SENSITIVITY AND COMPARISON EVALUATION OF SATURN V LIQUID PENETRANTS

By G. H. Jones
Quality and Reliability Assurance Laboratory

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Recognition is made to the following individuals, James B. Beal, Charlie Wages, and Gilbert Smith, for their contributions during the preparation of the work leading to the accomplishment of this study.  

This report describes a sensitivity and comparison evaluation performed on six liquid penetrants that were used on the Saturn V vehicle and other space hardware to detect surface discontinuities.

The relationship between penetrant materials and crack definition capabilities, the optimum penetrant materials evaluation method, and the optimum measurement methods for crack dimensions were investigated. A unique method of precise developer thickness control was envolved, utilizing clear radiographic film and a densitometer.

The method of evaluation included five aluminum alloy, 2219-T87, specimens that were heated and then quenched in cold water to produce cracks. The six penetrants were then applied, one at a time, and the crack indications were counted and recorded for each penetrant for comparison purposes. Measurements were made by determining the visual crack indications per linear inch and then sectioning the specimens for a metallographic count of the cracks present. This method provided a numerical approach for assigning a sensitivity index number to the penetrants.

Of the six penetrants evaluated, two were not satisfactory (one was not sufficiently sensitive and the other was too sensitive, giving false indications). The other four were satisfactory with approximately the same sensitivity in the range of 78 to 80.5 percent of total cracks detected.

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SENSITIVITY AND COMPARISON EVALUATION
OF
SATURN V LIQUID PENETRANTS

By

G. H. Jones

SUMMARY

Six liquid penetrants were subjected to a sensitivity and comparison evaluation. The liquid penetrants included four fluorescent penetrants and two dye (color contrast) penetrants. These penetrants were used for detecting surface discontinuities on the Saturn V vehicle and other space hardware.

For comparative purposes, five aluminum alloy, 2219-T87, specimens were selected, heated, and quenched in cold water to produce cracks. The penetrants were then applied, one at a time, to the specimens, and the crack indications were counted and recorded for each penetrant. Measurements were made by determining the visual crack indications per 2.54 cm (lineal inch) and then sectioning the specimens for a metallographic count of the cracks present. This provided a numerical method for assigning a sensitivity index number to the penetrants. A unique method of precise developer thickness control evolved from this program. Clear radiographic film, coated during developer application to specimen, was analyzed by densitometer to determine film light transmission as a function of developer thickness.

The results of this evaluation indicate that the method used to determine the sensitivity of the liquid penetrants was an effective approach for evaluating liquid penetrants.

Of the six penetrants evaluated, fluorescent penetrant P-545 was not satisfactory due to excessive sensitivity and many false indications. Dye penetrant SKL-HF had consistently poor sensitivity and was not satisfactory. The other four penetrants (ZL-44B, P-149, SKL-4, and ZL-22) were satisfactory with approximately the same sensitivity in the range of 78 to 80.5 percent of total cracks detected. Fluorescent penetrant ZL-22 should be evaluated with the developer wiped off.
SECTION I. INTRODUCTION

The objective of this program was to develop methods for determining the crack detection sensitivity of liquid penetrants used to locate surface discontinuities on all stages of the Saturn V vehicle and other space hardware. Comparisons were then made between penetrants, and their crack detection efficiency was established.

Six liquid penetrants were evaluated on five aluminum alloy specimens which were heated and then quenched in cold water to produce cracks. The liquid penetrants included four fluorescent penetrants and two dye penetrants. These penetrants, along with their respective developers and cleaners, are described in Section II.

The relationship between penetrant materials and crack definition capabilities, the penetrant materials evaluation method, and the measurement methods for crack dimensions are discussed in Section II. The evaluation was performed in accordance with the detailed evaluation procedure contained in Appendix A. The evaluation data sheets are presented in Appendix B. Appendix C defines the method which was evolved to control the penetrant developer thickness.

SECTION II. DISCUSSION

A. EVALUATION APPROACH

Fluorescent and dye liquid penetrant materials are used for detection of surface discontinuities on all stages of the Saturn V vehicle and other space hardware. The performance of the various types of liquid penetrant materials is well established and acceptable, although the relative sensitivities in determining width and depth of cracks in aluminum alloy weldments are not known. The degree of sensitivity is important when selecting penetrant materials.

An initial investigation determined that very few actual records of analysis have been produced for comparison or defining the absolute sensitivity (minimum crack dimensions) of penetrant materials. The relationship between penetrant materials and crack definition
The evaluation consisted of several phases as defined in the following paragraphs:

**Phase I.** Phase I consisted of the theoretical determination of an optimum penetrant material evaluation method for evaluating aluminum weldments. The optimum measurement method for crack width and depth dimensions was determined.

**Phase II.** Phase II consisted of the preparation of evaluation (test) specimens from aluminum alloy material.

**Phase III.** Phase III consisted of obtaining penetrant material samples from prime contractors or vendors.

**Phase IV.** Phase IV consisted of performing an analysis of the penetrant material capabilities and the evaluation specimens. Penetrant application was in accordance with the evaluation procedure given in Appendix A.

**B. LIQUID PENETRANTS**

1. **Penetrant Properties.** The mechanism by which a penetrant spreads into surface discontinuities was not investigated as a part of this project. The mechanism is very complex and is not completely known; however, several properties and characteristics do affect the penetrability. These properties and characteristics are discussed in the following paragraphs. It will be obvious that some of the properties and characteristics discussed will pertain to both fluorescent and dye penetrants while others will pertain to a single type of penetrant.

   a. **Penetrant Molecule.** Many measurements have been made of actual surface openings found by fluorescent and dye penetrant methods; however, experimental data have not been obtained to depict actual size of openings below the size of the penetrant molecule. It is believed that the size of the dye molecule is approximately 1000 angstrom units (A. U.), and that these molecules may be incapable of penetration into extremely narrow openings. The molecular size of typical dyes used in fluorescent penetrants is approximated at 10 x 10 x 2 A. U. For a quick comparison, 0.1 micron (\(\mu\)) equals 1000 A. U.
b. **Surface Tension.** Surface tension is defined as the cohesiveness of a liquid (the property that causes a drop of liquid to assume the shape of a sphere). Contrary to popular belief, there is no tension across the surface of a liquid; it is a mathematical device designed to describe the free-surface energy of a liquid. Surface tension resists capillary flow of a penetrant into a crack. At present, the exact effect that surface tension has on the ability of a penetrant to disclose a crack is not known.

c. **Contact Angle.** The contact angle is the angle measured through the liquid when the liquid makes contact with the surface of a material. (See figure 1.) Penetrant flow resistance is increased by a high contact angle, which indicates a low degree of wetting.

d. **Viscosity.** Viscosity is a dynamic force that is caused by molecular attraction. Viscosity makes a liquid resist a tendency to flow but has no effect on a static system; therefore, it will only affect the rate of spread of a penetrant into a crack. Many highly viscous materials are good penetrants and are economically important because of their relationship to the speed of drainage of excess penetrant from the surface of the part. An excessively rapid drainage of excess penetrant from the surface of the part would not allow sufficient penetration time while an excessively slow drainage of excess penetrant from the surface of the part would be time consuming.

e. **Wetting Ability.** Wetting ability of a penetrant is a classical term which is a function of the angle that the penetrant makes with the solid surface. The smaller the angle, the greater the wetting ability. (See figure 1.)

f. **Spreading Ability.** The spreading ability is a very important factor in liquid penetrants. It is assumed that the spreading of a liquid on a solid is dependent on some complex properties of the surface texture of the solid. Certain excellent penetrants on smooth surfaces fail on rough surfaces and vice versa; however, there is a lack of empirical data for substantiation. For a liquid to spread over a solid surface, the spreading coefficient ($S_{SL}$) must be positive.

$$S_{SL} = \gamma_{SG} - (\gamma_{L} + \gamma_{SL})$$
Figure 1. Contact Angles of a Good Penetrant and a Poor Penetrant
where:

\[ S_{SL} = \text{Spreading coefficient} \]
\[ \gamma_{SG} = \text{Surface energy of a solid-gas interface} \]
\[ \gamma_L = \text{Surface tension of liquid} \]
\[ \gamma_{SL} = \text{Surface energy of the solid-liquid interface} \]

A necessary, but not sufficient, condition for spreading requires that the surface energy of a solid-gas interface (\( \gamma_{SG} \)) exceed the surface energy of the solid-liquid interface (\( \gamma_{SL} \)).

\[ \gamma_{SG} > \gamma_{SL} \]

It is currently believed that spreading occurs by a pulling out and moving of the top portion of a liquid.

g. **Capillarity.** Capillarity is the ability of a liquid to rise in a capillary tube with one end immersed in the liquid. It was natural to try to use capillarity as a measure of penetrants, but it has not been successful because a natural long seam or crack is not a smooth round glass tube and the penetrants react differently. However, capillarity will be used in discussing the ability of penetrants to move over a rough surface.

h. **Penetrability.** Penetrability is the ability of a liquid penetrant to enter fine openings in reasonable time. One of the most probable causes of penetration is believed to be due to the difference in capillary pressure (\( P_c \)).

\[ P - P_o = P_c \]

where:

\[ P = \text{Final gas pressure} \]
\[ P_o = \text{Initial gas pressure} \]
\[ P_c = \text{Pressure difference (capillary pressure)} \]

The equilibrium position of the liquid in the crack tip (and the pressure difference, \( P_c \)) may be determined by equating the energy change due to an infinitesimal change in position (\( dy \)) to zero. (See figure 2.)
Figure 1. Contact Angles of a Good Penetrant and a Poor Penetrant
where:

\[ S_{SL} = \text{Spreading coefficient} \]
\[ \gamma_{SG} = \text{Surface energy of a solid-gas interface} \]
\[ \gamma_L = \text{Surface tension of liquid} \]
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Figure 2. Penetrant Crack Tip Configuration.
\[ 2 (\gamma_{SG} - \gamma_{SL}) \, ds - 2 (P - P_o) \, x \, dy = 0 \]

where:

- \( P_o \): Initial gas pressure
- \( s \): Perimeter of liquid-gas-solid
- \( P \): Final gas pressure
- \( x \): Width of tip interface
- \( y \): Position of interface

i. **Temperature.** The vast majority of liquids have a surface tension which decreases as temperature increases. If \( \gamma \) is surface tension at the temperature \( T \), its value is given by

\[ \gamma = a (T_c - T), \]

with \( T_c \) being the critical temperature (the temperature at which there is no surface tension) and "a" being a quantity independent of temperature but characteristic for every substance. Surface tensions become immeasurably small a few degrees below the critical temperature. Liquid penetrant evaluations should be performed at the same ambient temperature to eliminate any possible variance due to temperature.

ej. **Contamination of Penetrant.** Contamination by liquids usually results in an increase of surface tension and contact angle, thereby decreasing the wetting ability of the penetrant. Penetrants contaminated with dust display increased surface tension and anomalous contact angles. In general, oxides, carbides, and nitrides have higher surface energies at room temperature than their parent metals. Due to the higher surface energy of the oxide, the energy of the oxide-liquid interface and the oxide-gas interface will be greater than the values for the parent metal system. The oxides cause a smaller contact angle; therefore, oxides which do not physically block the crack or pore improve the driving force for penetration.

k. **Effect of Light.** Light does not affect penetrating ability of penetrants.
1. **Penetration Time.** Penetration time will vary according to viscosity of the penetrant and should be determined empirically for each penetrant used.

   m. **Other Factors.** The influence of factors other than temperature or surface tension is usually very small. The effects of electrostatic charges or magnetic fields on penetrant properties are not known, but should be investigated for plastics and magnetic materials.

2. **Penetrant Materials.** The penetrant materials evaluated were those used for detection of surface discontinuities on all stages of the Saturn V vehicle and other space hardware. In no way does this evaluation reflect the quality of the penetrant materials, but it does reflect the relationship between penetrant materials and crack definition capabilities, the optimum penetrant materials evaluation method, and the optimum measurement method for crack dimensions as applicable to space hardware. The penetrant materials evaluated are given in table 1.

### TABLE 1. LIQUID PENETRANT MATERIALS

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<thead>
<tr>
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<th>CLEANER</th>
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<tr>
<td>ZL-22</td>
<td>Fluorescent</td>
<td>ZP-9</td>
<td>ZC-7</td>
</tr>
<tr>
<td>ZL-44B</td>
<td>Fluorescent</td>
<td>ZP-45</td>
<td>ZC-7</td>
</tr>
<tr>
<td>P-149</td>
<td>Fluorescent</td>
<td>D-495A</td>
<td>Trichloroethylene</td>
</tr>
<tr>
<td>P-545</td>
<td>Fluorescent</td>
<td>D-495A</td>
<td>K-410</td>
</tr>
<tr>
<td>SKL-4</td>
<td>Dye</td>
<td>SKD-W</td>
<td>Demineralized Water</td>
</tr>
<tr>
<td>SKL-HF</td>
<td>Dye</td>
<td>ZP-4 or SKD-NF</td>
<td>SKC-NF</td>
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C. DEVELOPERS

After a crack has been filled with a penetrant, it is the purpose of the developer to overcome the equilibrium forces, draw the penetrant out of the crack, and spread the penetrant to magnify the surface opening of the crack. (See figure 3a.) There are several material characteristics that are very important in a good developer, such as particle size, efficiency of developer, contact angle, spreading ability, surface tension, and color. Manufacturers are aware of these factors and control the developer characteristics within narrow limits. Since the evaluator is assumed to use commercially available developers, he is primarily concerned about uniform application methods and thickness control.

Extreme care should be taken to ensure a uniform coating thickness of approximately 0.025 mm (0.001 inch). A developer that is too thick will absorb too much light and hinder observation of the penetrant. (See figure 3b.) Conversely, a developer that is too thin will not provide sufficient force to withdraw and spread the penetrant. Thus, a precise developer thickness control and application method was developed. Refer to Appendix C for the developer thickness control method used during this evaluation. The developers used in this evaluation are listed in table 1.

D. CLEANERS

Cleaners are used to remove the developers and liquid penetrants from the evaluation specimens. The types of cleaners for the developers and liquid penetrants are identified in table 1.

E. EVALUATION SPECIMENS

1. Type of Evaluation Specimens. Various types of materials have different surface energies; therefore, liquid penetrants behave differently on each surface. A good penetrant for a particular material will not necessarily be a good penetrant for other materials. Aluminum alloy blocks, 2219-T87, which were 7.6 cm (3.0 inches) long by 5.08 cm (2.0 inches) wide by 0.95 cm (0.375 inch) thick, were selected for this evaluation because of the wide use of this alloy in the Saturn V vehicle and other space hardware.
Figure 3. Developer Characteristics.

a. PENETRANT IN DEVELOPER

b. SCATTERING OF LIGHT PARTICLES IN THE DEVELOPER
2. **Condition of Evaluation Specimens.** The outcome of any liquid penetrant evaluation may be influenced by the condition of the evaluation specimen, including the dimensions and geometry of the cracks.

   a. **Surface Conditions.** The surface condition of the evaluation specimen is important because it influences the detectability of cracks visually after the liquid penetrant check. The evaluation specimens had a surface finish condition of 3.21 microns (125 microinches) root-mean-square average.

   b. **Dimensions of Crack.** The detection of cracks with the minimum dimensions is the goal of all liquid penetrant evaluations under a set of standard application, development, viewing, and analysis conditions.

   c. **Volume of Crack.** The volume of a crack is an important factor. Penetrant must enter a crack and later be pulled to the surface in sufficient quantity to be detectable. For a given material and crack volume, various penetrants have different detection capabilities; thus, selection of a penetrant should be the optimum available, based on laboratory and field evaluations.

   d. **Oxides and Nitrides.** Oxides and nitrides affect penetrability of liquid penetrants. These conditions produce higher surface energies and appear to aid penetration; however, they may also be detrimental by physically blocking a crack. Oxides and nitrides should be removed if they are suspected of blocking cracks.

   e. **Surface Contamination.** Contamination of the surface of the evaluation specimens by contaminants such as grease, dirt, and water tends to cause an increase in the contact angle which will lower the wetting ability of the liquid penetrant. Evaluation specimens should be thoroughly cleaned prior to the application of liquid penetrants.

F. **EQUIPMENT VARIABLES AND CHARACTERISTICS**

The equipment variables and characteristics must be considered if liquid penetrant evaluations are to be comparative. Optimum values should be selected and periodically verified throughout the evaluation. Most of the variables pertain to the black light (ultraviolet) sources, and several are discussed in the following paragraphs.
1. **Type of Black Light.** Mercury vapor (ultraviolet) black lights at a minimum of 100 watts each are considered the best choice for liquid penetrant evaluation.

2. **Filters.** Since ultraviolet black lights emit light of several frequencies, it must be filtered to pass only the usable wavelength of 3650 A.U. (See figure 4.) The most universal filter used is a dense red-purple (Kopp, or equivalent, filter) colored filter. This type filter effectively removes practically all visible light from the energy given off by the mercury arc. (See figure 5.) At the same time, this type filter removes most of the harmful shorter wavelengths and passes light in the range of 3200 to 3900 A.U. Also, nearly all fluorescent penetrants respond best to 3650 A.U. excitation and emit light in the yellowish-green spectrum. (See figure 6.)

3. **Intensity of Black Light.** Fluctuation in light intensity causes a corresponding fluctuation in the emitted light from fluorescent penetrants. Therefore, intensity of the filtered black light should be controlled and no less than 1076 lumen/meter\(^2\) (100 footcandles) at the point of inspection. The intensity may be measured with a Weston, Model 703, Type 3, light meter; a General Electric, Model No. 8DW40Y16, light meter; or equivalent. All evaluations should be made at the same intensity, with periodic checks for verification. If at any time the light is turned off during the evaluation, the evaluator should wait for 5 minutes after turning the light back on before he makes an evaluation. Similarly, any measurement with light meters should be made 5 minutes after the light is turned on.

4. **Photography.** The camera must be placed in the same viewing position as the evaluator. Any high-speed film should be sufficient; in this case type 58 Polaroid was used. Occasionally the lens should be cleaned of dust with a lens cloth.

5. **Viewing Room.** The viewing room should be as dark as possible.

G. **HUMAN FACTORS**

Since human factors play an essential part in the evaluation of liquid penetrants, it is imperative that steps be taken to ensure that human errors are minimized. The steps to be taken are given in the following paragraphs:
Figure 4. Spectrum of Output of High-Pressure Mercury Arc.
Figure 5. Transmission Curve of Black Light Filter Glass.
Figure 6. Emission Spectrum of Yellowish-Green Fluorescent Dye.
1. **Dark Adaptation of Eyes.** The human eye is a complex and adaptable organ. During a fluorescent penetrant inspection, the examination of the part is usually performed in a darkened area. The eye adapts to relative darkness by opening the pupil so more light may be admitted and adjusting chemically to see light of much less intensity than in normally illuminated rooms or in daylight. The human eye is a very sensitive receiver of light; in daylight it will perceive $2.8 \times 10^{-7}$ lamberts ($3 \times 10^{-4}$ foot-lamberts) of light, and while under conditions of full-dark it will perceive as little as $2.8 \times 10^{-10}$ lamberts ($3 \times 10^{-7}$ foot-lamberts). Therefore, it is very important to adapt the eyes to the light condition prior to any inspection. It has been determined that 30 minutes are required for full-dark adaptation of the eyes for an average person. A graphic representation for the perception of the human eye with decreasing illumination is shown in figure 7.

2. **Visual Acuity of Eyes.** The human eye, even in its nondark state, has no difficulty in perceiving brightness levels of $2.8 \times 10^{-6}$ lamberts ($3 \times 10^{-3}$ foot-lamberts) or greater. Most fluorescent indications exhibit brightness levels of approximately 0.046 lamberts (50 foot-lamberts). The seeability of indications is the degree of brightness contrast pertaining to the difference between the fluorescent indication and the background area around the indications. A brightness-to-background brightness ratio of 10 or more is considered sufficient for fluorescent inspection. The typical inspection booth area, which is properly draped and shielded from bright light, provides background brightness levels of about $9.3 \times 10^{-5}$ lamberts (0.1 foot-lambert) or less. Therefore, a person with good visual acuity should have no trouble seeing all but the smallest of indications. Color contrast dye penetrants are independent of background brightness, and their visibility increases with the intensity of illumination.

3. **Spectral Ability of Eyes.** Most fluorescent penetrants emit a yellowish-green light of approximately 5200 A.U. as shown in figure 6. The eyes of most persons perceive this color more readily than other colors; therefore, the evaluator should be able to perceive this frequency of light. Also, an evaluator should be free from color blindness to perform color contrast evaluations adequately.

**H. EVALUATION**

1. **Method.** Many approaches have been taken toward developing a method for evaluating liquid penetrants. Several of these methods presuppose one particular essential property of a good liquid.
Figure 7. Chart of Perception by the Human Eye.
penetrant and proceed to develop tests to measure that property. However, since the exact mechanism of liquid penetration into a crack is still not completely understood, these tests are subject to question. Some investigators have developed a variety of test specimens with simulated and actual cracks, but most of these are also subject to question. Any test method selected must include the actual application of the liquid penetrant since liquid penetration into a crack varies with composition and surface condition.

A simple method for evaluating fluorescent and dye penetrants was developed which employs blocks of 2219-T87 aluminum alloy that are heated and quenched in cold water to produce cracks. Like all other methods, this method also has its limitations. The cracks are of uncontrolled width and depth and only represent a single type of material and surface condition; however, a properly prepared evaluation specimen will yield a wide range of crack widths and depths.

The primary value of the aluminum alloy specimens is for comparison purposes. Absolute measurements can be made by determining the visible cracks per 2.54 cm (linear inch) and then sectioning the evaluation specimen for a magnified count of the cracks present. It will also provide a numerical method of assigning a number to penetrants for a particular evaluation specimen.

2. Procedure. The following general procedure was used to perform the sensitivity and comparison evaluation of the liquid penetrants. For a detailed procedure, refer to Appendix A.

a. Five evaluation specimens were selected, identified, and their surface finish determined. The specimens were heated over an open flame and then quickly quenched in water. The specimens were removed from the water, and heat was applied with a dry-air heat gun to remove the moisture from the cracks.

b. Each specimen was placed in a beaker containing enough trichloroethylene to cover the entire specimen. The specimen was vibrated with a sonic vibrator to ensure positive cleaning, then removed, and heat was applied to remove all traces of cleaner from the cracks.
c. The penetrant, developer, and cleaner to be evaluated were selected, and the penetrant was applied to the specimen. After 15 minutes penetrating time, the excess penetrant was removed with cleaner to eliminate false surface indications. The developer was then applied to the specimen, using the developer thickness control method of clear radiographic film around the periphery of the specimen (Appendix C).

d. Areas containing suitable numbers of cracks (20 to 25) along the top edge of the specimen were selected, and an area 2.54 cm (1.0 inch) in length was designated. The area was viewed at 9X magnification, and the penetrant indications (cracks) were counted.

e. A photograph of the top surface of each specimen with penetrant and developer was taken at normal size. The cleaner was then used to remove the developer, and another photograph was taken of the top surface of the entire specimen at normal size without the developer (fluorescent penetrants only).

**NOTE**

The lighting used for the fluorescent penetrants was ultraviolet with sufficient intensity for visibility of the X mark on the Uresco, or equivalent, inspectability scale. The non-fluorescent or dye penetrants were read with normal lighting sufficient for average reading.

f. The specimen was soaked in the penetrant cleaner, and then vibrated using a sonic vibrator until the penetrant was completely removed from the cracks in the specimen. The ultraviolet light was used to check removal of the fluorescent penetrants, and the developer was used to check the removal of the dye penetrants before the specimens were used for any subsequent penetrant evaluations.

M. The preparation and evaluation process outlined in paragraphs b through f was repeated for each specimen using the same penetrant. This process was used for all five evaluation specimens and the six liquid penetrants being evaluated, except the two dye penetrants which were photographed with the developer only.
h. Evaluation specimen Serial No. 2219-23 was selected from the group of five specimens for photographs of each of the six liquid penetrants at normal size and at 9X magnification of the 2.54 cm (1.0-inch) selected area. The one specimen selected for illustration in this document clearly indicates the evaluation method and data obtained.

i. The process outlined in paragraphs b through f was repeated for each of the six liquid penetrants on specimen Serial No. 2219-23, and photographs were taken each time at 9X magnification. The indications were counted and recorded from both the visual inspections at 9X magnification and the 9X magnification photographs; this included the four fluorescent penetrants with developers and without developers and the two dye penetrants with developers only. This direct visual comparison of penetrant sensitivities could be made by evaluation of the 10 photographs obtained. See figure 8 for typical 9X magnification photograph.

j. The 2.54 cm sections of the five specimens were cut out and identified.

k. The five specimen edges were then ground, and the face of each specimen was polished. A magnification of 1000X on the metallograph was used to count the number of actual cracks on each specimen, and the cracks were identified. Each crack depth was measured in mils and the width of the crack opening at the specimen surface was measured in microns.

l. The crack detection efficiency number was then calculated by dividing the observed cracks at 9X visual magnification by the actual cracks determined metallographically.

3. Results

a. The results of the sensitivity and comparison evaluation performed with the six liquid penetrant materials, using the quench-cracked aluminum alloy specimens, are illustrated in figures 8, 9, and 10, and tables 2 and 3.

b. As shown in table 2, false crack indications (7) were present with P-545 penetrant on specimens 21, 22, and 23 when developer was used; 3 false indications were present on specimen 22 when developer was wiped off.
1. Penetrant - P-545
2. Developer - D-495A
3. Cleaner - K-410
4. Photograph Exposure Time - 1 Minute
5. Light Source - Ultraviolet

Figure 8. Typical Evaluation Specimen, Serial No. 2219-23, 9X Magnification, (P-545 with Developer)
1. Penetrant - ZL-22
2. Developer - ZP-9 or ZP-45
3. Cleaner - ZC-7
4. Photograph Exposure Time - 1 Minute
5. Light Source - Ultraviolet

1. Penetrant - ZL-22
2. Without Developer
3. Cleaner - ZC-7
4. Photograph Exposure Time - 1 Minute
5. Light Source - Ultraviolet

1. Penetrant - ZL-44B
2. Developer - ZP-45
3. Cleaner - ZC-7
4. Photograph Exposure Time - 1 Minute
5. Light Source - Ultraviolet

1. Penetrant - ZL-44B
2. Without Developer
3. Cleaner - ZC-7
4. Photograph Exposure Time - 1 Minute
5. Light Source - Ultraviolet

Figure 9 Evaluation Specimen, Serial No. 2219-23
(Sheet 1 of 3).
1. Penetrant - P-149
2. Developer - D-495A
3. Cleaner - Trichloroethylene
4. Photograph Exposure Time - 1 Minute
5. Light Source - Ultraviolet

1. Penetrant - P-149
2. Without Developer
3. Cleaner - Trichloroethylene
4. Photograph Exposure Time - 1 Minute
5. Light Source - Ultraviolet

1. Penetrant - P-545
2. Developer - D-495A
3. Cleaner - K-410
4. Photograph Exposure Time - 1 Minute
5. Light Source - Ultraviolet

1. Penetrant - P-545
2. Without Developer
3. Cleaner - K-410
4. Photograph Exposure Time - 1 Minute
5. Light Source - Ultraviolet

Figure 9. Evaluation Specimen, Serial No. 2219-23 (Sheet 2 of 3).
1. Penetrant - SKL-HF
2. Developer - ZP-4 or SKD-NF
3. Cleaner - SKC-NF
4. Photograph Exposure Time - 7 Seconds

1. Penetrant - SKL-4
2. Developer - SKD-W
3. Cleaner - Demineralized Water
4. Photograph Exposure Time - 7 Seconds

Figure 9. Evaluation Specimen, Serial No. 2219-23 (Sheet 3 of 3).
Figure 10. Sensitivity and Comparison Evaluation.
### TABLE 2. LIQUID PENETRANT SENSITIVITY AND COMPARISON RESULTS - INDIVIDUAL SPECIMENS

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>Penetrant</th>
<th>Observed Cracks @ 9X - With Penetrants</th>
<th>Actual Cracks @ 1000 X</th>
<th>Sensitivity Index IS = Observed/Actual</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>W/dev</td>
<td>W/o dev</td>
<td>W/dev</td>
</tr>
<tr>
<td>21</td>
<td>ZL-22</td>
<td>17</td>
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<tr>
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<td>ZL-44B</td>
<td>20</td>
<td>19*</td>
<td>24</td>
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<tr>
<td></td>
<td>P-149</td>
<td>18*</td>
<td>26*a</td>
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</tr>
<tr>
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<td>P-545</td>
<td>28*b</td>
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<td>24</td>
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<td>-</td>
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<tr>
<td></td>
<td>SKL-4</td>
<td>14</td>
<td>-</td>
<td>24</td>
</tr>
</tbody>
</table>

*False Indication
a - 2 False Crack Indications (P-149, W/o dev)
b - 7 False Crack Indications (P-545, W/dev)
c - 3 False Crack Indications (P-545, W/o dev)
### TABLE 3. LIQUID PENETRANT SENSITIVITY AND COMPARISON RESULTS - TOTAL FOR ALL SPECIMENS

<table>
<thead>
<tr>
<th>Penetrant</th>
<th>All Specimens</th>
<th>All Specimens</th>
<th>Sensitivity Index</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Total Cracks</td>
<td>Total Actual</td>
<td>Total Ig (%)</td>
</tr>
<tr>
<td></td>
<td>W/dev</td>
<td>W/o dev</td>
<td>Cracks</td>
</tr>
<tr>
<td>ZL-22</td>
<td>87</td>
<td>91</td>
<td>118</td>
</tr>
<tr>
<td>ZL-44B</td>
<td>94</td>
<td>92</td>
<td>118</td>
</tr>
<tr>
<td>P-149</td>
<td>95</td>
<td>105*</td>
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<td>P-545</td>
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<td>107*</td>
<td>118</td>
</tr>
<tr>
<td>SKL-HF</td>
<td>61</td>
<td>-</td>
<td>118</td>
</tr>
<tr>
<td>SKL-4</td>
<td>92</td>
<td>-</td>
<td>118</td>
</tr>
</tbody>
</table>

*False Indications Included in These Figures (more observed cracks than actual cracks)*
c. False crack indications (2) were present with P-149 penetrant on specimen 21 when developer was wiped off.

d. Figure 10 indicates that four of the six penetrants have approximately the same crack detection efficiency (CDE) and could be used interchangeably depending on availability and specification requirements. Fluorescent penetrants ZL-44B, P-149, and dye penetrant SKL-4 give optimum sensitivity (79.7, 80.5, and 78 percent respectively) when used with developer. Fluorescent penetrant ZL-22 gave optimum results (77.1 percent) when developer was wiped off before evaluation.

e. Dye penetrant SKL-HF was consistently low in sensitivity (51.7 percent) compared to the other five penetrants which had sensitivity indexes no less than 73.7 percent.

SECTION III. CONCLUSIONS AND RECOMMENDATIONS

A. CONCLUSIONS

1. General

a. No single property of a liquid penetrant is dominant.

b. Liquid penetrants are capable of penetrating extremely small cracks. Other factors such as cleanliness, washing technique, and viewing technique are equally important in disclosing minute cracks.

c. The penetrant evaluation method discussed in this document will not be universally accepted because it describes a particular type of crack in a particular material of a specified surface finish.

d. Sensitivity of a penetrant, for the purposes of this evaluation, has been defined as the numerical results of the number of visual crack indications obtained at 9X magnification (with the penetrant
applied) divided by the actual number of cracks determined metallographically. The numerical results are quoted as the sensitivity index number of the penetrant.

e. The process of penetration into a crack has not been completely explained theoretically and verified by experimentation. It is probably a complex interaction of forces caused by both penetrant and material properties.

f. Several investigators have devised tests for evaluating penetrants that require the simultaneous consideration of several penetrant properties. However, the application of these approaches to actual cracks is not straightforward. The final application to an actual crack represents a considerable departure from simulated conditions. Nevertheless, some of the approaches appear promising.

g. Several investigators have undertaken to explain the process of penetration by relating it to an observable property of penetrants. Among them is the immersional free energy approach, which attempted to relate a theoretical factor called static penetrability parameter (SPP) to the crack detection efficiency (CDE) for several penetrants, but was not successful.

h. Other investigators maintain that penetration is based on the interaction of the penetrant with the walls of the crack and not merely their separation. They associate the flow of penetrant into the crack and along its walls with the process of spreading. The process of spreading depends on how much the wetting tension, \( \gamma_S - \gamma_{SL} \), exceeds the surface tension, \( \gamma_L \). There is no experimental evidence to verify this premise.

2. Specific

a. Based on the results shown in table 2 and Section II.H.3, fluorescent penetrant P-545 is too sensitive for the aluminum alloy and surface finish investigated herein. False indications were present, giving a crack count higher than actual cracks present. If, in an emergency, there is no other penetrant present, then evaluations with P-545 should be performed without developer (developer wiped off).
b. If fluorescent penetrant P-149 is used it should be evaluated with the developer; false crack indications were obtained when the developer was wiped off.

c. Of the six penetrants evaluated, four have approximately the same sensitivity and can be used interchangeably. Fluorescent penetrants ZL-44B, P-149, and dye penetrant SKL-4 gave optimum results when used with developer. Fluorescent penetrant ZL-22 gave best results when developer was wiped off.

d. Dye penetrant SKL-HF had consistently poor sensitivity.

B. RECOMMENDATIONS

1. It is recommended that the evaluations of liquid penetrants intended for use on aluminum alloys be performed utilizing the method discussed in Section II, paragraph H. The number of visible crack indications per 2.54 cm should be counted and divided by the number of actual cracks. This provides a numerical crack detection efficiency or sensitivity index number with which to compare penetrants. After obtaining the number of detectable cracks per 2.54 cm, the same specimen should be examined metallographically to ascertain the exact number of cracks present. The metallographic count should be made at 90 degrees to the direction in which the visible indications were made. Five evaluation specimens should be sufficient for the penetrant sensitivity and comparison evaluation.

2. The use of fluorescent penetrant P-545 is not recommended with the alloy and surface finish specified herein. It is too sensitive and consistently gives false crack indications in excess of actual cracks present. If used at all it should be evaluated without developer (developer wiped off).

3. If fluorescent penetrant P-149 is used with the alloy and surface finish specified herein, it is recommended that it be evaluated with developer only, otherwise false indications may be obtained.
4. It is recommended that fluorescent penetrant ZL-44B or P-149, or dye penetrant SKL-4 be used for evaluation of the 2219-T87 aluminum alloy of 3.21 micron (125 microinch) surface finish specified herein. These three penetrants should be used only with developer. Fluorescent penetrant ZL-22 is recommended for use also, but with the developer wiped off.

5. The use of dye penetrant SKL-HF is not recommended for evaluation of the alloy and surface finish specified herein. Crack sensitivity is not adequate.
REFERENCES


APPENDIX A

DETAILED EVALUATION PROCEDURE

A. OBJECTIVE

The objective of this detailed procedure is to provide an effective preparation and evaluation method for determining the sensitivity and making a comparison of liquid penetrants used on the Saturn V vehicle and other space hardware.

B. SCOPE

This procedure describes the sequence, methods, and equipment used to perform the sensitivity and comparison evaluation of liquid penetrants used on the Saturn V vehicle and other space hardware.

C. PROCEDURE

1. Evaluation Specimen Preparation. A total of five evaluation specimens shall be prepared from 2219-T87 aluminum alloy. These specimens shall be cut to 7.6 cm (3.0 inches) long by 5.08 cm (2.0 inches) wide by 0.95 cm (0.375 inch) thick and shall exhibit a surface finish condition of 3.21 microns (125 microinches) root-mean-square average.

2. Liquid Penetrants. A total of six liquid penetrants shall be used in this evaluation, as specified in table 1 of the report.

3. Evaluation Procedure. The following detailed procedure shall be used to perform the sensitivity and comparison evaluation of the liquid penetrants.

   a. Select the five evaluation specimens, determine their surface finish, and identify. Record information.

   b. Using a propane gas flame, heat the evaluation specimens to 1000°F, with the open flame impinging on the center of the underside. On the center of the topside, apply a 1000°F Tempilstik in
a circular motion to an area about the size of a dime. The heating shall be accomplished at a rate to cause the Tempilstik mark to melt in approximately 4 minutes.

c. As soon as the Tempilstik mark melts, quench the evaluation specimens in water. For best crack results, the water temperature should be between 68° and 72°F; knife edge the evaluation specimens into the water to one-half width of the specimens and then turn the specimens flat into the water. Remove evaluation specimens from water after quenching. Record information.

**NOTE**

The quenching process must be accomplished very quickly.

d. Select one specific evaluation specimen from the group of five specimens. Record identifying number of specimen.

e. Select penetrant, cleaner, and developer to be evaluated. Record information.

f. Using a heat gun, apply heat for 30 minutes at a temperature between 150° and 200°F to the evaluation specimen to remove moisture from cracks. Record information.

g. Place evaluation specimen in a beaker containing enough trichloroethylene to cover the entire evaluation specimen.

h. Using an ultrasonic cleaner, vibrate evaluation specimen for 1 hour; remove evaluation specimen from trichloroethylene. Record information.

i. Using a heat gun, heat evaluation specimen for at least 15 minutes to completely remove all traces of trichloroethylene; allow evaluation specimen to cool to room temperature. Record information.

j. Apply penetrant to evaluation specimen in accordance with manufacturer's recommendations; allow a minimum of 15 minutes penetrating time. Record information.

A-3
k. Using penetrant cleaner, thoroughly remove excess penetrant from evaluation specimen to eliminate false surface indications. Record information.

- 1. Using the developer thickness control method of clear radiographic film around the periphery of the evaluation specimen, apply developer to evaluation specimen and adjacent film. (Refer to Appendix C.) Allow 10 minutes developing time. Record information.

m. Using a standard radiograph densitometer, Macbeth-TD-102 or equivalent, measure clear radiographic film density and the density of the developer-coated film after the developer has been applied to the film and evaluation specimen. Record information.

n. Within developer time limits, select areas of precise crack density along top edge of evaluation specimen. Establish a 2.54 cm (1.0 inch) edge length where gage marks can be applied for metallographic purposes. Make suitable gage marks perpendicular to specimen edge and place a centerpunch mark in from the edge 1.59 mm (0.0625 inch). Count penetrant indications (cracks) between gage marks at 9X visual magnification. Record crack indications.

o. Photograph entire top surface of evaluation specimen in color at normal size. Record information.

**NOTE**

Lighting for the fluorescent penetrants should be ultraviolet with sufficient intensity for observing at the X mark on the Uresco, or equivalent, inspectability scale. The X mark should be readily visible down to 30 percent relative brightness. The nonfluorescent or dye penetrants should be read with normal lighting sufficient for average reading.

p. For the fluorescent penetrant evaluation remove developer from specimen with cleaner. Record information.
q. Photograph entire top surface of evaluation specimen in color at normal size without developer (fluorescent penetrants only). Record information.

r. Soak evaluation specimen in cleaner for a minimum of 8 hours. Record information.

s. Place evaluation specimen in a beaker containing enough cleaner to cover the entire evaluation specimen.

t. Using ultrasonic cleaner, vibrate evaluation specimen for 2 hours or until the penetrant is completely removed from the cracks. Record information.

**NOTE**

Use the ultraviolet light for checking removal of fluorescent penetrants and the developer for checking removal of dye penetrants.

u. Repeat operations outlined in paragraphs C. 3. d through C. 3. t for each of the remaining specimens using the same penetrant, developer, and cleaner.

v. Select one typical evaluation specimen from the group of five for photographs of each of the six liquid penetrants at 9X magnification of the 2.54 cm (1.0 inch) selected edge length (between gage marks).

**NOTE**

Evaluation specimen, Serial No. 2219-23, was selected from the group of five specimens. The selection of one specimen for photographs will give an adequate evaluation sample for the record. This method will save time in cleaning processes, camera focal setups, and cost of color film.

w. Repeat operations outlined in paragraphs C. 3. e through C. 3. t using evaluation specimen, Serial No. 2219-23,
and the six penetrants. All photographs will be of the 2.54 cm (1.0 inch) selected edge length at 9X magnification.

**NOTE**

The crack indications shall be counted and recorded by both visual inspection at 9X magnification and from the 9X magnification photographs; this includes the four fluorescent penetrants with developers and without developers and the two dye penetrants with developers only. By using this process, direct visual comparison can be made of the penetrant sensitivities.

x. Using an abrasive cutting wheel, cut out the 2.54 cm (1.0 inch) section of the five evaluation specimens between the gage marks allowing 3.2 mm (0.125 inch) on the outer side of each gage mark. Identify the five specimens and record the information. Mount the specimens in transparent plastic per standard metallographic specimen preparation procedures.

y. Select one of the five specimens for metallographic inspection. Record specimen identification number.

z. Using a belt sander, grind off 1.59 mm (0.0625 inch) of material from specimen edge. (This will be at the center of the centerpunch marks, between which the visual crack count was taken at 9X magnification). Polish specimen down to 1.0 micron finish.

**NOTE**

The surface which was penetrant checked is at right angles to the surface being ground.

aa. Using a magnification of 1000X on the metallograph, count the number of cracks between the gage marks on the evaluation specimen. Record information.

ab. Identify cracks from left to right, viewing edge of evaluation specimen with gage marks on top. Record information.
ac. Measure individual crack depth in mils from top surface of evaluation specimen to crack tip. Record information.

ad. Measure individual crack width in microns at crack opening in the top surface of evaluation specimen. Record information.

ae. Calculate crack detection efficiency number for all six penetrants on each of the five specimens by dividing the observed cracks between gage marks at 9X visual magnification by the actual cracks determined metallographically.

NOTE

Ensure that the serial number of the evaluation specimen inspected metallographically matches the serial number of the evaluation specimen inspected at 9X visual magnification.
APPENDIX B

DATA SHEETS
FIGURE B-1. CRACK DETECTION.
APPENDIX C

DEVELOPER THICKNESS CONTROL
APPENDIX C

DEVELOPER THICKNESS CONTROL

Use a commercially available light density measuring device such as an X-ray densitometer. Perform the spraying operation with the part placed over and as close as possible to a clear glass plate or clear photographic film (developed unexposed photo film). The spray will coat the part and glass or photofilm to the same depth. See figure C-1 for illustration of this technique. Test with various spray applications until a thickness is applied which can be determined by simple mechanical inspection tools such as micrometers and depth gages. Place the coated glass plate or photo film on the densitometer and record the indicated number. Compare the reading with a zero reading taken on the glass plate or photo film prior to application of sprayed film. The net reading will be the accurate density reading. Perform sufficient tests to enable the preparation of a graph of densitometer readings versus coating thickness which can be used for actual final spray application of the thin film.

The method just described can be automated by developing a mechanical or electrical control which would shut off the film applicator at the desired densitometer reading. This could be an adjustable limit switch setup on the densitometer so as to be activated when the indicator needle reached the desired reading, at which point the switch would shut off the film applicator.

Another control could be electrical, whereby when the voltage created from the light density measuring mechanism was sufficient to cause the needle to deflect to the desired reading, a cutoff switch would activate and shut down the applicator. This voltage would have to be known from calibration tests and an adjustable dial used to set it.

While the technique explained above is most applicable to a manually operated film spray device, used with a contact type densitometer, such as the usual X-ray densitometers, it could conceivably be developed for an automated operation where a continuous density reading light meter device would be employed.

Figure C-2 is a plot of typical densitometer versus thin film thickness readings obtained using an X-ray type densitometer and liquid penetrant developer as the thin film.
Figure C-1. Technique of Controlling Developer Thickness Using Clear Glass Plate or Clear Photographic Film.
Figure C-2. Typical Densitometer Reading Versus Thin Film Thickness Reading.
APPROVAL

SENSITIVITY AND COMPARISON EVALUATION
OF
SATURN V LIQUID PENETRANTS

By

G.H. Jones

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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