D1141 – 41.95 grams/liter, 900 mL with 2 mL sulfurous acid (not less than 6.0% assay as SO₂).

5.3 Aluminum Oxide Cloth – 240 grit.

5.4 1, 1, 1-Trichloroethane (MIL-T-81533).

5.5 Distilled Water (ASTM D1193 – Type III).

6. PROCEDURES

6.1 Prepare test specimen as follows:

- a. Degrease test specimens in 1, 1, 1-trichloroethane (MIL-T-81533).
- b. Hand polish surface to be tested with 240 grit aluminum oxide cloth to a surface finish of (0.25 to 0.50 μ m).
- c. Repeat Step a.
- d. Allow specimens to dry and store in a dust-free container.
- e. Apply solid-film lubricant to surface of steel specimen by spraying to produce a film thickness between 0.0005 cm – 0.0013 cm and cure film in accordance with specification requirements.
- f. Handle the specimen at all times with a hook.

6.2 Prepare the apparatus (Figure A-1) as follows:

- a. Full bottom 5cm of jar with the synthetic sea water-sulfurous acid solution and then lower the turntable assembly into jar.
- b. Place disk in specimen holder (Figure A-3) with ice jacket on top.
- c. Mount the complete specimen assembly in one of the holes of the turntable and start the turntable.
- d. Turn on the air and regulate rate of flow to 472 cm³/s.
- e. Two minutes after the spray has started, place ice in ice jackets in order to cause a fine condensate to form on the surface of disks.

6.3 Perform test as follows:

- a. Continue spraying for two hours with a rate of flow of approximately 0.4 mL/s.
- b. Discontinue the spray at the end of two hours; remove the ice jackets; and raise the turntable above the jar to allow specimens to dry for twenty-two hours at room temperature. The two-hour spray and twenty-two-hour drying period shall constitute one cycle.
- c. Rinse the test specimens after each full cycle, while still retained in the holder, with distilled water (ASTM D1193 – Type III) and dry with a stream of air.
- d. After each cycle examine test specimen for evidence of visible corrosion, pitting or staining and replace synthetic sea water-sulfurous acid solution with a fresh batch.
- e. Repeat the test cycle until failure occurs. Failure is defined as the first visible sign of corrosion, pitting or staining.

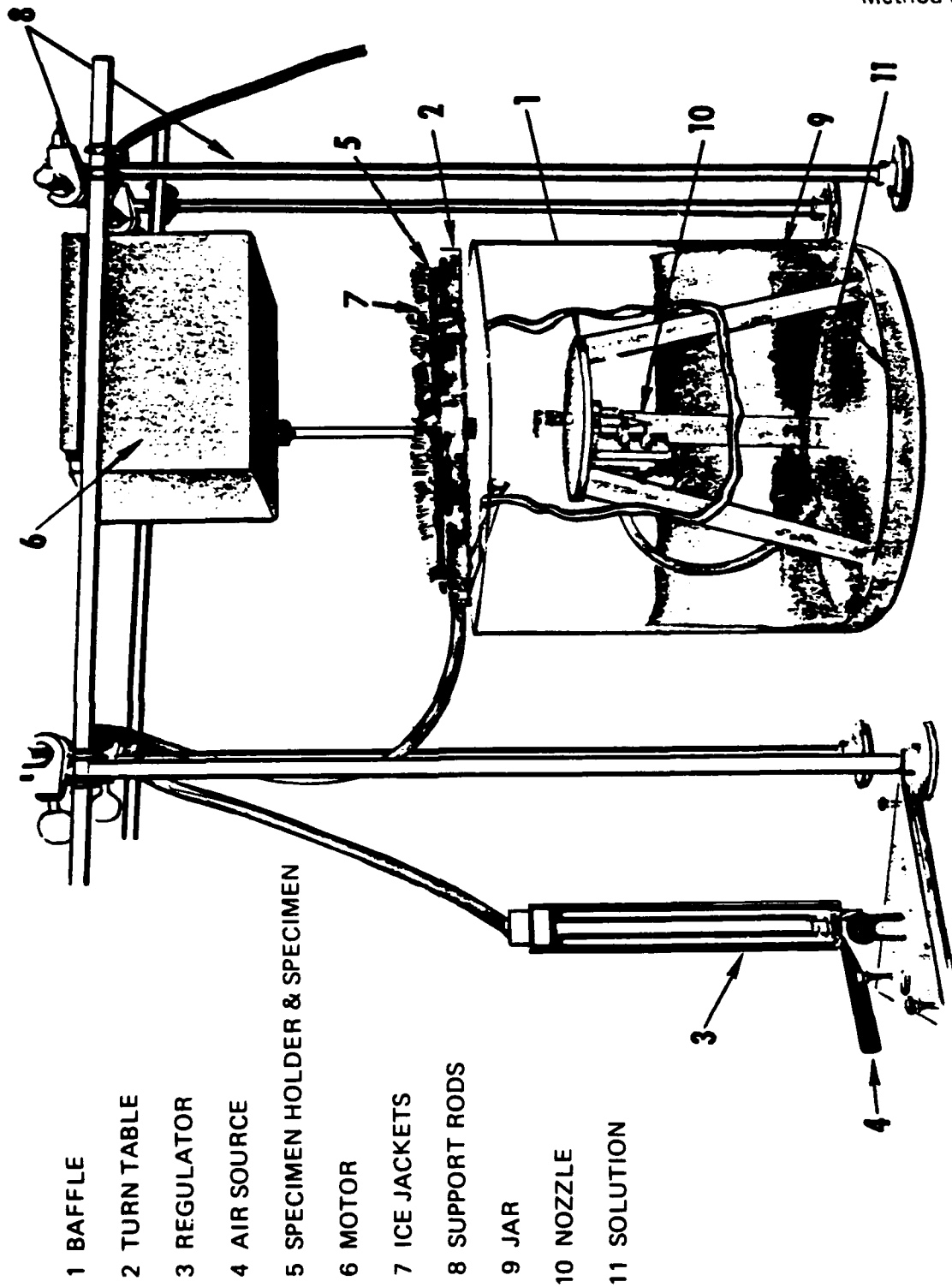
7. REPORTING

Report the number of test cycles completed prior to failure.

8. PRECISION

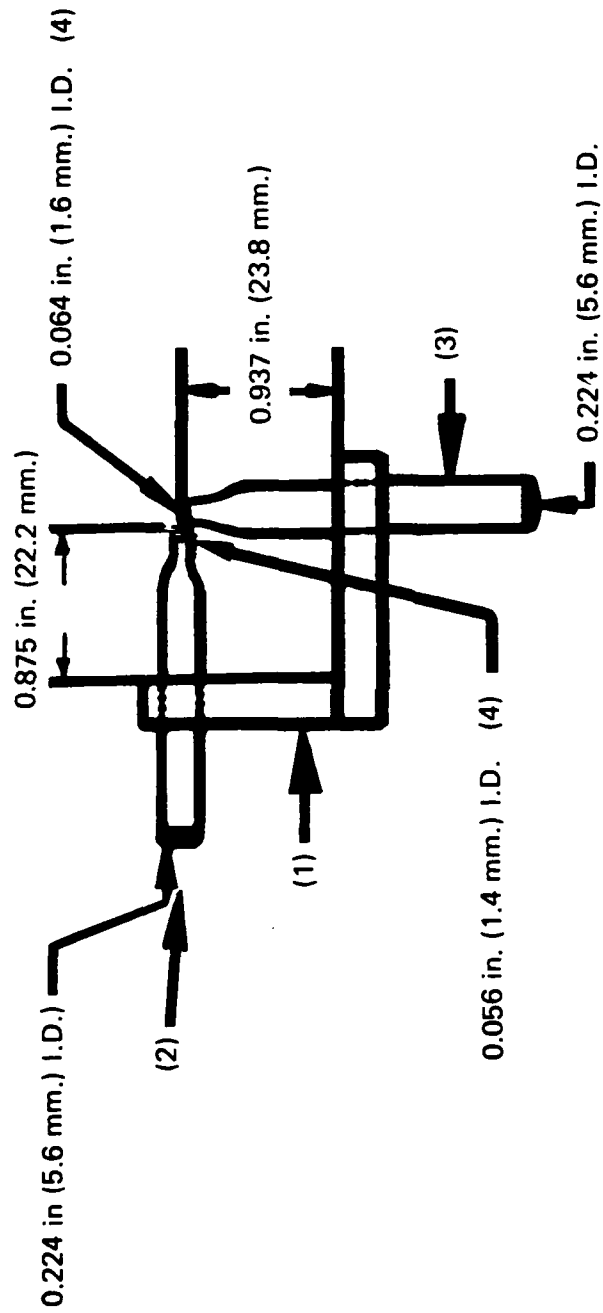
No precision data has been established for this method.

Method prepared by:
Navy-NADC-1984



- 1 BAFFLE
- 2 TURN TABLE
- 3 REGULATOR
- 4 AIR SOURCE
- 5 SPECIMEN HOLDER & SPECIMEN
- 6 MOTOR
- 7 ICE JACKETS
- 8 SUPPORT RODS
- 9 JAR
- 10 NOZZLE
- 11 SOLUTION

Figure A-1. Synthetic Sea Water — Spray Test Apparatus



NOTES:

1. 0.25 in. (6.37 mm.) Acrylic Material
2. Filtered Air Source
3. Liquid Pick-Up Tube
4. Orifice

Figure A-2. Spray Nozzle

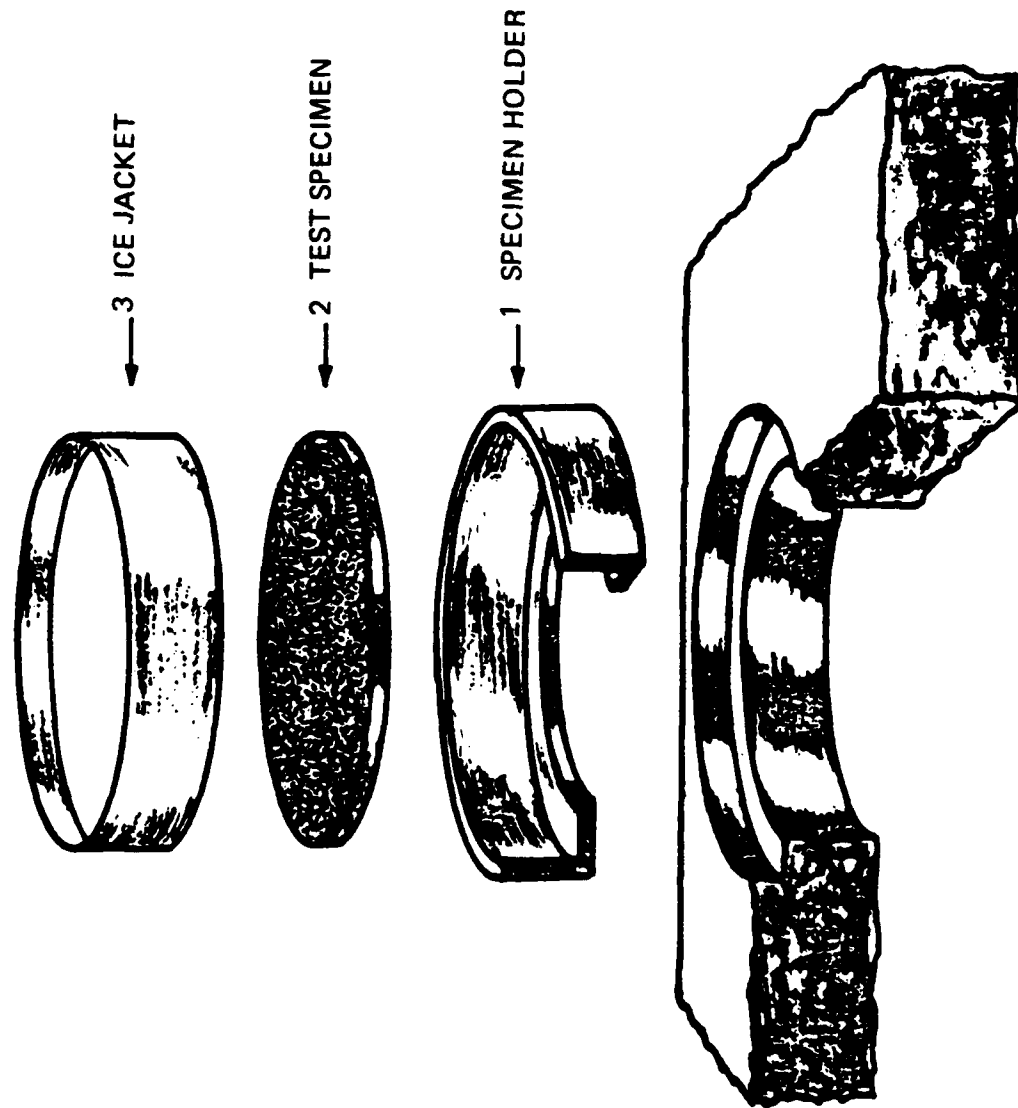


Figure A-3. Test Specimen Holder Assembly