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HIGH TEMPERATURE ELASTOMERIC INTEGRAL FUEL TANK SEALANT EVALUATIONS

TECHNICAL DOCUMENTARY REPORT NO. ML-TDR-64-207

September 1964

Air Force Materials Laboratory
Research and Technology Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

Project No. 7340, Task No. 734005

(Prepared under Contract AF33(657)-10962, by The Boeing Company, Airplane Division, Wichita Branch, Wichita, Kansas; Lyle G. Middleton, author)

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FOREWORD

This report was prepared by The Boeing Company, Airplane Division, Wichita Branch, under AF33(657)-10962. The work was initiated under Project No. 7340, "Nonmetallic and Composite Materials", Task No. 734005, "Elastomeric and Compliant Materials". The contract was administered under the direction of the Nonmetallic Materials Division, Air Force Materials Laboratory, Research and Technology Division, with T. L. Graham acting as project engineer.

This report covers the work conducted from 15 April 1963 to 15 July 1964.

This program was conducted under the technical direction of Mr. Charles E. Johnson, the supervisor of the Sealants, Adhesives, and Plastics Group of the Materials Technology Unit. The work was conducted by Lyle G. Middleton, Loyd H. Church, Wayne Brainerd, and Joe Minge.

ABSTRACT

Several experimental sealants were evaluated to determine their suitability for sealing fuel tanks of supersonic aircraft exposed to temperatures up to 650°F. Sealants were initially screened by standard material specification test methods, followed by the evaluation of promising sealants in small titanium simulated fuel tanks. These tanks were subjected to realistic fuel tank environments in conjunction with cyclic structural loading at temperatures up to 450°F on a dynamic test device developed under a previous Air Force contract.

A total of five sealants were evaluated in five tanks. A proprietary sealant (EC-2288) was considered marginal for sealing fuel tanks exposed to 300°F. An experimental fluorocarbon sealant prepared by the Air Force Materials Laboratory was found to be unsatisfactory for sealing fuel tanks exposed to 450°F. This sealant was modified by AFML and evaluated in another tank at 450°F. Although an improvement was noted, the sealant was still judged to be unsatisfactory. An experimental fluorosilicone sealant (Q-95-500) was considered satisfactory for fillet sealing fuel tanks exposed to temperatures up to 450°F. Another fluorosilicone sealant (Q-2-0046) was found unsuitable for sealing faying surfaces of fuel tanks exposed to 450°F.

This technical documentary report has been reviewed and is approved.

J. M. KELBLE, Chief
Elastomers and Coatings Branch
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INTRODUCTION

Fuel tanks of current subsonic (and some supersonic) aircraft are generally sealed with polysulfide based sealants which have a service temperature limit of about 275°F. Fuel tanks of supersonic aircraft will be subjected to temperatures ranging from -65°F up to +650°F, which is generally believed to be beyond the temperature range capabilities of currently available fuel tank sealants.

The objective of this research program is to evaluate new experimental or commercial sealants to determine their suitability for sealing fuel tanks of supersonic aircraft exposed to temperatures in the range of -65°F to +650°F. All sealants evaluated under this program were specified and furnished by the Air Force Materials Laboratory (AFML). Table 1 is a list and description of each sealant evaluated.

The evaluation of sealants was divided into two phases:

Phase I - Laboratory Evaluation of Sealants

Phase II - Tank Evaluation of Sealants on Dynamic Test Device

The Phase I laboratory evaluations were performed to become familiar with the properties and to determine the temperature capabilities of each sealant. Standard sealant evaluation procedures based upon current sealing material specifications (modified as necessary) were used. Specimens were exposed to liquid fuel at temperatures up to 400°F, and to fuel vapor and nitrogen at temperatures up to 500°F. Pertinent physical properties were measured before and after such exposures at temperatures from -65°F to +550°F.

It is believed that the best approach to date for determining whether a promising sealant will actually seal a fuel tank in a given system, is to use the proposed sealant to seal a small scale fuel tank. The sealed tank is then exposed to simulated fuel tank environments (fuel, temperature extremes) and cyclic structural loading. Under Phase II, five small titanium tanks were sealed with the sealants from Phase I. These tanks were environmentally conditioned at the maximum temperature extremes determined from Phase I, and load cycled on the Dynamic Test Device developed under another contract (Reference A.).

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PHASE I

LABORATORY EVALUATION OF SEALANTS

A. General

1. Laboratory evaluations were conducted initially when each sealant was received in order to become familiar with the basic physical properties of each material and to determine their temperature capabilities. This information was needed to set the temperature limits (upper and lower) that each sealant could be expected to withstand in the Phase II fuel tank evaluations.

2. The following standard sealant test methods provided the basis for the laboratory evaluation of each sealant:

   a. Low Temperature Flexibility (MIL-S-8802B and MIL-S-8802C).

   b. Peel Strength (MIL-S-8802C).

   c. Tensile Strength and Elongation (ASTM D412).

3. In addition to the three test methods listed above, a Tensile-Shear Strength Test was devised to evaluate sealants intended for the faying surface method of sealing. Several other test methods were developed to evaluate specific properties of certain of the sealants. These test methods are described in Section B.

4. Initial samples of the AFML fluorocarbon sealant were received packaged in standard paint cans. It was found that these samples had very short storage stability and were partially polymerized when received. Samples were subsequently shipped frozen in standard Semco sealant cartridges. This material was kept frozen (-40°F) until it was ready to use, and no further storage stability problems were evident. The material, when thawed to room temperature, was easy to work with and to apply. There were no difficulties due to short work life, high viscosity and/or flow. When applied to titanium panels the sealant did not immediately wet the substrate; however, good initial tack was obtained after the sealant was in contact with the substrate for a few minutes.

B. Test Procedures and Results

1. General

a. All titanium was vapor degreased in trichloroethylene, acid etched (nitric-hydrofluoric), and finally hand cleaned with methyl-ethyl-ketone (MEK) prior to applying sealant.

b. Unless otherwise specified, the sealant specimens were cured as follows:

(1) EC-2288: 4 hours at 200°F, plus
2 hours at 300°F.

(2) Q95-500: 10-14 days at 77±2°F and 50±5% relative humidity.

(3) AFML Fluorocarbon Sealant:
168 hours at 77±2°F and 50±5% relative humidity, plus

48 hours at 120°F, plus

8 hours at 150°F, plus

24 hours at 200°F, plus

91 hours at 250°F, plus

24 hours at 300°F.

The above curing conditions are not intended to represent optimum cure conditions. These cure times were used to insure that the sealants were completely cured; however shorter cures may be suitable.

c. All specimens were environmentally exposed to fuel and heat using the Environmental Test Device illustrated in Figure 1. This test device is suitable for conducting liquid fuel exposures at temperatures up to the boiling point of the fuel (392°F in the case of the F63-18 fuel used herein), and fuel vapor exposures (inerted with nitrogen) at temperatures up to 650°F. The liquid fuel exposures were conducted with the specimens immersed in fuel in the environmental chamber heated by a fire-brick insulated electric oven. To conduct fuel vapor exposures all but a small portion of fuel was forced into the fuel reservoir from the environmental chamber, using nitrogen pressure. The system was generally operated at just enough positive nitrogen pressure to exclude air (oxygen). Fuel vapors were condensed by a water condenser and returned to the environmental chamber. The entire system, including the electric oven, was continuously purged with a small amount of nitrogen.

d. The fuel used for all environmental exposures is a special aviation kerosene blend similar to MIL-J-5624 JP-5. The fuel was furnished by AFML and was labeled F63-18. Figure 2 is an ASTM distillation curve of this fuel.

2. Low Temperature Flexibility per MIL-S-8802B

a. Initial testing of the first two sealants evaluated, EC-2283 and Q-2-0046*, was conducted using the MIL-S-8802B Low Temperature Flexibility Test using MIL-T-7993 class II titanium panels (1.0 x 6.0 x .040 inches).

b. Six panels were coated with a layer of EC-2288 on one side of each panel to obtain a cured sealant thickness of 0.15-0.17 inches. Sealant was cured 20 hours at 77°F, plus 4 hours at 200°F, plus 2 hours at 300°F (followed by an additional 5 days at 77°F prior to environmental conditioning).

c. Six panels were primed with RTV1200 (thin brush coat) and dried 30-60 minutes. A layer of Q-2-0046 was applied to one side of each panel to obtain a cured sealant thickness of approximately 0.08 inches. Specimens were cured 3 days at 77°F plus 4 days at 140°F (followed by an additional 3 days at 77°F prior to environmental conditioning).

d. The specimens were exposed per Table 2 (3 specimens of each sealant per condition).

e. Specimens were then cooled to -65°F and bent over a 2-inch radius flexibility jig (as specified in the MIL-S-8802B Low Temperature Flexibility Test).

f. Results are tabulated in Table 2.

*EC-2288 was the first sealant received for evaluation. Q-2-0046 was a promising fluorosilicone previously evaluated by Boeing for high temperature fuel containment. Evaluation tests were initiated to compare these two sealants.

3. <u>Low Temperature Flexibility per MIL-S-8802C</u>

a. MIL-S-8802C low temperature flexibility specimens were prepared using 2.875 x 6.0 x .040 inches MIL-T-7993 titanium panels. Sealant thicknesses ranged from .06 to .10 inch. AFML fluorocarbon sealant specimens (Figure 3) and EC-2288 specimens were prepared by injection in a closed mold (AFML fluorocarbon sealant was removed from the mold for curing). Q95-500 specimens were prepared in an open face mold. Sealants were cured per B.1.b.

b. EC-2288 and Q95-500 specimens were exposed to Table 3 Conditions as follows:

EC-2288: Conditions 1 and 2 (3 specimens each)

Q95-500: Condition 1 (1 specimen)
Conditions 5 and 9 (2 specimens each)

c. AFML fluorocarbon sealant was exposed to the conditions specified in Table 4.

d. All specimens were subjected to 130 cycles on the MIL-S-8802C Low Temperature Flexure Tester. All specimens were tested at -65°F, except the AFML fluorocarbon sealant which was tested as specified in Table 4.

e. All EC-2288 and Q95-500 specimens passed with no specimens cracking or losing adhesion. The AFML fluorocarbon sealant specimens failed as shown in Table 4 and Figure 4.

4. <u>Peel Strength</u>

a. Peel strength specimens were based on the MIL-S-8802C Peel Strength Test, using 1.0 x 6.0 x .040 inches MIL-T-7993 titanium panels.

b. Five EC-2288 and six Q95-500 peel strength specimens were prepared and exposed per Table 5 (one specimen per condition). Satisfactory peel strength specimens could not be prepared with AFML fluorocarbon sealant, due to poor adhesion to the wire screen tab.

c. Specimens were peel tested per MIL-S-8802C at 77°F at a jaw separation rate of 2 inches per minute.

d. Results are tabulated in Table 5.

5. Tensile Strength and Elongation

a. Tensile Strength and Elongation Specimens were based on ASTM D412 (Reference B) using die C (1/2 scale) specimens prepared from cast or injection molded sheets of sealant of .100-.125 inches in thickness. Figure 3 shows injection molded sheets of AFML fluorocarbon sealant after curing.

b. Specimens were exposed to the conditions and tested at the temperatures shown in Table 6 (3 specimens per condition). Jaw separation rate was 20 inches per minute.

c. Average test values (including Rex hardnesses) are presented in Table 6.

6. Tensile-Shear Strength

a. In order to evaluate the sealants in a manner which simulates their usage as faying surface sealants, tensile-shear specimens (Figures 3 and 5) were used. These specimens were made from 1 inch wide "L" sections, fabricated from 6Al-4V titanium extrusions (this is the same material used in the titanium tanks in Phase II).

b. In addition to the AFML fluorocarbon sealant, tensile-shear specimens (Figure 5) were prepared with several fluorosilicone sealants in an effort to evaluate a suitable fluorosilicone faying surface sealant. Fasteners were torqued to 20 inch-pounds and specimens were cured 14 days at 77±2°F and 50±5 percent relative humidity. One to three specimens of each sealant were exposed to the conditions specified in Table 7.

c. Specimens were tested in tension at a jaw separation rate of 1 inch per minute (fasteners removed) at the temperatures indicated in Table 7.

d. Results are presented in Table 7.

7. Tear Resistance of EC-2288

a. Tensile strength and elongation control specimens were bent 180 degrees over 1/8 inch diameter glass rods and retained in this position. The specimens were then "nicked" slightly with a knife on the outside radius.

b. When heated to 140°F these specimens spontaneously tore completely through within 2 hours.

8. Evaluation of Q95-500 As An Injection Sealant

a. Sealant was injected into five, 3/16 inch I.D. by 2.0 inches long, primed aluminum tubes and cured 14 days at 77°F and 50% relative humidity.

b. The tubes were exposed to Table 3 Conditions as follows:

Condition No. 1 (two tubes)

Condition No. 6 (two tubes)

Condition No. 7 (one tube)

c. Tubes were pressure tested at 77°F by applying 10 psi air pressure to one end.

d. All tubes leaked upon application of pressure. Sealant had cured for a length of about .25 inches in the ends of each tube, the interior being uncured. Tubes exposed to fuel and heat showed extrusion of uncured sealant which was forced out the ends of the tubes (apparently due to thermal expansion).

9. Flow Tests

Each sealant was evaluated qualitatively to determine its resistance to flow when applied as fillets from standard Semco sealant cartridges. EC-2288 flowed excessively, making the forming of satisfactory fillets in one application impossible. Q95-500 and the AFML fluorocarbon sealant could be readily formed into fillets which held their shape satisfactorily.

C. Summary of Results

1. EC-2288:

   a. has satisfactory low temperature flexibility at -65°F after liquid and vapor fuel exposures at 300°F, but not at 450°F.

   b. hardens excessively (70 Rex durometer) when exposed to vapor fuel exposures at 350°F to 400°F.

   c. has marginal to unsatisfactory tensile strength (values less than 100 psi) when tested at 350°F or higher after liquid and vapor fuel exposures above 300°F.

   d. has unsatisfactory to marginal percent elongation (values less than 100) when tested at 300°F or higher.

   e. has poor adhesion to titanium after immersion in salt water.

   f. has very poor tear resistance at temperatures above 140°F.

   g. has excessive flow, making application and forming of fillets very difficult.

2. Q95-500:

   a. has satisfactory low temperature flexibility at -65°F after liquid and vapor fuel exposures up to 500°F.

   b. has satisfactory room temperature peel strength (at least 20 lb./in.) after liquid and vapor fuel exposure cycles at 450°F and 500°F (some reversion was observed in the centers of peel panels exposed to these temperatures). Peel strength is also satisfactory after salt water exposures at 140°F.

   c. generally has satisfactory tensile strength and elongation (values above 100) when tested at 450°F, after liquid and vapor fuel exposures up to 450°F.

   d. has marginal to unsatisfactory tensile strength and elongation (values less than 100) when tested at 500°F after liquid and vapor fuel exposures up to 500°F.

e. is unsatisfactory in enclosed applications because it will not cure completely. Also thick sections (above .5 inch) will not cure readily. It is unsatisfactory in faying surface seals--it appears to cure adequately but depolymerizes when exposed to fuel and heat.

3. AFML Fluorocarbon Sealant:

a. has the following low temperature flexibility properties after liquid and vapor fuel exposure cycles up to 450°F and 500°F:

(1) marginal (slight cracking of fillets) at -20°F.

(2) failing (major cracks and loss of adhesion) at -30°F.

b. has unsatisfactory tensile strength and elongation (values less than 100) when:

(1) control specimens are tested at 450°F - 550°F.

(2) specimens exposed to liquid and vapor fuel cycles up to 450°F are tested at 450°F.

(3) specimens exposed to liquid and vapor fuel cycles up to 500°F are tested at 500°F.

c. has virtually no tear resistance when tested at elevated temperatures (450°F) after the above exposures.

d. has marginal tensile-shear strength when specimens are tested at 450°F - 500°F after liquid and vapor fuel exposures up to 500°F.

4. Fluorosilicones Evaluated As Faying Surface Sealants

a. Q-2-0046 has satisfactory low temperature flexibility at -65°F after liquid and vapor fuel exposures up to 450°F. In addition, Q-2-0046 was the only fluorosilicone sealant tested that did not depolymerize (revert) in a faying surface (tensile-shear strength test) after liquid and vapor fuel exposures up to 450°F.

Discussion of Results

1. EC-2288 has suitable physical properties for usage as a faying surface sealant; however the low viscosity causes excessive flow when used as a filleting sealant. This sealant has a maximum fuel environmental temperature resistance of about 300°F. Above this temperature the material hardens excessively, resulting in unsatisfactory elongation and low temperature flexibility. The material has very poor tear resistance at temperatures as low as 140°F. After discussing these results with AFML, the decision was made to seal one Phase II test tank with EC-2288 used both as a faying surface and a filleting type sealant for fuel tank exposures to 300°F.

2. Q95-500 has satisfactory curing properties only when used where a major portion of its surface is exposed to air (such as in filleting sealing). It does not cure adequately in enclosed or very thick applications, since it requires air and moisture to properly cure. This sealant appears to be suitable for exposures to fuel tank environments up to 450°F (including at least 332 hours in fuel vapor at 450°F). A small amount of depolymerization (softening, reversion to an uncured state) was obtained in the centers of .25 inch thick peel strength specimens exposed to 450°F fuel temperatures, indicating some degradation at this temperature. However the sealant was well cured and appeared undamaged in the outer .125 inch thick layer of sealant. After exposures to fuel vapor temperatures at 500°F, depolymerization was somewhat more severe, and tensile strength and elongation properties were considered marginal to unsatisfactory. The decision was made to fillet seal one tank with Q95-500, and to evaluate this tank at an upper temperature of 450°F and a lower temperature of -65°F.

3. The AFML fluorocarbon sealant has satisfactory physical properties for usage as a filleting or faying surface sealant. When the frozen sealant is thawed to room temperature, the viscosity is low enough to make satisfactory applications with standard sealant injection guns, yet the sealant does not flow but remains in the applied configuration very well. Work life was more than adequate, and a stepped heat cure to 300°F is specified to accomplish a complete cure. The sealant does not readily wet a surface to which it is applied, and consequently several minutes are required to pick up adequate adhesion. The sealant was evaluated after liquid fuel exposures at 350°F, followed by fuel vapor exposures in the 450°F - 500°F temperature range. The sealant is considered unsatisfactory for exposures to these temperatures. The sealant hardened excessively, resulting in poor elongation, and marginal low temperature flexibility as high as -20°F. The sealant has essentially no cohesive strength (tear resistance) at 450°F - 500°F after the above mentioned exposures. Even control specimens (receiving only the specified step cure to 300°F) have unsatisfactory tensile strength and elongation when tested at 450°F - 550°F. These results indicate that this sealant would not be satisfactory in a fuel tank at 450°F. However, the degree of correlation between laboratory specimen test results and the evaluation of a sealant in a simulated fuel tank is still subject to some speculation; consequently the decision was made to seal two tanks with the AFML fluorocarbon sealant--one tank to be faying surface sealed and the other to be fillet sealed. In order to compare this sealant with Q95-500, the specified upper temperature exposure was the same as for Q95-500 (450°F).

4. Q-2-0046, although subjected to only a minimum amount of testing, appeared promising as a faying surface sealant for fuel tanks exposed to temperatures up to 450°F. However, there was not enough time left in the contract period to conduct further evaluation tests on this sealant.

PHASE II

TANK EVALUATION OF SEALANTS ON DYNAMIC TEST DEVICE

A. General

1. The original design for the tanks was based on aluminum tanks evaluated under a previous Boeing-Air Force research contract (Reference A). Optimum design of the tank required that it be large enough to simulate actual fuel tank structure and stress loads, and at the same time small enough to be economical to evaluate. The tank design is a semi-monocoque box beam of 6Al-4V titanium sheet skins and webs, and extruded titanium chords and stiffeners. The end plates and test jig attach fittings are 4130 steel. Drawings for the test tanks are in the Appendix. Photographs of the tank structure, obtained during the assembly of one tank, are presented in Figures 6 through 9. Overall dimensions are approximately 9 by 14 by 50 inches. This type and size of tank is believed to be the most economical and the least complex construction with which significant and reliable results may be obtained.

2. The following five tanks were sealed and evaluated:

Tank No.	Sealant	Method of Sealing	See Figure No.
1	EC-2288	Fillet and Faying Surface (in separate areas)	10
2	AFML Fluorocarbon	Fillet	14
3	AFML Fluorocarbon	Faying Surface	15
4	Q95-500	Fillet	14
5	AFML Fluorocarbon (modified)	Fillet and Faying Surface (in all areas)	Figures 14 and 15 combined

B. Sealing of Test Tanks

1. General

a. All test tank detail parts were fabricated per the appended drawings. All parts were vapor degreased in trichloroethylene. Titanium parts were then acid etched in nitric-hydrofluoric acid. Steel parts for the first three tanks were descaled in nitric acid. Steel parts for the last two tanks were sandblasted. All steel parts were then spray-painted with an epoxy coating (Reference C) for corrosion protection.

b. All parts were assembled using chromate passivated, cadmium plated, steel lockbolts with silver plated monel collars.

c. All parts to be sealed were hand cleaned with methylethylketone (MEK) immediately prior to applying sealant.

d. After each tank was sealed and cured, it was leak tested at room temperature by applying 5 - 10 psi air pressure to the tank interior, and then brushing the exterior with a soap bubble solution to indicate leakage.

2. Sealing of Tank No. 1 with EC-2288

a. EC-2288 was mixed on a Semco SP-1350 sealant mixer, extruded into polyethylene sealant cartridges, and stored at -40°F.

b. The tank was sealed using both faying surface and fillet type seals in separate areas, as shown in the Figure 10 cross-sectional view of the tank. Note that fasteners were fillet sealed in fillet sealed joints but not in faying surface sealed joints. The center rib and the ends were completely faying surface and fillet sealed (including fasteners) as shown in Figures 12 and 13.

Faying surface seals were applied during the tank assembly to the areas shown in Figure 10. In addition the spar web stiffeners and the access door stiffeners were faying surface sealed. Sealant was applied to one faying surface using a rubber r er to roll on a continuous coating of sealant. Ma..ng surfaces were joined and all fasteners installed within three hours after thawing the sealant. Faying surface "block-off" seals (approximately 1 inch wide) were applied during tank assembly to the mating surfaces which were not faying surface sealed as shown typically in Figure 17. This was to prevent traveling leaks in areas to be subsequently fillet sealed. The tank access door was also completely faying surface sealed.

d. Fillet seals were applied during and after the tank was assembled to the areas shown in Figure 10. Fillets were applied by injecting thawed sealant from polyethylene cartridges fitted with nozzles cut to form the desired fillet shape. Fillets on fasteners were formed with a fairing tool. Because of the high flow of EC-2288 due to its low viscosity, it was necessary to build up fillets in two or more applications to obtain the desired fillet size. Final fillet dimensions approximated those shown in Figure 11. However it was observed later that fillet dimensions over the tops of fasteners were less than Figure 11 due to the high flow of EC-2288.

e. The completely sealed tank was cured 4 hours at 200°F followed by 2 hours at 300°F.

f. The tank was then pressure tested per Section B.1.d. No leakage was observed.

3. Sealing of Tank No. 2

a. This ..nk was completely fillet sealed with the AFML fluorocarbon sealant (Figure 14). The frozen sealant cartridges were thawed to room temperature and fillets were applied using the equipment shown in Figure 16. Fillets were applied in two applications. A small first fillet was applied, followed by the final full bodied fillet using the nozzle shown in Figure 16. Final fillet dimensions were per Figure 11. Fasteners were sealed by running a continuous fillet over a row of fasteners, or by applying a ball of sealant to a fastener head and pressing it against the structure with flared tubing tools (see Figure 16). In a few areas fasteners were brush coated with sealant thinned with MEK, prior to applying the fillets. Final sealant fillet dimensions were per Figure 11.

b. Faying surface "block-off" seals (approximately 1 inch wide) were applied during tank assembly as shown in Figure 17, to prevent traveling leaks. The tank ends were completely faying surface and fillet sealed as shown in Figure 13. The access door was also completely faying surface sealed. Faying seals were applied by smoothing the sealant out with a spatula to form a continuous coating of sealant on one mating surface. The faying surface was assembled and all fasteners installed within 2 hours. Four injection seals were also used on the access door of each tank as shown in Figure 9.

c. Details of the sealing of Tank No. 2 are shown in Figure 17 (tank partially assembled), Figure 18 (center rib area), and Figure 19 (tank assembled except for access door).

d. The sealant was cured as follows:

77°F for 3-7 days,
20-40% relative humidity, plus
100°F for 3 hours, plus
120°F for 24 hours, plus
160°F for 65 hours, plus
200°F for 7 hours, plus
250°F for 17 hours, plus
300°F for 4 hours.

The access door was not installed during the above curing.

e. The tank was then pressure tested for leaks per Section B.1.d. There was considerable seam leakage on the top and bottom skins. For leakage evaluation, each skin (and also the spar web) can be divided into 8 fillet sealed seam lengths (each about 10 inches long), isolated from each other by faying surface "block-off" seals. These 8 seam lengths can be seen in Figure 17 as the lengths between block-off seals, before the lower skin is installed. The upper and lower skins were leaking as follows:

Lower skin - 7 out of the 8 seam lengths leaking

Upper skin - 6 out of the 8 seam lengths leaking

f. To find the source of these leaks on the tank interior the tank was leak tested by the vacuum method. About 1 gallon of water was placed in the tank, a transparent (plexiglas) door was sealed in place where the access door is normally installed, and 2 psi vacuum was pulled on the tank. Leakage was indicated by air bubbles coming through the water from the source of a leak. Only about half of the areas leaking were traced to their source by this method.

g. Some additional leak sources were located by applying air pressure locally to the tank exterior and soap bubble solution locally to the tank interior.

h. Most of the leaks were caused by poor adhesion of sealant fillets over fasteners. The leaks located were repaired by applying new sealant over the areas leaking. There were three areas where the source of the leaks could not be located. These areas were "repaired" by applying additional brush coats of sealant over the fastener lines.

i. The sealant was cured as follows: Sixty-six hours ambient conditions, plus 2 hours at 120°F, 2 hours at 160°F, 1 hour at 200°F and 2 hours at 250°F.

j. The tank was pressure tested again, and leaks were again obtained:

Lower skin - 4 out of 8 seam lengths leaking

Upper skin - 2 our of 8 seam lengths leaking

Spar web - 1 out of 8 seam lengths leaking

k. The tank was again leak tested by the vacuum method:

Lower skin - found leak source in 1 of 4 areas leaking, and found 2 leaks in areas not leaking.

Upper skin - no leak sources found, 1 fillet leak found on center rib.

Spar web - no leak sources found.

l. All leaks were repaired with new applications of sealant. It was believed that the source of the leaks in the four leaking areas (where no leak source could be found) was the fasteners in these areas. For this reason the AFML fluorocarbon sealant on the approximately 60 fasteners in these four areas was removed, and two fluorosilicone sealants were applied. Two adjacent areas in the upper skin were sealed with Q-2-0046, and the other two adjacent areas on the lower skin were sealed with Q95-500. These fillet seals were applied to meet Figure 11 dimensions. These leaking areas were all adjacent to the access door on the upper and lower skins. The two fluorosilicone sealant applications were cured 7 days at ambient conditions.

m. The tank was pressure tested again and the lower skin was still leaking in the area where the Q95-500 sealant had been applied. The leak was subsequently traced to its source, which was a leaking fillet of AFML fluorocarbon sealant in the area of the center rib. This fillet was repaired.

n. The access door was then installed with a faying surface seal. The sealant was cured by exposing the tank to the following cure cycle: Four to five days at ambient conditions, plus 110°F for 24 hours, 130°F for 3-1/2 hours, 160°F for 2 hours, 200°F for 2 hours, 250°F for 16-1/2 hours, and 300°F for 3 hours.

4. <u>Sealing of Tank No. 3</u>

a. This tank was completely faying surface sealed with the AFML fluorocarbon sealant (Figure 15). Thawed sealant was applied to one faying surface from a Semco cartridge and the surface was completely covered with a thin continuous coating using a spatula (Figure 16). The mating surface was applied and fasteners installed. All assembly was completed within about four hours after thawing the sealant.

b. In addition to the faying surface seals, fillets were installed in the ends of the tank. since these areas were not properly designed for faying surface seals alone (faying seals were not wide enough, gaps between parts were too large). Figures 7, 8, and 9 show three views of this tank.

c. The tank was cured together with Tank No. 2 per Section A.3.d.

d. The tank was pressure tested per Section B 1.d. There was no leakage.

5. Sealing of Tank No. 4

a. This tank was completely fillet sealed with Q95-500 (Figure 14). Fillets were applied in two applications to meet Figure 11 dimensions, using the equipment shown in Figure 16. All fasteners were fillet sealed. All parts to be sealed were primed with a thin coat of RTV 1200 primer (Dow Corning) which was dried at least 30 minutes prior to applying the sealant.

b. All fillets on the spar only were sealed with Lot E108-68, while the remainder of the tank was sealed with Lot E108-92. The spar was sealed with the same lot of sealant which was observed to revert in Tank No. 2 (see Section C.2.e.). When this reversion was obtained in Tank No. 2, a new lot of sealant was obtained to seal the remainder of the tank. Typical fillets of Q95-500 are shown in Figure 20.

c. Q-2-0046 was used for faying surface "block-off" seals and for faying surface sealing the tank ends and the access door. This sealant was evaluated in Phase I and appeared promising for faying surface sealing, since it was the only fluorosilicone evaluated which did not revert in a faying surface when exposed to fuel and heat. Faying seals were applied as previously specified in Section B.3.b.

d. The sealant was partially cured at 75-78°F as follows:

Sealant	Location of Sealant	Days Cure	Percent Relative Humidity
Q-95-500	Spar	21	20-35
		plus 28	35-55
Q-95-500	Lower Skin	28	35-55
Q-95-500	Upper Skin	21	35-55
Q-2-0046	"Block-Off" seals and Attach Fitting Faying Seals	21-48	20-55
Q-2-0046	Access Door	5*	35-55

*Humidified air circulated through tank continuously.

In addition to the above room temperature cure, the entire tank received the following heat cure after the access door was installed:

Hours	Temperature	Percent Relative Humidity on Tank Interior
24	100°F	70*
24	120°F	90*
24	138°F	90*
48	138°F	No air circulation**
24	300°F	Dry air circulation

* Humidified air circulated through tank continuously.
** Due to failure of plant air supply over week end.

e. Before installing the access door, the tank was pressure tested per Section B.1.d., after the Q-95-500 sealant had cured at least 7 days. Several fastener leaks were found and were caused by small pinholes in sealant fillets over fasteners. These were repaired and the curing of the sealant was continued. After the access door had been installed and the sealant had cured 5 days at 77°F, the tank was again pressure tested and two faying surface leaks on the access door were found. These were repaired by applying a fillet of Q-2-0046 on the tank exterior. There were no other leaks.

6. Sealing of Tank No. 5

a. This tank was both faying surface and fillet sealed in all areas with the modified AFML fluorocarbon sealant. This sealant contained additional Viton B to increase tensile strength and elongation. The sealant was initially shipped without freezing and when received the extrusion rate was too low to use. It was determined that the sealant was apparently beginning to cure during shipment and storage at room temperature. Subsequent lots were shipped and stored frozen. The sealant was thawed in a 140°F water bath until its temperature was slightly above room temperature. By thawing in this manner the extrusion rate was high enough to use.

b. Faying surface seals were applied during the tank assembly as detailed in Section B.4. for Tank No. 3. Fillet seals were applied as detailed in Section B.3. for Tank No. 2, except that a brush coat of sealant thinned with MEK was used in all areas to be fillet sealed. This was done in order to promote better wetting of the heavier, modified AFML sealant, which was slower to wet the tank structure than the AFML sealant used previously. In addition all fasteners were sealed individually with a fillet over each fastener.

c. The sealant was partially cured at 75-78°F and 40-58 percent relative humidity as follows:

Location of Sealant	Lot No. of Sealant	Days Cure
Spar	185A	11
Upper Skin	185B 187A	7
Fillets in Tank Ends	187A	5
Lower Skin	185D 185E	1

The access door was installed and the tank was heat cured as follows with humidified air circulating through the tank:

Hours	Temperature	Percent Relative Humidity in Tank Interior
24	100°F	70
24	120°F	90
24	160°F	90
24	200°F	No Air Circulation*
24	250°F	No Air Circulation*
24	300°F	25

*Due to failure of plant air supply over week end.

d. The tank was pressure tested per Section B.1.d. and no leakage was observed.

C. Environmental Conditioning of Tanks

1. Tank No. 1 (EC-2288)

a. This tank was filled with AFML test fuel F63-18 and conditioned at 300°F for 24 hours.

b. All but approximately 2 quarts of fuel was removed and the tank was exposed to fuel vapor at 300°F for 24 hours.

c. The tank was pressure tested. No leakage was observed.

2. Tank No. 2 and No. 3 (Sealed with AFML Fluorocarbon Sealant)

a. These two tanks were filled with AFML test fuel F63-18 and conditioned at 350°F for 24 hours.

b. All but approximately two quarts of fuel was removed from each tank. The tanks were purged with nitrogen and placed in an oven. It was planned to condition the tanks at 450°F for 24 hours. However due to difficulties encountered in raising the oven temperature to 450°F, the following conditioning was actually used:

400 - 410°F for approximately 4 hours
410 - 440°F for approximately 16 hours
450°F for 20 hours.

Tanks were at atmospheric pressure.

c. Following the conditioning of these two tanks, both were cooled to room temperature and pressure tested. The tanks were leaking badly on both ends (around the end plates).

(1) Tank No. 3 (faying surface sealed) had a few fastener leaks on two attach fittings, and two injection seal leaks (see Figure 9) on the access door, but there was no leakage in the center areas where the major faying surface seals were to be evaluated.

(2) Tank No. 2 (fillet sealed) was leaking badly:

Lower skin - 3 out of 8 seam lengths leaking

Upper skin - 3 out of 8 seam lengths leaking

Spar web - 2 out of 8 seam lengths leaking

Access door - 4 out of 4 injections leaking.

d. The access door was removed from Tank No. 2 and approximately 15 cracks were observed on the fillets throughout the tank, as shown in Figures 21, 22, and 23. The sealant fillets were hard and somewhat embrittled, with very littl elongation or tear strength (this correlated with the results of the Phase I tests). The sealant in this tank was considered to have failed badly due to the environmental exposures used, and no additional testing was conducted.

e. The fasteners in Tank No. 2 that had been sealed with the two fluorosilicone sealants (Q-2-0046 and Q95-500) were badly reverted (sticky, gummy) as shown in Figures 23 and 24.

3. <u>Tank No. 4 and No. 5</u>

a. These two tanks were completely filled with AFML test fuel F63-18 PF-1 and conditioned at 350°F for 24 hours.

b. All but approximately 2 quarts of fuel was removed from each tank. The tanks were positioned with the ram ends (ends adjacent to the test device ram) down and conditioned at atmospheric pressure for 24 hours at 450°F. Nitrogen was continuously circulated through each tank during this exposure.

c. Following the conditioning of these two tanks, both were cooled to room temperature and pressure tested.

d. Tank No. 4 was leaking as follows:

Lower Skin	- 1 out of 8 seam lengths leaking
Upper Skin	- 1 out of 8 seam lengths leaking
Spar Web	- 2 fasteners leaking at center rib
Access Door	- 27 fasteners leaking
Attach Fittings	- 18 seam leaks 27 fastener leaks
Tank Ends	- both ends leaking badly around end plates.

e. Examination of the sealant through the access door plumbing fittings revealed no reversion or degradation. However, some loss of adhesion of fillets of Q-95-500 was observed on the steel end plates which were coated with an epoxy coating. Extrusions of Q-2-0046 sealant were observed around the attach fittings and end plates. This extrusion was apparently caused by thermal expansion of cured sealant, and/or extrusion of uncured sealant from the faying surface. This extrusion of Q-2-0046 sealant, together with the loss of adhesion of Q-95-500 sealant to the epoxy coating, are believed to be the reasons for the leaks in the tank ends and around the attach fittings. The reason for the access door fastener leaks (sealed with Q-2-0046) was not apparent. Although the reasons for the leaks found in the upper and lower skins and the spar web were not apparent, these leaks were minor and the tank was considered suitable for testing on the dynamic test device.

f. Tank No. 5 exhibited only one leak--an air bubble in a fillet on the tank exterior adjacent to the access door. This leak was not significant and this tank was considered ready for testing on the dynamic test device.

D. Evaluation of Sealed Tanks on Dynamic Test Device

1. Description of Dynamic Test Device

a. The Dynamic Test Device (Figure 25) was developed under a previous Boeing-Air Force research contract (Reference A). The test device is capable of applying torsional and/or bending loads to the integral test tank at temperatures ranging from -100°F to +800°F. Torsional loads are applied by mounting a tank horizontally in the test jig as a cantilever beam, with one end firmly attached to the steel frame and the other end subjected to dynamic loading using a hydraulic ram. Cyclic loading is accomplished by a timer which operates a valve causing the ram to cycle.

b. To evaluate a tank at elevated temperatures, an insulated chamber attaches to the test jig, completely enclosing the test tank. The chamber is a stainless steel cylinder, consisting of two half-sections with fiberglass insulation, as shown in Figure 26. Heating of the tank is accomplished by blowing hot air between the tank and the insulated chamber.

c. To evaluate a tank at low temperatures, cold fuel is circulated through the tank. The fuel is cooled to the desired temperature by pumping it through a coil immersed in a mixture of trichloroethylene and dry ice.

General Procedure For Dynamic Evaluation of Tanks

a. The Dynamic Test Device is capable of applying both bending and torsional loads. However, to insure a wide distribution of stress loads in the tank structure, and to induce greater joint deflections, only the torsional loading capability was used in the tank evaluations. Stress calculations were made to determine the torsional loads necessary to produce permanent deformation in the tank structure. The load level which would just produce permanent deformation was called the test tank "yield point". It was believed that a realistic dynamic evaluation of the sealants in the tanks would be obtained by dynamic loading of the tanks at a load level approximating 90 percent of the "yield point". This load level was used to evaluate each tank, and the actual torsional loads used are presented in Table 8 as a function of the tank temperature during dynamic cycling.

b. In general, the tanks were exposed to 1000 cycles at the upper service temperature (from Phase I), followed by exposure to 1000 cycles at the minimum service temperature (from Phase I). Each cycle consisted of loading the tank from zero load to the desired load level in one direction, back to zero and to the desired load level in the opposite direction. Loading rate was 6 cycles per minute unless otherwise specified.

c. Each tank tested at elevated temperatures contained approximately 2 quarts of fuel. The tank was pressurized to 5 psi with nitrogen and continuously purged during dynamic cycling. The tank was cooled to room temperature for pressure (leak) testing per Section B.1.d., since it could not be tested for leaks until the insulated chamber surrounding the tank was removed.

d. Each tank tested at low temperatures was completely filled with JP-4 fuel which was circulated at -65°F. No pressure was applied during dynamic cycling. After the completion of dynamic cycling the tank was pressurized to 10 psi and examined for fuel leaks.

3. <u>Dynamic Evaluation of Tank No. 1 (Sealed with EC-2288)</u>

a. This tank was initially cycled for 50 cycles at 77°F (empty) at a load level of 270,000 inch-pounds (Table 8), to check out the operation of the Dynamic Test Device. Leakage was obtained as shown in Table 10.

b. The tank was subsequently subjected to 1000 cycles at 300°F per Section D.2.c. at a load level of 210,000 inch-pounds.

<u>NOTE</u>: This load level is less than that specified in Table 8 for a tank tested at 300°F, because, at the time, it was believed the Table 8 values were in error; however, this was not found to be the case.

(1) Considerable leakage was obtained (Table 10). The tank access door was removed and it was determined that most of the leaks, with the exception of those in the access door, were caused by insufficient sealant over fasteners (due to the flow of EC-2288). To trace the leaks to their source on the tank interior, air pressure was applied locally to the tank exterior, and soap solution was brushed on the sealant inside the tanks. This method indicated that pinholes or small air bubbles in the thin layer

c ... ant over the tops of fasteners, were causing the leaks. The fillets over fasteners were built up with new applications of EC-2288 to meet the fillet dimensions of Figure 11.

(2) The reason for the leaks in the access door was not too clear. The access door on this tank had been divided in the middle, making two separate access doors. This may have caused the mating faying surfaces to work (more in relation to each other) excessively during dynamic cycling, and together with the hardening of the sealant due to the environmental exposures, considerable leakage resulted when the tank was cycled at 300°F.

(3) The slight leakage in the ends of the tank was repaired by applying sealant to the tank exterior in the areas leaking.

(4) The access door was reinstalled and the sealant cured.

c. The tank was then subjected to 1000 cycles at -65°F per Section D.2.d. at a load level of 280,000 inch-pounds (Table 8). Subsequent pressure testing at -65°F produced no fuel leakage.

4. Dynamic Evaluation of Tank No. 3 (Faying Sealed with AFML Fluorocarbon Sealant)

a. This tank was subjected to 1000 cycles at 450°F per Section D.2.c. at a load level of 210,000 inch-pounds. After pressure testing at 77°F per Section B.1.d. (with the tank loaded in both directions per Table 8), leakage was obtained as follows in the faying surface sealed areas of the tank:

Location		Number of Seam Leaks	Number of Fastener Leaks
Upper skin		14	71
Lower Skin		29	47
Spar web		26	39
	Total	69	157

Several individual seam leaks were actually continuous leaks for several inches of seam length. In addition to the above leaks, the leaks reported after the Section C. environmental conditioning were still present (leaks around end plates, fastener leaks on attach fittings, and injection leaks in access door). The access door leaked at approximately 80 percent of the total seam length, and most of the fasteners were also leaking.

b. The cause of the leaks reported above is believed to be due to the poor physical properties of the sealant at 450°F as summarized in Section D.3. of the Phase I laboratory evaluations.

5. Dynamic Evaluation of Tank No. 4 (Fillet Sealed with Q-95-500)

a. This tank was tested as specified for tank No. 3 in Section D.4.a. Leaks were obtained as follows:

Area of Tank	Seam Lengths Leaking After Environmental Conditioning (Section C.3.d.)	New Seam Lengths Leaking After Dynamic Cycling	Total Seam Lengths Leaking (Out of 8 Total Per Area)
Upper skin	1	1	2
Lower skin	1	1	2
Spar web	0	2	2

In addition to the above leaks three fastener leaks were found in the area of the center rib (two previously leaking fasteners on the spar web, and one new leak on the lower skin).

b. The access door, attach fittings and tank ends, which were leaking badly after environmental conditioning, were now much worse with many seams and fasteners leaking in these areas. The apparent causes of these leaks were discussed previously in Section C.3.e.

c. The access door was removed and the tank interior was examined. The Q-95-500 fillets in general were in excellent condition. Leakage reported above in paragraph 5.a. was generally traced to fillets over fasteners. Some fillets over fasteners were removed and found to be undersize (less than the thickness specified by Figure 11.). In addition some of these fillets contained small pinholes and/or void spaces which were apparently large enough to cause leakage. Some of this type of fastener leakage was found when the tank was first pressure tested after sealing (Section B.5.e.). The reason all of these inadequately sealed fasteners did not leak earlier is not clear, unless the subsequent environmental conditioning caused a small amount of shrinkage which resulted in leakage of a few inadequately sealed fasteners (it should be emphasized that all of the leakage obtained could have been caused by as few as 6 to 10 leaking fasteners). The leaks were not believed to be due to degradation of the sealant, as the sealant appeared to be completely undamaged by the environmental conditioning and dynamic cycling. However, in the ends of the tank the Q-95-500 fillets exhibited loss of adhesion to the epoxy coating, and some reversion of Q-95-500 was observed where it was in contact with reverted or uncured Q-2-0046 which extruded from the faying surfaces (after the Q-95-500 fillets had been applied).

d. Normally, the next step with this tank would be to dynamically test it filled with fuel at -65°F. However, the major leakage in the tank ends made it impossible to fill the tank with fuel. The Q-95-500 appeared to be undamaged by the environmental conditioning and dynamic cycling at 450°F. Since this sealant was found to have excellent low temperature flexibility in Phase I, it is considered probable that the sealant would not have failed if it could have been dynamically cycled at -65°F.

6. <u>Dynamic Evaluation of Tank No. 5</u> (Fillet and Faying Surface Sealed with Modified AFML Fluorocarbon Sealant)

a. This tank was tested as specified for tank No. 3 in Section D.4.a. Leaks were obtained as follows:

Location		Number of Seam Leaks	Number of Fastener Leaks
Upper skin		39	17
Lower skin		11	3
Spar web		16	4
	Total	66	24

Several of the above individual leaks were actually continuous seam leaks for several inches of seam length.

b. In addition to the above leaks, the access door contained in excess of 43 seam leaks and at least 19 fastener leaks. Both ends of the tank were leaking generally around both end plates, and the attach fittings contained 13 seam leaks and 10 fastener leaks.

c. The access door was removed and the sealant examined. There were no cracks in any of the fillets and the sealant generally appeared to be in good condition, except for pinholes and void spaces (air bubbles) in some fillets. After the tank was removed from the test device, attempts were made to determine the cause of the extensive leakage. Bubble solution was brushed on the interior fillets and air pressure was applied locally to the tank exterior. This method was generally not successful in pointing to the causes of the leakage. Pinholes on the fillet surfaces and void spaces (air bubbles) in fillets were believed most likely to have caused the fillet leaks, particularly over fasteners where the sealant thickness was smaller. Also the marginal adhesion of this sealant to fasteners and to the structure around the base of fasteners was believed to be another cause of the leakage. A better method of applying sealant over fasteners is badly needed. Air bubbles in fillets over seams were believed to be due mainly to entrapped air from the injection gun during application of the fillets (these air bubbles apparently expand considerably during subsequent heat exposures).

d. Fillets of the modified AFML fluorocarbon sealant appeared to be in better condition than was the original AFML fluorocarbon sealant used to fillet seal tank No. 2. The sealant was softer and appeared to have better elongation, although the tear strength was very poor.

E. Summary and Conclusions

1. A summary of the results of the environmental conditioning of each tank is presented in Table 9. The results of the dynamic cycling evaluations are summarized in Table 11.

2. Tank No. 1, fillet and faying surface sealed with EC-2288, passed the environmental conditioning and dynamic cycling evaluations with no major leakage directly attributable to degradation of the sealant. Although there was no major leakage after exposure to fuel tank environments at 300°F and -65°F, this sealant is considered marginal at best for sealing fuel tanks exposed to these temperatures. The physical properties of EC-2288 (especially the tear strength) are generally low compared with those of currently used polysulfide sealants, and the temperature advantage of EC-2288 at best is only 50°F higher than polysulfides. In addition the flow is much too great for a filleting sealant.

3. Tank No. 2, fillet sealed with the AFML fluorocarbon sealant, leaked badly after environmental conditioning at 450°F. Sealant fillets were badly cracked consequently evaluation of this tank was terminated. It was concluded from the Phase I laboratory evaluations of this sealant that it would fail in a tank that was exposed to fuel and heat at 450°F, and then stressed at 450°F. It is believed that the cracking of the fillets were probably the result of internal stresses in the sealant, caused by shrinkage of the sealant and leaching action of the fuel. The sealant from the tank was hard and had very poor cohesive (tear) strength and elongation. The sealant is considered unsatisfactory for fillet sealing of fuel tanks exposed to 450°F.

4. Tank No. 3, faying surface sealed with the AFML fluorocarbon sealant, passed the environmental conditioning with no leakage in the faying surface sealed areas. However, after dynamic cycling at 450°F the tank was leaking extensively and generally in all faying surfaces. It was concluded from the Phase I laboratory evaluations that this sealant was unsatisfactory for the environmental exposures used, due to the poor physical properties of the sealant at 450°F. This is believed to be the reason for the extensive leakage obtained when this tank was stressed at 450°F. The poor physical properties (tensile-shear and tear strength, elongation, and adhesion) of the sealant at 450°F resulted in sealant failure (rupture). This sealant is considered unsatisfactory for faying surface sealing of fuel tanks exposed to 450°F.

5. Tank No. 4, fillet sealed with Q-95-500 sealant, was leaking in 25 percent of the exterior seam lengths after environmental conditioning and dynamic cycling at 450°F. However, the sealant was in excellent condition and appeared to be undamaged. The cause of the leakage was traced to small holes or void spaces in sealant fillets applied over fasteners and, in a few cases, to an inadequate thickness of sealant over fasteners. Thus it is evident that the leakage of this tank was not the fault of the sealant, but was caused by an inadequate method of sealing fasteners. The Q-2-0046 faying surface sealant used in the tank ends and access d[illegible] either reverted badly or did not cure adequately, and subse[illegible]tly extruded from the faying surfaces, causing extensiv[illegible]kage of the tank ends and access door. It is thus co[illegible]d that:

   a. Q-95-500 is satisfactory for fillet sealing fuel tanks exposed to temperatures up to 450°F. It is believed that this sealant will adequately seal fasteners if larger fillets are applied and a better application method is developed.

   b. Q-2-0046 is unsatisfactory for faying surface sealing fuel tanks exposed to 450°F.

6. Tank No. 5, fillet and faying surface sealed with AFML fluorocarbon sealant (modified), passed the environmental conditioning with no significant leakage. After dynamic cycling at 450°F the tank exhibited extensive seam leakage and moderate fastener leakage. The fillets of AFML fluorocarbon sealant (modified) generally appeared to be in good condition except for some pinholes and void spaces, and some marginal adhesion of fillets, particularly over fasteners. The AFML fluorocarbon sealant (modified) appeared to be in better condition than was the original AFML fluorocarbon sealant used in tank numbers 2 and 3. There were no fillet cracks, the sealant appeared to be softer and to have better elongation; however, the tear strength was very poor. The leakage of fillets appeared to be due in part to the method used to apply the sealant, which resulted in some pinholes and air bubbles in the fillets, and poor adhesion of fillets over fasteners (a better method of applying the sealant over fasteners is needed). The leakage of the faying surfaces (and also some of the fillets) was believed to be due to the poor physical properties (tensile-shear and tear strength, elongation, and adhesion) of the sealant when tested at 450°F. The AFML fluorocarbon sealant (modified) is considered unsatisfactory for fillet sealing and/or faying surface sealing of fuel tanks exposed to 450°F.

TABLE 1

LIST OF SEALANTS EVALUATED

A. EC-2288, Minnesota Mining and Manufacturing Co., formerly designated XS-1293646. It is a gray two-part, 100 percent solids, chemically-curing sealant of an unspecified polymer type. A heat cure to 300°F is required.

B. AFML Fluorocarbon Sealants. These are high solid content sealant systems containing a curing agent which can be activated by moisture or heat. The compositions of these experimental sealants are as follows:

Component	Parts By Weight AFML Fluorocarbon Sealant (1)	Parts By Weight AFML Fluorocarbon Sealant (modified)(2)
Viton B	40	45
Viton LM (extracted)	60	55
Thermax	15	15
Maglite Y	15	15
FS 1265 (1000 CS)	8	8
MEK	20	20
Epon H-2	2.5	1.5

(1) Used in Phase I, and in Phase II Tank Numbers 2 and 3.
(2) Used in Phase II Tank Number 5.

C. Q-95-500, Dow Corning Corporation. This is a one-part fluorosilicone sealant with a moisture-sensitive curing agent. A room temperature cure of 10 to 14 days is specified. A primer (RTV 1200) is also specified to promote adhesion.

D. Q-2-0046, Dow Corning Corporation. This sealant is similar to Q-95-500. It was used only as a faying surface sealant in conjunction with fillet seals of Q-95-500.

TABLE 2

MIL-S-8802B LOW TEMPERATURE FLEXIBILITY TEST RESULTS

Environmental Exposure	Test Results (3 specimens per condition)	
	EC-2288	Q-2-0046
Immersion in liquid fuel at 300°F for 168 hours.	One specimen cracked. Hardness increased from 42 to 65 Rex.	All specimens passed. (35-40 Rex)
Immersion in liquid fuel at 300°F for 168 hours, plus 5 exposure cycles per [1]	All 3 specimens cracked [2] Hardness increased from 42 to 75-80.	All specimens passed. (35-40 Rex)

[1] Immersion in fuel at 300°F for 21 hours, followed by fuel vapor and nitrogen at 450°F for 2 hours.

[2] One specimen cracked at -65°F; the other two cracked later (18-50 hours) at room temperature. The bent specimens apparently broke spontaneously due to residual stresses.

Fuel was F63-18 fuel from AFML.

TABLE 3

ENVIRONMENTAL EXPOSURE CONDITIONS
FOR LABORATORY EVALUATION OF SEALANTS

Condition No.	Environmental Exposures [1]
1	None (control specimens)
2	Immersion in liquid fuel at 300°F for 24 hours, followed by fuel vapor and nitrogen at 300°F for 24 hours.
3	Immersion in liquid fuel at 300°F for 24 hours, followed by fuel vapor and nitrogen at 350°F for 24 hours.
4	Immersion in liquid fuel at 300°F for 24 hours, followed by fuel vapor and nitrogen at 400°F for 24 hours.
5	Immersion in liquid fuel at 350°F for 24 hours, followed by fuel vapor and nitrogen at 450°F for 24 hours.
6	Two exposures per Condition No. 5.
7	Two exposures per Condition No. 5 plus exposure to fuel vapor and nitrogen for an additional 284 hours at 450°F.
8	Immersion in liquid fuel at 350°F for 24 hours, followed by fuel vapor and nitrogen at 500°F for 24 hours.
9	Immersion in liquid fuel at 400°F for 24 hours, followed by fuel vapor and nitrogen at 500°F for 24 hours.
10	Immersion in liquid fuel at 140°F for 168 hours.
11	Immersion in liquid fuel (saturated with water) at 140°F for 168 hours.
12	Immersion in 3 percent aqueous sodium chloride at 140°F for 168 hours.
13	Dry heat at 350°F for 24 hours.

[1] Fuel was AFML Test Fuel F63-18.

TABLE 4

MIL-S-8802C LOW TEMPERATURE FLEXIBILITY TEST RESULTS - AFML FLUOROCARBON SEALANT

Table 3 Condition No.	Environmental Exposure	Number of Environmental Exposure Cycles	Number of Specimens Tested [2]	Test Temp. °F	Number of Specimen Failures
5	Immersion in liquid fuel at 350°F for 24 hours, followed by fuel vapor and nitrogen at 450°F for 24 hours.	1	3	-20°F	1, cracked slightly
				-30°F	3, [1]
		2	3	-20°F	3, cracked slightly
				-30°F	3, [1]
8	Immersion in liquid fuel at 350°F for 24 hours, followed by fuel vapor and nitrogen at 500°F for 24 hours.	1	3	-10°F	None
				-20°F	1, cracked slightly
				-30°F	3, [1]
		2	3	-10°F	None
				-20°F	None
				-30°F	2, [1]

[1] On first few bending cycles, large sections of sealant cracked and separated adhesively from panels.

[2] Each set of 3 specimens was progressively tested at lower temperatures unitl major failures were obtained.

TABLE 5
PEEL STRENGTH TEST RESULTS
(All Specimens Were Tested At 77°F)

Table 3 Condition No.	Environmental Exposure	EC-2288		Q95-500	
		Average Peel Strength, lb. per in.	Percent Cohesive Separation	Average Peel Strength, lb. per in.	Percent Cohesive Separation
1	None (control specimens)	67	100	46	100 △3
4	Immersion in liquid fuel at 300°F for 24 hours, followed by fuel vapor and nitrogen at 400°F for 24 hours.	△1	---		
5	Immersion in liquid fuel at 350°F for 24 hours, followed by fuel vapor and nitrogen at 450°F for 24 hours.	---	---	22 △4	100
6	2 exposures per Condition No. 5.	---	---	22 △4 △5	100
7	2 exposures per Condition No. 5 plus exposure to fuel vapor and nitrogen for an additional 284 hours at 450°F.	---	---	22 △4	100
9	Immersion in liquid fuel at 400°F for 24 hours, followed by fuel vapor and nitrogen at 500°F for 24 hours.	---	---	20 △4	100
10	Liquid fuel at 140°F for 168 hours.	46 △2	100		
11	Liquid fuel (saturated with water) at 140°F for 168 hours.	48 △2	100		
12	3 percent aqueous sodium chloride at 140°F for 168 hours.	47 △2 △6	25	>25 △7	100

△1 Sealant brittle, separates from wire screen, poor tear strength (70 Rex hardness).

△2 Poor tear strength.

△3 Also on MIL-S-5059 Type 302 stainless steel panel.

△4 Some reversion in center of panel (soft and somewhat gummy).

△5 Center portion pulled 10-24 lb. per in.

△6 Peel strength during cohesive separation only.

△7 Sealant separated from wire screen.

Fuel is F63-18 Fuel from AFML.

TABLE 6
TENSILE STRENGTH AND ELONGATION TEST RESULTS

Table 3 Condition No.	Environmental Exposure	Test Temperature	EC-2288 Average Tensile Strength psi	EC-2288 Average Percent Elongation	EC-2288 Rex Hardness @ 77°F	Q95-500 Average Tensile Strength psi	Q95-500 Average Percent Elongation	Q95-500 Rex Hardness @ 77°F	AFML Fluorocarbon ▷1 Average Tensile Strength psi	AFML Fluorocarbon ▷1 Average Percent Elongation	AFML Fluorocarbon ▷1 Rex Hardness @ 77°F
1	None (control specimens)	77°F 300°F 350°F 400°F 450°F 500°F 550°F	1400 390 382 378	254 59 86 55	50-64	515 31	467 173	40	524 23 22 20	367 83 50 50	68-70
2	Immersion in liquid fuel at 300°F for 24 hours, followed by fuel vapor and nitrogen at 300°F for 24 hours.	77°F 300°F	771 376	106 53	64						
3	Immersion in liquid fuel at 300°F for 24 hours, followed by fuel vapor and nitrogen at 350°F for 24 hours.	77°F 350°F	951 49	153 27	70						
4	Immersion in liquid fuel at 300°F for 24 hours followed by fuel vapor and nitrogen at 400°F for 24 hours.	77°F	760	90	70						
5	Immersion in liquid fuel at 350°F for 24 hours followed by fuel vapor and nitrogen at 450°F for 24 hours.	77°F 450°F				574 124	225 50	55	522(479) 0(0)	63(67) 41(29)	72(75)
6	Two exposures per Condition No. 5.	77°F 450°F				560 145	233 150				
7	Two exposures per Condition No. 5 plus exposure to fuel vapor and nitrogen for an additional 284 hours at 450°F.	77°F 450°F 500°F				417 111 85	170 117 100	53			
8	Immersion in liquid fuel at 350°F for 24 hours, followed by fuel vapor and nitrogen at 500°F for 24 hours.	77°F 500°F							489(430) 0(0)	68(25) 10(0)	78(79)
9	Immersion in liquid fuel at 400°F for 24 hours, followed by fuel vapor and nitrogen at 500°F for 24 hours.	77°F 500°F				481 48	270 47	55			
13	Dry heat at 350°F for 24 hours.	77°F 450°F 500°F				662 83 108	358 88 100	43			

▷1 Values in parentheses are after two cycles at the environmental exposure indicated.

Fuel was F63-18 Fuel from AFML.

Each value is an average of 3 specimens.

TABLE 7
TENSILE-SHEAR STRENGTH TEST RESULTS

Table 3 Condition No.	Environmental Exposure	Number of Exposure Cycles	Test Temperature °F	Average Tensile-Shear Strength, psi				
				AFML Fluorocarbon	Q95-500	Q-2-0046	Q94-002	Q94-501
1	None (control specimens)	-	77°F		188	195	93	149
5	Immersion in liquid fuel at 350°F for 24 hours, followed by fuel vapor and nitrogen at 450°F for 24 hours.	1 2	77°F 450°F 77°F	203 34 146	45 [1]	210	48 [1]	25 [1]
8	Immersion in liquid fuel at 350°F for 24 hours, followed by fuel vapor and nitrogen at 500°F for 24 hours.	1	77°F 500°F	198 40		184		
9	Immersion in liquid fuel at 400°F for 24 hours, followed by fuel vapor and nitrogen at 500°F for 24 hours.	1	77°F		44 [1]			

[1] Sealant depolymerized (soft and gummy).

TABLE 8

TEST TANK CALCULATED TORSIONAL LOAD LEVEL VERSUS TEMPERATURE

Test Tank Temperature °F	Load at Ram, lbs.	Torsional Load Level in. - lbs. [1]
-65	17,500	280,000
-20	17,400	278,000
+77	16,800	270,000
+300	15,000	240,000
+450	13,100	210,000

[1] This is the calculated torsional load required to produce a stress level in the tank structure approximating 90% of that required to obtain yield. Torsional load equals load at ram times the effective lever arm length (16 inches).

TABLE 9
SUMMARY OF RESULTS OF ENVIRONMENTAL CONDITIONING OF TANKS

Tank No.	Sealing System	Environmental Exposure	Results After Environmental Exposure
1	Fillets and faying surface seals (in separate areas) with EC-2288.	300°F for 24 hours filled with fuel, plus 300°F for 24 hours with fuel removed (except for 2 quarts).	No leakage.
2	Fillet seals with AFML fluorocarbon sealant.	350°F for 24 hours filled with fuel, plus 400-450°F for 40 hours (450°F for 20 hours) with fuel removed (except for 2 quarts).	General leakage over entire tank due to cracks in fillets. Testing of this tank terminated. Sixty fasteners fillet sealed with two fluorosilicones were badly reverted.
3	Faying surface seals with AFML fluorocarbon sealant.	Same as Tank No. 2	No leaks in faying surface sealed areas.
4	Fillet seals with Q-95-500 sealant.	350°F for 24 hours filled with fuel, plus 450°F for 24 hours with fuel removed (except for 2 quarts). Continuous nitrogen circulation through tank.	Minor leakage in upper and lower skins and spar web (2 seams and 2 fasteners). Reasons not apparent. Tank ends and attach fittings leaking due to: (1) loss of adhesion of Q-95-500 to epoxy coated steel end plates, and (2) extrusion of Q-2-0046 faying sealant. Many access door fasteners leaking.
5	Fillet and faying surface seals (in all areas) with AFML fluorocarbon sealant (modified).	Same as Tank No. 4	No significant leakage.

TABLE 10

LEAKAGE OF TANK NO. 1 AFTER DYNAMIC CYCLING

Dynamic Cycling Conditions				Leaks on Tank Exterior After Cycling							
				Through Paying Surfaces		Through Fillets		Access Door		Ends of Tank	
Condition of Tank	Load Level, in.-lbs. Torque	Cycling Rate, Cycles per Min.	Total No. of Cycles	Fasteners	Seams	Fasteners	Seams	Fasteners	Seams	Fasteners	Seams
Empty [1] at 77°F	270,000	3	50	0	0	3	5	4	3	0	1
Empty [1] at 300°F	210,000	4	1000	1	0	3	14	26	12	1	5
Filled with JP-4 at -65°F	280,000	6	1000	0	0	0	0	0	0	0	0

[1] Two quarts of fuel in tank.

TABLE 11
SUMMARY OF RESULTS OF DYNAMIC CYCLING OF TANKS

Tank No.	Sealing System	Condition No.	Tank Condition During Cycling [1]	Summary of Results After Dynamic Cycling
1	Fillet and faying surface sealed (in separate areas) with EC-2288	1	77°F with tank empty [2], (only 50 cycles run).	Leakage per Table 10. Leaks in fillet sealed areas due to pinholes and under-sized fillets over fasteners (too much sealant slump). More sealant was applied to all fasteners. Leaks in access door due to too much movement in 2-part access door and to hardening of sealant.
		2	300°F with tank empty [2] (load level under 90%)	
		3	-65°F with tank filled with circulating JP-4	No fuel leakage
3	Faying surface sealed with AFML fluorocarbon sealant	1	450°F with tank empty [2], and with continuous purging with nitrogen.	Extensive leakage of seams and fasteners due to poor physical properties (tensile, tensile-shear, tear, elongation) at 450°F after Table 9 environmental exposures.
4	Fillet sealed with Q-95-500	1	450°F with tank empty, and with continuous purging with nitrogen.	Six seam lengths out of 24 were leaking (25%), and also 3 fasteners in the center rib. These leaks were not believed to be due to degradation of Q-95-500, but to an unsatisfactory method of applying sealant fillets over fasteners. The Q-95-500 was in excellent condition. The tank ends and attach fittings leaked badly due to loss of adhesion of Q-95-500 to the epoxy coating on steel end plates, and due to reversion and extrusion of Q-2-0046 sealant in faying surfaces. Reversion and extrusion of Q-2-0046 also caused major leakage of the access door (both seams and fasteners).
5	Fillet and faying surface sealed (in all areas) with AFML fluorocarbon sealant (modified)	1	450°F with tank empty, and with continuous purging with nitrogen.	Extensive leakage of seams. Moderate fastener leakage. Fillet leakage appeared to be due to (1) pinholes or voids caused by entrapped air, and (2) marginal adhesion, particularly over fasteners. Faying surface leaks were believed to be due to poor physical properties of the sealant at 450°F after Table 9 environmental conditioning.

[1] Unless otherwise specified, for each run the tank was subjected to 1000 load (torque) cycles at a load level equal to 90% of yield at the temperature of the tank. Actual loads per Table 8.

[2] Except for about 2 quarts of fuel in tanks.

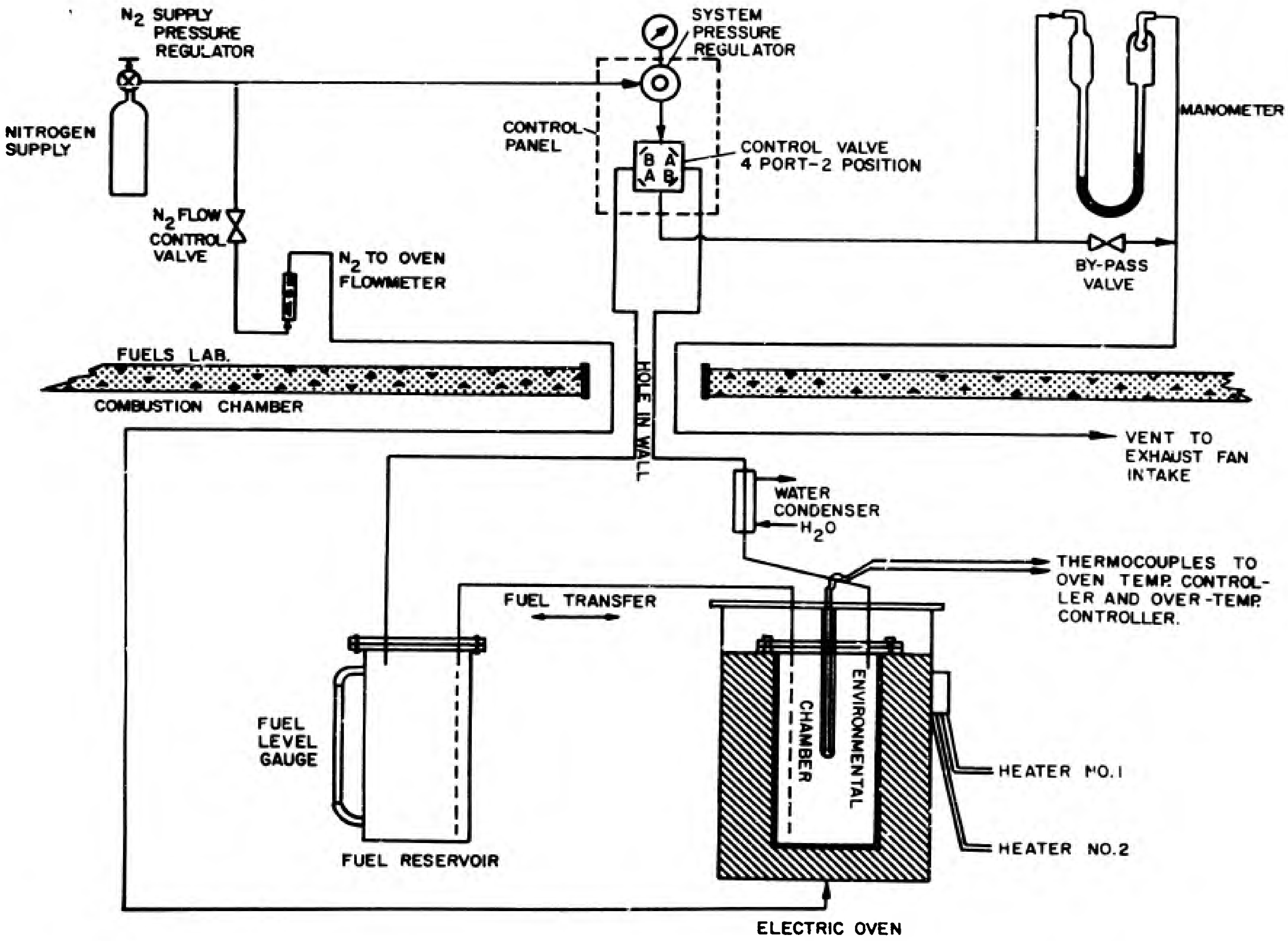
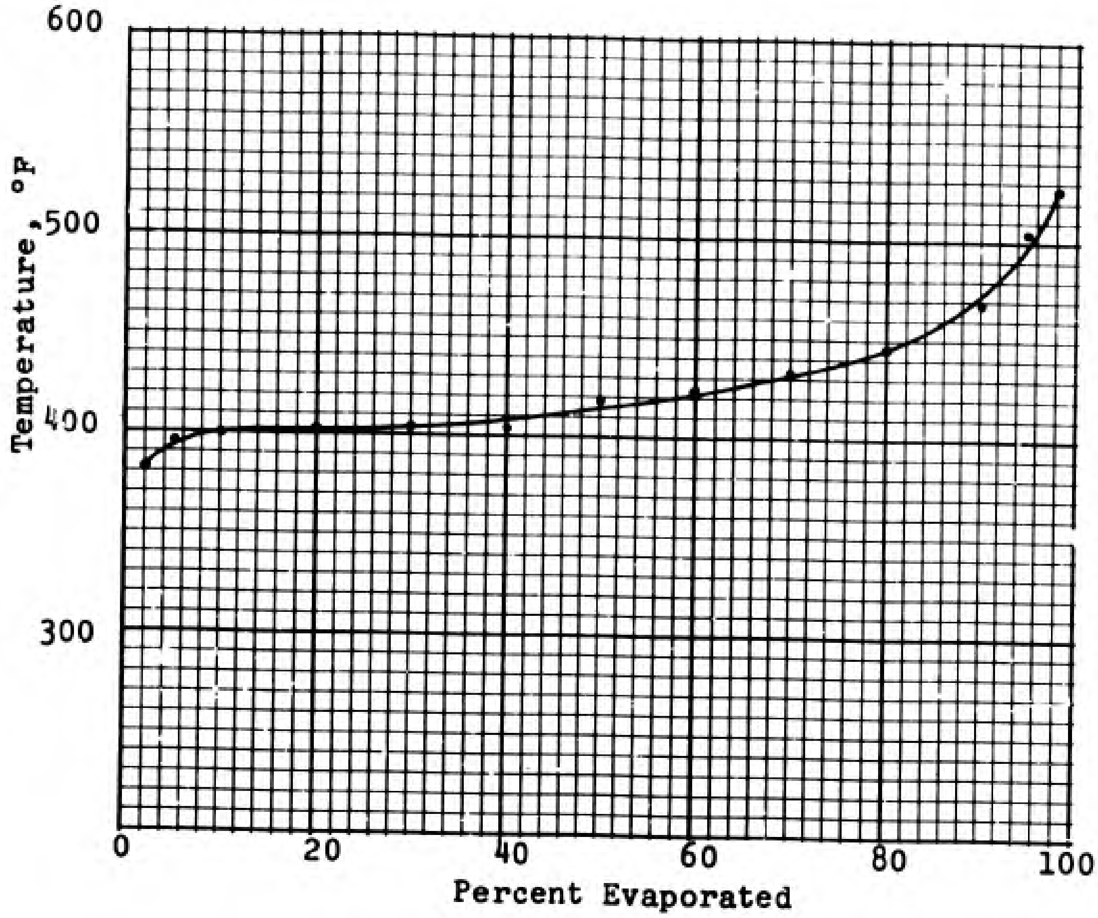


FIGURE 1
ENVIRONMENTAL TEST DEVICE



Tested Per ASTM D 86-54

FIGURE 2

ASTM DISTILLATION OF AFML
FUEL (F18-63)

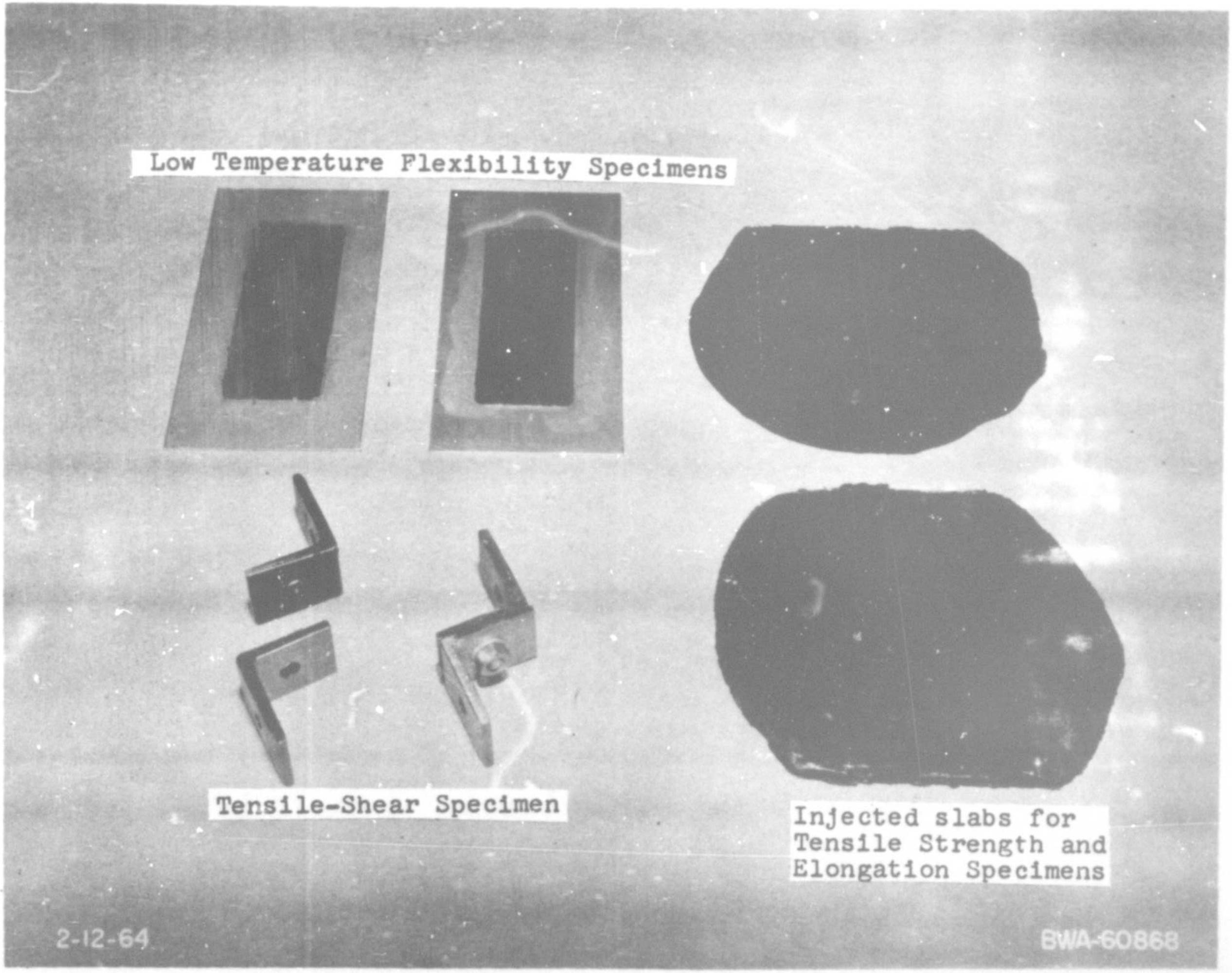


FIGURE 3
AFML FLUOROCARBON SEALANT TEST SPECIMENS

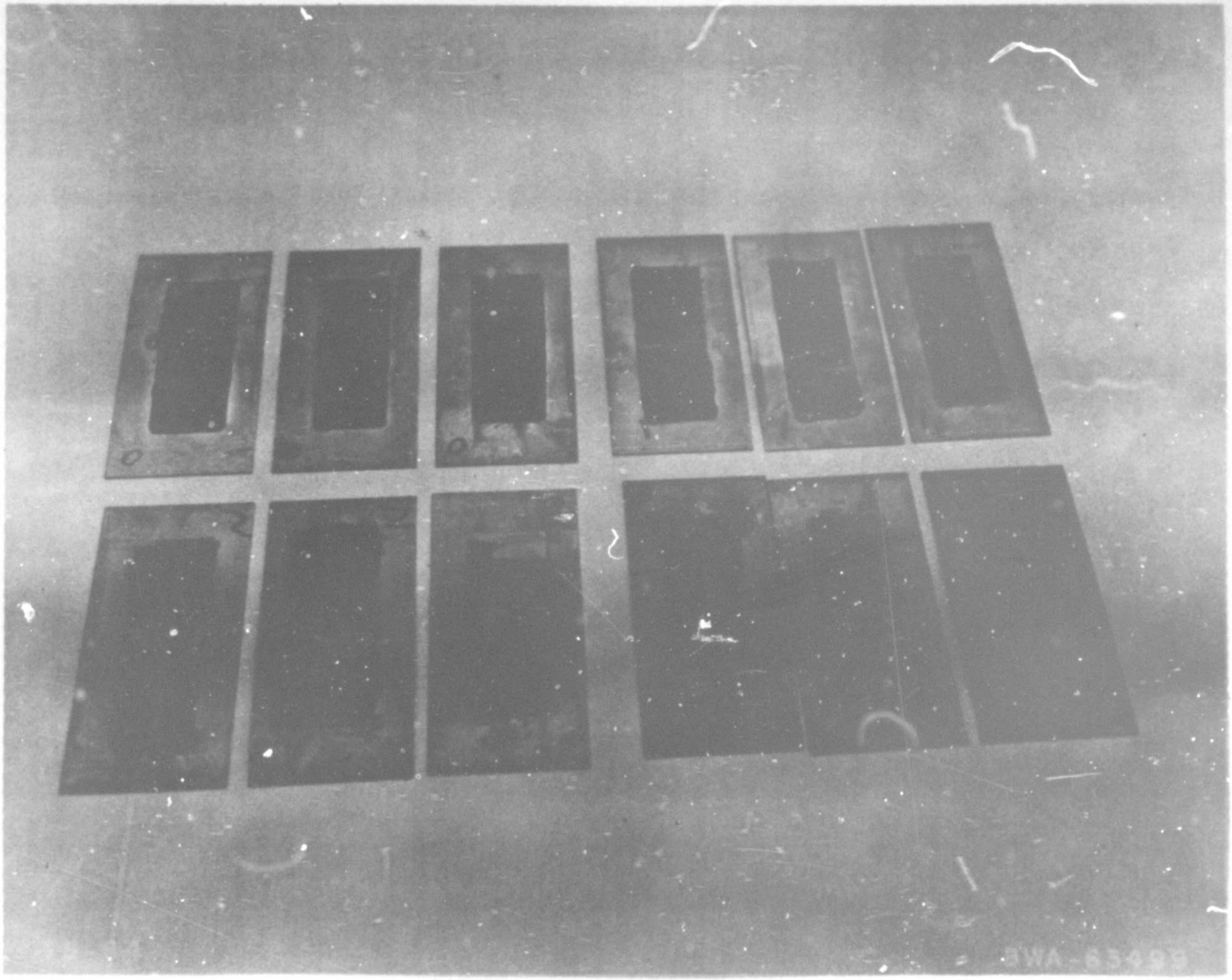
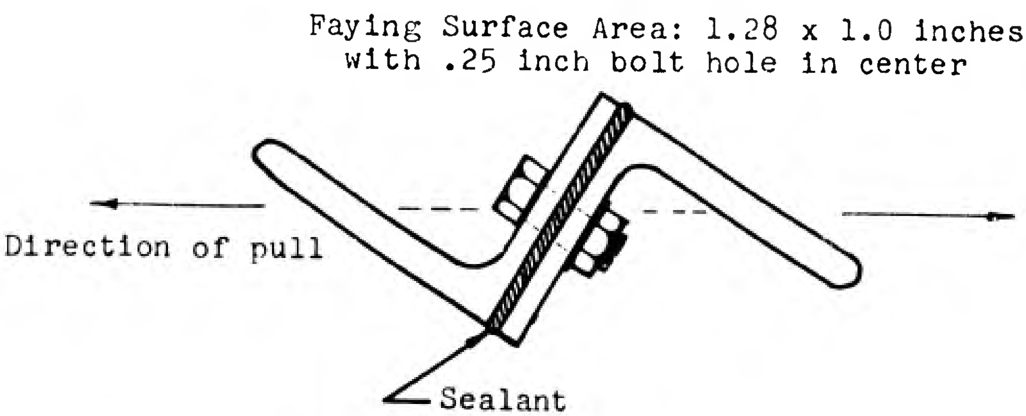


FIGURE 4
LOW TEMPERATURE FLEXIBILITY SPECIMENS -
AFML FLUOROCARBON SEALANT



Tensile-shear specimen is made from two 1-inch long "L" sections fabricated from BMS 7-44A Condition 1 extrusions (6Al - 4V titanium, annealed).

FIGURE 5

TENSILE-SHEAR SPECIMEN

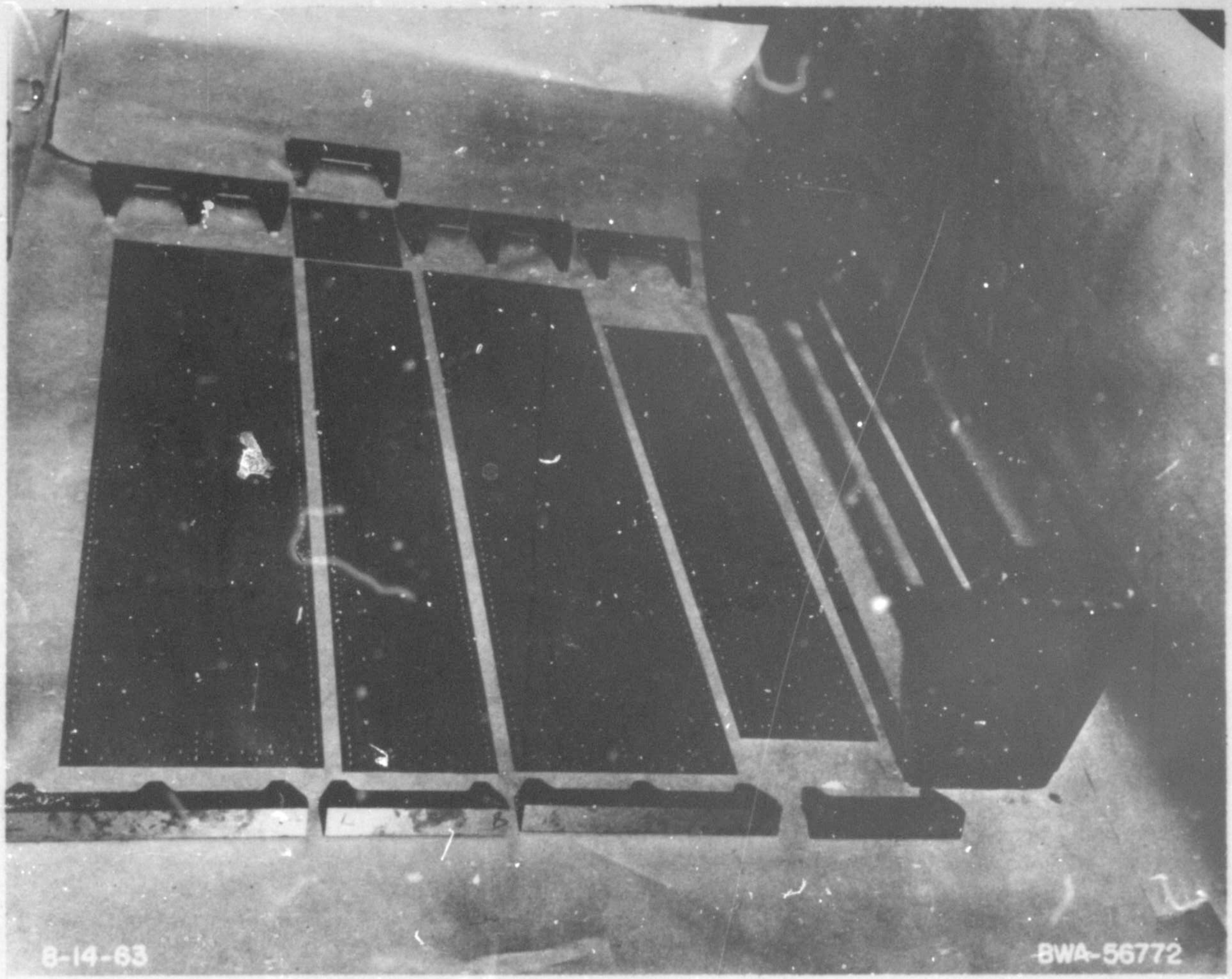
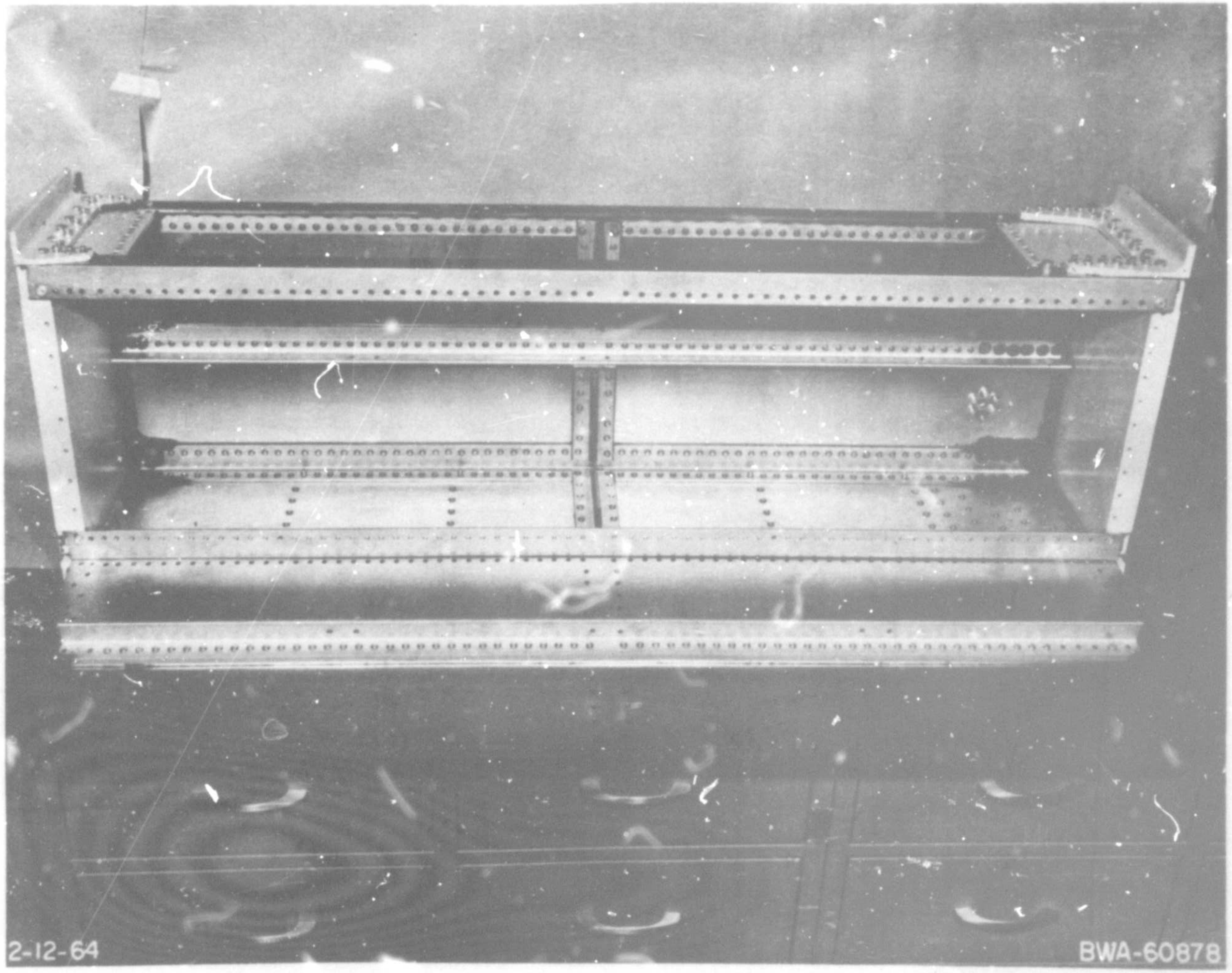


FIGURE 6

TEST TANK DETAIL PARTS



NOTE: This tank is completely faying surface sealed with the AFML fluorocarbon sealant. In addition tank ends were fillet sealed with the same sealant.

FIGURE 7

TEST TANK NO. 3 PARTIALLY ASSEMBLED (LOWER SKIN NOT INSTALLED)

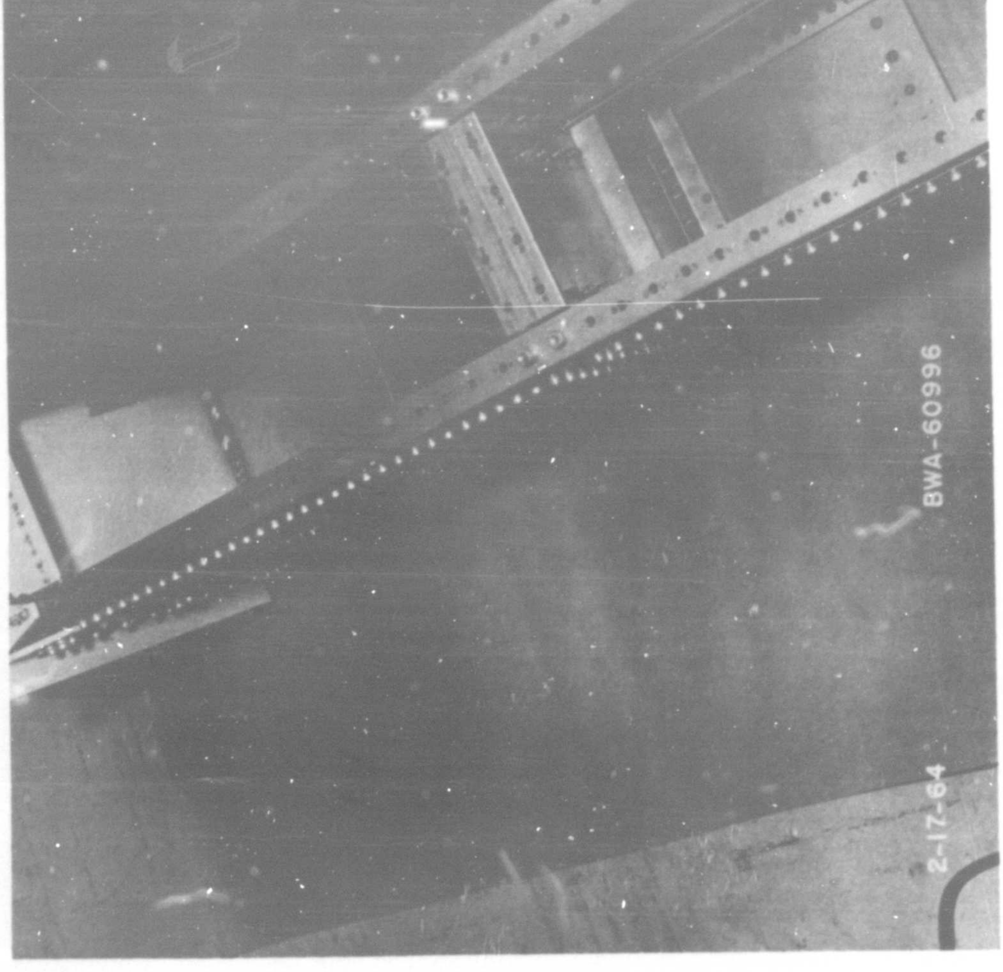
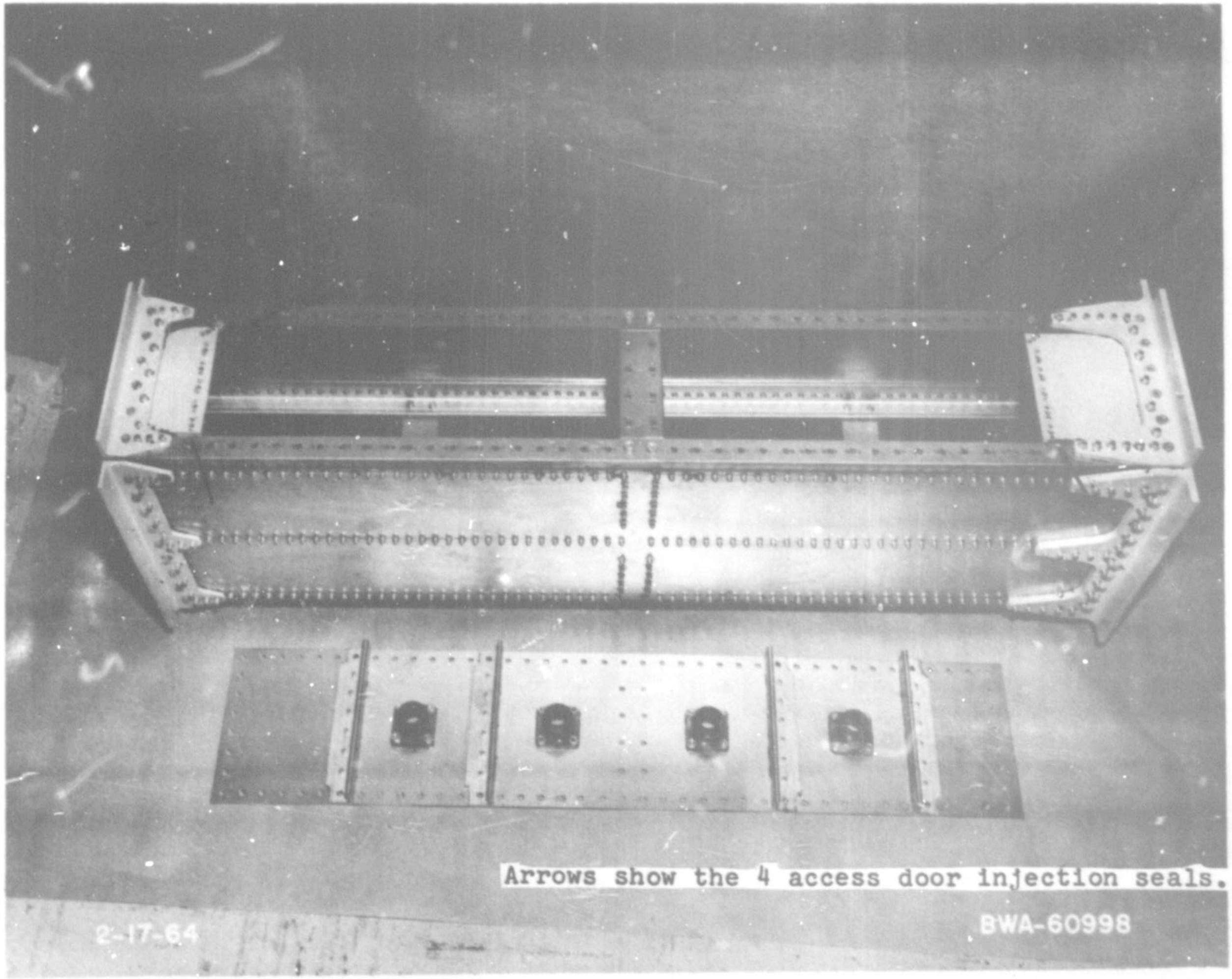


FIGURE 8
ASSEMBLED TANK NO. 3 - CENTER RIB DETAILS



<u>NOTE</u>: This tank is completely faying surface sealed with the AFML fluorocarbon sealant. In addition the tank ends were fillet sealed with the same sealant.

FIGURE 9

ASSEMBLED TANK NO. 3 (ACCESS DOOR NOT INSTALLED)

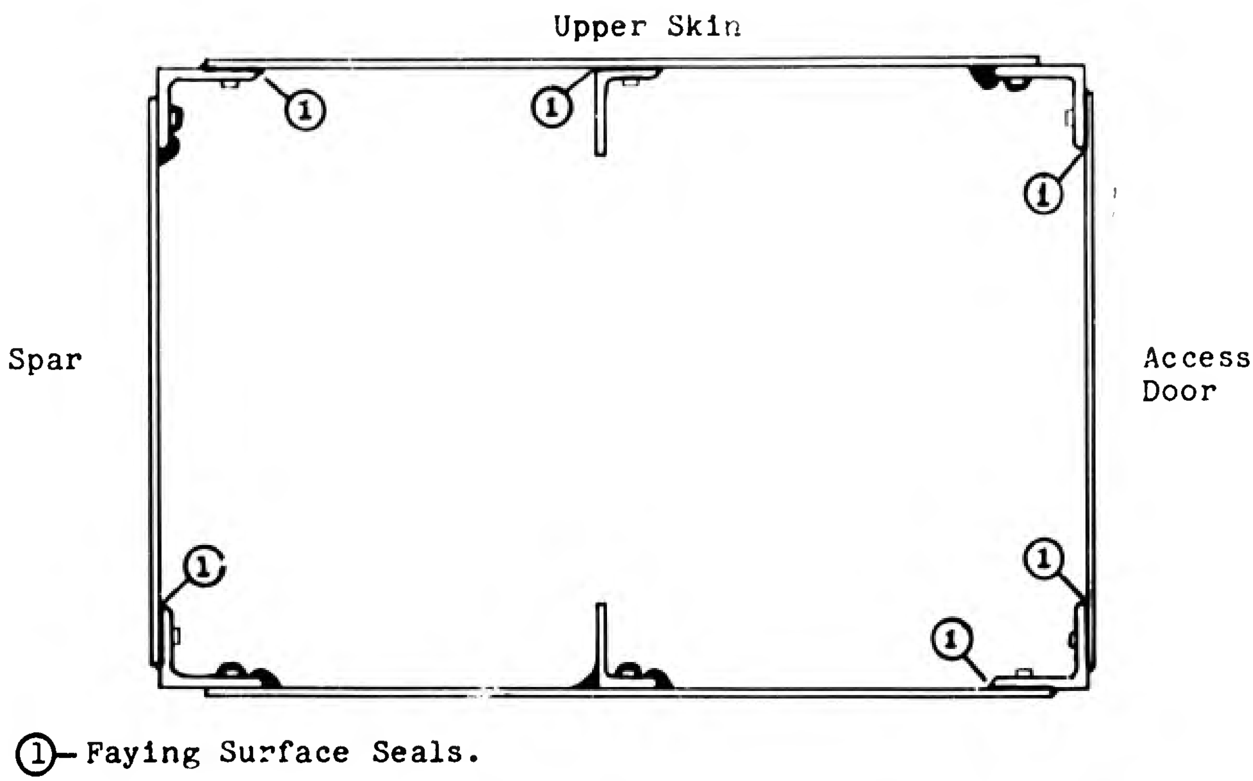
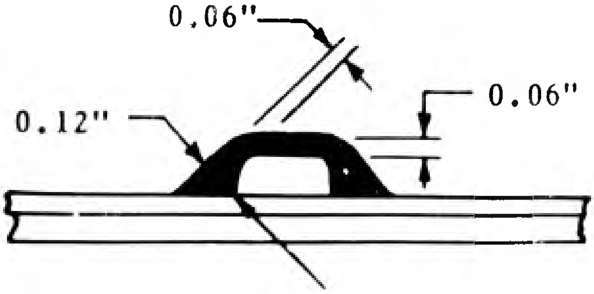
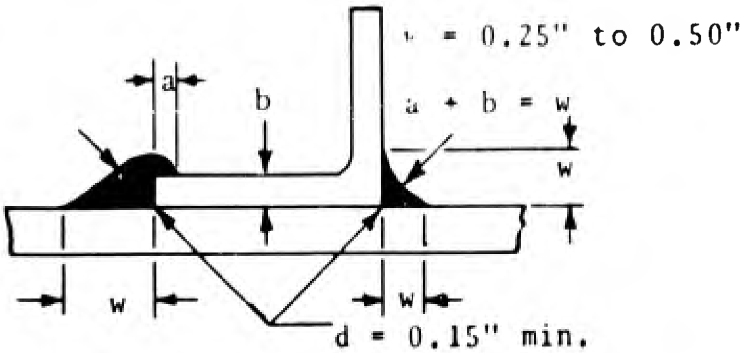


FIGURE 10

TYPICAL CROSS-SECTIONAL VIEW OF TANK NO. 1 (EC-2288)



Fillet Dimensions
Over Fasteners



Fillet Dimensions

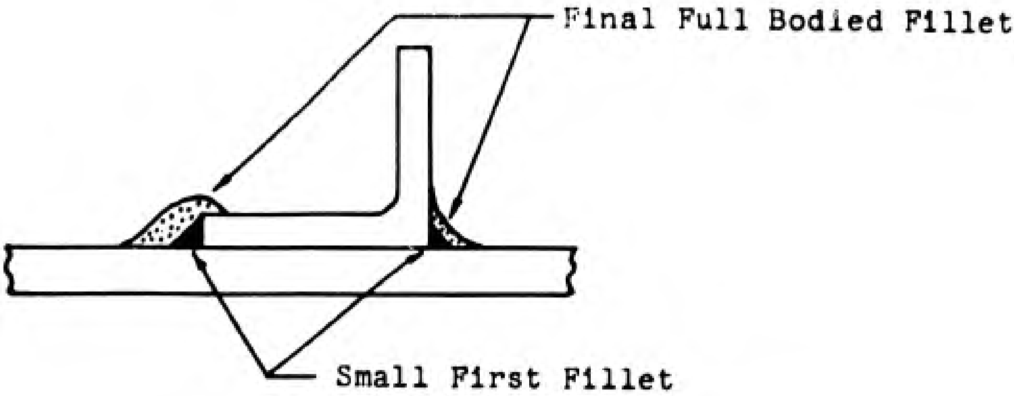


FIGURE 11
TYPICAL FILLET DIMENSIONS FOR TEST TANKS

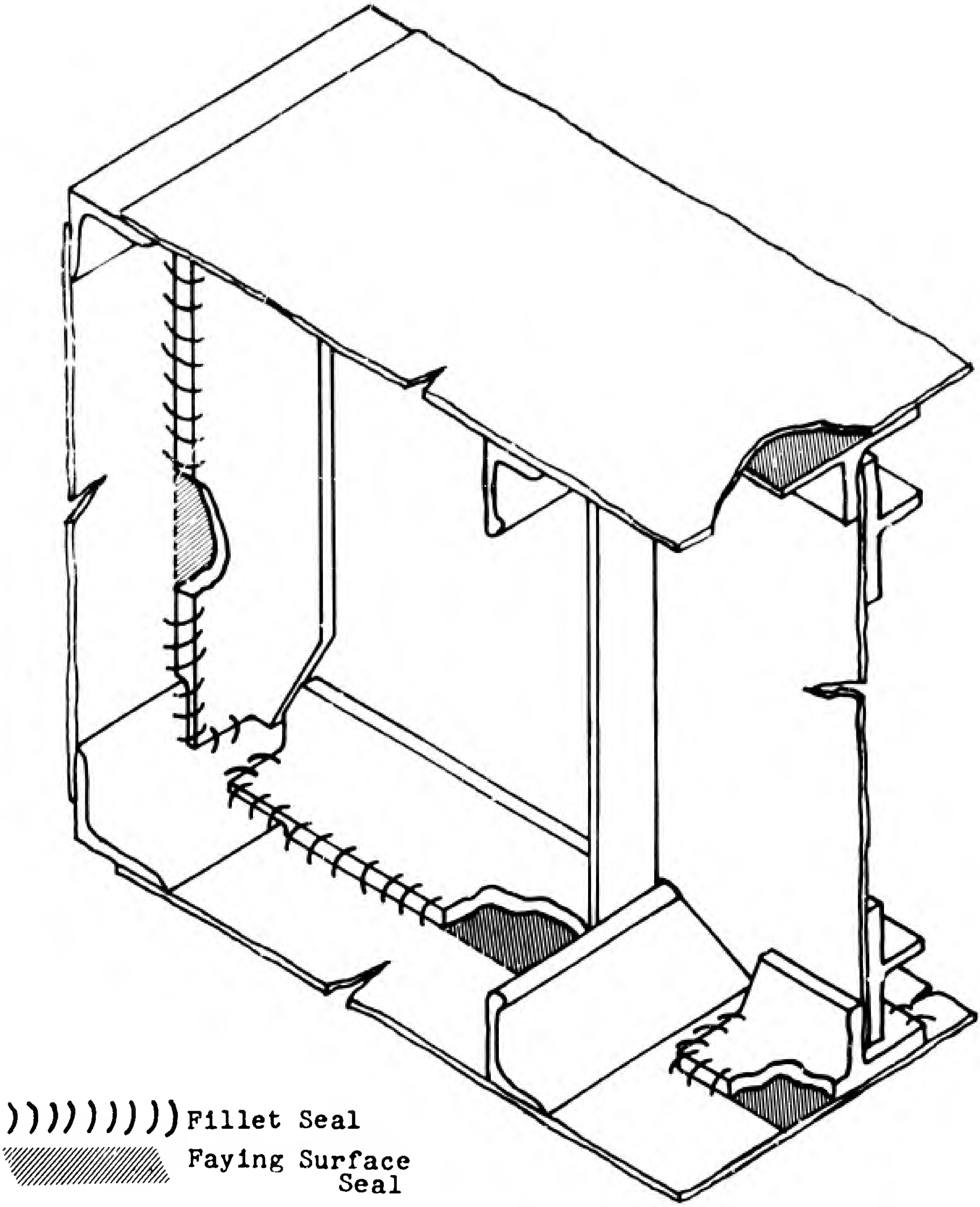
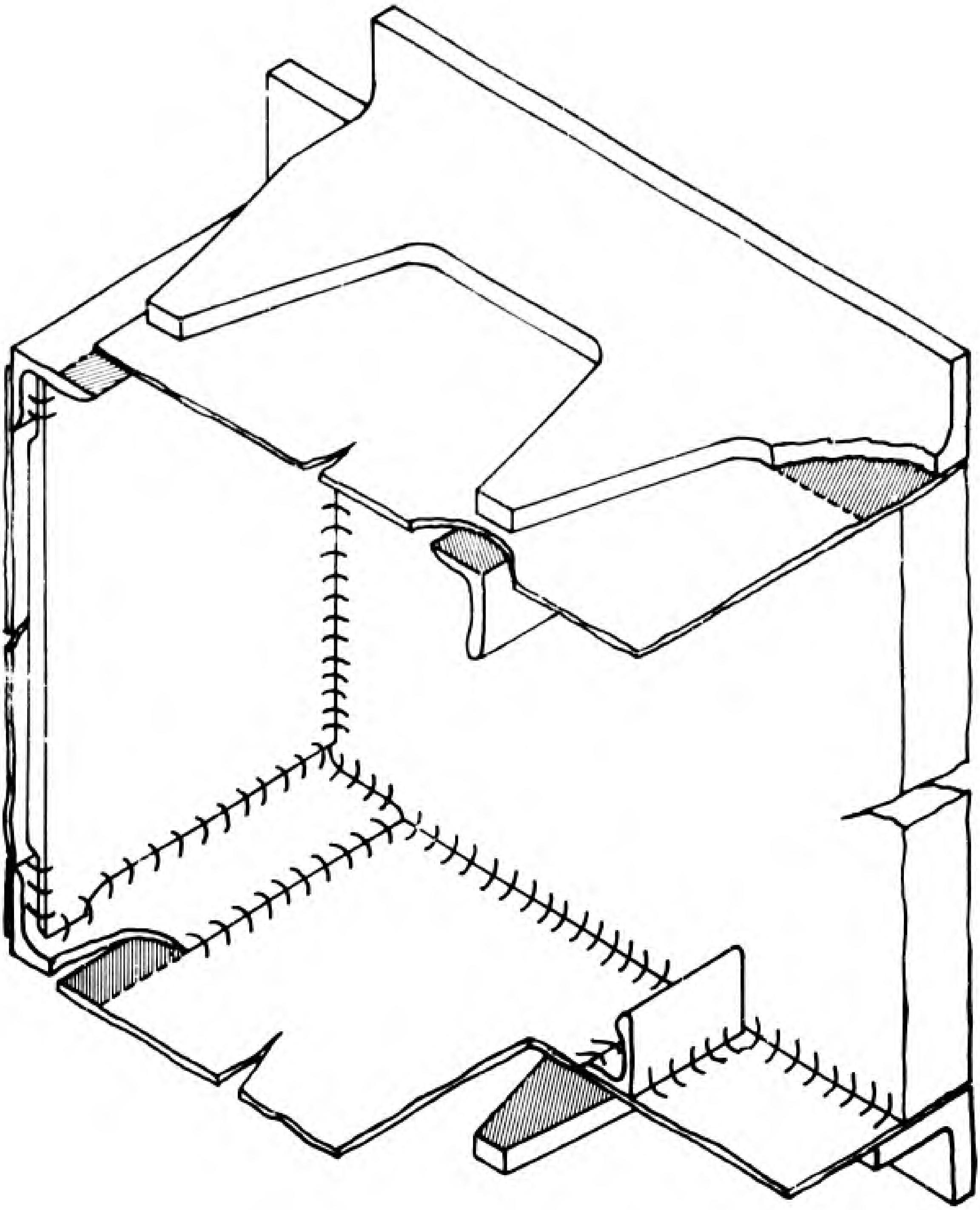


FIGURE 12

SEALING OF CENTER RIB OF TANK NO. 1 (EC-2288)



Faying Surface Seal

))))))) Fillet Seal

FIGURE 13

TYPICAL SEALING OF TANK ENDS

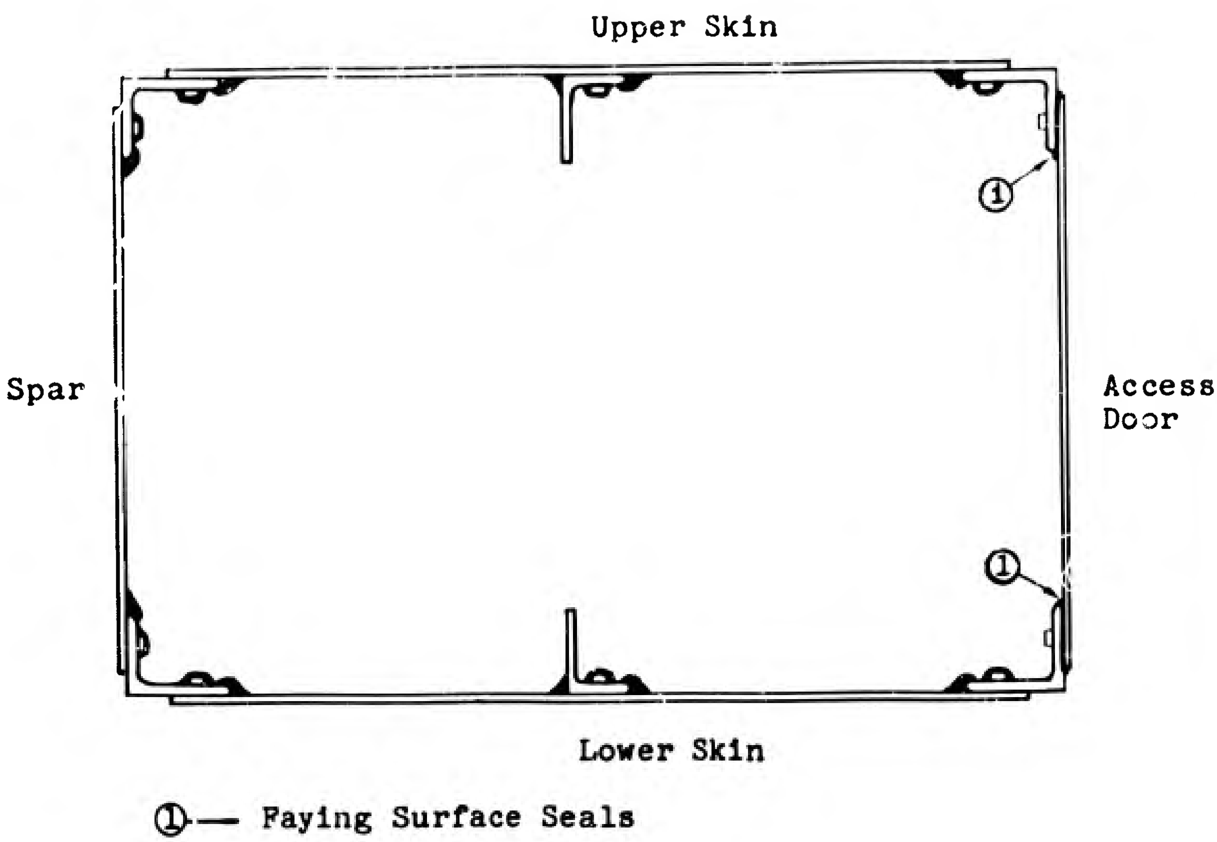


FIGURE 14

TYPICAL CROSS-SECTIONAL VIEW OF FILLET SEALED TANKS
(Numbers 2 and 4)

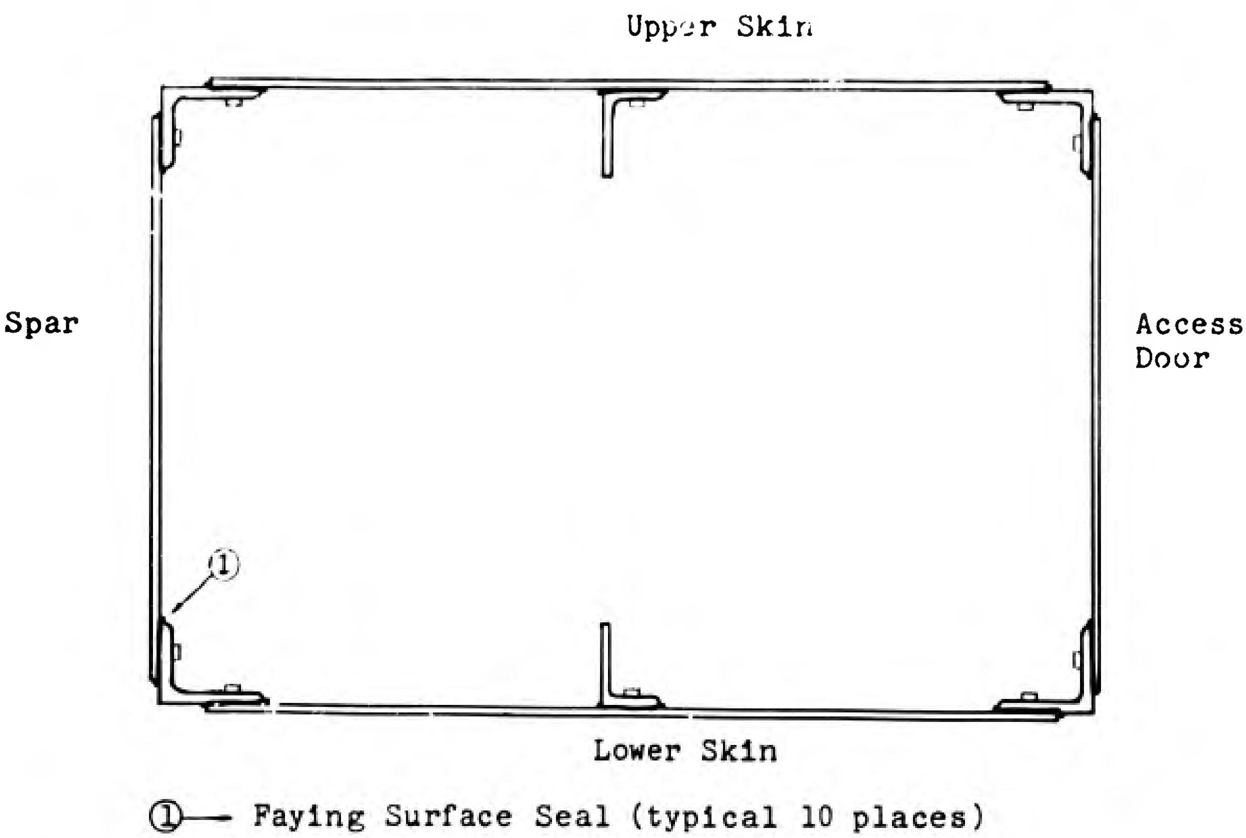


FIGURE 15

TYPICAL CROSS-SECTIONAL VIEW OF FAYING SURFACE SEALED TANK NO. 3

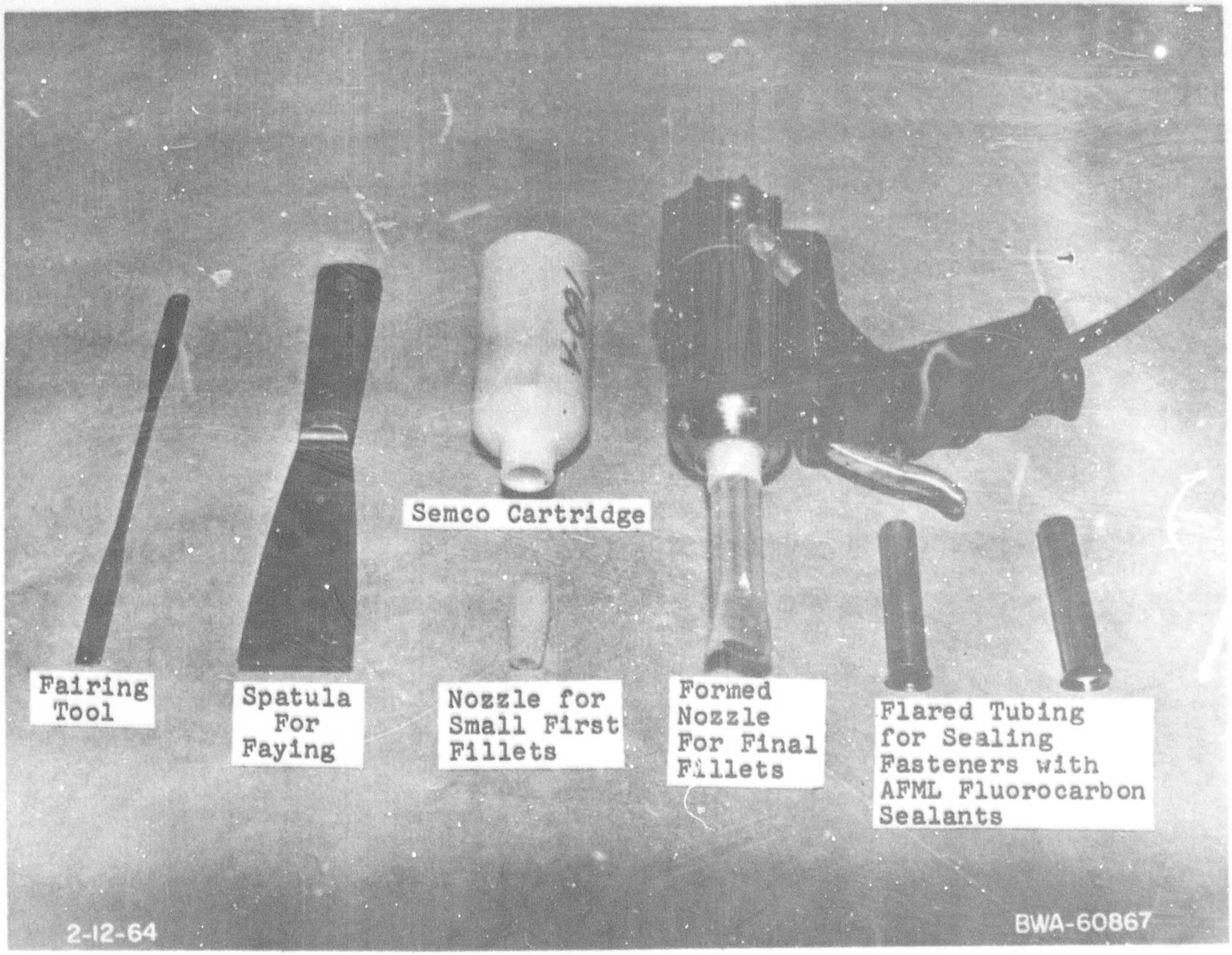
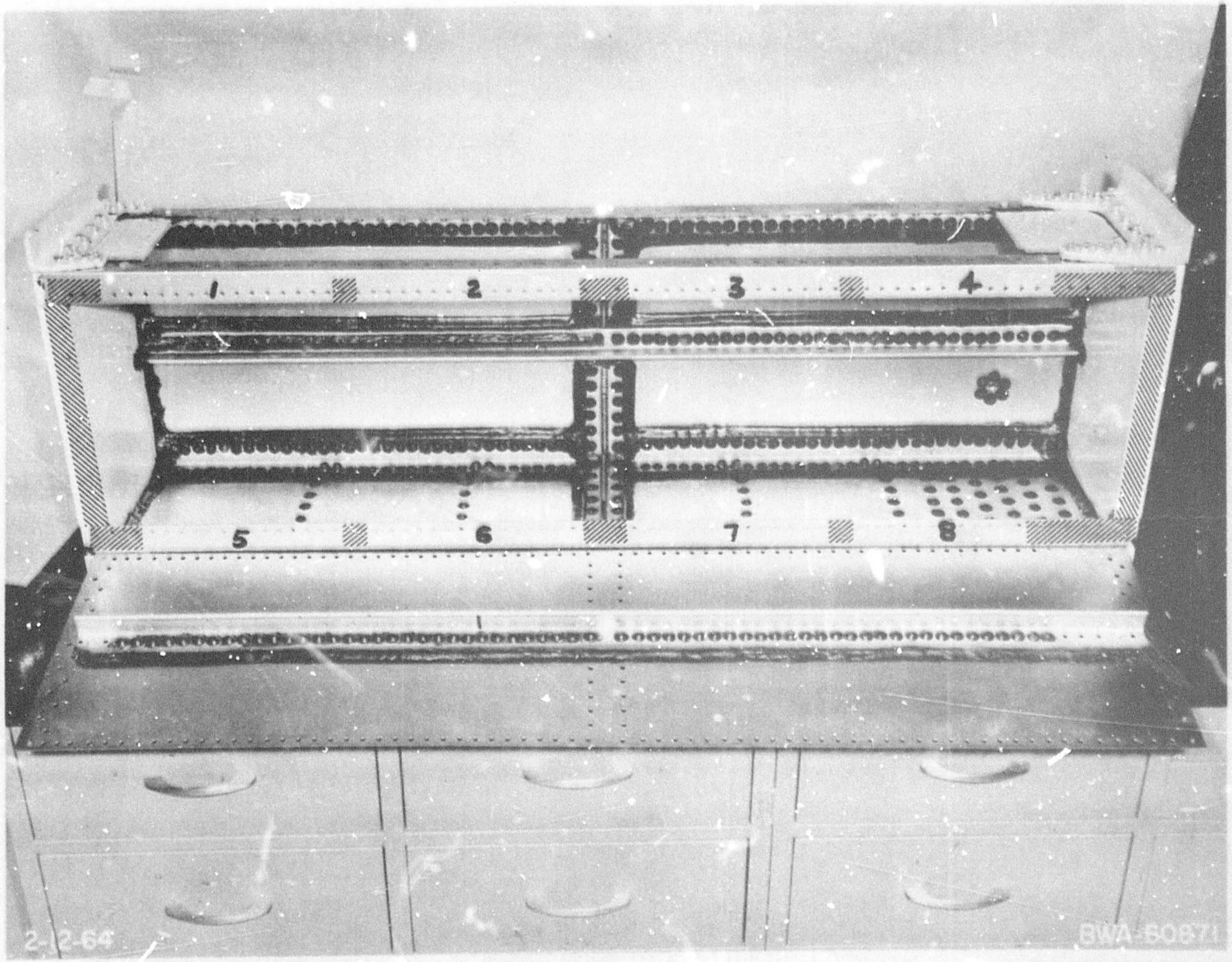


FIGURE 16
TOOLS FOR APPLYING SEALANTS



NOTE: "Block-off" faying surface seals were added to upper and lower skin and the spar web, as shown typically by ▨▨▨,prior to the installation of the lower skin. The numbers indicate the 8 areas which were isolated from each other by these block-off seals.

FIGURE 17

TANK NO. 2 PARTIALLY ASSEMBLED

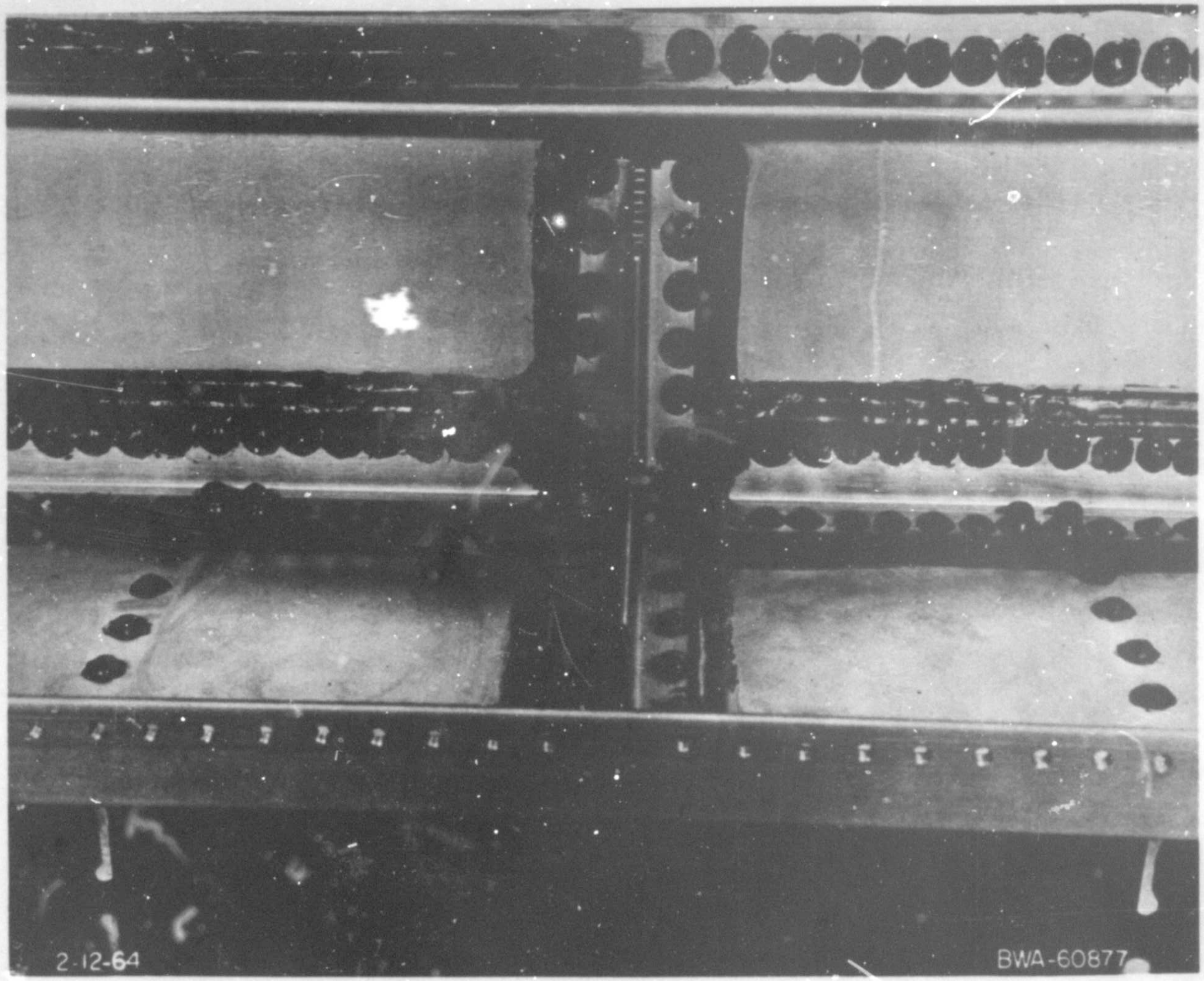


FIGURE 18

TANK NO. 2 CENTER RIB SEALING DETAILS

FIGURE 19
ASSEMBLED TANK NO. 2
(Access Door Not Installed)

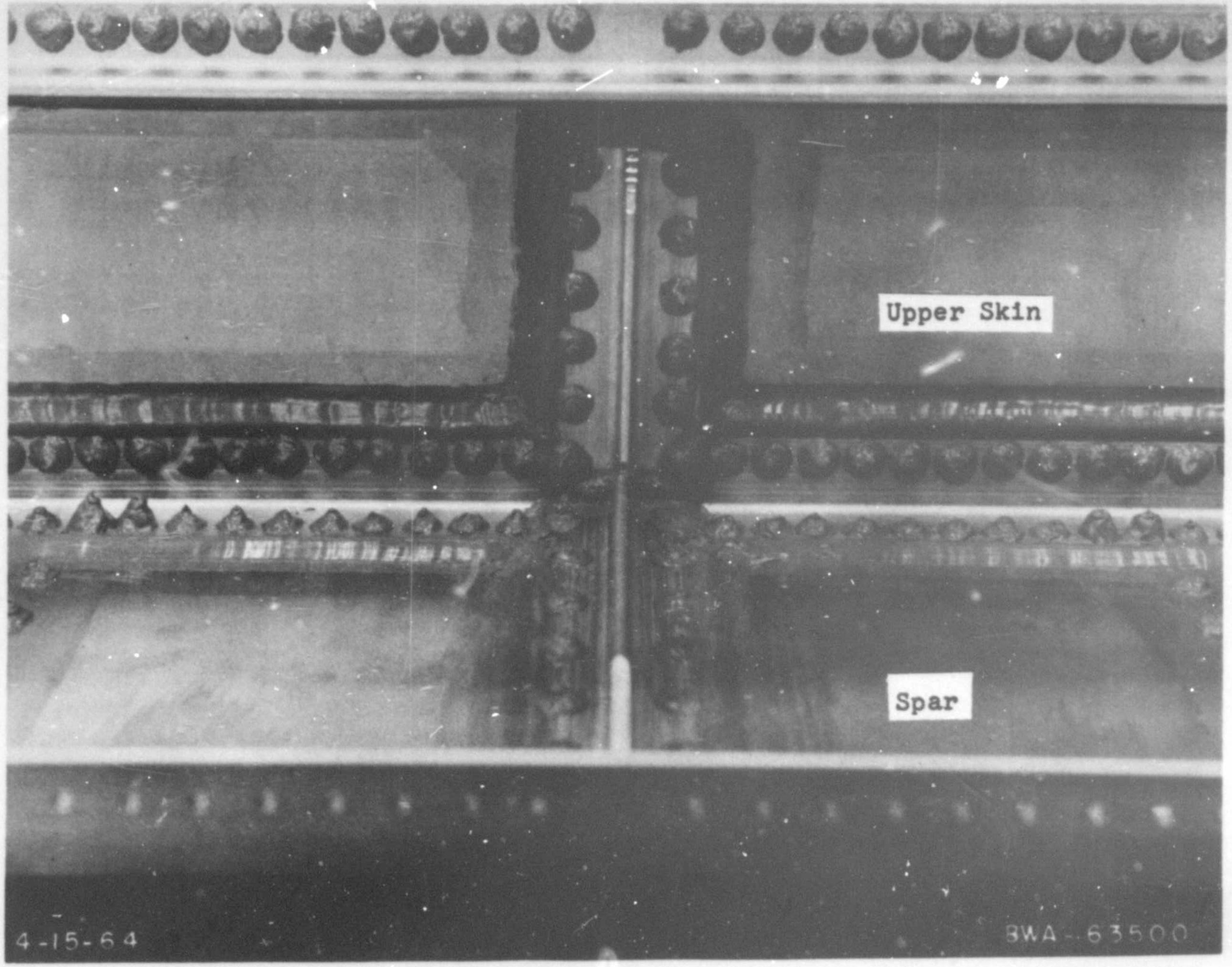


FIGURE 20
FILLETS OF Q-95-500 IN TANK NO. 4

FIGURE 21
CRACKS IN AFML FLUOROCARBON FILLETS (TANK NO. 2)

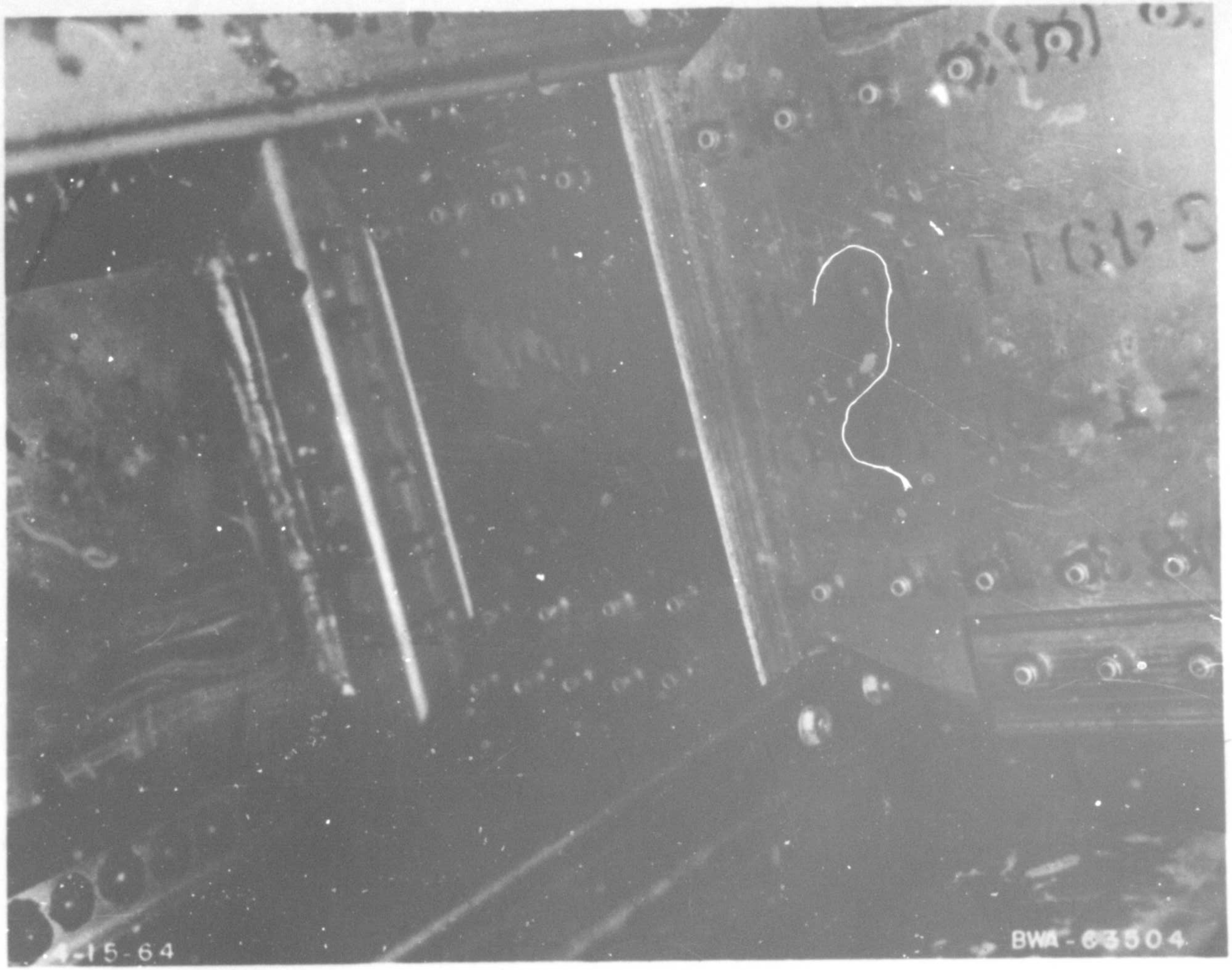


FIGURE 22
CRACKS IN AFML FLUOROCARBON FILLETS (TANK NO. 2)

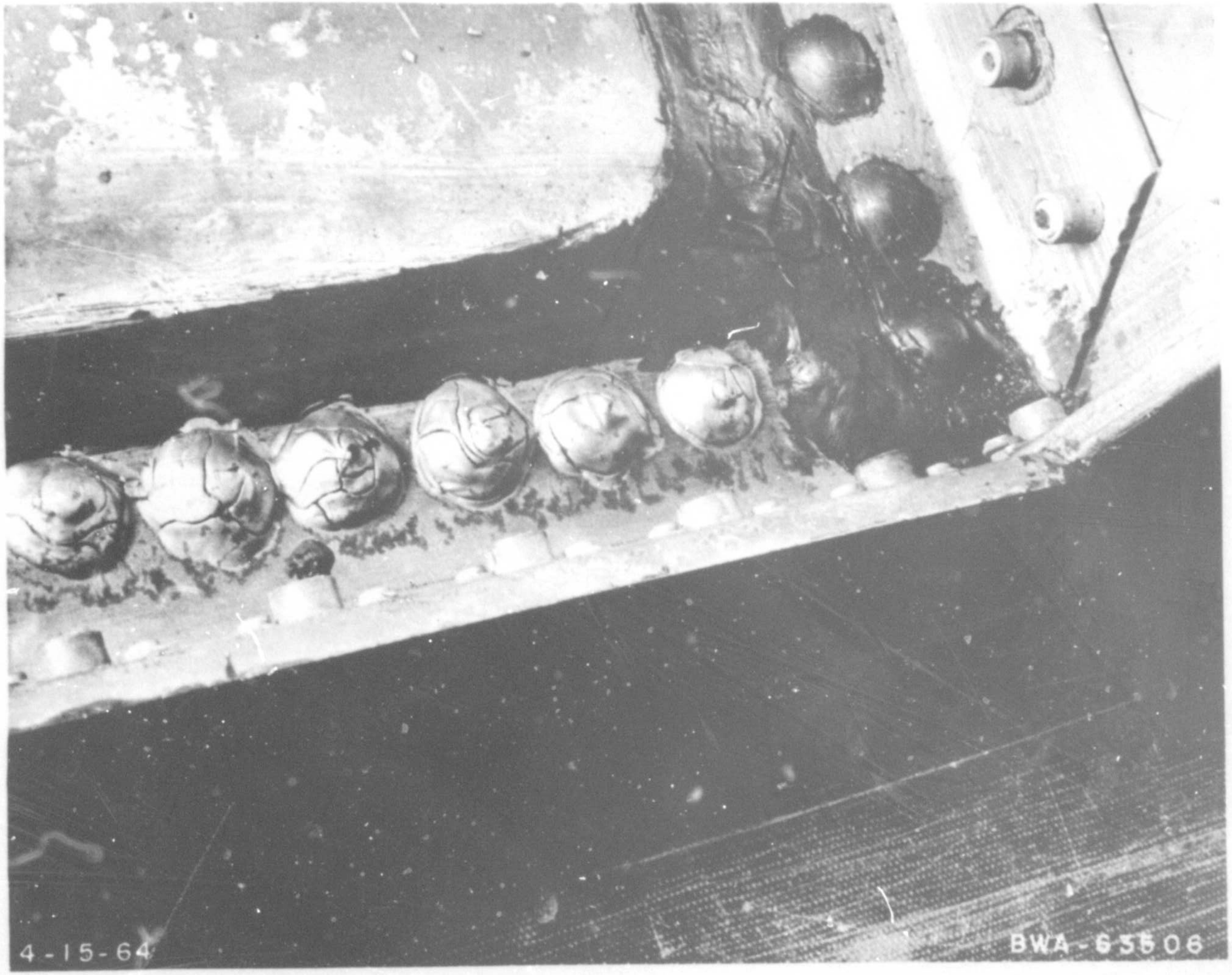


FIGURE 23
CRACK IN AFML FLUOROCARBON FILLET AND
REVERTED Q-2-0046 OVER FASTENERS (TANK NO. 2)

FIGURE 24
REVERTED Q95-500 SEALANT OVER FASTENERS (TANK NO. 2)

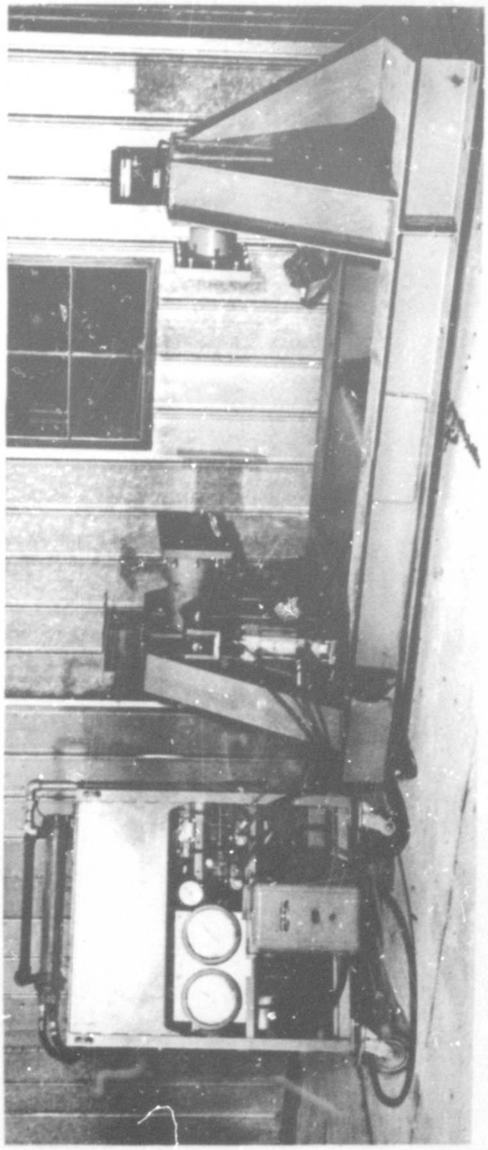


FIGURE 25
DYNAMIC TEST DEVICE

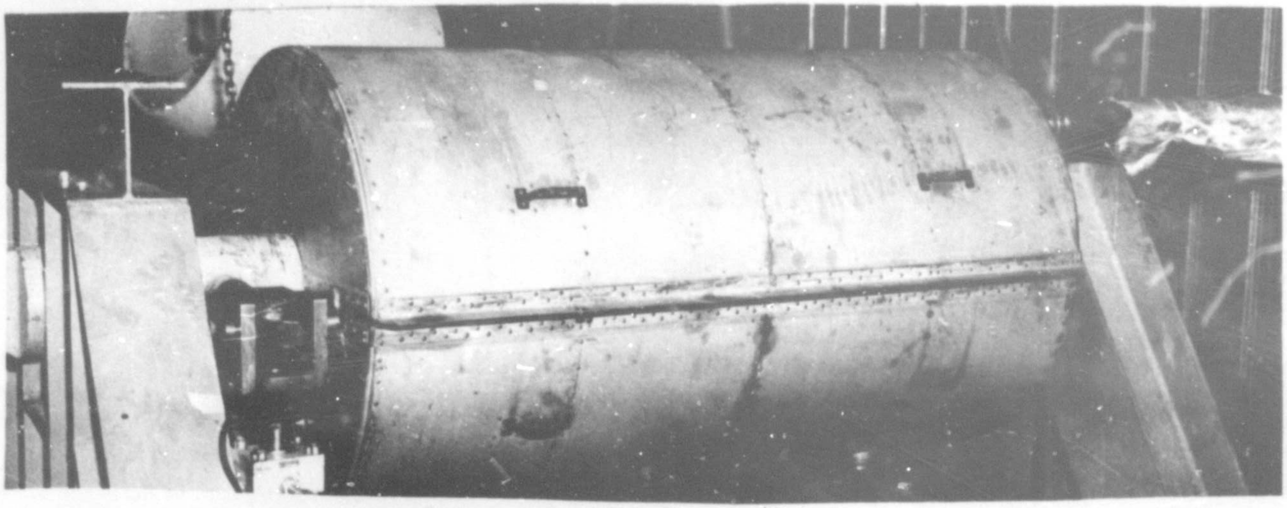


FIGURE 26

DYNAMIC TEST DEVICE WITH INSULATED CHAMBER

REFERENCES

A. ASD-TDR-62-91, "Test Device for the Dynamic Evaluation of Aircraft Fuel System Materials and Structures".

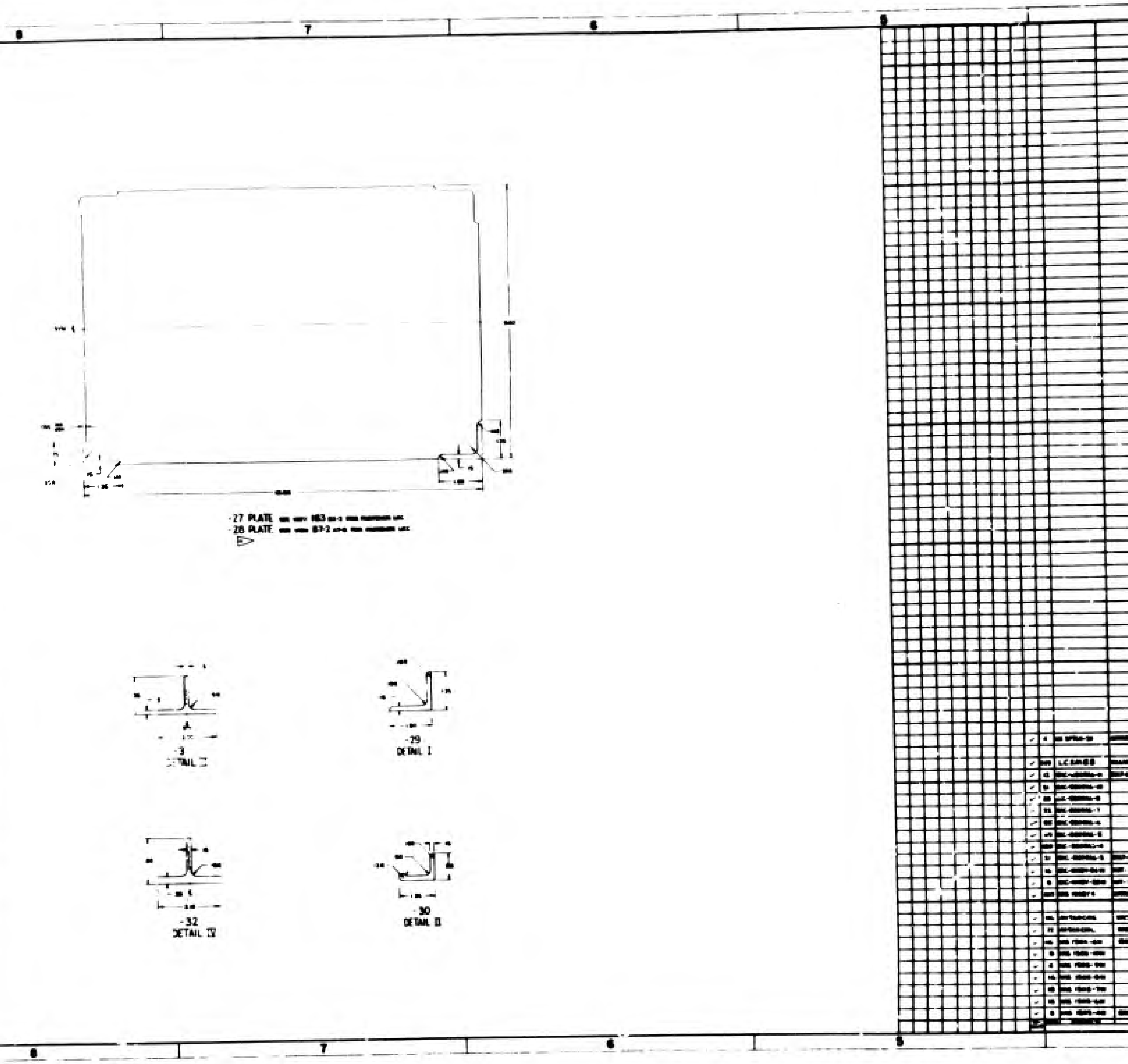
B. ASTM D412, "Tension Testing of Vulcanized Rubber".

C. Boeing Material Specification 10-39, "Fuel and Moisture Resistant Finish for Fuel Tanks".

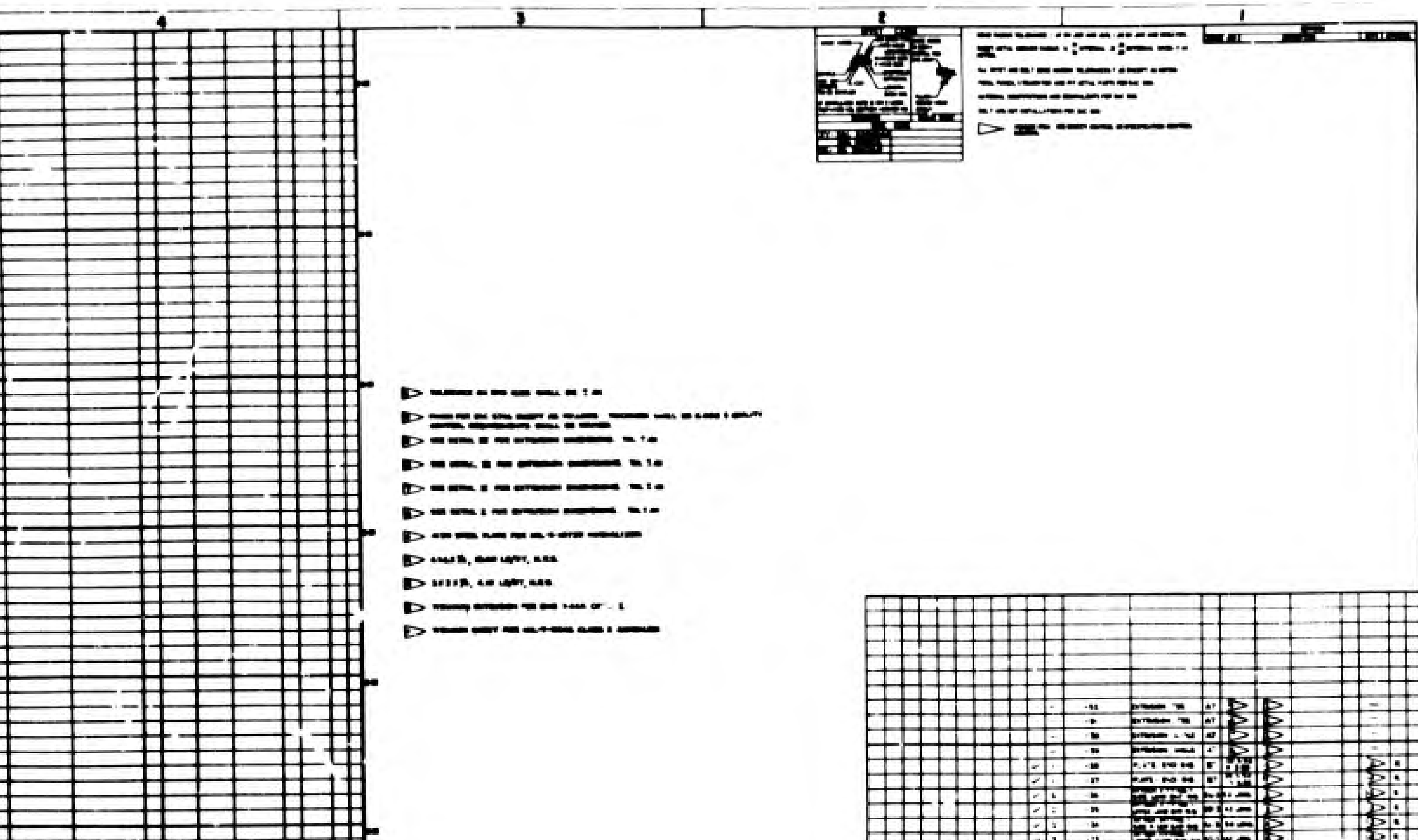
D. MIL-S-8802, "Sealing Compound, Temperature-Resistant, Integral Fuel Tanks and Fuel Cell Cavities, High-Adhesion".

APPENDIX

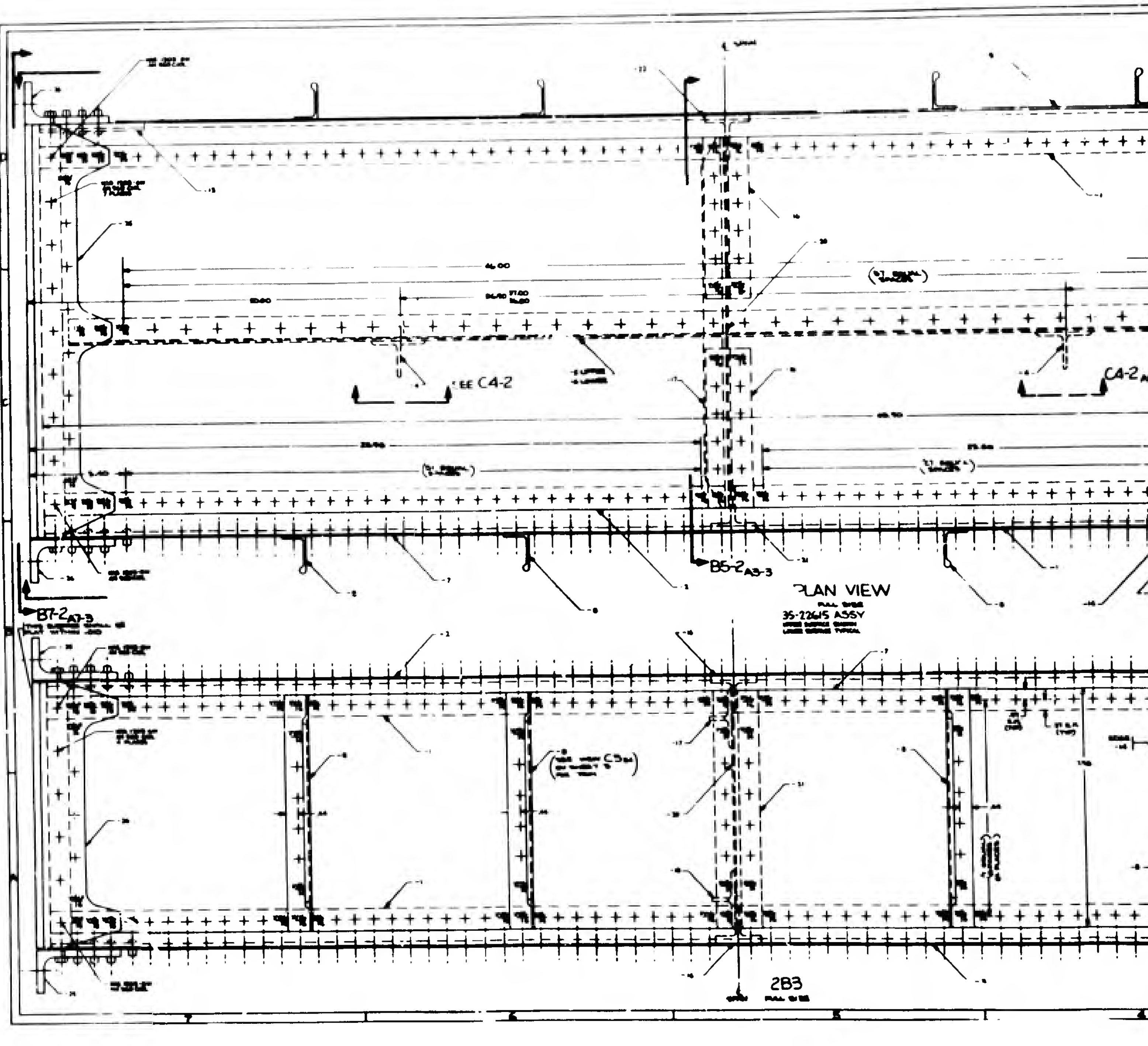
BOEING FUEL TANK DRAWINGS



A



B



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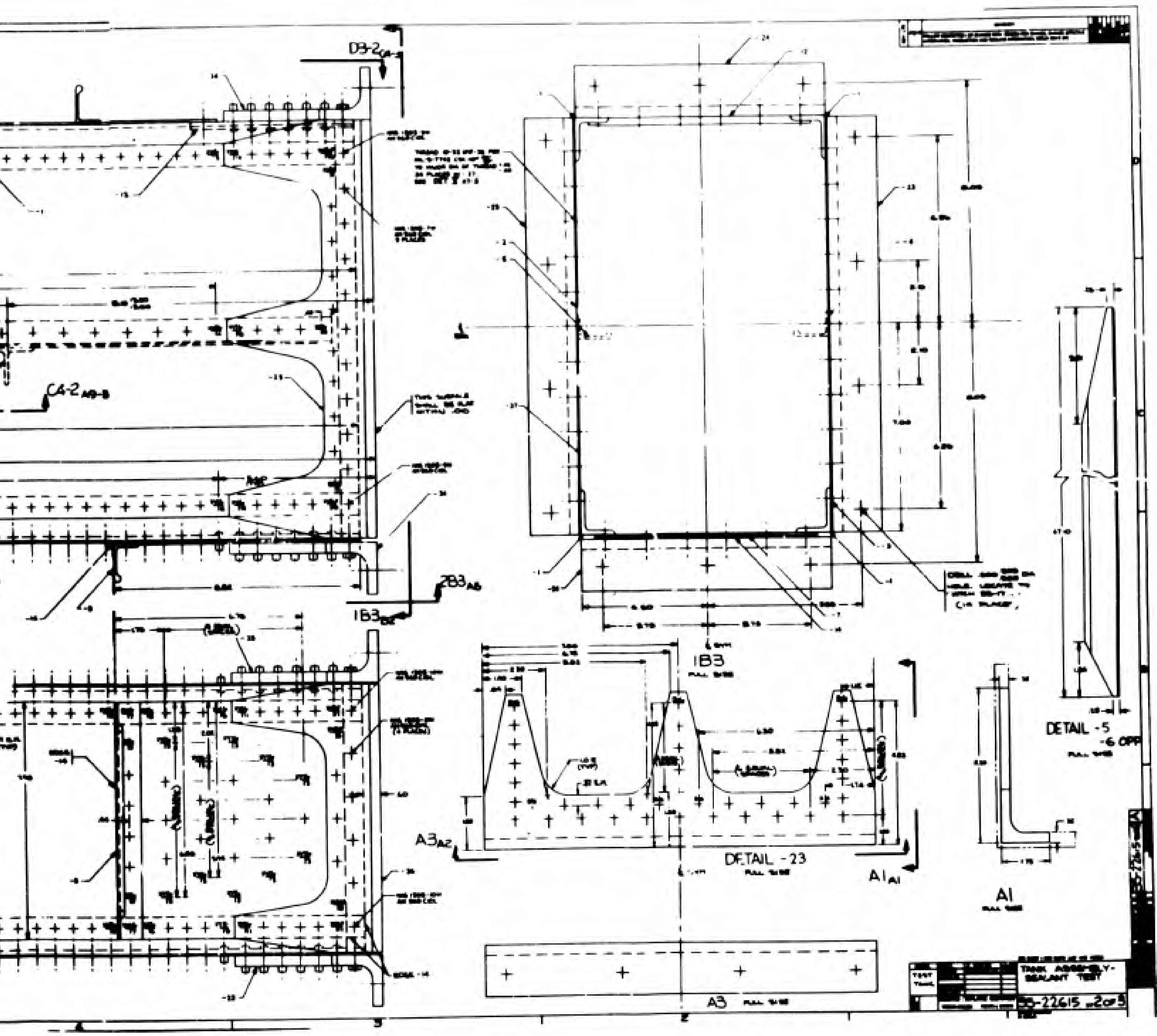
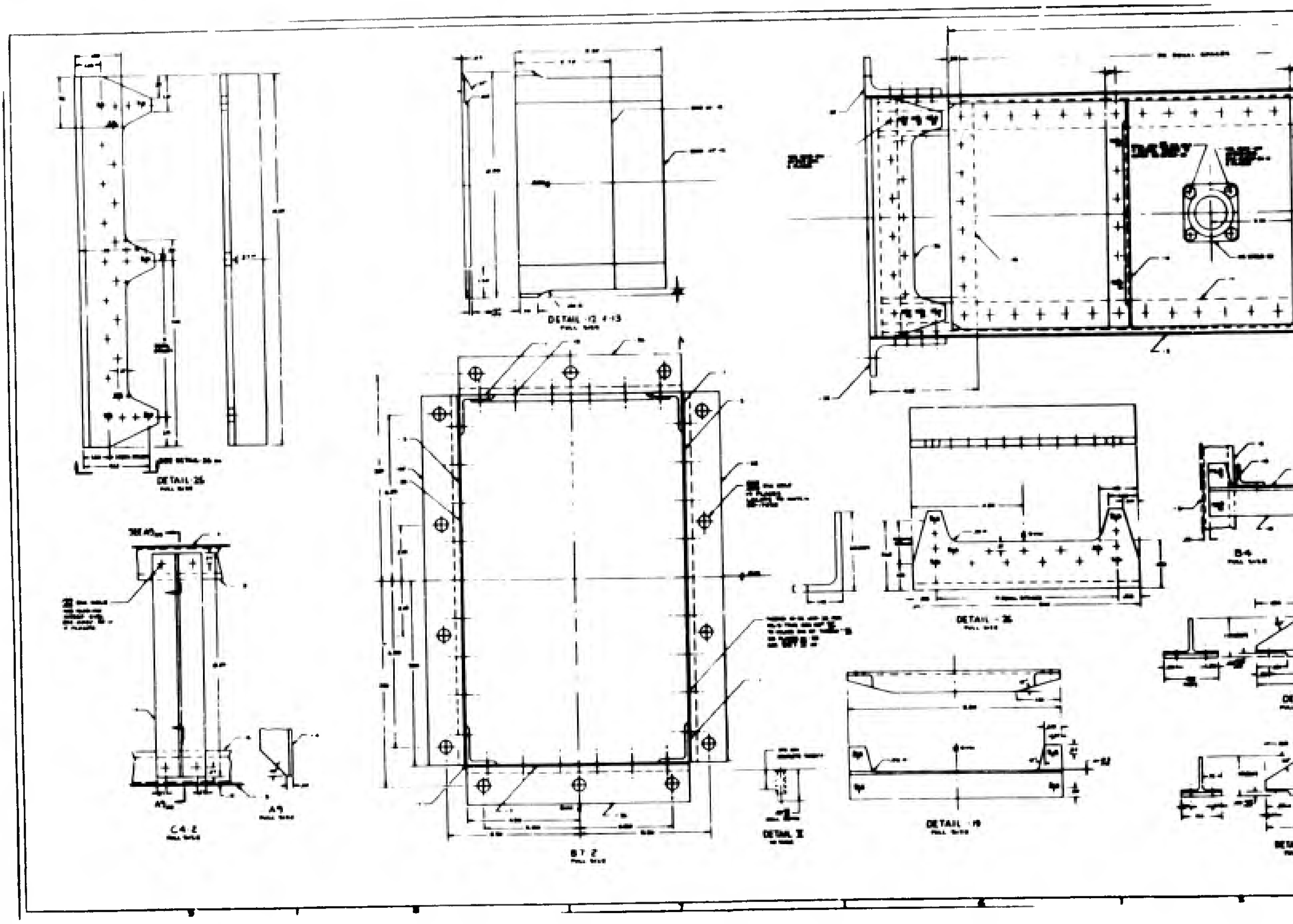
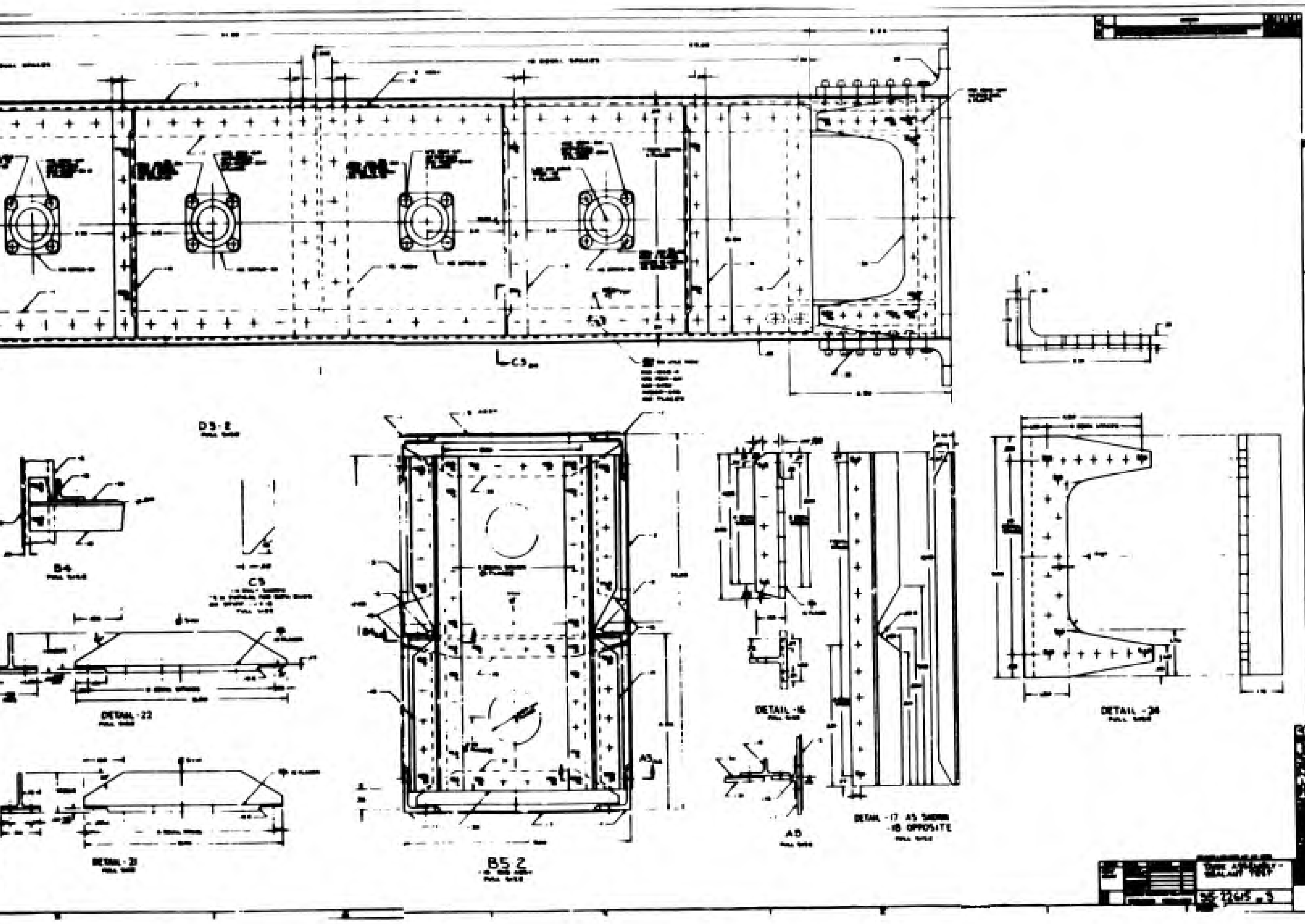


D3-2
C4-2
2B3
1B3
DETAIL -23
FULL SIZE
A3
A1
DETAIL -5
-6 OPP
TANK ASSEMBLY-
SEALANT TEST

B



A


D5-2
B4
C5
DETAIL-22
DETAIL-21
B5-2
DETAIL-16
A5
DETAIL-17 AS SHOWN
-18 OPPOSITE
DETAIL-24

B 73