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PROCEDURE FOR CASUAL EXPOSURE OF MATERIALS TO HYPERGOLIC FLUIDS.

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1.0 SCOPE

1.1 This procedure provides a means to determine the effects of minor amounts of hypergolic fluids, such as a splash, on materials such as plastics, foams, adhesives, coatings, composites, elastomers, fabrics, coated fabrics, films, supported films, lubricants, metals, etc. This procedure is not applicable for determining the prolonged effects of hypergolic fluids on materials used in the design and operation of hypergol storage and transfer systems. Materials that are in constant exposure to hypergolic fluids should be evaluated per NHB 8060.1, latest revision.

1.2 This procedure may also be applicable for the testing of materials that may similarly be exposed to other chemicals.

2.0 APPLICABLE DOCUMENTS

2.1 ASTM STANDARDS

D412 Test Methods for Rubber Properties in Tension
D638 Test Method for Tensile Properties of Plastics
D751 Test Methods for Coated Fabrics
D882 Test Methods for Tensile Properties of Thin Plastic Sheeting
D1117 Test Methods for Nonwoven Fabrics
D1623 Test Method for Tensile Properties of Rigid Cellular Plastics
D1682 Test Method for Breaking Load and Elongation of Textile Fabrics
D1683 Test Method for Seam Breaking Strength (Load) of Woven Textile Fabrics
D1708 Test Method for Tensile Properties of Plastics by use of Microtensile Specimens
D1876 Test Method for Peel Resistance of Adhesives (T-Peel Test)
D2240 Test Method for Rubber Property-Durometer Hardness
D2261 Test Method for Tearing Strength of Woven Fabrics by the Tongue Method
D3574 Test Method for Flexible Cellular Materials-Slab, Bonded, and Molded Urethane Foams
D3787 Test Method for Bursting Strength of Knitted Goods-Constant-Rate-of-Travel Ball Burst Test
F739 Test Method for the Resistance of Protective Clothing Materials to Permeation by Hazardous Liquid Chemicals

2.2 MILITARY STANDARDS

MIL-P-25604 Propellant, Unsymmetrical Dimethylhydrazine
MIL-P-26536 Propellant, Hydrazine
MIL-P-26539 Propellant, Nitrogen Tetroxide

MIL-P-27402 Propellant, Hydrazine-Unsymmetrical Dimethylhydrazine; (50 percent N_2H_4 - 50 percent UDMH)

MIL-P-27404 Propellant, Monomethylhydrazine

2.3 NASA STANDARDS

NHB 8060.1 Flammability, Odor, Offgassing, and Compatibility Requirements and Test Procedures for Materials in Environments that Support Combustion

3.0 FOREWORD

3.1 This procedure evaluates the effects on materials from casual exposure to hypergolic fluids such as nitrogen tetroxide, hydrazine, monomethylhydrazine, unsymmetrical dimethylhydrazine, and various mixtures of the three fuels. This procedure will provide methods to determine if a fluid could react exothermally or spontaneously ignite on contact with a material, cause changes in the mechanical properties of a material, induce environmental stress cracking in a material, cause a material to become shock sensitive, and whether it could penetrate or permeate a material.

3.2 The compatibility of materials with other chemicals may also be evaluated using similar methods that would take into consideration the characteristics of the particular chemicals being used, and of the materials being evaluated.

4.0 TEST CONDITIONS

4.1 TEMPERATURE - Unless otherwise specified, the test temperature shall be 23± 2°C. When the temperature is other than the above standard temperature, the test temperature shall be reported.

4.2 PRESSURE - Unless otherwise specified, the test pressure shall be conducted at ambient pressure. When the pressure is other than ambient, the test pressure shall be reported.

4.3 EXPOSURE TIME - For the purpose of this procedure, casual exposure times are considered to be of any duration less than or equal to 240 minutes.

The choice of the exposure time shall be specified by the requester based upon the intended use of the material and the expected duration of field exposure. The actual test exposure time shall be reported.

4.4 SAMPLE THICKNESS - The thickness of the sample may be determined using a standard micrometer. If the thickness of the

sample is reported, it should be reported to the nearest 0.025 mm (0.001 inch).

4.5 SAMPLE WEIGHT - The weight of the sample may be determined using a standard balance. If the weight of the sample is reported, it should be to the nearest 0.1 mg.

5.0 SAFETY PRECAUTIONS

5.1 Hypergolic fluids are considered toxic chemicals. These chemicals shall only be exposed to room atmosphere inside an approved laboratory hood. Separate, dedicated hoods shall be used for the oxidizer and fuels.

5.2 Personal protective clothing shall be worn by personnel when performing these tests. The minimum protection required are hypergol compatible gloves, lab apron, and face shield or goggles.

6.0 SAMPLE PREPARATION

6.1 The material shall be properly identified when submitted. The information provided should conform to the requirements of NHB 8060.1. See Figure 1 for a form suitable for recording the necessary information. The information on this form, or a copy of the completed form, shall be included in the test report.

6.2 Films, fabrics, sheets, metals, and composites shall be cleaned and dried to the end-use specification. Contamination on the surfaces of solid, nonporous samples should be removed by washing with deionized water and mild detergent, rinsing with deionized water, and drying with filtered gaseous nitrogen. Particulate matter on the surfaces of solid, porous samples should be removed with filtered gaseous nitrogen. Flaws and any residual contamination shall be noted. If the flaws resulted from sample preparation at the test facility, new samples shall be prepared. After cleaning, the test samples shall not be handled with bare hands. Samples shall be weighed, measured, and individually identified.

6.3 Materials being tested for chemical reactivity shall be cut in the form of a 4-inch square sample in the use thickness (see Figure 2). To determine changes in the mechanical properties of a material, the sample shall be cut in the form of a 4 by 6-inch rectangle (see Figure 2). For nonisotropic materials, the 4 by 6-inch samples shall be cut from both the machine (warp) and transverse (fill) directions.

6.4 Materials such as adhesives and coatings shall be applied in a thickness equivalent to normal use on aluminum foil and cured, if necessary, in accordance with the manufacturer's instructions.

6.5 Materials such as tapes shall be applied on either aluminum foil, a watch glass, or petri dish in the as-received condition and thickness.

6.6 Greases and gels shall be applied on aluminum foil in a thickness equivalent to normal use and cured, if required, in accordance with the manufacturer's instructions.

6.7 Liquids shall be tested by placing 10 ml in the bottom of a 100 ml glass laboratory beaker.

6.8 Complex shapes such as O-rings, cables, pipes, etc., shall be tested in a configuration consistent with the intended use. Samples shall be cleaned as specified in 6.2.

7.0 TEST METHODS

7.1 REACTIVITY TEST METHOD - This test is used to determine a possible material reaction and/or degradation when exposed to hypergolic fluids or other chemicals of interest.

7.1.1 Test Procedure

Take an appropriately sized sample of the test material (see Figure 2) and place it on a watch glass, or petri dish.

Add the test fluid, approximately 1 ml of the specified test fuel or oxidizer, to the center of the sample, taking care not to expose the edges of the sample to the fluid.

Allow the test fluid to stand on the sample for the specified exposure time. Add test fluid as required to maintain a liquid film on the test sample during the specified exposure time.

Carefully observe the test sample throughout the duration of the test.

At the end of the specified exposure time, carefully blot the liquid from the sample and rinse the sample with running water for 60 seconds. Allow the test sample to air dry for 24 hours prior to final evaluation.

7.1.2 Reporting

The report shall consist of the following as a minimum (an example of a suitable form for reporting the result of this test is shown in Figure 3, KSC FORM 3-539NS):

a. The name of the test material, supplier, and manufacturer.
b. The test temperature, pressure, duration, and sample thickness before and after the test.

c. Any reactivity observed during the exposure such as burning, smoking, bubbling, frothing, charring, solubility, swelling, or fracture of the sample.
d. Any changes in the condition of the sample after the exposure such as color, flexibility, rigidity, surface condition, transparency, pitting, hardness, tackiness, friability, or powder formation.
e. Test conclusions as defined in Section 8.0.

7.2 PENETRATION TEST METHOD - This test is used to determine both the possible penetration and chemical reactivity of materials when exposed to hypergolic fluids or other chemicals of interest.

7.2.1 Test Procedure

Take an appropriately sized sample of the test material (see Figure 2) and place it over a beaker.

Add the test fluid, approximately 1 ml of the specified test fuel or oxidizer, to the center of the sample, taking care not to expose the edges of the sample to the fluid.

Allow the test fluid to stand on the sample for the specified exposure time. Add test fluid as required to maintain a liquid film on the test sample during the specified exposure time.

Carefully observe for the first fallen droplet at the bottom of the beaker, and note the time of occurrence.

For materials used for protective garments, observe for initial wetness underneath the test sample, and note the time of occurrence.

NOTE:

Atmospheric condensation could occasionally form underneath a sample during a test, giving a false indication of penetration. In such an event, verification can be made by applying a hypergol compatible blotter that is known to discolor when in contact with a hypergolic fluid.

At the end of the specified exposure time, carefully blot the liquid from the sample and rinse the sample with running water for 60 seconds. Allow the test sample to air dry for 24 hours prior to final evaluation.

7.2.2 Reporting

The report shall consist of the following as a minimum (an example of a suitable form for reporting the results of this test is shown in Figure 3, KSC FORM 3-539NS):

a. The name of the test material, supplier, and manufacturer.
b. The test temperature, pressure, duration, and sample thickness before and after the test.
c. Any penetration observed during the exposure, and the elapsed time of occurrence.
d. For materials used for protective garments, any wetness observed underneath the test sample during the exposure, and the elapsed time of occurrence.
e. Any reactivity observed during the exposure such as burning, smoking, bubbling, frothing, charring, solubility, swelling, or fracture of the sample.
f. Any changes in the condition of the sample after the exposure such as color, flexibility, rigidity, surface condition, transparency, pitting, hardness, tackiness, friability, or powder formation.
g. Test conclusions as defined in Section 8.0.

7.3 MECHANICAL EVALUATION TEST METHOD - This test is used to determine possible changes in the mechanical properties of a material due to exposure to hypergolic fluids or other chemicals of interest.

7.3.1 Test Procedure

Select the desired mechanical property to be evaluated.

Determine the value of that property on unexposed samples as a control.

Place a 4 by 6-inch test specimen on a flat Teflon or stainless steel base plate (see Figure 4).

Take a template, shown in Figure 4, and apply a bead of fluid compatible grease around the 3-inch opening (to prevent wicking under the template). Then clamp both the template (with the grease against the test specimen) and test specimen to the base plate. The test specimen shall be sandwiched between the template and the base plate.

Place sufficient test fluid in the slot to wet the full 3-inch wide exposed strip of test specimen.

Allow the test fluid to stand on the test specimen for the specified exposure time. Add test fluid as necessary to maintain a uniform liquid film across the width of the test specimen during the specified exposure time.

At the end of the specified exposure time, carefully remove the test fluid and rinse with running water for 60 seconds. Disassemble the test fixture, wipe the grease off the test specimen, and allow the test specimen to air dry for 24 hours.

Determine the value of the mechanical property to be evaluated using one of the typical test methods listed in Section 2.0. If a test method is other than those listed in Section 2.0, a copy of the desired test procedure shall be supplied by the requester.

7.3.2 Reporting

The report shall consist of the following as a minimum:

a. The name of the test material, supplier, and manufacturer.
b. The name of the test chemical.
c. The test temperature, pressure, duration, and sample thickness before and after the test.
d. Any reactivity observed on the test specimen in accordance with Section 7.1.
e. The value of the mechanical property obtained for the exposed and unexposed test material, and the method used to measure that property.

7.4 SHOCK SENSITIVITY TEST METHOD - The shock sensitivity of a material exposed to these fluids shall be assessed using mechanical impact test procedures similar to NHB 8060.1, Test 13, Part 1.

7.5 EXOTHERMIC REACTION TEST METHOD - This test is used to measure the temperature rise of a material when exposed to hypergolic fluids or other chemicals of interest. An excessive temperature rise could suggest an impending, spontaneous ignition of the material.

NOTE:

Since Nitrogen Tetroxide has a boiling point of 20°C, this method would not be applicable for N_2O_4 since its evaporative cooling effect would make any results obtained inconclusive.

7.5.1 Test Method

Take an appropriately sized sample of the test material (see Figure 2) and place it on a watch glass or petri dish.

Position a sheathed thermocouple or resistance thermometer such that it touches the center of the sample.

Add 0.5 ml of the specified test fuel to the center of the sample making sure that the sheath is in the liquid.

Allow the test fluid to stand on the sample for the specified exposure time while monitoring the temperature. At the end of the specified exposure time, carefully blot the sample dry, rinse with running water for 60 seconds, and air dry for 24 hours.

7.5.2 Reporting

The report shall consist of the following as a minimum (an example of a suitable form for reporting the results of this test is shown in Figure 3, KSC Form 3-539NS):

a. The name of the test material, supplier, and manufacturer.
b. The name of the test chemical.
c. The starting temperature of the test fluid, the maximum fluid temperature observed during the test and the time of occurrence, and the test fluid temperature at the end of the test.
d. The duration of the test and sample dimensions.
e. Any reactivity observed in the test specimen in accordance with Section 7.1.
f. Test conclusions as defined in Section 8.0.

7.6 PERMEABILITY TEST METHOD - This test is used to determine the vapor or liquid permeation rate of a material when exposed to hypergolic fluids or other chemicals of interest as specified by the requester.

Two kinds of permeability tests may be performed: conditioned and/or unconditioned. The conditioned test is performed on specimens that were previously exposed to hypergolic fluids prior to a permeability test in order to simulate used materials. The unconditioned test is performed on new, unused specimens in the as-received condition. The duration of the permeability test shall be 120 minutes.

Configuration Requirements - The fuel permeability tests shall be performed in accordance with ASTM F-739 using the standard 2-inch diameter test cell. Oxidizer permeability tests shall also be performed in accordance with ASTM F-739 except that the above cell shall be oriented horizontally with the oxidizer containing side down. Materials used for enclosed, pressurized protective garments (such as SCAPE suits or PHE's) shall always be tested with the pressure on the collection side of the test cell maintained between 0.5 and 1.0 Inches Of Water above the pressure applied on the test fluid side of the cell. If conditioned test specimens are used, the conditioned side shall be facing the test fluid side of the cell.

7.6.1 Test Procedure for Preparing Conditioned Test Specimens

Place a 3-inch diameter test specimen (Figure 2) on a flat Teflon or stainless steel base plate (see Figure 5). The side of the material that is normally exposed in service shall be in the up position.

Take a template (see Figure 5) and apply a bead of fluid compatible grease around the opening (to prevent wicking under the template). Then clamp both the template (with the grease against

the test specimen) and test specimen to the base plate. The test specimen shall be sandwiched between the template and the base plate.

Place sufficient test fluid on the specimen to wet the entire surface.

Allow the test fluid to stand on the test specimen for 60 seconds. Carefully remove the test fluid, rinse the test specimen with running water for 60 seconds, and disassemble the test fixture.

Wipe the grease off the test specimen taking care not to contaminate the propellant exposed area of the test specimen.

Allow the test specimen to air dry for 24 hours.

Perform this process with the same sample using, in order, monomethylhydrazine, nitrogen tetroxide, and hydrazine.

Condition the test specimen a second time applying the same above order of fluids.

7.6.2 Test Procedure for Oxidizer Permeability Test

This test shall be conducted with the test cell oriented horizontally, allowing only vapor contact with the test specimen.

Fill the bottom of the assembled test cell approximately half full with liquid oxidizer.

Allow the oxidizer vapors to vent freely for approximately 60 seconds, then close the stopcocks on the test cell.

After 120 minutes, open the stopcocks and drain the oxidizer from the test cell.

Disassemble the test cell and decontaminate the test specimen by gently blotting any liquid from the sample, and rinsing it with running water for 60 seconds. Allow the test sample to air dry for 24 hours.

7.6.2.1 Reporting

The report shall consist of the following as a minimum:

a. The name of the test material, supplier, and manufacturer.
b. The test temperature, pressure, duration, and sample thickness before and after the test.
c. Any differential pressure maintained across the test specimen.
d. The condition of the test specimen at the conclusion of the test.
e. The breakthrough time.
f. The steady state permeability rate.

g. The threshold detection level of the measuring system to the specific oxidizer used in the test.

7.6.3 Test Procedure for Fuel Permeability Test

This test shall be conducted as in 7.6.2 but with the test cell oriented in the vertical position, allowing liquid contact with the test specimen.

7.6.3.1 Reporting

The report shall consist of the following as a minimum:

a. The name of the test material, supplier, and manufacturer.
b. The test temperature, pressure, duration, and sample thickness before and after the test.
c. Any differential pressure maintained across the test specimen.
d. The condition of the test specimen at the conclusion of the test.
e. The breakthrough time.
f. The steady state permeability rate.
g. The threshold detection level of the measuring system to the specific fuel used in the test.

7.7 ENVIRONMENTAL STRESS CRACKING TEST METHOD - This method determines the susceptibility of a material to environmental stress cracking (ESC) when placed in contact with a test fluid. This procedure utilizes a two-point buckling load technique, which would generate a bending stress at the midsection of a specimen. The deformation required on the specimen in order to achieve the desired bending stress can be estimated from the following equations:

$$I = \frac{Wt^3}{12} \qquad M = \frac{2SI}{t} \qquad V = \frac{ML^2}{8EI}$$

where S is the desired bending stress, L is the specimen length, t is the specimen thickness, V is the maximum distance from the specimen holder base to the top surface of the specimen (see Figure 6), E is Young's modulus for the material, W is the specimen width, I is the moment of inertia of the cross-sectional area, and M is the bending moment. The required dimensional units for each are as follows:

S = psi	t = inches	W = inches	M = in.-Lb
L = inches	E = psi	I = $in.^4$	V = inches

7.7.1 Test Procedure

Select the desired bending stress.

Calculate the distance V using the above equations.

Set the thumb screw blocks of the specimen holder to opposite ends of the specimen holder and place an environmental stress cracking test specimen (see Figure 2) in the holder.

Adjust the thumb screw blocks as required to produce the distance V on the test specimen (see Figure 6).

If the specimen exhibits cracking or crazing prior to the test, the desired stress is too high. Choose a lower stress value, and insert a new specimen.

Carefully apply solvent dropwise with a stirring rod to the upper surface of the specimen at the apex of the bend, taking care not to touch the specimen with the stirring rod. Do not allow the test fluid to touch the edges of the specimen. If either the test specimen surface is touched with the stirring rod or the solvent wets the edges of the test specimen, discard the test specimen and insert a new one.

While still mounted in the holder, carefully observe the test specimen with the naked eye or with a 4 or 8 diopter magnifier for crack formation.

Determine the time to crack or craze by measuring the time required for the first visible crack or craze to appear.

NOTE:

The relative opacity of a material will increase the difficulty in discerning when cracking or crazing has commenced.

While still mounted in the holder, gently blot the liquid from the specimen, and rinse the specimen with running water for 60 seconds. Allow the specimen to air dry for 24 hours before removing it from the holder.

Preliminary qualitative tests at relatively high stress levels may be performed to identify materials with a tendency to undergo rapid ESC or crazing and to reduce the total number of tests required to characterize a material.

Perform a series of tests that would determine the time to crack or craze at different stress levels.

Plot the stress level versus the time to crack or craze. An L shaped curve should be obtained.

Draw straight lines through the points that make up the vertical and horizontal legs of the L. The intersection of the lines will determine the critical stress level for that particular solvent/material combination.

Repeat the procedure for each different solvent/material combination.

7.7.2 Reporting

The report shall contain the following information as a minimum:

a. The name of the test material, supplier, and manufacturer.
b. The name of the test chemical.
c. The test temperature, pressure, duration, and sample thickness (at the exposed area) before and after the test.
d. The length, thickness, and width of the overall test sample.
e. The critical stress level as obtained from the plot.

8.0 TEST CONCLUSIONS

For those results that are reported on KSC Form 3-539NS (Ref. Figure 3), test data conclusions shall be based on the following definitions:

A. No Significant Reactivity Observed

When test data based on visual observations with the unaided eye reveal no reactivity and no changes in the visual characteristics, bulk characteristics, and surface characteristics of the test sample.

B. Slight to Moderate Reactivity Observed

When test data based on visual observations with the unaided eye reveal reactivity (but no ignition) and/or any changes in the visual characteristics, bulk characteristics, and/or surface characteristics of the test sample.

C. Sample Shows Indications of Gross Incompatibility

When ignition of the test sample was observed with the unaided eye.

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GRAPHICS: Laura Bales

TEST MATERIAL

MANUFACTURER DESIGNATION_____

COMPOSITION_____

SPECIFICATION_____

MATERIAL CODE_____

GENERIC ID_____

APPLICATION_____

USE TEMPERATURE (MIN)_____ USE TEMPERATURE (MAX)_____

HYPERGOLIC FLUID EXPOSURE TIME (FIELD USE)_____

MANUFACTURER

NAME_____

ADDR L1_____

ADDR L2_____

CITY_____

STATE_____

COUNTRY_____

SUPPLIER

NAME_____

ADDR L1_____

ADDR L2_____

CITY_____

STATE_____

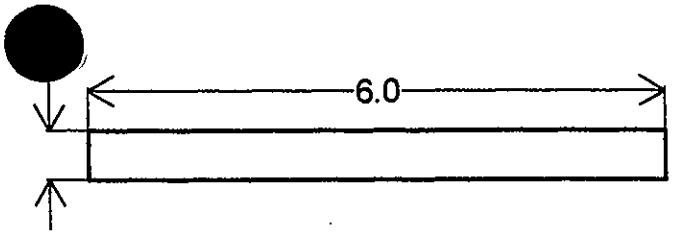
COUNTRY_____

REMARKS_____

_____

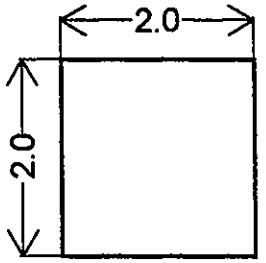
_____

FIGURE 1
MATERIAL IDENTIFICATION

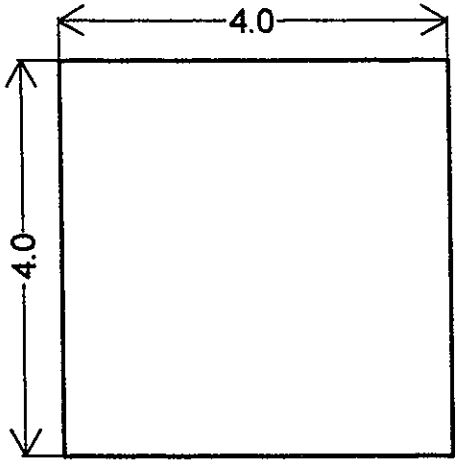


Environmental Stress Cracking

Test Specimen

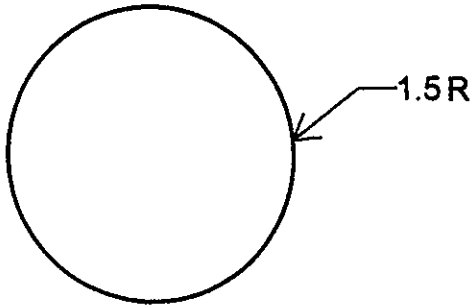


Exothermic Test Specimen

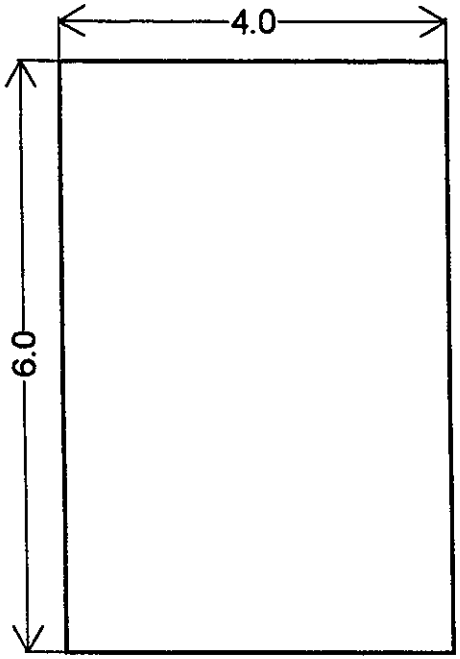


Reactivity/Penetration

Test Specimen



Permeability Test Specimen



Mechanical Test Specimen

FIGURE 2

TEST SPECIMEN DIMENSIONS (INCHES)

CHEMICAL ANALYSIS LABORATORY COMPATIBILITY REPORT

DATE	LAB WORK ORDER NO. G-
TELEPHONE NO.	REFERENCE NO.

REQUESTING ORGANIZATION	REQUESTOR

[redacted]CLE | SYSTEM | REFERENCE DOCUMENT

MATERIAL NAME OR MFGR'S ID

CHEMICAL CLASS OF MATERIAL

GENERIC NAME OF MATERIAL

SPECIAL INSTRUCTIONS

TEST CONDITIONS

Test 1 Sample per: _____

Test Name: _____

Test Fluid: _____

TEST DATA

Test Sample Description: _____

Material Quantity (gms): _____

Media Volume (ml): _____

Container Volume (ml): _____

Media Exposure Time (Hrs): _____

OBSERVATIONS

[redacted] _____ Temp. Change _____

[redacted]e _____ Soluble _____

Froth _____ Fracture _____

Bubble _____ Swell _____

Char _____

Remarks: _____

VISUAL CHARACTERISTICS/	Pre-Test	Post-Test
Color		
Opaque		
Translucent		
Transparent		

Remarks: _____

BULK CHARACTERISTICS/	Pre-Test	Post-Test
Shape		
Flexible		
Rigid		
Soft		
Hard		
Friable		
Powder		

Remarks: _____

SURFACE CHARACTERISTICS/	Pre-Test	Post-Test
Smooth		
Rough		
Wrinkled		
Pitted		
Woven		
Matted		
Tacky		

Remarks: _____

OTHER OBSERVATIONS

[redacted]

CONCLUSIONS

- ☐ NO SIGNIFICANT REACTIVITY OBSERVED
- ☐ SLIGHT TO MODERATE REACTIVITY OBSERVED
- ☐ SAMPLE SHOWS INDICATIONS OF GROSS INCOMPATIBILITY

ANALYST:	DATE:	APPROVAL:

FIGURE 5

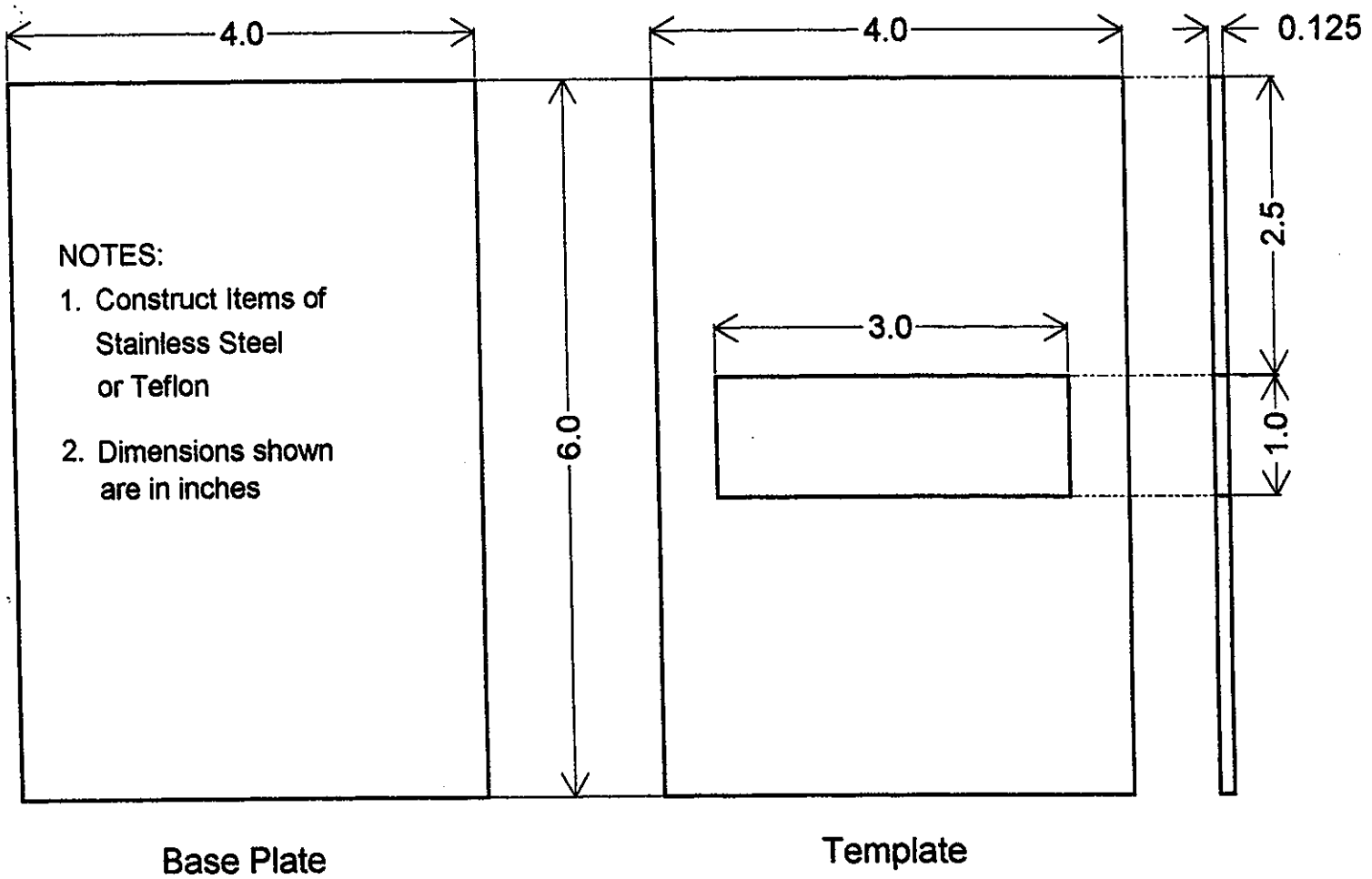
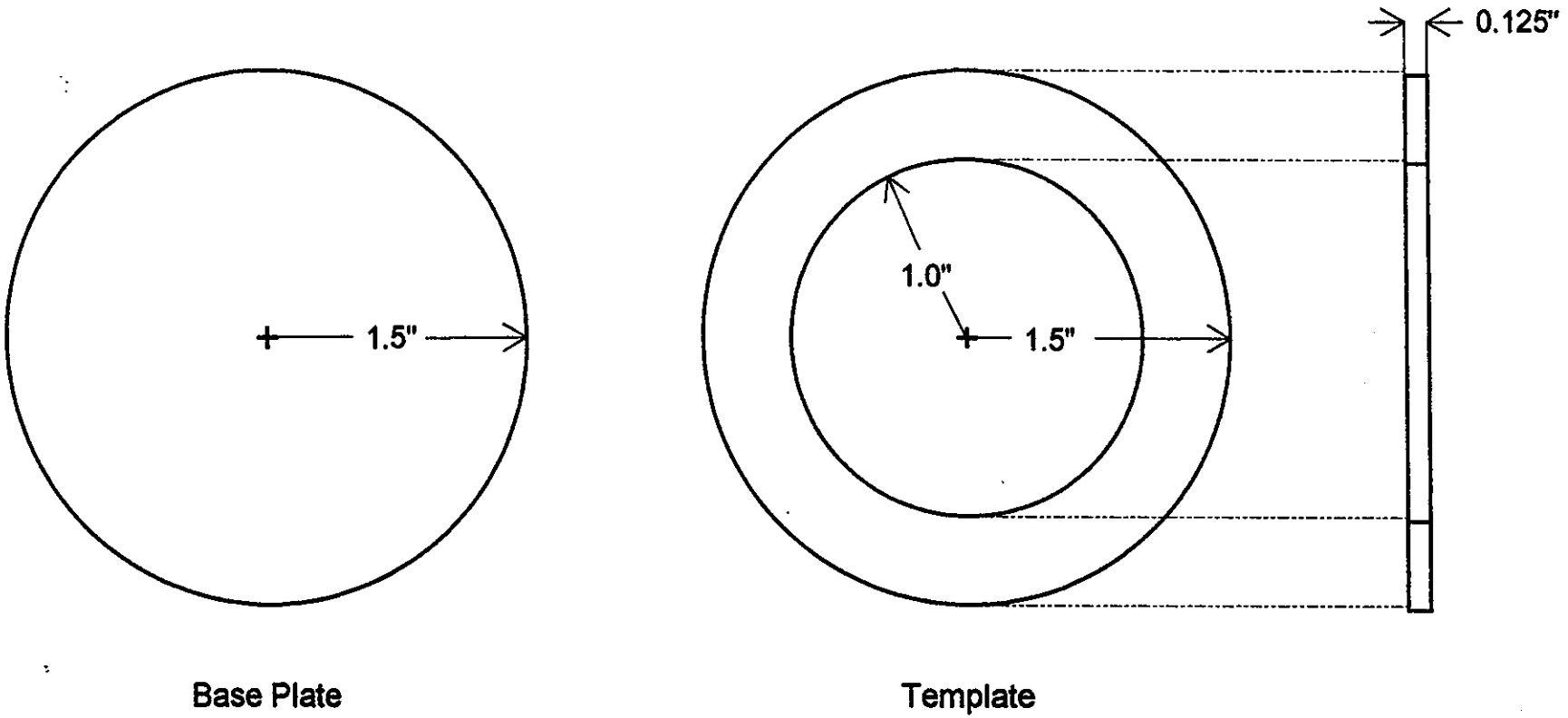


FIGURE 4

EXPOSURE FIXTURE FOR MECHANICAL TEST



NOTE:
Construct Items of
Stainless Steel or Teflon

FIGURE 5

EXPOSURE FIXTURE FOR PERMEABILITY TEST

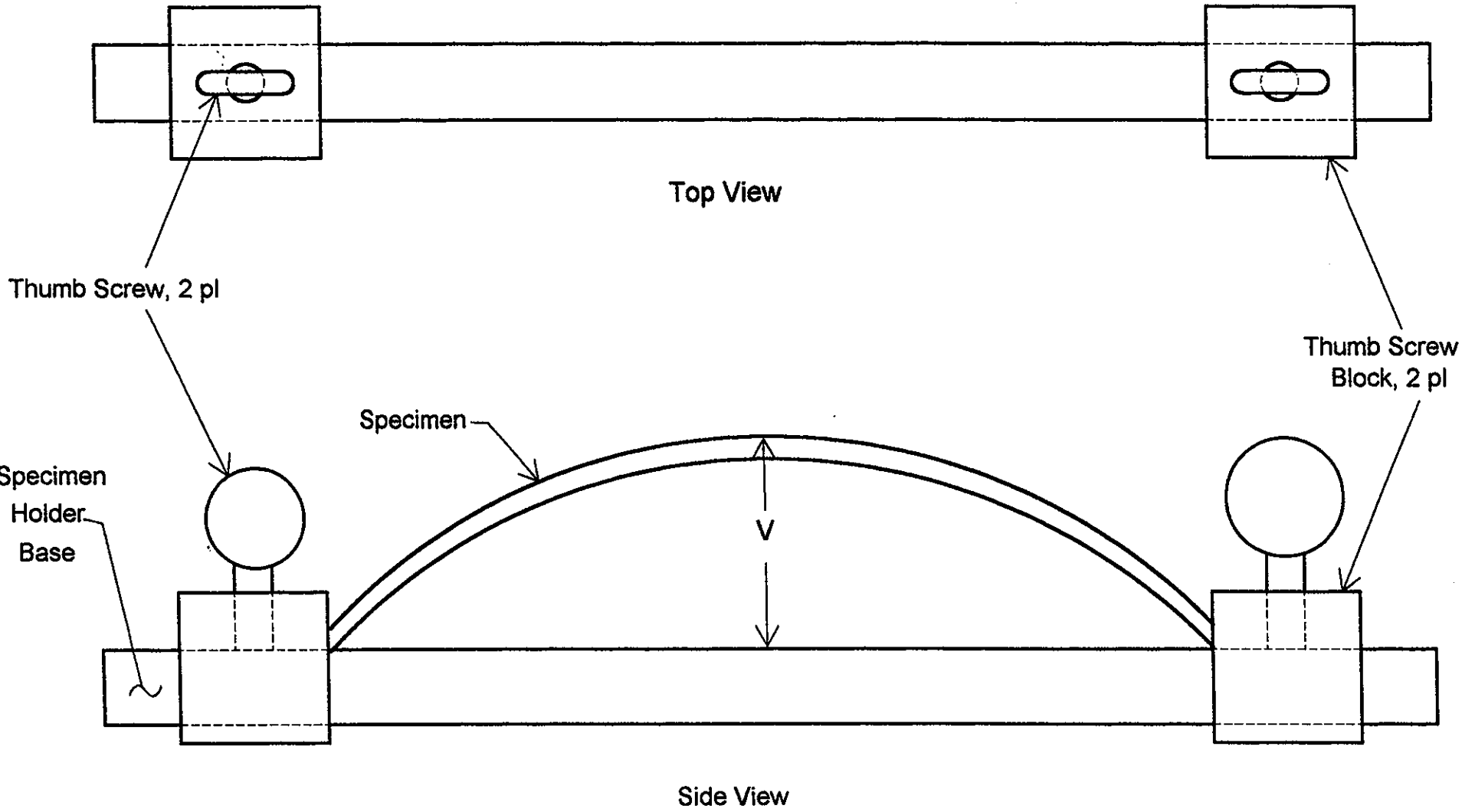


FIGURE 6

ENVIRONMENTAL STRESS CRACKING TEST CONFIGURATION