WADC TECHNICAL REPORT 54-527
SUPPLEMENT 1

THE DEVELOPMENT OF A PROTECTIVE COATING RESISTANT TO NITRIC ACID AND HYDROCARBONS

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FOREWORD

This report was prepared by The United States Stoneware Company under USAF Contract No. AF 33(616) - 150. The contract was initiated under Project No. 7312, "Finishes and Materials Preservation", Task No. 73121, "Organic Protective Coatings", and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Mr. Sam Collis as Project Engineer.

The initial contract period covered from May 15, 1952 to May 15, 1954. The background information is completely reported in WADC Technical Report No. 54-527, dated February 1956. The work reported in this addenda covers two contract extensions dated May 15, 1954 to May 15, 1955, and May 15, 1955 through July 15, 1955.

The investigation covered by the complete report includes all phases of the development of a suitable lining procedure for coating ATO compartments employing coating and filleting materials specifically compounded to give resistance to White Fuming Nitric Acid (WFNA). On April 22, 1954 per Change Order C-3(54-1525) this program was limited to coating and filleting materials based specifically on fluorocarbon resins.

Following the development of a soluble fluorocarbon resin by the M. W. Kellogg Company designated resin X-200, an intensive program was initiated to explore the properties of X-200 resin for WFNA resistance. Due to the experimental nature of this resin, the investigation was of necessity limited to those data which were of immediate importance in the most severe service requirements as specified in the contract. Initially, quantities of the resin were limited, and only through the complete cooperation of The M. W. Kellogg Company were we able to arrive at a material specification which appeared to offer a satisfactory balance of required resistance and application properties.

ABSTRACT

X-200 (Kel-F 800) resin, a new fluorinated fuming nitric acid resistant material was formulated as a lacquer and a filleting putty. Application procedures were developed, and resistance of the lacquer coating, the filleting putty, and the complete system in FNA and JP-4 fuel were determined.

Results indicate that the X-200 system is suitable for application on aircraft metals. When force dried at 300° F, FNA resistance approaches that of fused Kel-F. Resistance of the system when air dried is considerably lower but improves slowly over a long period of time as residual solvent evaporates. The coating system softens in JP-4 fuel after 3 days at 125° F.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. R. WHITMORE
Technical Director
Materials Laboratory
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I. INTRODUCTION

Background:

In May, 1952, the Research and Development Laboratories of the United States Stoneware Company began a study of film-forming coating systems. The purpose of this investigation was to develop a protective coating for exposed metal surfaces in the Nitric Acid Tank and ATO (Assist Take Off) compartments of the B-47 aircraft or similar applications in other Air Force equipment.

Initially, the problem was divided into two phases as follows:

Phase I - To develop a comparatively nitric acid resistant primer which could be used as an adhesive base for sheet or solution applications of a completely resistant coating.

Phase II- To develop a coating system completely resistant to white and red fuming nitric acid; this coating to be based on a fluorocarbon type resin.

With the revision of the contract per Change Order C-3 (54-1525) the investigation program was again divided into two parts but revised as follows:

Phase I - To develop a filleting material which shall be suitable for filling and feathering such joints, cracks or normal surface irregularities as would be encountered in the B-47 ATO compartment, over which the topcoat as defined in Phase II would be applied. The filleting material shall not detract from the metal protection normally afforded by the topcoat.

Phase II - To develop a protective coating material completely resistant to white and red fuming nitric acid capable of being bonded satisfactorily to aluminum and steel. This material shall not lose its adhesion due to white or red fuming nitric acid vapor transmission. The coating material shall be of the Kel-F or Teflon type; a polymeric fluorinated hydrocarbon derivative with properties enabling practical application.

II. DISCUSSION

A. Coating

A. 1 Summary of Former Work

The initial work on this program was of an exploratory nature. A thorough screening of all possible coating raw materials which might be used for nitric acid resistance included materials of recent development as well as those of earlier origin. This investigation pointed out the need for a completely new material, and indicated the need for reliable test methods. The tests adopted as standard include the Pfaudler Test (See Photos 1 and 2), and the H-Cell (See Photo No. 3). Subsequent work has indicated that both methods have merit in specific cases, but that neither has more than comparative value in a program of this type. The Pfaudler Unit has been used to check the resistance of films which were solvent applied to standard aircraft Alclad Aluminum 2024, meeting Specification QQ-A-362. For the evaluation of free films of the pure or compounded resin, the H-Cell is an effective method of testing WFNA resistance.

The results of the foregoing work indicated that the only material applicable by practical methods, which exhibited reasonably satisfactory resistance to WFNA was the polymer X-200 resin. Consistent results could not be obtained with all batches of this material. In addition, it soon became evident that the strength of the resin was, in a sense, its weakness. Kel-F X-200 provided a dense, impermeable film - not appreciably affected by nitric acid. However, as in the case of other synthetic polymeric materials,

appreciable solvent is retained even after extended air dry periods. This is probably due to the dense, impermeable film restricting the flow of any gas, including solvent vapor. Ordinarily solvent retention is not too serious for mild exposure conditions, but in the case of WFNA the solvent retained will be attacked by the acid as would any non-resistant modifying ingredient in the formula. The amount of retained solvent appears to be in direct relationship to the weakened resistance of the film to WFNA.

From the initial work on this contract, it was concluded that a material could be developed for WFNA resistance. Since few materials of a compatible nature could be added to improve application properties without detracting from resistance, the project entered a new phase. Rather than continue basic formulation work, it was decided that X-200 could be used unmodified if the application and drying technique were strictly maintained. The formulation problems were, therefore, resolved to achieving proper solvent balance, and utilization of compatible fluorocarbon plasticizers.

The subsequent investigation under the contract has proceeded along the channels outlined above. A detailed report follows covering the period from May 15, 1954 to July 15, 1955.

A. 2 Formulation

At the conclusion of the two year contract period the first encouraging results of exposure tests were being accumulated.

Room temperature tests in WFNA at 77°F. indicated that the major problem was to obtain a blister-free film for testing. Blistering occurred erratically during the graduated force-dry schedule. This testing work was somewhat handicapped by the fact that the quantities of resin were limited, and different batches did not give reproducible results.

Since M. W. Kellogg was producing only small pilot plant batches of Resin X-200 at this time, there was mutual concern over the lack of correlation in results of the exposure tests. Understandably there was a difference, in some cases, in viscosities of identical solutions made with different lots of the resins. The variations may have been due to the polymer molecular weight distribution. However, subsequent work has indicated that other factors involving technique, and the normal degree of error inherent in working with limited quantities of this material could explain some inconsistencies.

The details of the formulations are reported in Table 1.1 through 1.12. In addition, there is listed a resume of the results of the specific work on the particular formulas pertinent to this report.

The main objective at this time was to obtain a formula which would lend itself to consistent application and drying without blistering. It was apparent that Resin X-200 did not completely follow the pattern expected of a polymeric material of this type.

Solvent balance was important in obtaining, by multiple coats, a film which would be free from blisters and imperfections. When baked at 300°F. the fusion which occurs gives a reasonable degree of mechanical adhesion, but this type of bond is easily ruptured by underfilm creepage and expansion of corrosive gases or liquids. Any blisters present were potential weak points through which the acid penetrated. It was therefore, essential that a balanced solvent blend be used. This blend was selected to release solvents fast enough to prevent sagging, and slowly enough to eliminate surface drying which was usually accompanied by small blisters. All coatings were sprayed, using a Binks Model 80 spray gun. Sprayed films were satisfactory, but occasionally blisters developed during the application or drying period. Since we have not experienced blistering in recent work involving a variety of solvent blends, we believe the primary cause of these defects was related to the varying resin lots used in the earlier work. The original materials were apt to give hazy solutions, and viscosity variations. This would be closely related to the rate of solubility and solvent release. When a panel is sprayed, the amount of cooling at the film surface due to solvent evaporation holds back the incorporated solvents temporarily. On returning to room temperature or during initial force dry temperatures, the faster evaporating solvents in the body of the film are released too quickly through a partially dry surface, causing blisters.

There were two logical approaches to the solvent release

problem; one being the addition of a plasticizer which would hold the film open; a second corrective measure would be pigmentation with acid resistant pigments which would help in solvent release. The only materials available in the plasticizer line which would not degrade the X-200 resin were Kel-F TR-Wax, Kel-F 150 Wax, and Kel-F Plasticizer 3-200. Table 1.1 through 1.3 lists the details of these formulation modifications and the results obtained.

At this time we were using Lot J-4652 of X-200 resin. This was the most promising lot of resin we had received to date, and a graduated temperature baking cycle gave a reasonably good film, although there was still some blistering due to application. The solvent balance used was a blend of fast evaporating solvents and diluents.

It appeared that it might be possible to partially disperse the Kel-F X-200, and plan on a 300°F. fusion temperature. The formulations AF-81A, through AF-84 all gave rather poor results. However, on the basis of our present knowledge of the resin, this may be a worthwhile avenue of approach for future work with this material.

When the supply of X-200 resin lot J-4652 was depleted, we were advised that M. W. Kellogg was temporarily unable to supply a material which duplicated this lot. They supplied an "off-grade" material designated as Lot J-4832 on a no-charge basis for whatever work we might be able to accomplish with "questionable" resin. The

initial tests on this lot were conducted using formulas AF-88 through AF-96, and AF-99-A. While checking this material, it was decided that the solvent balance should be revised to include enough slow solvent to keep the film "open", and plan on using a gradually increasing temperature force-dry schedule.

The revised solvent balance permitted good spray application without blistering. However, with this lot of resin the previously used force-dry schedule did not give a blister-free film after baking. It was possible that the off-grade resin was at fault. Subsequent work indicated that 72 hours air dry at 77°F. was essential prior to starting the force-dry schedule at elevated temperatures. The Pfaudler test results were not encouraging, even at room temperature exposure to WFNA.

It was thus necessary that some control procedure be set up for testing X-200 resin prior to further development work with any new lot. It was also essential that we determine accurately and quantitatively the amount of solvent remaining in the 8-15 mil film at any time during the force-dry period. These phases of this program will be discussed individually in subsequent sections of this report.

A.3 Control Procedure

A development resin such as X-200 is normally available in unblended lots. When sufficient quantities are manufactured, it is customary to blend batches or lots on a selective basis to obtain

the solution viscosity desired for a given resin specification. Since X-200 was in the initial stages of development, it was necessary to consider some preliminary standard procedure for quality control of incoming resin batches.

The following method is suggested for the control testing of X-200 resin. Practical limits have been established to check solution and resistance characteristics of subsequent batches.

Item (1) Retain Samples

On lots of new resin of 5 pounds or more, an 8 ounce sample shall be retained for a period of at least twelve months. Retained samples shall be coded with the lot number, and other pertinent information furnished by the manufacturer. They shall be marked with the date of receipt, and shall be stored at 77°F. until such time as materials produced from the lot have been used in a satisfactory manner within 12 months.

Item (2) Records

A special form shall be used by the laboratory responsible for control testing, with this record available on request. The details considered advisable are listed on page (12) in the proposed X-200 Batch Control Record.

Item (3) Manufacturer's Product Specifications

X-200 resin, Lacquer Grade

(Supplied by M. W. Kellogg Company to U. S. Stoneware Company for inclusion in the latter company's report to WADC, WCRTH-4).

1. Scope

These specifications cover X-200 Resin for use in the manufacture of lacquers, sealants, etc., where a fine particle, ketone-soluble resin of low molecular weight is required.

2. Properties

X-200 Resin, lacquer grade, shall conform to the following requirements:

2.1 Solubility: The resin shall be completely soluble in methyl ethyl ketone at a weight concentration of 20% resin solids at room temperature.

2.2 Viscosity: The viscosity of a 20 weight percent solution of the resin in methyl ethyl ketone at 77°F. shall be B-F on the Gardner-Holdt scale.

2.3 Physical Form: The resin shall be supplied as a fine, white, granular powder, free from hard lumps, dirt, or other contamination.

Item (4) Resistance to White Fuming Nitric Acid

4.1 Make up a test solution of clear lacquer as follows:

X-200 Resin	- 80 grams	(From Lot of Resin to be tested)
Methyl Ethyl Ketone	- 160 grams	
Methyl Isobutyl Ketone	- 160 grams	
Methyl Cellosolve Acetate	- 100 grams	
	500 grams - Total	

4.2 Apply by spray to a 6" x 6" glass panel in such a manner as to give the least amount of material loss from overspray. Apply single coats with a 15 minute drying time between coats. When the entire 500 grams have been applied in this manner, the resulting film should range from 14 - 18 mils (dry thickness).

4.3 Air dry at 77°F. for a minimum of one week in a well ventilated area.

4.4 Force dry in accordance with the following schedule. Keep panels in a horizontal position during the baking schedule. Use a circulating air oven which will maintain a uniform temperature to plus or minus 5°F.

24 hours at 140 - 155° F.
24 hours at 175 - 195° F.
8 hours at 250 - 275° F.

4.5 Carefully strip the film from the glass panel and select a circular area 2-3/4" in diameter for standard H-Cell test. Measure and record film thickness.

4.6 Expose the prepared sample to WFNA in the H-Cell at 130°F. Maintain this temperature by the use of Infra-Red lamps if a suitable constant temperature cabinet is not available. In order to prevent the loss of fumes a suitable water condenser shall be adapted for continuous operation during the test. See Photo No. 3.

4.7 The pH of the water on the water side of the H-Cell shall not fall below 4.5 after four weeks exposure to WFNA.

4.8 Control Procedure

There were several methods used in the preparation of K-200 in film form for the H-Cell tests. The previously described method is most representative of the actual application, and is therefore preferred to pressing the dry powder into sheet form by milling or between heated platens in a laminating press. The Pfaudler test is not recommended in this case to evaluate the suitability of the resin since it does not give a quantitative measure of the impermeability of the film. It will be required that the person conducting the panel preparation for the H-Cell test become proficient through practice. If this test is considered for incorporation into a military specification, it will be necessary to expand on the details of panel preparation, film application, drying and testing.

X-200 BATCH CONTROL RECORD

Lot Number _____ Date Received _____

1. Manufacturer's Specification

a) Solubility	_____	20% Solution of X-200 in Methyl Ethyl Ketone.
b) Viscosity	_____	B-F on Gardner-Holdt Scale at 77°F on solution (a) above.
c) Physical Form	_____	Fine, white granular powder free from hard lumps, dirt, or other contamination.
d) Color	_____	The solution prepared for viscosity tests shall be clear, free from contamination or suspended matter, and shall be substantially colorless.

2. Resistant to White Fuming Nitric Acid (Optional) See Item 4

a) Application _____ Satisfactory or Unsatisfactory.

b) Air Dry Time _____ One week minimum at 77°F.

c) Force Dry _____ hours at _____ Note time and temperature if blisters occur.

_____ hours at _____

_____ hours at _____

d) Average Film Thickness (DRY) of Test Specimen _____.

e) H-Cell Test

	Temperature (125-130°F.)	pH (minimum 4.5) Water side of cell
Start	_____	_____
1 week	_____	_____
2 "	_____	_____
3 "	_____	_____
4 "	_____	_____

A.4 Drying of X-200 Film

Possibly the most important phase of this work has been the investigation of the application and drying of the X-200 film. It was desirable that unmodified X-200 Resin be deposited as a lining on the metal. It was, therefore, necessary to apply this sheet lining from a lacquer base, similar to a sheet lining used in tank lining procedure. The complexity of compartment construction makes sheet lining impossible. In practice, complete drying is difficult to achieve, since the solvents are retained to a greater extent than with other polymeric materials. It appears that if solvents are present in trace quantities, or if blisters and foreign matter are present after the coating application, the breakdown is greatly accelerated. The ultimate objective of the bake is to fuse the coating in place at 15 or more mils with no breaks or blisters in the film.

In recent work it has been found that the actual drying schedule is not limited to that used in panel work. This phase will be further discussed. Heavier films require longer exposure to the lower temperatures in the schedule. Any convenient schedule which increases the temperature at such a rate as to provide a blister-free film after fusion (275 - 300°F.) is considered satisfactory.

Table II lists the formulations tested for drying and the percentage of solvent remaining after each stage of drying. It will be noted that this quantity is appreciable even after 1 week

air dry time, and/or considerable time at elevated temperatures up to 200°F. In order that blisters can be avoided, all of the solvent of a given type must be removed before the temperature is raised above the boiling point of that solvent.

The method for determining solvent release was simple, and believed to be reliable. Small brass panels one inch square were weighed and mounted on 6" x 12" steel panels by use of an adhesive film. Two sets of Pfaudler test panels were also mounted in the same manner, and the entire assembly sprayed in the customary fashion. After one week air dry time at 77°F. all of the panels were removed from the template, and force dried in a circulating air oven. After each stage of the bake, the small panels were accurately weighed to determine the solvent loss. An extended bake at 300°F. gave a reasonably constant weight loss which was later determined to be volatility of the resin, and/or resin-wax blend. For practical purposes, it was assumed that if no blistering occurred at 300°F., all the solvent had been removed, since all solvents used will boil in this range.

It will be noted (See Table 3) that AF-102-4 gave the least solvent retained after baking. This formula contained a blend of fast and slow solvents, but did not lend itself to the subsequent work on "hot-spray" technique. Formulations AF-115, 116 and 117 contained no MEK, and were selected as the preferred formulations for application by hot-spray at 160°F. Actually, the AF-102 series gave excellent results in the Pfaudler test

units considering the fact they were applied at dry film thicknesses of 8-9 mils. However, AF-117 containing no slow solvents did not release solvents as well as AF-102-4 containing Methyl Cellosolve Acetate. This was attributed to the fact AF-115, 116 and 117 were all applied in the 13 - 15 mils thickness range. Subsequent work with the compartments indicated that a much longer drying schedule, particularly in the low temperature range, was necessary to prevent blistering of the film when the thickness exceeded 20 mils.

A.5 Application

The investigation of this phase of the project has been continuous throughout the contract period. A summary of the results will be discussed in this section of the report. All application of X-200 lacquer solutions has been by conventional spray methods. By adjusting the drying time between coats, a wide range of solvents may be used.

Continuous Spray. If very fast solvents are used, it is possible to spray a continuous film in a small confined area. This is not generally recommended since blistering may occur. However, for larger areas it is possible to apply a coating continuously if adequate ventilation is provided. In using standard spray techniques, it was necessary to thin the formulations in varying amounts with the solvent balance used. Since most of our work has been in a spray booth, it is probable that adjustment will have to be made in spray methods

in the application of these coatings to the interior of compartments.

Hot-Spray (160°F.) The recent interest in hot spray techniques for the application of maintenance coatings led to a thorough investigation of hot spray techniques for the application of X-200 coatings. Several formulations were developed based on workable solvent blends for the possible application of X-200 coatings heated to 160°F in a commercial paint heater. For test purposes, PolyFlo heated cups were used, the only basic difference being suction instead of pressure in carrying the hot coating to the atomizing head of the spray gun. The formulation work on this phase is listed on Table I. Panels prepared in this manner were tested in the Pfaudler Unit and were very satisfactory.

It was decided that the hot-spray process would be used in the work being planned regarding the application of test coatings to mock-up compartments designed for test purposes. The advantages were increased "build" per coat and the fact that thinner was not necessary. AF-115 was the best formulation from an application standpoint. Based on previous test work, it had been established that both Chrome Oxide Green and Ultrox were suitable for WFNA resistance. AF-115 was pigmented in a green formulation (AF-119) and a white formulation (AF-120). These were checked by alternating coats to

insure even coverage, and preparing Pfaudler panels for WFNA test. The test results were excellent for films in the 20 mil thickness range, applied by hot-spray. It was, therefore, decided that this system would be used in the final test program.

A. 6 Resistance Test - JP-4 Fuel.

In previous sections of this report the emphasis has been on the development work of a coating for WFNA resistance. The coating developed should also be resistant to JP-4 Fuel in order to completely satisfy the requirements of this contract. Before conducting compartment tests, Pfaudler Units were used to test panels of various X-200 formulations in JP-4 Fuel. These panels were duplicates which had previously been prepared for tests in WFNA in the event a recheck was necessary. Table 3 lists the results of this work. Actually, our final conclusions are based on the compartment tests. It appears that resistance to JP-4 is not completely satisfactory, and that better coating systems can be made available for this purpose.

A. 7 Combined Coating - Filleting

At this point in the program it was necessary to check the coating applied over the filleting material. Several panels were prepared by riveting aluminum strips to standard 6" x 12" aluminum panels. A gap of 1/8" between the strips allowed space for a representative fillet. This work is discussed in detail in Section B - Filleting. We did find that aluminum pigmentation

of the X-200 coating (AF-115) gave improved adhesion to alodized aluminum and provided an interfacial bond for the putty. Following the fusion of the putty and coating at 300°F. there were sufficient blisters to indicate that entrapped air would have to be more completely removed from the putty.

B. Filleting

B. 1 Summary

The former work on the filleting compound closely parallels the investigation of the coatings phase. It consisted of evaluation of various organic polymers as base resins for the filleting compound, modifying ingredients such as plasticizers, and fillers having basic resistance to WFNA in both compounded and uncompounded states. The results of these experiments showed that Resin X-200 was the only logical choice for this application. The only difficulty encountered was the non-uniformity of the resin.

A number of batches of "off-spec" resins were evaluated to determine what manufacturing process would result in a good resin for a filleting compound. These tests were quite exhaustive and indicated that a relatively insoluble resin would be needed. The formulation of the putty was dependent upon the resin to such a great degree that formulation work had to be done on the basis of each individual resin lot. Processing of the putty appeared to be expensive and investigations of suitable methods to make the process more efficient,

simpler and less costly from an equipment standpoint were made.

B. 2 Compounding

Using the J-4322 resin which, at the end of the two year period was found to be the best of the "off-spec" X-200 resins, the formulation developed was:

59-2

J-4322	- 100
Kel-F 150 Wax	- 100
Kel-F Plasticizer Grade Oil	- 50
Ultrox	- 90

This compound exhibited the best resistance to WFNA that had yet been experienced but was still subject to cracking due to the insolubility of the resin. The compound had a good putty viscosity and was resistant to softening and deformation at 300°F. This type formula when used with other more soluble resins resulted in excessive hot flow (See Table I). This same putty, when air dried, was resistant to WFNA at room temperature for 32 days, at which time the test was discontinued. An air dried putty of this formulation was not resistant to WFNA at 160°F. This same material was filled with 1/32" long glass fibers to improve the lateral strength of the fused fillet to prevent cracking, and to eliminate the blistering due to acid penetration of an unfused fillet. Both experiments with the glass fiber were not successful.

During the time when other resins were under test, a different approach to the filleting problem, based on a suggestion by M. W. Kellogg, was made. This involved making a gel of the X-200 resin in a Kel-F Oil and compounding as a rubbery type filleting material. The formulation was:

63-1

X-200 Resin (Blend J-4652 and J-4709)	- 100
Kel-F 1-3 Oil	- 160
Kel-F 10-200 Wax	- 140
*Ultrox	- 100

*Ultrox milled in on cold rubber mill.

This material, when exposed to WFNA at 160°F. for two days, was totally destroyed. The softening point of this mixture was far too low for such accelerated exposure.

When it was discovered that there was no possibility of securing a special resin for filleting work, further compounding was based upon the standardized solution grade resin. As previously mentioned, the 59-2 formulation would result in a filleting compound with excessive vertical flow at elevated temperatures when used with a solution grade resin. The plasticizer ratio was, therefore, lowered:

66-1

X-200 (J-4948-3)	- 100
Kel-F 10-200 Wax	- 107.15
Ultrox	- 71.43

This material, when processed in the normal mixing and extrusion cycle, was far too soluble and the viscosity of the putty was

extremely high. It was too rubbery and stiff for effective deaeration.

The plasticizer content was once again raised with an increase in the filler content to offset any flow at elevated temperatures, as follows:

66-2

X-200 (J-4948-3)	- 100
Kel-F 10-200 Wax	- 140
Ultrox	- 100

This compound also exhibited the stringy rubbery consistency of the former compound. On the basis of these and some hand mixing tests, it was decided that a filleting compound of a true putty-like consistency at room temperature could not be made with the solution grade X-200 resin.

In order to reduce this nerve and to provide filling without sacrifice of WFNA resistance, some Kel-F 300 grade resin was incorporated:

66-3

X-200 (J-4948-3)	- 50
Kel-F 300 grade	- 50
Kel-F 10-200 Wax	- 50
Kel-F Plasticizer grade oil	- 100
Ultrox	- 100

The consistency of this compound was at first quite satisfactory but after one week's aging, it, too, became quite rubbery.

The WFNA resistance of 66-1 and 66-2 was quite good with no evidence of decomposition after five days immersion

at 160°F. Compound 66-3 for the same conditions was badly cracked and blistered.

The softening or hot flow at elevated temperatures seemed satisfactory for the 66-3 compound but was excessive for the 66-1 and 66-2 compounds. This was illustrated by the results of the first test compartment fusion in which the 66-2 formulation was used. (See Section C). It was felt that in a thin fillet there would not be the large mass of material inducing vertical flow and that a compound such as 66-2 would suffice. We found this to be in error and that the thin fillets in the compartments showed excessive flow during the fusion cycle of the coating. A stiffer putty was needed. A stiffer putty would, however, necessitate drastic changes in the processing and application originally decided upon. The following formulation is the recommendation based upon the conclusions of the testing program. It exhibits good resistance to flow at elevated temperatures, has good resistance to WFNA at 160°F. in Pfaudler column tests and is applied in a very simple manner. Its viscosity and shelf life stability are satisfactory and it is the most effectively deaerated compound of any thus far produced.

68-1

X-200 (J-5274) Solution grade	- 100
Kel-F 10-200 Wax	- 120
Ultrox	- 140

This is the composition of the filleting compound finally decided upon as being the best that could be devised for this project.

B. 3 Resins

At the end of the two year reporting period, the Kellogg X-200 resins tested for filleting compounds could be classified into two categories - soluble and very insoluble - on the basis of Kellogg solubility determinations. The soluble resins exhibited the following behavior in a filleting compound:

A - Resulted in high putty viscosity and short shelf-life aging period.

B - Had low deformation and softening temperatures during baking cycles or accelerated exposures.

C - Were supplied in a chunk form and were very difficult to grind to a fine particle size.

The insoluble resins were not satisfactory in a filleting compound because they did not fuse into a continuous fillet and would exhibit cracking when exposed to WFNA at elevated temperatures.

A total of nineteen different "off-spec" X-200 resin batches were evaluated as filleting compounds (See Table I). Of these, only six were found to be promising. The results of our testing on the resins were forwarded to M. W. Kellogg in hopes that through the results, they would be able to select the process responsible for these resins and be able to duplicate them. M. W. Kellogg replied that there was no correlation between our results and their polymerization data or test results. The whole program of resin selection for filleting work was, therefore, discontinued. During this period of evaluation, there had been developed a standardized solution grade X-200 resin. This material was supplied in a

fine flake form which did not require excessive grinding. Since no resin ideally suited for filleting could be had, we were by necessity forced to use the standardized solution grade resin for filleting work. By compound modification, this solution resin has given satisfactory results.

B. 4 Processing

At the expiration of the two year period, the method of processing the filleting compound had been as follows:

1. The ingredients were dispersed in a cooled sigma blade mixer. This necessitated the use of a mixer with capacity to overcome the very high viscosities obtained.
2. At the same time, the compound was being mixed, a vacuum of 24-26 inches was being applied to the mixing chamber.
3. After mixing, the compound was placed into a modified ceramics extruder and extruded into a rod, under vacuum.

The disadvantages of this system were:

1. The effectiveness of the deaeration was questionable. The viscosity of the putty was high and the 24-26 inches of vacuum very probably did not break the solid mass surrounding the air pocket. This was demonstrated by Pfaudler exposures which showed the presence of entrapped air in the vacuumed and extruded product.
2. The process would be quite expensive as it would necessitate the design of new equipment, especially for the filleting compound. All of this equipment would be heavy-duty type.

The new and present methods of processing are as follows:

1. The dry components of the filleting compound are pre-blended.
2. The liquid or wax is pre-chilled and added to the dry preblend.
3. The mixing equipment used is a Hobart N-50 mixer equipped with a pastry knife. This pastry knife cuts the liquid into the dry mix so as to form a granular dispersion. Care must be exercised not to subject the mixture to too long a mix cycle as pasting will occur. The appearance of the mass after mixing is similar to a lumpy vinyl dry blend. (See photo No. 18).
4. This crumbly mix is then transferred to the hopper of an ordinary commercial meat grinder which has been vacuum equipped (See photo No. 19).
5. A vacuum of 24-26 inches is applied to the mix for 20-30 minutes.
6. At the end of this period the extrusion is started, the vacuum continuing.
7. Extrusion can be of any shape or size of rod.

The advantages of this system are:

1. The deaeration of this crumbly mix is very effective- the best that could be accomplished in the time required.
2. The equipment needed would cost in the range of five hundred dollars.

3. The dispersion attained should be quite adequate. The preblending, the compression mixing achieved in the extruder cylinder, and the intense shear at the face of the die will insure adequate dispersion.

The effectiveness of deaeration of the new process was demonstrated by high temperature baking cycles which showed very little thermal expansion and no blistering of the filleting compound.

B. 5 Application

Prior to the usage of the solutiongrade X-200 resin, the application procedure had been merely to putty the crevices, coat with the X-200 lacquer, and put the whole system through the baking cycle.

The usage of the solution grade resin in the filleting compound has resulted in some change in the procedure.

Since the extruded X-200 filleting rod is a rubbery, tough, stiff composition, it cannot be puttied in the usual manner. It is necessary to first soften the filleting material by the moderate application of heat. We have found that a hot air gun is ideal for this purpose.

1. Place the extruded rod into the joint to be filleted.
2. By use of a hot air gun (an ordinary hair dryer has been found quite satisfactory), soften the compound until it is soft and workable.

3. For straight long seams, compressing the soft filleting material into the seam by the use of a roller has been found to be the quickest and neatest method of application.

4. For fillets other than straight seams, the heated putty may be shaped and compressed with other tools.

5. After the fillet has cooled and is hard, the flash or excess may be trimmed off with a knife and reused.

6. The coating may then be applied over the cooled fillet.

B. 6 JP-4 Fuel Tests on Filleting Compounds

Formulation 66-1 and 66-2 were tested in JP-4 fuel for one week at 160°F. The surface effects of the fuel on the filleting compound were not discernable. Upon cutting open the filleting compound, it was apparent that the fuel had penetrated the fillet to some extent. There appeared to be some leaching of the plasticizer from the fillet mass, but there seemed to be no resultant stiffening of the compound. The overall resistance of the filleting compound to JP-4 fuel was good.

C. Compartment Testing

C. 1 Description and Purpose of Test

The background work on this project could not provide a sound basis for a specific recommendation. It was essential that some application tests be run on a larger scale than provided by test panels. The purpose of testing compartments was to determine

a practical method of applying coating system and a filleting material in such a manner to provide protection to a complex structure embodying the essential features of aircraft construction.

Four special compartments were designed for the final test. These were fabricated from .064" - 2024 Alclad aluminum meeting Specification QQ-A-362. A fifth compartment was later fabricated as an extra in case the results indicated a necessity for retesting. These mock-up compartments included:

1. A representative extruded section with bead weld, and extruded angle section.
2. A top-hat section.
3. A "Z" section in vertical position, and also one positioned in the acid container tray.
4. An integral cross-bracing section.
5. A cable housing mount.
6. Fabricated angle sections - in horizontal and verticle positions.
7. Compartment fabricated to comply with standard aircraft structures employing riveted, and mechanical screw fastening devices.

Photographs included with this report show illustrations of the disassembled compartment, and close-up shots of the critical sections which, from a coating standpoint, would be

the most difficult areas to obtain complete protection. Detail drawings are included of both the test compartments and the outer "safety shell" which was used in testing with WFNA. This was merely a protective housing for the test cell while it was being tested at 160°F. in a commercial type oven.

This test was designed to simulate as closely as possible the difficulties which might be encountered in coating an ATO aircraft or rocket compartment. The compartments which have been tested, and the extra compartment, will be placed at the disposal of WADC for their examination, and further use.

The test conducted on these cells were as follows:

Cell A - Tested with JP-4 Fuel for 10 days at 160°F.

Cell B - Tested with WFNA for 5 days at 160°F.

Cell C - Tested with WFNA for 10 days at 160°F.

Cell D - Tested with JP-4 Fuel for 10 days at 160°F.

Cells C and D were first cycled on the following schedule:

160°F. in oven for 3 hours)
-65°F. in cold box for 3 hours) This cycle was repeated for 10 days allowing 2 hours per day for the transfer between oven and cold-box.

This test, therefore, included two cells which were subjected to heat and cold extremes, and two control cells which were not cycled.

C. 2 Metal Preparation and Priming

All cells were prepared in the same manner prior to coating. The aluminum cell sections (disassembled) were first solvent cleaned with methyl ethyl ketone. An alodizing solution was prepared from

*Alodine Liquid No. 500 (1 quart) mixed with *Alodine "Make-Up Powder No. 5 (2 pounds) diluted with 6 quarts of distilled water. This material was brushed on and rinsed off after 3 minutes contact at 77°F. The surface was then thoroughly washed with clean cold water and dried.

AF-121 X-200 Aluminum Primer was applied by spray to attain a thickness of 0.5 - 1.0 mils over all parts of the compartment. Following a 24 hour air dry period, the compartments were filleted in accordance with C.3 which follows:

C. 3 Filleting

Compartments D, A and B were filleted with Compound 66-2. The putty was softened by a hot air blower until workable and rolled or compressed into the seams by a hand roller. The joints or seams which could not be rolled were filleted by pressing the heated, softened putty into place by hand or putty knife. After cooling, the flash was trimmed off and re-used. The 66-2 putty proved to have too low a softening point, resulting in excessive blistering and vertical sagging.

Compartment C was filleted in the seams with 68-1 formulation in the above manner to reduce the blistering and vertical flow.

*American Chemical Paint Company
Ambler, Pennsylvania

C. 4 Coating Application - Discussion

Following the filleting operation, all compartments were spray coated with a second coat of AF-121 Aluminum Primer over the entire surface area, including the putty joints. (All loose putty and other foreign particles were first removed by light sanding, followed by dusting with air pressure.)

Initially, we had planned to use the procedure outlined in A.5 (Hot-spray at 160°F.) for the application of coating. Since this was a test application, it was decided to check formulations which were identical except for the fact that one contained 10-200 Wax. The second formulation contained no wax. The 10-200 wax has some advantages in this case in that it permits higher "build" per coat, and controls solvent release. It was possible that the "cold test" at -65°F. would indicate the need for some plasticizer. Chart V lists the coating formulations tested, and parts of the cell to which they were applied.

All coating operations were essentially the same. Alternate coats of green and white were applied by spray. In coating the first compartment, it became apparent that the "hot-spray" process would not be satisfactory. At any pressure setting, or fluid-air mixture, the dry spray which resulted was detrimental to good coating procedure. Panels which had been coated previously had not occupied a large area in the spray booth, and for that reason we did not experience difficulty with ventilation. If the hot-spray formulas were thinned to the viscosity at which spraying was

satisfactory, the cold-spray technique was equally effective. For this reason it was decided that the best procedure would be to spray by the conventional method, using a suction cup and 35 - 50 lbs. atomizing pressure. There were certain areas such as edges, corners and structural joints to which the material was difficult to apply. The best procedure was to narrow the fan width on the gun until a stream of material could be directed conveniently, and in effect, flowed on. Regardless of sags, it was decided that appearance could be sacrificed for complete coverage.

The procedure of application was as follows:

(1) One compartment was coated at a time, using two men, each coating half of each cell.

(2) 15-30 minutes minimum were allowed for tack-free time between coats. When 6-8 coats had been applied in one 8 hour period, the parts were allowed 16 hours to dry overnight. The temperature varied from 55°F. to 70°F. during the drying period between coats.

(3) The coverage on the white formulation was low, and in order to cover the green, it was necessary to use excessive amounts of pigment in the formulation. Actually, it is easy to judge when a complete cover coat has been applied, and no attempt was made to cover the green color completely.

(4) The odor of the di-isobutyl ketone in the formula was very objectionable and as previously stated, it is planned that we will use the former solvent balance containing ethyl amyl ketone as the residual slow solvent.

(5) The coating application was scheduled to allow at least one week air dry time before the force dry schedule was started. Twenty coats were applied to the first 3 compartments and 18 to the last cell ("C"). It was found, after baking the first compartment, that we had obtained over 20 mils on the application with the pigmented materials.

After the first compartment had been force dried, it was obvious, that the baking schedule previously used for test panels was not satisfactory for applications in which a heavier coating was applied. The oven was designed for a higher temperature range, and could not be accurately maintained constant in the 115-200°F. range. Compartment D was processed first, followed by A, B and C. Compartment D blistered badly on baking in the 250°F.-300°F. range. This was used in the JP-4 Fuel Test, and the blisters were not detrimental to the test. It was probable that this blistering occurred because of the following reasons:

1. Film was very heavy in certain areas which, of necessity, had to be excessively coated in order that adjacent areas received sufficient coating.
2. The solvent balance used was designed for "hot" spray rather than for cold application, and the wet material did not lose as much solvent between the gun and target to be coated.

Our primary interest in baking was, of course, to obtain a solvent-free film regardless of what solvent balance was used. Therefore, the same materials were applied to the remaining cells, and the baking schedule modified. Compartment "A" was slightly blistered, and compartments "B" and "C" may be considered representative insofar as the baking schedule for this particular series of coatings is concerned.

When Compartment "A" was tested in WFNA, it was found that the seams and bolt-heads were points of severe attack. (See Sec. III - Conclusions). Therefore, a filleting and touch-up procedure was used after the initial force dry schedule. This was performed on the assembled compartment, contrary to the previous procedure of force drying coated parts, and then assembling them for the corrosion test. A second (shorter) bake cycle

was used to remove solvents introduced during the touch-up (or repair) stage. (See Table 4 - Baking Cycle - Compartment C).

C. 5 Results

C.5.1 JP-4 Fuel

Compartments A and D were exposed to JP-4 Fuel for 10 days at 160°F. Compartment D was cycled in accordance with C.1, 3 hours in oven at 160°F, 2 hours to transfer to the cold box and 3 hours in the cold box at -65°F. The compartment was allowed to stand overnight at room temperature. This cycle was repeated once each day for 10 days. In no case was there any indication of failure due to the cycling. Both the plasticized and unplasticized X-200 films are unchanged by the cycling process. The X-200 film was softened by the JP-4 Fuel, and in Cell A there was some blistering in the pan which contained the JP-4 Fuel. The coatings were not solvated in any case, but were softened sufficiently to cause loss of adhesion. After 30 days, there appears to be an appreciable amount of JP-4 Fuel remaining in the film. See Photos No. 25, 26, 39, 40.

C.5.2 White Fuming Nitric Acid

Compartments B and C were exposed to WFNA. Compartment B was not cycled, and was exposed to WFNA for 5 days at 160°F. The initial observations on the results of this test indicated the need for better sealing of the edges and

seams after assembly of the compartment. At any point when there was acid penetration, blistering and lifting occurred. Steel bolts were used to fasten the compartment sections together. These were brush coated with AF-120 and air dried before exposure. This procedure was not adequate, and, therefore, Compartment C was treated in a different manner. Photos of Compartment B before and after exposure are included in Photo No. 27-30. The tray (containing the WFNA) was badly attacked at several points such as edges, and places where the putty had blistered. After 5 days test, the tray was too badly attacked to re-expose, and the test was discontinued. Other points of attack were under the tray where spillage had occurred, and on the cable housing mount (from fumes). At points where the sides and bottom were assembled after coating, there was corrosion starting where the coating had been compressed (and possibly cut) during assembly.

Compartment C was cycled in accordance with the schedule described for Compartment D. Following this cycle, the results of the test on Compartment B indicated the need for better sealing along the edges which were butted during assembly. This compartment was further

filleted and 5 heavy brush coats of AF-120 Green applied over the filleting. All bolt heads were heavily capped with putty and recoated by brush. The compartment was then rebaked in accordance with the cycle listed in C. 4, Chart V.

The results of this exposure indicate that at 5 days continuous exposure to WFNA, moderate breakdown occurred at several points, principally along the Z section fabricated into the acid tray. At 10 days exposure, the underfilm creepage was more severe, but the coating itself was not attacked. Photos No. 31-38 show the results of this test. There was no attack from vapor except at points where the coating application was thin.

Note: All four compartments will be forwarded to WADC for examination.

C.5.3 Panel Tests

There was some question regarding the advisability of force drying X-200 coatings, particularly since the tests we were conducting were severe, and indicate the worst which might be expected. It was, therefore, decided that panels would be exposed to WFNA vapor only in conjunction with the test on Compartment C. In addition, an adhesive priming system had been investigated for possible

use with X-200 coatings. The details and results of this test are given below. No conclusions have been indicated since the results have not been rechecked, and therefore, are open to question.

All panels were given an identical primer system as follows: 1 coat of wash primer, 1 coat of Tygonite 8A*, 1 coat of Tygonite 8B* and 1 coat of AF-121. Topcoats as indicated were applied over the primer.

Panel No.	Topcoat	Coats	Cure	Results
1	AF-119 and 120 (alternating)	16	Baked	Blistered
2	"	17	Air dry	Bad blistering
3	"	17	Baked	Blistered
4	AF-120	13	Air dry	Bad blistering
5	AF-122 and 123 (alternating)	16	Baked	Good
6	"	10	Air dry	Blistered
7	"	16	Baked	Excellent
8	"	10	Air dry	Blistered

The adhesives Tygonite 8A and 8B gave excellent results in maintaining the bond and preventing creepage under the film. An even line of penetration of about 1/4" in 10 days gave the first indication that the rate of creepage may be held to a minimum. This will require further test before any recommendation can be made.

*Tygonite 8A and 8B are proprietary products manufactured by U. S. Stoneware Co.

III. CONCLUSIONS

A. Coatings

1. An X-200 coating system has been developed which, when properly applied, will give substantially better resistance to WFNA than any materials heretofore tested.
2. This coating system is softened by JP-4 fuel, but is not otherwise attacked. The fuel is retained for an appreciable time in the coating film, and adhesion is reduced.
3. A system composed of an aluminum primer (AF-140A) and alternate white (AF-140C), and green (AF-140B) provides the most convenient method of obtaining a uniform adherent film.
4. The coating system must be force dried to achieve maximum FNA resistance. A graduated schedule of increasing temperature must be followed to prevent blistering. Extended air drying may be used when maximum resistance is not required.
5. Adhesion of dry (solvent free) X-200 film is sufficient for normal abrasive forces. However, creepage occurs under the film at any point of acid or acid fume penetration. An adhesive system used in tank lining application appears to materially reduce this creepage. This material is produced by U. S. Stoneware Co. as Tygonite 8A Primer and Tygonite 8B, and is used over wash primer. Test data on this material are insufficient to warrant any definite conclusions at this time.

6. The X-200 coating system may be applied by conventional methods of brush, spray, or dip. However, spray coating is recommended as the most appropriate method of application to compartments.
7. All cracks, crevices and joints will require careful filleting and sealing. Any breaks in the coating allow severe blistering to take place.
8. Coating production and application must be done under controlled conditions of cleanliness. Dirt, lint and contamination of X-200 lacquers influence the degree of protection afforded by the applied coating.
9. Manual stripping of the primer-topcoat system causes cohesive failure within the primer. Additional work, beyond the contract, will be undertaken to improve the coating system adhesion.

B. Filleting

1. Resin X-200 as supplied by the M. W. Kellogg Co. was the only material which was sufficiently inherently resistant to WFNA to perform as a base for the filleting compound.
2. A putty-like material was the best form to insure filling instead of bridging of seams and voids.
3. Studies of "off spec" X-200 resins which showed decided advantages for usage in a putty were fruitless in that the resins could not be reproduced.

4. The resistance of the putty to WFNA was primarily dependent upon the amount of free air space in the putty or the extent of deaeration, and the resistance to softening and vertical flow when temperatures in excess of 160°F. were encountered.

5. The standardized solution grade X-200, though not an ideal resin for a filleting compound, has been satisfactorily compounded.

6. Processing of the filleting compound will not involve costly or special equipment but can be done in conventional inexpensive equipment.

7. The processing used insures the best possible deaeration of the putty.

8. Application of the putty, on articles of small mass, can be accomplished by heating with a portable hot air blower during application. Larger structures may require preheating of the area to be filleted and the filleting putty in addition to hot air heating during application. For air dry applications, where resistance to flow at 300° F force dry temperature is not important, a softer, more workable putty can be prepared.

9. Resistance to JP-4 Jet Fuel of the filleting compound has been good, with no apparent solvation or extraction, but the fuel does penetrate the fillet.

10. Resistance to vertical sag or flow has been quite satisfactory.

11. A repeated cycling from 160°F. to -65°F. has not affected the filleting.

12. The blistering occurring at 300°F. in the fillet has been due to:

    (a) Improper drying of the overcoat of X-200 lacquer.

    (b) Entrappment of air under the fillet in the seam which expands on heating and may force the fillet out of the seam at its weak point.

IV RECOMMENDATIONS

A. 1 Coatings Formula

The following formulas have been adopted for recommendation:

Material	AF-140 Aluminum	AF-140B Green	AF-140C White	AF-140 Thinner
X-200 Resin	15.50	18.24	18.24	—
10-200 Wax	2.74	—	—	—
Alcoa 422 Aluminum Flake	9.00	—	—	—
Chrome Oxide Green	—	6.70	—	—
Zirconium Silicate	—	2.16	8.86	—
Methyl ethyl ketone	18.20	18.25	18.25	31.25
Methyl isobutyl ketone	18.20	18.25	18.25	25.00
Ethyl butyl ketone	13.60	13.65	13.65	18.75
Ethyl amyl ketone	4.56	4.55	4.55	—
Xylol	9.10	9.10	9.10	12.50
Toluol	9.10	9.10	9.10	12.50
(Total)	100.00	100.00	100.00	100.00
Wt/gal.	8.28 lbs.	8.55 lbs.	8.46 lbs.	6.76 lbs.
No. 4 Ford cup visc. at 77°F.	47 Sec.	67 Sec.	67 Sec.	—
Fineness of grind (North Standard)	(Mix only)	8 NS	8 NS	—

A. 2 Application Procedure - Coatings

The following procedure is recommended for the application of X-200 coatings to ATO Cells, or to test panels used in preliminary investigation of the coating.

1. Thoroughly wash (or degrease) with methyl ethyl ketone or other suitable solvent all metal be coated.
2. Application of a chemical chromate pretreatment, such as covered by MIL-C-5541, will improve metal-coating adhesion.
3. Apply one coat of AF-140-A Aluminum Primer thinned 4 to 1 with AF-140 Thinner.

Note: Preliminary test results indicate that use of a "wash" primer followed by a vinyl tie-coat applied over the metal pretreatment and prior to the X-200 aluminum primer will give excellent coating adhesion. The "wash" primer also appears to retard corrosion undercutting of the film where there is a break in the film.

Note: At this point, if filleting is to be used, follow the instructions in (B. Filleting). Clean and sand lightly after filleting is applied, and apply one full coat of AF-140-A Aluminum Primer over the filleting prior to using the alternate topcoats.

4. Apply alternate coats of AF-140-B Green, and AF-140-C White reduced 4 to 1 for spray. Apply a total of 20 coats allowing a minimum of 15 minutes air dry between coats, and 16 hours after the 8th and 16th coat. The pigmentation is intended for identification convenience only, and it is not essential that complete hiding be obtained on each coat.

5. For spray application, 35 - 50 lbs. is used on atomizing air. As the application proceeds, it will be advisable to lower the pressure and apply slightly less per coat.

6. Allow one week air-dry time at 77°F. or higher.

7. For force dry, bake in an accurately controlled oven with full air circulation, or use heated air in the compartment, thermostatically controlled to maintain the required temperature to plus or minus 5°F. The air circulation should be baffled so as not to impinge directly on a coated surface, or blistering will occur.

THE FOLLOWING SCHEDULE MUST BE FOLLOWED:

24 hours at 100 - 114°F.
24 hours at 120 - 125°F.
8 hours at 150°F.
8 hours at 170°F. (Do not exceed 176°F. on this portion of cycle)
8 hours at 200°F.
4 hours at 250°F.
1 hour at 275°F. (Coated work should fuse and become glossy at this temperature.)

8. Check baked material for pin-holes, blisters which penetrate to an appreciable depth, or other flaws in the coating which might expose bare metal.

9. Repair any damaged areas with several brush coats of unthinned AF-140-B Green or AF-140-C White.

10. If the amount of brushed-on coating is neglible, the work may be dried for a sufficient length of time to allow the additional solvent to migrate evenly throughout the coating (24-48 hours) and the coated metal rebaked (starting the cycle at 8 hours at 170°F.) and continuing on the same schedule as previously used.

B. 1 Formula - Filleting

The following formula, 68-1, was found to have satisfactory flow resistance at force dry temperatures (For air dry systems softer modifications of this formula may be used).

X-200 (J-5274) Solution Grade	- 100
Kel-F 10-200 Wax	- 120
Ultrox	- 140

This material is produced in the form of an extruded rod. It necessitates heat softening during application. It is the most effectively deaerated of all the putties thus far produced. Its resistance to WFNA has been satisfactory.

B. 2 Application of X-200 Filleting Material

The X-200 filleting material is supplied in an extruded form. This material has been extruded under vacuum and is relatively free of entrapped air. The formulation has a high softening point and requires heat to make it soft and workable. The following procedure is recommended:

1. Allow AF-140-A Aluminum Primer to air dry 18-24 hours.
2. Place extruded rod in seam to be filleted.
3. By using a hot air blower, apply heat until the putty is soft and workable. An ordinary small electric hair drier is completely satisfactory.
4. When putty is soft, compress into the seam or joint by the use of a small roller. A roller of about 1/4" width with a semi-circular rolling edge is recommended.
5. After the putty is pressed into the seam and has cooled, trim off excess and use excess for other seams.
6. Seam should be examined to see if it is pressed tightly against the primer.
7. For fillets other than seams where a solid lump of fillet is needed, preheat the putty until soft and press into the cavity with a putty knife or similar tool.

8. The fillet is now ready for X-200 lacquer coating.

The reason for the stiffness of the filleting compound is to prevent sagging on vertical surfaces when temperatures of 300°F. are encountered.

V SERVICE TEST APPLICATION

A service test application of the recommended X-200 coating system was accomplished at North American Aviation, Inc., International Airport, Los Angeles, California, in November 1955. Laboratory testing of the material prior to application and shop scheduling indicated that the following changes, as compared to the recommended procedure, would be desirable.

1. Laboratory testing of the coating system revealed that when the cured coating was stripped from a panel, separation always occurred within the aluminized primer. The major part of the primer remained on the panel but the stripped topcoat always carried a thin film of aluminum pigment. This occurred on both air-dried or force-dried coatings applied over both solvent cleaned or chemically treated aluminum. Overall adhesion of the coating system was very good. In view of the primer being the weakest link the aluminum was not chemically treated prior to coating. Work to improve the cohesion of the primer is continuing.
2. The filleting material used, Formula 68-1, was designed for force dry temperatures and was found to be rather difficult to apply. It was found necessary to preheat the filleting material, preheat the metal with heat lamps or, for smaller parts, by conditioning in an oven and apply hot air on the fillet during forming. A softer formulation has been prepared for use in air-dry applications.
3. The lacquer was applied at the rate of 6 to 8 coats per 8-hour day with a 16 hour, over night, air-dry after each 8-hour painting period.
4. Results of the service test should be available by September 1956.

TABLE 1.1 FORMULATION - DETAILS AND RESULTS OF TESTING - PFAUDLER EXPOSURE TO WFNA

FORMULA NUMBER	COMPOSITION		VISCOSITY 4 FORD CUP	PURPOSE OF FORMULATION	Type of Test Conducted: Film Thickness	Drying Schedule	Results	PHOTO REF.
AF-78A	X-200	15.0						
	3-200		43 Sec.	Check new lot of X-200 Resin for solution properties	No further tests			None
	Oil	5.0						
*J-4652	MEK	30.0						
	MIBK	30.0	Slight cloud					
	Toluol	20.0						
		100.0						
AF-78B	X-200	15.0						
	3-200							
	Oil	5.0	48 Sec.	Check new lot of X-200 Resin for solution properties	No further tests			None
*J-4709	MEK	30.0						
	MIBK	30.0	Slight cloud					
	Toluol	20.0						
		100.0						
AF-79A	X-200	20.0				100°F.-24 hrs.		
	MEK	30.0	197 Sec.	Control with no wax - to be used in checking TR-wax in formula	Top-10 mil	125°F.-24 "	Exposed 43 days at 77°F. Blistered	
*J-4652	MIBK	30.0				150°F.- 8 "		
	Toluol	20.0	Clear		Bottom- 9 mil	175°F.- 8 "		
		100.0				200°F.-16 "		
						250°F.- 4 "		
						300°F.- 1 "		
AF-79B	X-200	17.0						
	TR-Wax	3.0	156 Sec.	Check TR-Wax at 15% of Resin X-200	Top-16 mil			
*J-4652	MEK	30.0				Same as above	Exposed 106 days- at 77°F. Blistered- some occurred in bake cycle	
	MIBK	30.0	Clear		Bottom-11 mil			
	Toluol	20.0						
		100.0						

*Indicates lot number of X-200 used in formulation.

TABLE 1.2 FORMULATION - DETAILS AND RESULTS OF TESTING - PFAUDLER EXPOSURE TO WFNA

FORMULA NUMBER	COMPOSITION		VISCOSITY # FORD CUP	PURPOSE OF FORMULATION	Type of Test Conducted: Film Thickness	Drying Schedule	Results	PHOTO REF.
AF-79-C *J-4652	X-200 TR-Wax MEK MIBK Toluol	16.0 4.0 30.0 30.0 20.0 100.0	75 Sec. Clear	Check TR-Wax at 20% of Resin X-200	Top-11 mil Bottom-11 mil	Same as above	Exposed 94 days at 77°F. Blistered. This occurred in bake cycle and was worse after exposure	
AF-80A *J-4652	X-200 3-200 MEK MIBK Toluol Ultrox	18.0 2.0 30.0 30.0 20.0 8.7 108.7	146 Sec.	Check increased X-200 resin content vs. 3-200 Kel-F plasticizer	1-22 mil. 2-23 " 3-22 " 4-22 " 5-22 " 6-23 "	100°F-24 hrs. 125°F.-8 " 150°F.-16 " 175°F- 8 " 200°F- 16 " 250°F- 4 " 300°F- 1 "	First set of panels blistered badly on bake. 2nd set dupl. these results. Blistering too heavy for test in Pfaudler	
AF-81A *J-4652	X-200 MEK Methylene chler. Ultrox	15.0 75.0 10.0 8.7 108.7	46 Sec.	Check appl. of X-200 dispersed rather than dissolved	1-41 mil. 2-37 " 3-38 " 4-42 "	100°F-24 hrs. 125°F- 8 " 150°F-24 " 175°F- 8 " 200°F-16 " 250°F. 4 " 300°F- 1 "	2 sets prepared and both blistered badly No test	
AF-82 *J-4652	X-200 Acetone	15.0 75.0 90.0	32 Sec. (dispersed)	Check acetone as a dispersant		Air dry 2 weeks	Panels prepared-tested No. 4 24 days at 77°F-No good	

TABLE 1.3 FORMULATION - DETAILS AND RESULTS OF TESTING - PFAUDLER EXPOSURE TO WFNA

FORMULA NUMBER	COMPOSITION		VISCOSITY 4 FORD CUP	PURPOSE OF FORMULATION	Type of Test Conducted: Film Thickness	Drying Schedule	Results	PHOTO REF.
AF-82A	Same as AF-82 with Ultrox added for hiding						Panels prepared and tested 24 days at 77°F. No good	
AF-83	X-200	15.0						
	Acetone	75.0	Insoluble	To check weaker solvent-dispersant				
*J-4652	Methylene chlor.	10.0					No tests	
		100.0						
AF-84	X-200	15.0						
	3-200	2.0	40 Sec.	Solvent bal. check			Poor solubility- No test	
*J-4652	Acetone	65.0						
	MEK	18.0						
		100.0						
AF-86 AF-86A AF-87	These materials were prepared, using Ultrox Fiberglass, and Chrome Oxide Pigments in X-200 - MEK solution				Panels blistered on bake		No tests	
AF-88	X-200	15.0		Check new lot of X-200 in balanced solvent blend for appl. and dry	5-11mils	100°F-24 hrs. 125°F- 8 hrs.		
	3-200				6-11 mils	150°F-24 hrs. 176°F- 8 hrs.		
	Wax	5.0			7-12 mils	200°F-16 hrs. 250°F- 4 hrs.	Exposed to WFNA for 28 days - at 77°F.	No.5
*J-4832	MEK	20.0	43.5		8-11 mils	300°F- 8 hrs.		
	MIBK	27.0						
	EAK	13.0						
	Toluol	10.0						
	Xylol	10.0						
		100.0						
AF-89	Slight change in solvent blend - See AF-90 with TR-Wax replacing 3-200							No.6

TABLE 1.4 FORMULATION - DETAILS AND RESULTS OF TESTING - PFAUDLER EXPOSURE TO WFNA

FORMULA NUMBER	COMPOSITION		VISCOSITY 4 FORD CUP	PURPOSE OF FORMULATION	Type of Test Conducted: Film Thickness	Drying Schedule	Results	PHOTO REF.
AF-90	X-200	17.0		Check in AF-90	1- 12 mils	20 days Air Dry	Pfaudler panels	No. 7
	TR-Wax	3.0		AF-91, AF-92	2- 16 mils	at 77°F.	failed 1 day	
*J-4832	MEK	20.0		available	3- 12 mils	100°F-24 hrs.	Panels blistered	
	MIBK	20.0	58 Sec.	plasticizers	4- 15 mils	125°F- 8 "	on bake	
	EBK	15.0		for X-200		150°F-24 "	Tested in	
	EAK	5.0				176°F- 8 "	Pfaudler at	
	Toluol	10.0		Also-further		200°F-16 "	125°F.for 1	
	Xylol	10.0		check solvent		250°F- 4 "	day-complete	
		100.0		revision		300°F- 1 "	failure	
AF-91	X-200	17.0					Exposed to WFNA 23 days at 77°F.	
	Kel-F 150	3.0		Same as above	Same as above			
*J-4832	MEK	20.0	64 Sec.					
	MIBK	20.0						
	EBK	15.0						
	EAK	5.0						
	Toluol	10.0						
	Xylol	10.0						
		100.0						
AF-92	X-200	17.0			1- 20 mils		Blisters started	
	3-200	3.0			2- 13 mils	Air dry-14 days	in 1 day at 77°F. Removed in 7 days	
*J-4832	MEK	20.0	62 Sec.	Same as above				
	MIBK	20.0						
	EBK	15.0			3- 13 mils	Air dry-14 days	Tested at 125°F.	
	EAK	5.0			4- 18 mils		Failed in 1 day	
	Toluol	10.0			5- 11 mils	Same schedule	Tested at 125°F.	
	Xylol	10.0			6- 11 mils	as AF-90 above	Failed in 1 day-removed in 4 days	
		100.0						

Note- Lot J-4832 reported Off spec
No others available

TABLE 1.5 FORMULATION - DETAILS AND RESULTS OF TESTING - PFAUDLER EXPOSURE TO WFNA

FORMULA NUMBER	COMPOSITION		VISCOSITY 4 FORD CUP	PURPOSE OF FORMULATION	Type of Test Conducted: Film Thickness	Drying Schedule	Results	PHOTO REF.
AF-93 AF-94 AF-95 AF-96	Special series used for checking solvent release. The AF numbers refer to pages of discussion in Lab. notebook rather than actual formulation work.						This work covered by AF-102-1,2,3,4 and details covered in this report.	
AF-97	X-200	17.0		Check new batch of resin and standard plasticizer 10-200 wax. Solvent bal. is also standard.	1- 7 mils	100°F.-24 hrs.	Set 1 - exposed in Pfaudler at 77°F-48 days to complete failure	No. 8
	10-200 Wax	3.0			2- 9 mils	125°F- 8 "		
*J-4948-1 (Match for Lot J-4652	MEK	20.0	35 Sec.			150°F- 24 "		
	MIBK	20.0				176°F- 8 "		
	EBK	15.0				200°F- 16 "		
	EAK	5.0			3- 9 mils	250°F- 4 "		
	Toluol	10.0			4- 9 mils	300°F- 1 "	Set 2-exposed in Pfaudler at 125°F. for 6 days to complete failure	No. 9
	Xylol	10.0			Note: All panels blistered slightly before exposure			
		100.0						
AF-97 White	AF-97	100.0	—	Check pigmented coating on same schedule as above.	All over 15 mils	Same as AF-97	Set 1- exposed in Pfaudler at 77°-8 months-OK	No. 10
	Ultrox	8.7					Set 2 - exposed in Pfaudler at 125°F-blistered in 14 days	No. 11
		108.7						
AF-98A	X-200	20.0		Check solvent retention - help establish control procedure	4 lbs/sq.ft.	3 coats-continuous to give 8 mils/coat	Glass panel coated to measure 1.Coverage/lbs. of material 2.Revision of bake 3.Unsupported film for H-cell	
	MIBK	40.0						
*J-4948 -1	MEK	40.0	45 Sec.		About 25 mils avg.	1/2 hr.dry-30' at 60°C		

TABLE 1.6 FORMULATION - DETAILS AND RESULTS OF TESTING - PFAUDLER EXPOSURE TO WFNA

FORMULA NUMBER	COMPOSITION		VISCOSITY 4 FORD CUP	PURPOSE OF FORMULATION	Type of Test Conducted: Film Thickness	Drying Schedule	Results	PHOTO REF.
AF-96 J-4832	Same as above except for resin(lot) of X-200			Submitted for H-cell tests with same material made from Lot J-4832		Bake 16 hrs. at 140-150°F. 16 hrs at 176°-194°F. 4 hrs. at 250°-275°F.	Some large scattered blisters but good application	
AF-99A J-4832 (Off spec)	X-200 MEK MIBK Meth. Cell. Acetate	16.0 32.0 32.0 20.0 100.0	54 Sec.	Same as above	Same as above		Same as above Application webbed somewhat. Results of H-cell test reported in detail. No apparent difference between lots.	
AF-101-1 *J-4948 -1	X-200 10-200 Wax MEK MIBK EBK Toluol Xylol EAK	17.0 3.0 20.0 20.0 15.0 10.0 10.0 5.0 100.0	37 Sec.	1)This will be a detailed test to determine solvent retention at various stages of bake. 2)Pfaudler panels will be prepared for future test (Control formula)	Air Dry- 13 Mils Baked 9 Mils	114°F-22 hrs. 125°F- 8 " 150°F-12 " 176°F- 8 " 250°F- 4 " 300°F-14 " Air dry time before bake (1 week)	1)No blistering on bake. 2)Solvent retention-See chart- "evaporation rate" Exposed in Pfaudler-WFNA 27 days at 77°F.- O.K. 7 days at 125°F.- O.K. 8 days at 160°F.- Blistered	No.12

TABLE 1.7 FORMULATION - DETAILS AND RESULTS OF TESTING - PFAUDLER EXPOSURE TO WFNA

FORMULA NUMBER	COMPOSITION		VISCOSITY 4 FORD CUP	PURPOSE OF FORMULATION	Type of Test Conducted: Film Thickness	Drying Schedule	Results	PHOTO REF.
AF-101-2	X-200	17.0		Same as above except	Air dry		(1) and (2) Same as AF101-1 above	
	10-200							
*J4948-1	Wax	3.0			13.5 Mils	Dried with AF-101-1	Exposed in Pfaudler-WFNA	
	MEK	20.0	38 Sec.	Check EAK against methyl cellosolve acetate as residual slow solvent.			27 days at 77°F- C.K.	
	MIBK	20.0			Bake 8-9 Mils		6 days at 125°F- Edge	
	Meth. Cell.Ac.	5.0						
	EBK	15.0						
	Xylol	10.0						
	Toluol	10.0						
		100.0						
AF-101-3	X-200	20.0		Same as above except	Air dry 11 mils		(1) and (2) Same as AF101-1 above.	See Chart III
	MEK	20.0	72 Sec.			Dried with AF-101-1		
*J4948-1	MIBK	20.0					Exposed to JP-4 Fuel	
	EBK	15.0		Check against control (AF101-1 only 10-200 Wax removed.	Bake 8 mils		3 days at 77°F.- O.K.	
	Toluol	10.0					3 days at 125°F- Softened	
	Xylol	10.0					1 week at 160°F- Very soft	
	EAK	5.0						
		100.0						
AF-101-4	X-200	20.0			Air dry		(1) and (2)	
	MEK	20.0	72 Sec.	Same as above except	11 mils	Dried with AF-101-1	Same as AF101-1 above.	No.13
*J4948-1	MIBK	20.0					Exposed in Pfaudler WFNA	
	EBK	15.0			Bake 8 mils		7 days at 125°F.- OK	
	Toluol	10.0		Check against AF101-2 which has 10-200 wax			7 days at 160°F- Blistered	
	Xylol	10.0						
	Methyl cello acetate	5.0						
		100.0						

TABLE 1.8 FORMULATION - DETAILS AND RESULTS OF TESTING - PFAUDLER EXPOSURE TO WFNA

FORMULA NUMBER	COMPOSITION		VISCOSITY 4 FORD CUP	PURPOSE OF FORMULATION	Type of Test Conducted: Film Thickness	Drying Schedule	Results	PHOTO REF.
Note - Subsequent work was directed toward the development of a satisfactory solvent balance for "Hot Spray" of these materials. This was intended as a method of application by which more solids could be applied per coat.								
AF-105	X-200	17.0	38 Sec.	Check hot spray characteristics	Hot sprayed at 160°F.		No feathering 5-7 passes could be applied before sags developed	
	10-200							
	Wax	3.0						
*J-5175	MIBK	30.0						
	EBK	15.0						
	EAK	5.0			No further tests			
	Toluol	15.0						
	Xylol	15.0						
		100.0						
AF-106 *J4948-3	Same as AF-105 using different lot of resin		73 Sec.	Same as above	Same as above - AF-105			
AF-107 *J4948-1	Same as AF-105 using different lot of resin		43 Sec.	Same as above	Same as above - AF-105			
AF-108 *J4948-3	Same solvent balance as AF-97 - Control		53 Sec.	Check solution viscosity	Slightly heavier than control formula but in range for use			
AF-109 *J-5175	Same solvent balance as AF-97 - control		29 Sec.	Same as above	Almost identical to Lot J-4948-1 established as standard for control purposes.			

TABLE 1.9 FORMULATION - DETAILS AND RESULTS OF TESTING - PFAUDLER EXPOSURE TO WFNA

FORMULA NUMBER	COMPOSITION		VISCOSITY 4 FORD CUP	PURPOSE OF FORMULATION	Type of Test conducted: Film Thickness	Drying Schedule	Results	PHOTO REF.
AF-110	X-200	17.0		Check "fast solvent system applied by hot spray at 160°F.	18 mils	114°F-22 hrs. 125°F- 8 " 150°F- 8 " 176°F- 8 " 200°F- 8 " 250°F- 4 " 300°F-15 "	Panels were weighed to "No loss". AF 15 hrs. at 300°F. there was still a trace of solvent remaining. No further test	
	10-200							
*J5175	Wax	3.0	38 Sec.					
	MIBK	40.0						
	Xylol	20.0						
	Toluol	20.0						
		100.0						
AF-111	X-200	17.0	45 Sec.	Check hot spray characteristics	18 mils	Not baked	Blistered on spraying - small blisters	
	10-200							
*J4948-1	Wax	3.0						
	MIBK	40.0						
	Xylol	20.0						
	Toluol	20.0						
		100.0						
AF-112	X-200	20.0		Check hot spray characteristics with no 10-200 wax	18 Mils	Not baked	Poor spraying- too heavy and blistered on spray application. Feathered	
	MIBK	40.0	72 Sec.					
*J-5175	Xylol	20.0						
	Toluol	20.0						
		100.0						
AF-113	X-200	20.0		Control for AF-111 with 10-200 wax deleted	No panels prepared		Too heavy for spray	
	MIBK	40.0	89 Sec.					
*J4948-1	Xylol	20.0						
	Toluol	20.0						
		100.0						
AF-114	Methyl Cellosolve checked in formula - No solution (Incompatable)							

TABLE 1.10 FORMULATION - DETAILS AND RESULTS OF TESTING - PFAUDLER EXPOSURE TO WFNA

FORMULA NUMBER	COMPOSITION		VISCOSITY 4 FORD CUP	PURPOSE OF FORMULATION	Type of Test Conducted: Film Thickness	Drying Schedule	Results	PHOTO REF.
AF-115A	X-200	17.0		Check spray	10-13 Mils	114°F-22 hrs.	Set A	
	10-200		44 Sec.	characteristics	(2 sets	125°F- 8 "	7 days at 125°F-	
	Wax	3.0		and solvent	of panels)	150°F- 8 "	WFNA	
*J-5175	MIBK	40.0		evaporation at		176°F- 8 "	7 days at 160°F-	
	DIBK	10.0		various stages		200°F- 8 "	WFNA	No.14
	Methyl Cello.			of bake		250°F- 4 "	Blistered but gave good protection-creepage from edges.	
	Acetate	10.0			Air Dry	300°F- 1 "	Set B	
	Xylol	20.0		Test in	132 hrs.		Unsatis. in JP-4 at elevated temp.	See Chart 3
		100.0		Pfaudler	before bake			
AF-116A	X-200	17.0	42 Sec.	Same as AF-115A	Same as AF-115A		Same as AF-115A	
	10-200	3.0						
*J-5175	MIBK	50.0			Air Dry 108 hrs. before bake			
	Xylol	30.0						
		100.0						
AF-117A	X-200	17.0	32 Sec.	Same as AF-115A	Same as AF-115A		Same as AF-115A	
	MIBK	50.0						
*J-5175	Xylol	33.0			Air Dry 84 hours before bake		Note-See solvent release Chart on AF-115, 116 and 117	
		100.0						

TABLE 1.11 FORMULATION - DETAILS AND RESULTS OF TESTING - PFAUDLER EXPOSURE TO WFNA

FORMULA NUMBER	COMPOSITION		VISCOSITY 4 FORD CUP	PURPOSE OF FORMULATION	Type of Test Conducted: Film Thickness / Drying Schedule	Results	PHOTO REF.
AF-119	X-200 10-200	15.50		Milled green for alternate color coats to facilitate multi-coat application using hot spray process.	Set 1 - 20 Mils: 100-114°F-32 hrs.; 125°F. - 8 "; 150°F. - 8 " Set 2 - 20 Mils: 176°F. - 8 "; 200°F. - 8 "; 250°F. - 4 "; 300°F. - 1 " 48 hrs. Air dry at 77°F. Prior to bake	1)On baking there was sl. spotty blistering. 2)Both sets OK after 5 day exposure to WFNA at 125°F. 3)Set 1 failed at edges and blisters(solvent) after 5 days at 150°F. expos. to WFNA	
*5175	Wax	2.74					
	MIBK	36.50					
	DIBK	9.10					
	M.Cell.		47 Sec.				
	Ac.	9.10					
	Xylol	18.20					
	Ultrox	2.16					
	Chrome oxide	6.70					
		100.00					
AF-120	X-200 10-200	15.50		Milled white (opaque) for alternate color coats to facilitate multi-coat application	See above. Both materials used as alternate coats in preparing 2 sets of Pfaudler panels for exposure to WFNA	4)Set 2 OK after 8 days at 150°F. and failed at edges after 3 addl. days expos. to WFNA at 160°F.	No.15
*5175	Wax	2.74					
	MIBK	36.50					
	DIBK	9.10					
	M.Cell.		51 Sec.				
	Ac.	9.10					
	Xylol	18.20					
	Ultrox	8.86					
		100.00					
AF-121	X-200	15.50	48 Sec.	Alum. prime coat- gave better adhesion to alodized aluminum	1)Tested adhesion only on aluminum panels. Stand. AF-119 and AF-120 were used as topcoats. Improved adhesion. 2)Tested as sealer over putty - OK 3) Tested as primer under putty - OK		
	10-200	2.74					
*5175	MIBK	36.39					
	DIBK	9.09					
	M.Cell.						
	Ac.	9.09					
	Xylol	18.19					
	422 Alum flake	9.00					
		100.00					

TABLE 1.12 FORMULATION - DETAILS AND RESULTS OF TESTING - PFAUDLER EXPOSURE TO WFNA

FORMULA NUMBER	COMPOSITION	VISCOSITY 4 FORD CUP	PURPOSE OF FORMULATION	Type of Test Conducted: Film Thickness	Drying Schedule	Results	PHOTO REF.
AF-123 *5175	Same as AF-120 without 10-200 Wax		These and above formulations are used in final with compartment tests - no further Pfaudler tests				
AF-124 *5175	Same as AF-121 without 10-200 wax		Conducted.				

TABLE II

SOLVENT EVAPORATION RATE

% SOLVENT RETAINED IN FILM AFTER EACH STAGE

Formula	72 Hrs. at 77°F.	22 Hrs. at 114°F.	8 Hrs. at 125°F.	8 Hrs. at 150°F.	8 Hrs. at 176°F.	8 Hrs. at 200°F.	4 Hrs. at 250°F.	*1 Hr. at 300°F.	Remarks
AF-102-1	10.82	6.97	5.96	4.79	3.29	2.36	0.92	Theoretically none	Contains 10-200 wax and EAK-Slow solvent
AF-102-2	10.41	6.84	5.91	4.73	3.35	2.32	0.90	"	Contains 10-200 wax and methyl cellosolve acetate Slow solvent
AF-102-3	8.36	4.90	4.01	3.04	1.75	0.91	0.21	"	No 10-200 wax. EAK-Slow solvent
AF-102-4	8.28	4.67	3.75	2.76	1.57	0.82	0.13	"	No 10-200 Wax. Methyl cellosolve acetate-Slow solvent
AF-115	(136 Hrs.) 10.46	(Combined bake) 9.96		8.66	7.00	4.58	1.54	"	Contains 10-200 Wax and DIBK Slow solvent
AF-116	(108 Hrs.) 9.98	9.46		8.30	6.76	4.75	1.49	"	Contains 10-200 Wax and MIBK Slow solvent
AF-117	(84 Hrs.) 5.23	4.72		3.81	2.84	1.48	0.46	"	No 10-200 Wax. MIBK in slow solvent

TABLE II (Contd.)

Note: For purposes of the above calculation, the weight loss was figured to zero after 1 hour at 300°F. Continuing the bake at 300°F. results in a loss which cannot be attributed to solvent alone. At this point both X-200 and 10-200 wax volatize in an erratic manner as indicated below.

Rate of Volatilization - X-200 and 10-200 Wax

(1)	X-200 (85%) and 10-200 wax (15%)	Loss - 4.82% at 300°F. in 5 hours.
(2)	X-200 (100%)	Loss - 0.337% at 300°F. in 5 hours.
(3)	10-200 Wax (100%)	Loss - 11.60% at 300°F. in 20 hours (3.90% at 300°F. in 5 hours)

X-200 COATINGS TESTED IN JP-4 FUEL

TABLE III

FORMULA	SOLIDS CONTENT	TIME	TEMP.	RESULTS	
AF-101-3 Panel A and C	100% X-200	3 days	77°F.	Possible softening	
		3 days	125°F.	Definite softening	
		7 days	160°F.	Very soft - loose from panel	
AF-101-3 Panel B and D	100% X-200	3 days	77°F.	Possible softening	
		3 days	125°F.	Definite softening	
		7 days	160°F.	Very soft - loose from panel	
AF-115	80% X-200 20% 10-200 Wax	3 days	77°F.	Softened	
		3 days	125°F.	Very soft	Photo 16
		7 days	160°F.	Wrinkled and swollen	
AF-116	80% X-200 20% 10-200 Wax	3 days	77°F.	Softened	
		3 days	125°F.	Very soft	
		7 days	160°F.	Wrinkled and swollen	
AF-117	100% X-200	3 days	77°F.	Possible softening	
		3 days	125°F.	Definite softening	Photo 17
		7 days	160°F.	Very soft	

FILLETING FORMULATION

TABLE IV

Batch No.	Resin Appearance	Ease of grind	Spec. gravity	(1) Aver. particle size Microns.	Putty Viscosity	Putty texture	(2) Hot flow at 300°F.	(3) Fusion Temp.	160°F. Resist. Pfaudler column WFNA Resistance	Remarks
J-4294	Med. size granules	Good	1.94	1.30	Good-Sl.stiff-pliable	Good	Good	265	Extensive large cracks 5-8 days	
J-4311	Med. hard pdr.	Fair-good	1.87	25.0	Fair	Good	Fair		—	Too soluble
J-4321	Lg. hard lumps	Good	1.92	2.83	Good	Good	Good	>325	Edge cracks-V. good - 12 days	Good
J-4322	Fine pdr.	Good	1.92	8.08	Good	Good	Poor	>325	V.Sl.Cracking - 4 days	Hot flow
J-4323	Lg. hard lumps	Good	1.93	5.28	Good	Good	Bad	>325	Hairline cracks - 2 days*	Hot flow
J-4352	Lg. waxy lumps	Good	1.95	20.5	Good	Good	Good	> 325	V.Sl.cracking - 1 day*	Particles too lg.
J-4354	Lg. spongy lumps	Good	1.98	3.50	Dry powder	—	—	265	—	Too spongy
J-4410	Mod.waxy granules	Fair	1.92	40.0	Good	Good	Poor		—	Hot flow-particle size
J-4623	Fine pdr.	Good	1.91	1.34	Good-sl.stiff	Good	Good	> 325	Extensive lg.cracks-12 days	Too insoluble
J-4625	Fine pdr.	Good	1.77	1.17	Dry powder	—	—		—	—
J-4630	Fine pdr.	Good	1.83	1.22	Dry powder	—	—		—	—
J-4817	Med.size pdr.	Fair	1.96	21.1	Good, sl.	Sl.grainy stiff	Good		—	—

TABLE IV (Cont.)

Batch No.	Resin Appearance	Ease of grind	Spec. gravity	(1) Aver. particle size Microns.	Putty viscosity	Putty texture	(2) Hot flow at 300°F.	(3) Fusion Temp.	160°F. Resist. Pfaudler column WFNA Resistance	Remarks
J-4818	Lg. hard lumps	Bad	1.94	25.0	Stick- soft	Sl. grainy	Good	>325	Blistered-penetration-2 days	Too soluble-particle size
J-4820	Lg. waxy lumps	Fair	1.91	24.1	Good- sl. stiff	Good-Sl. grainy	Good		—	Particle size
J-4822	Lg. waxy lumps	Fair	1.87	32.3	Fair	Sl.grainy	Good		—	Particle size
J-4826	Fine waxy pdr.	Bad	1.84	34.6	Fair	Grainy	Good		—	Particle size
J-4828	Lg. hard lumps	Bad	1.95	30.9	Sticky-soft	Sl. grainy	Good	>325	Blistered cracked-1 day	Too soluble-particle size
J-4835	Lg. rubbery lumps	Could not grind				—	—		—	—
J-4948 -1	Fine pdr.	Bad	1.83	1.83	Very stiff-rubbery	—	Bad	> 325	—	Too soluble
J-4383	Lg. waxy lumps	Fair	1.91	32.5	Good	Good	Bad		—	Hot flow

(1) As determined by Fisher Sub-Sieve Sizer
(2) 1/8 in. plug of putty - baked for 1 hr. at 300°F. at a 30° angle to the horizontal.
(3) Temperature at which uncompounded X-200 resin was fused:
  a. 1/16" press polish platens.
  b. 10 min. at noted temperature at 1000 psi.
  Degree of flexibility and extent of flow were noted.
* Water leaked into Pfaudler Unit - ruining test.

TABLE V
COMPARTMENT COATING APPLICATION

COMPARTMENT	SECTION OF COMPARTMENT	COATINGS USED AND NUMBER OF COATS	DATE S-Start C-Complete	BAKING CYCLE
D	Top	AF-121(2) AF-122 and 123 (10) each	Start 5/5/55	1 wash - Air dry
	Side (Horizontal bracing)	" " "	Complete 5/11/55	24 hrs. at 131°F. 24 hrs. at 150°F.
	Side (vertical bracing)	AF/121(2) AF 119 and 120(10)each		18 hrs. at 176°F. 8 hrs. at 215°F.
	Trays	" " "		4 hrs. at 250°F. blistering
	Corners	" " "		1 hr. at 300°F. bad blistering
A	Top	AF-121(2) AF-119 and 120 (10 each)	Start 5/12/55	5 days Air dry at room T°.
	Side (horizontal bracing)	" " "	Complete 5/17/55	48 hrs. at 135°F. 24 hrs. at 150°F.
	Side (vertical bracing)	AF-121(2) AF-121 and 122(10) each		18 hrs. at 176°F. 8 hrs. at 210°F.
	Trays	" " "		4-1/2 hrs. at 275°F.
	Corners	" " "		(Some blistering-not as bad as "A")
B	Top	AF-121(2) AF-122 and 123(10 each)	Start 5/17/55	1 wk. Air dry at room temp.
	Side(Horizontal bracing)	" " "	Complete 5/24/55	48 hrs. at 133°F. 72 hrs. at 150°F.
	Side (vertical bracing)	AF-121(2) AF-119 and 120(10 each)		24 hrs. at 180°F. 24 hrs. at 200°F.
	Trays	" " "		4 hrs. at 260°F.
	Corners	" " "		(No blisters)
C	Top	AF-121(2) AF-119 and 120(8) each	Start 6/10/55	1 wk. air dry at rm. T°. First cycle-same as "B" above
	Side(horizontal bracing)	AF-121(2) AF-122 and 123 (8) each	Complete 6/15/55	48 hrs. at 133°F. 72 hrs. at 150°F.
	Side (vertical bracing)	AF-121 AF-119 and 120(8) each		24 hrs. at 200°F. 4 hrs. at 260°F.
	Tray	AF-121 AF-122 and 123(8) each		(2nd cycle)
	Corners	AF-121 AF-122 and 123 (8) each		(No blisters)

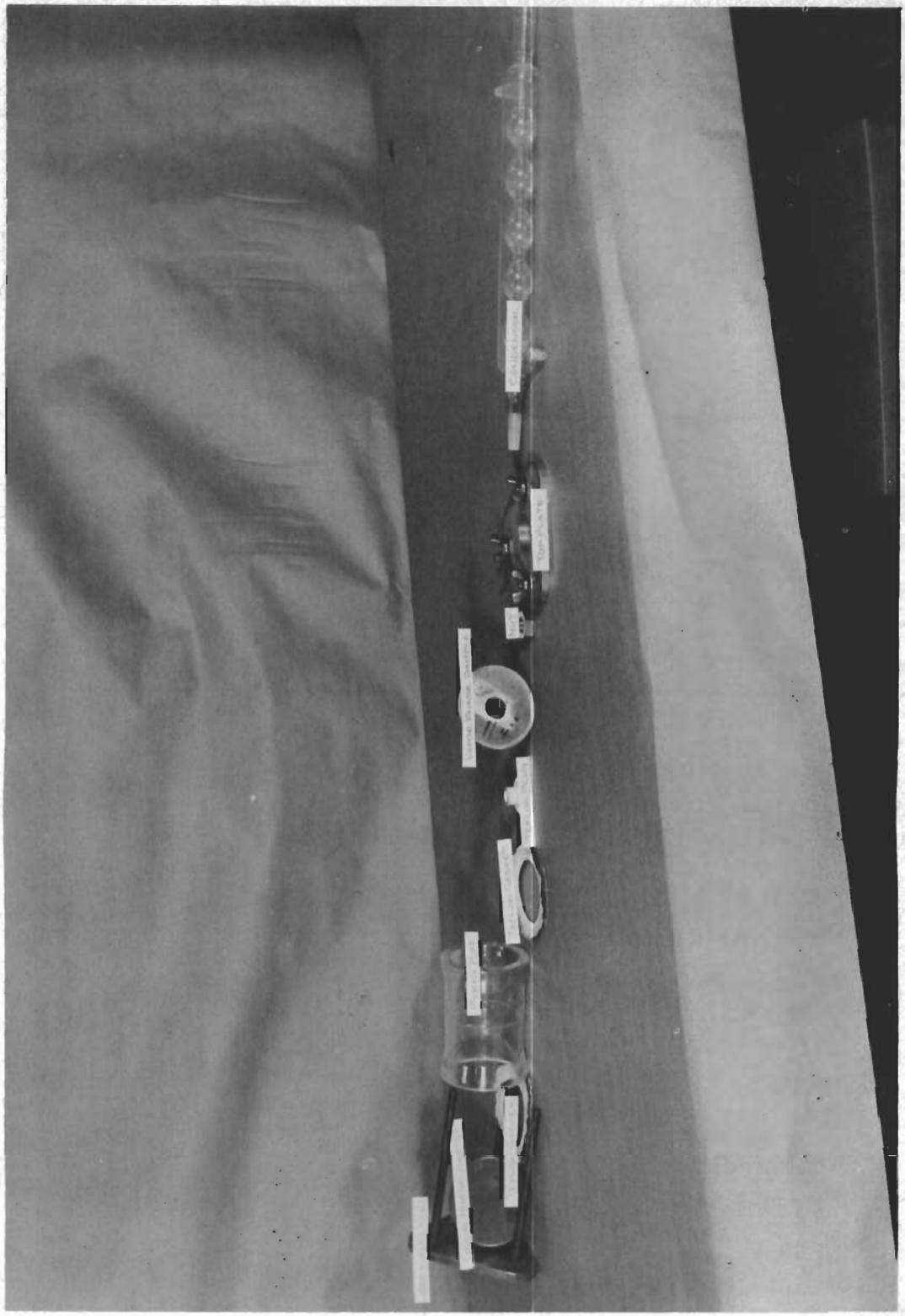

Figure No. 1 Pfaudler Unit - Disassembled

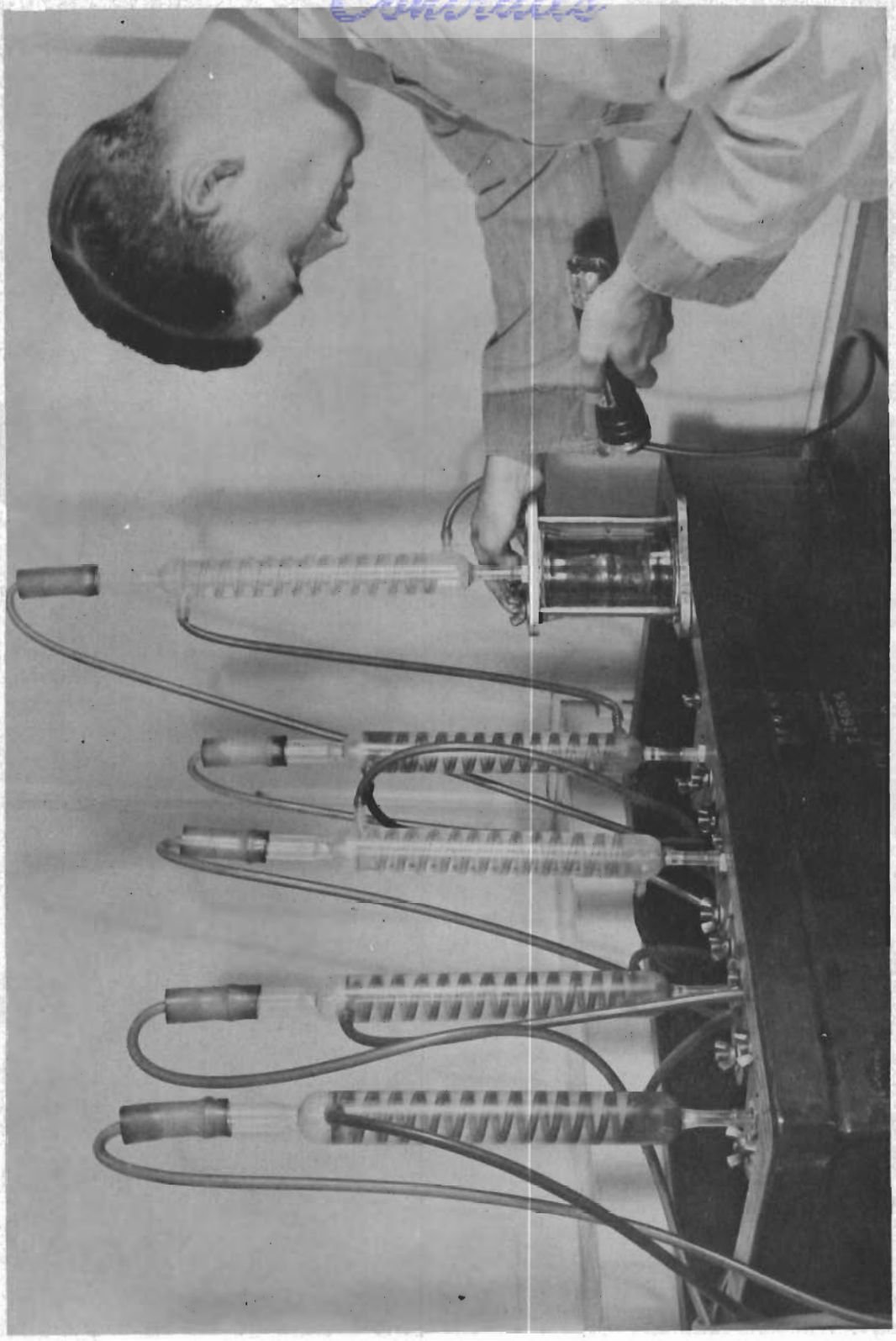

Figure No. 2 Pfaudler Unit - Set up for Test

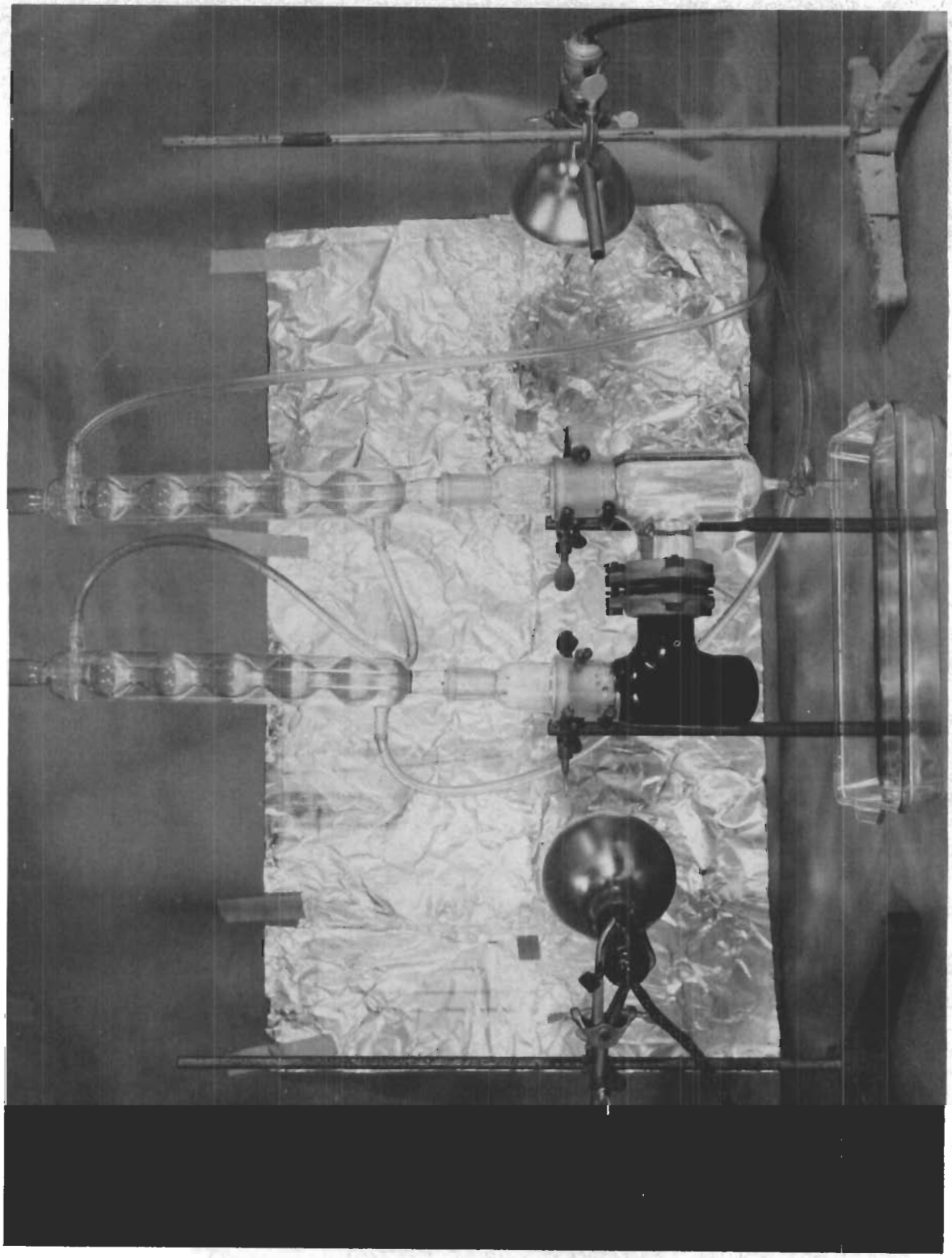

Figure No. 3 H-Cell Unit - Set up for Test

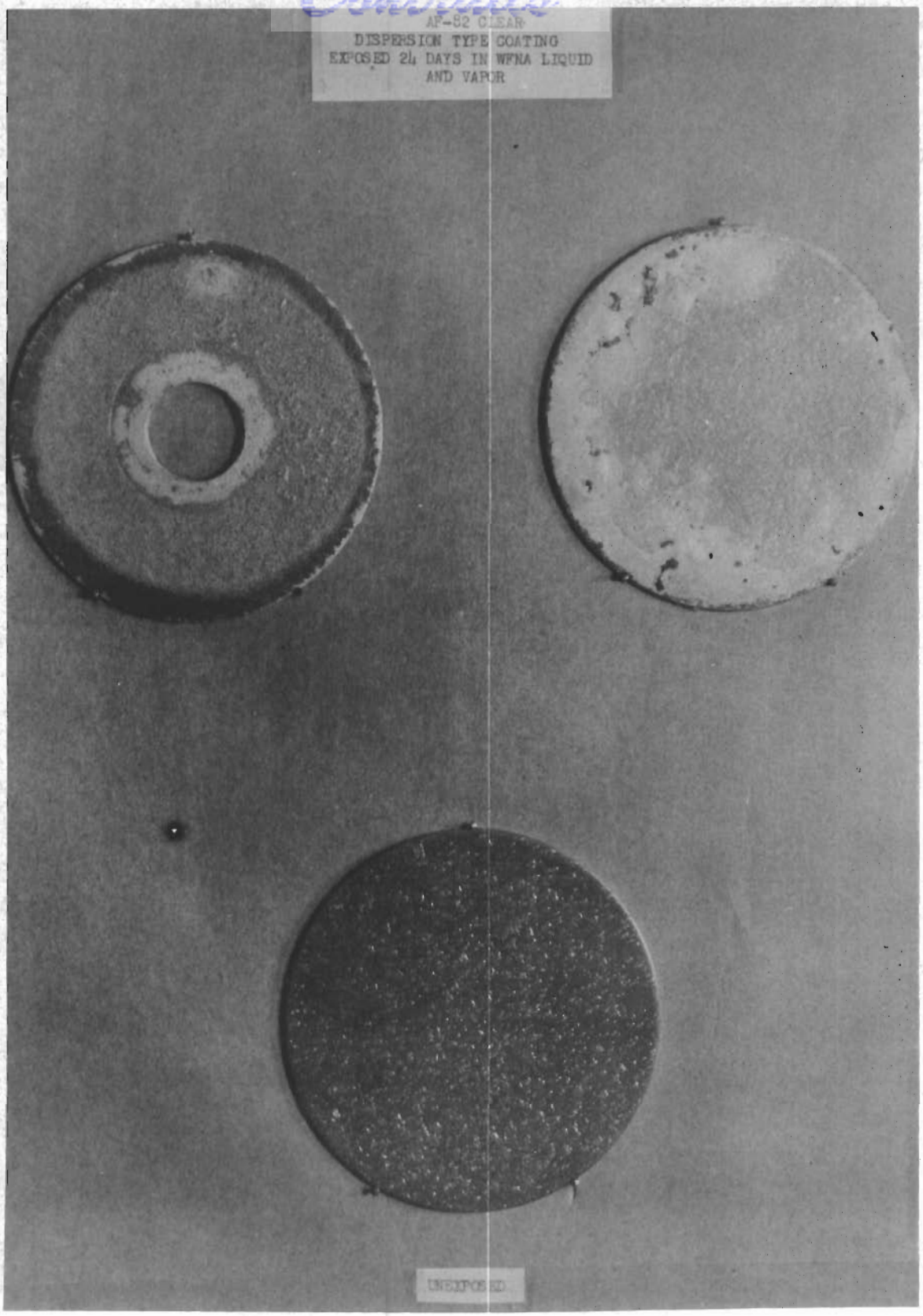

Figure No. 4 AF-82 Exposed in Pfaudler Unit at 77°F. to WFNA.

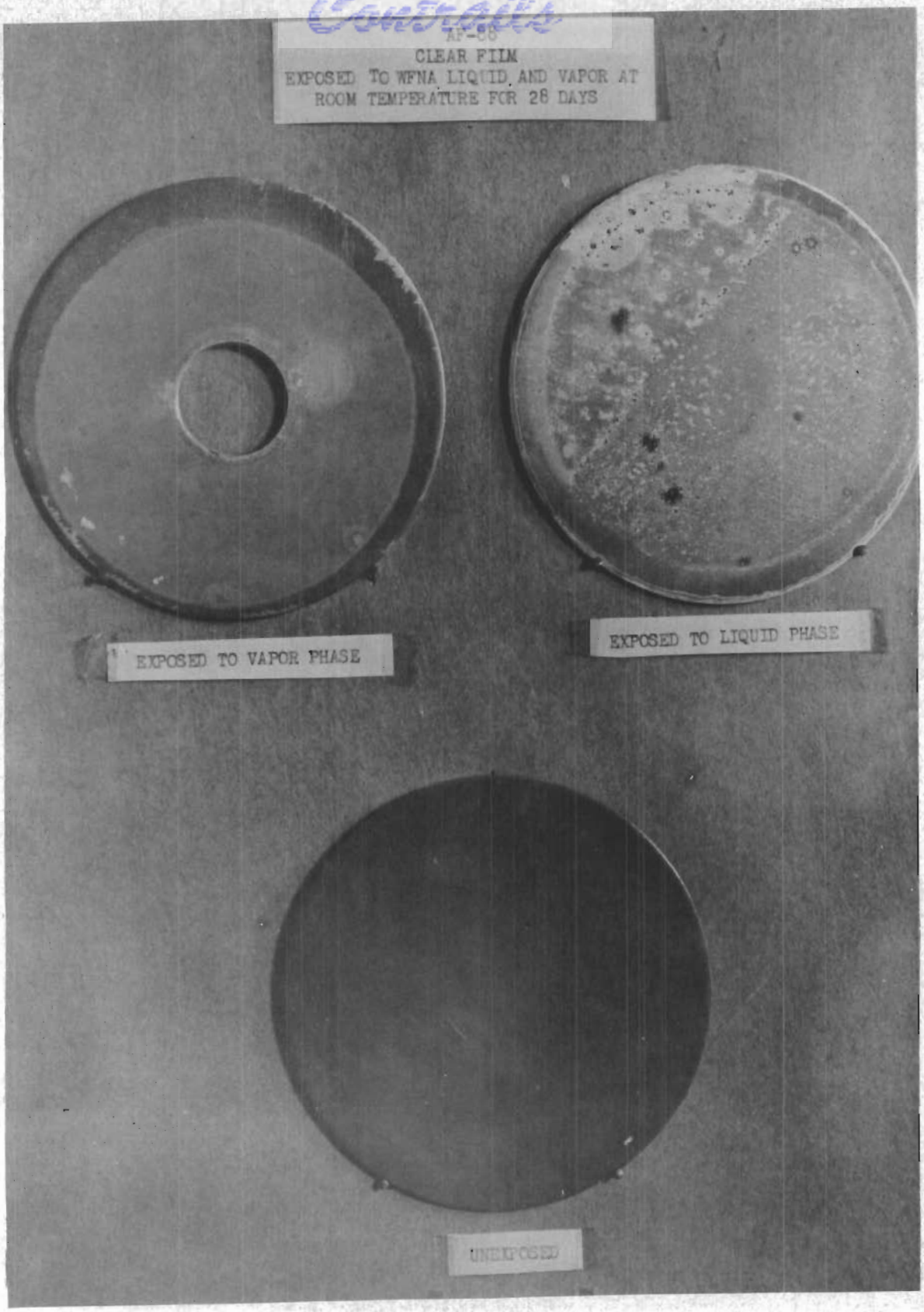

Figure No. 5 AF-88 Exposed in Pfaudler Unit at 77°F. to WFNA.

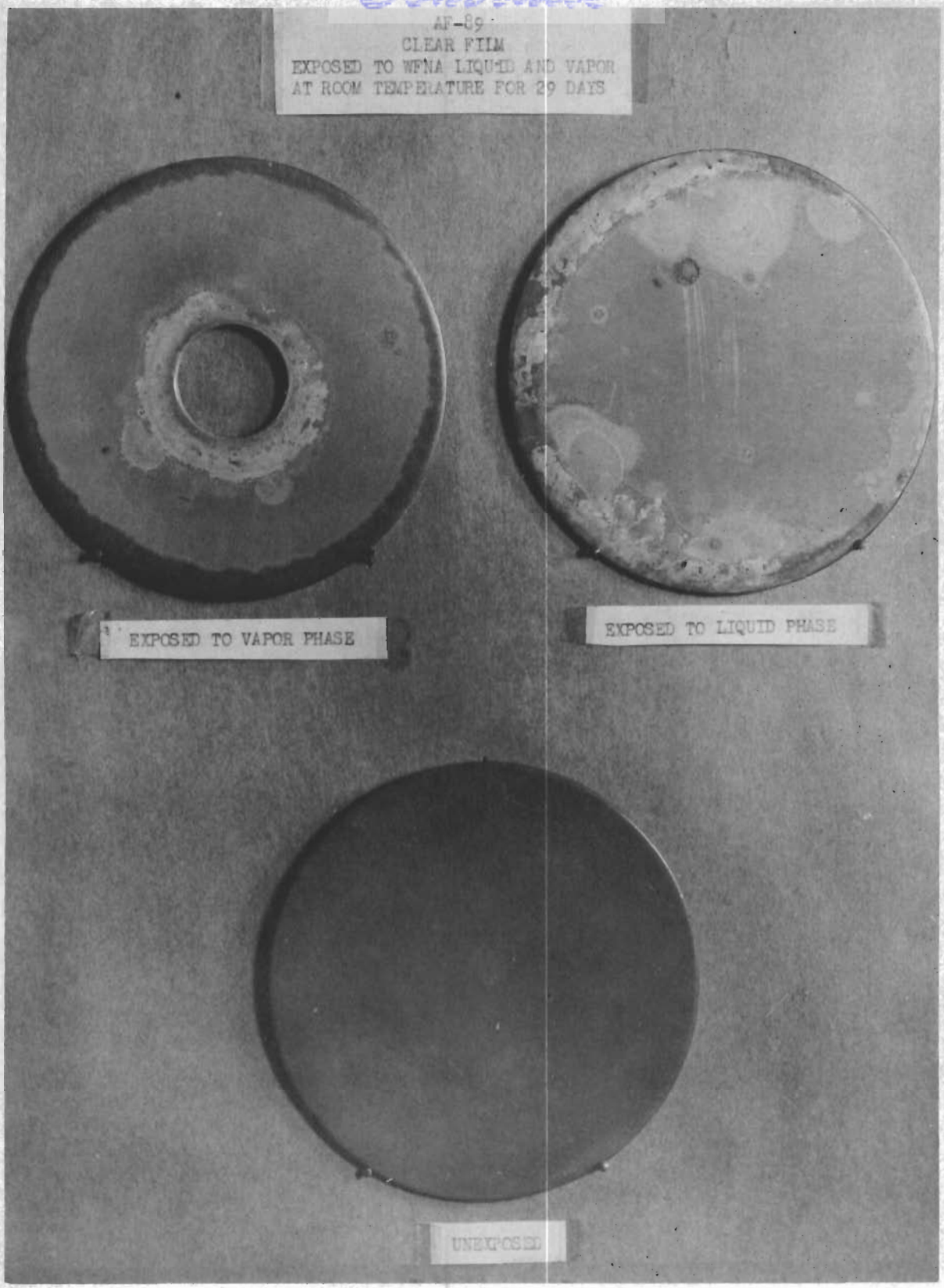

Figure No. 6 AF-89 Exposed in Pfaudler Unit at 77°F. to WFNA.

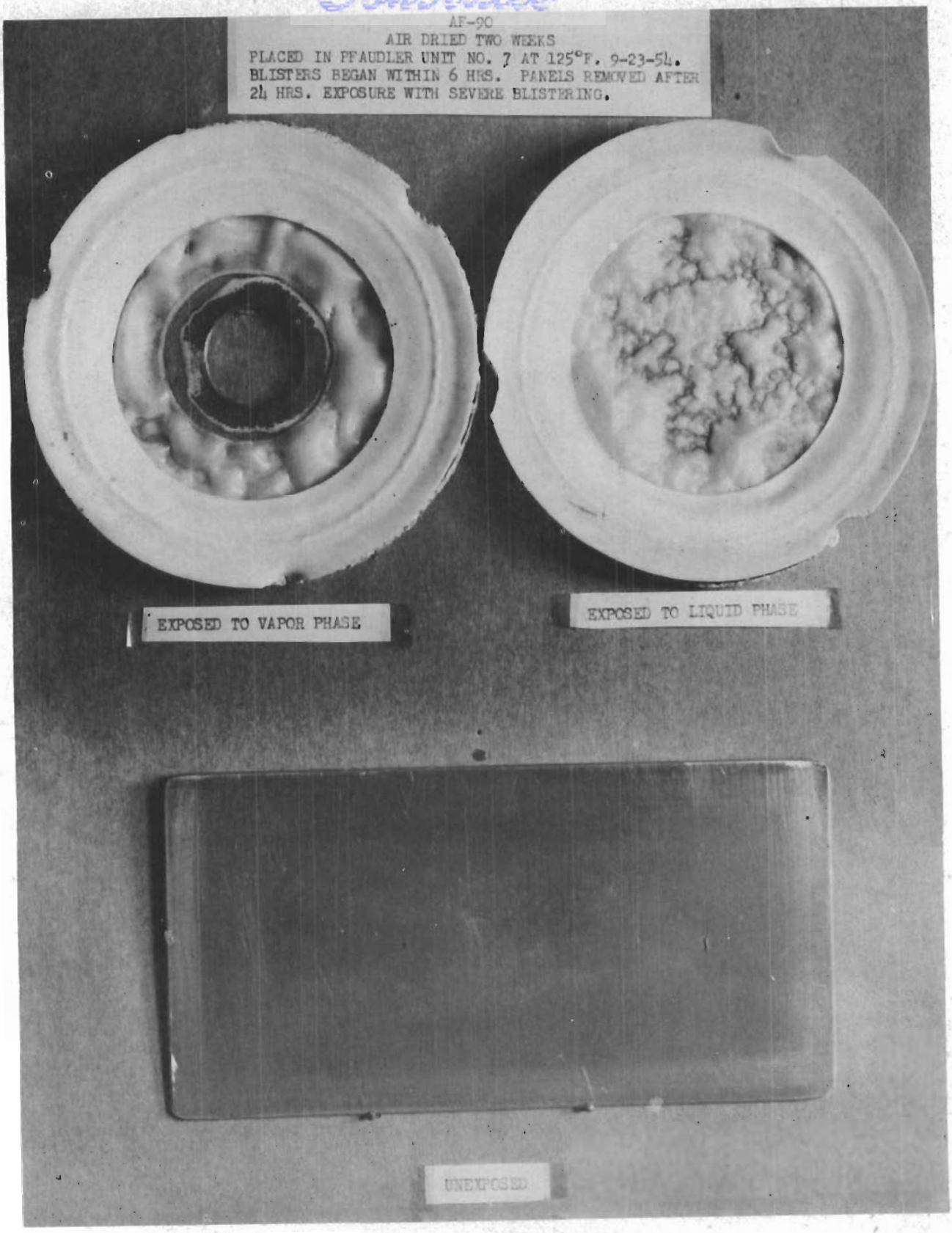

Figure No. 7 AF-90 Exposed in Pfaudler Unit at 77°F. to WFNA.

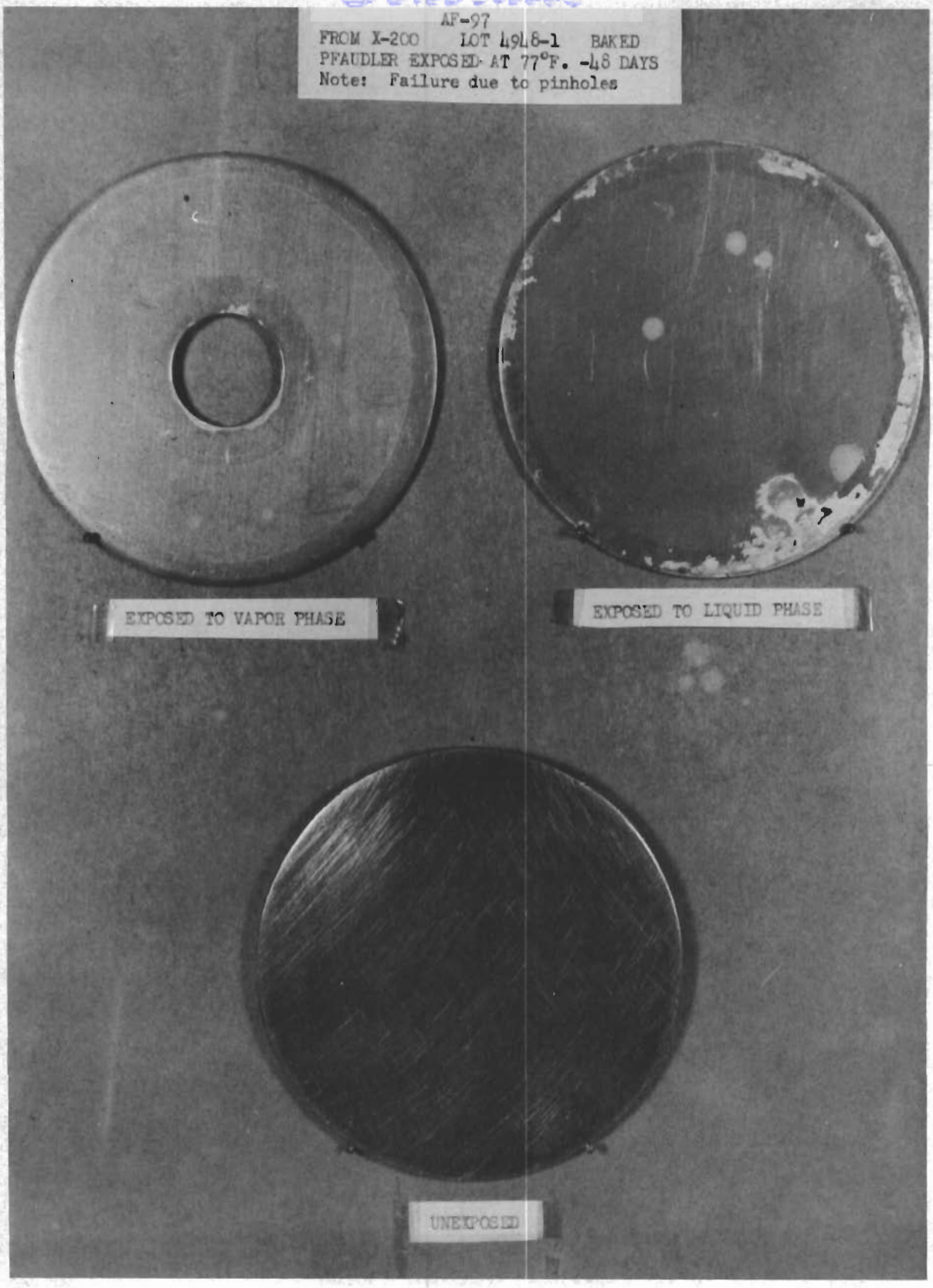

Figure No. 8 AF-97 Exposed in Pfaudler Unit at 77° F. to WFNA.

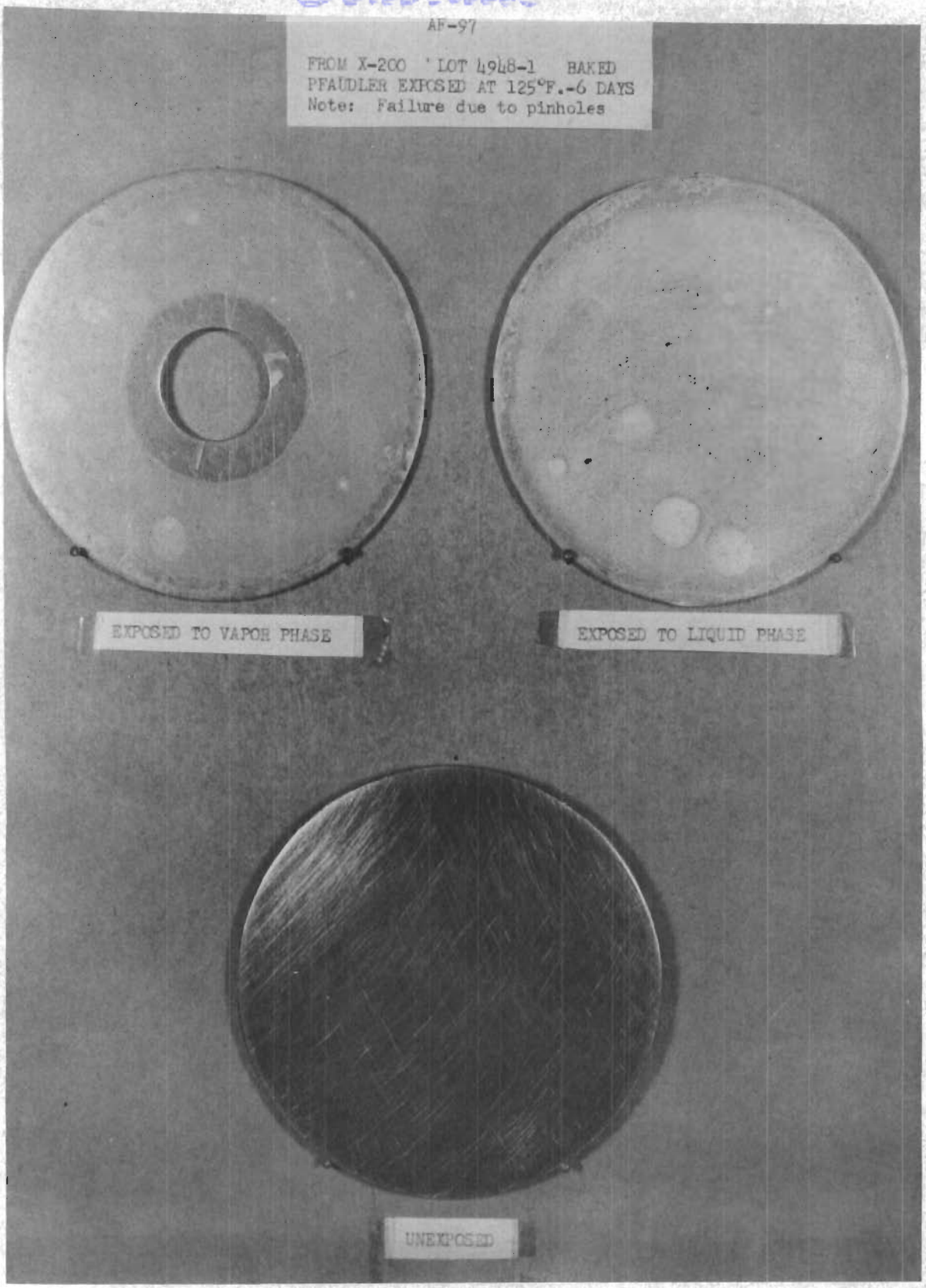

Figure No. 9 AF-97 Exposed in Pfaudler Unit at 125°F. to WFNA.

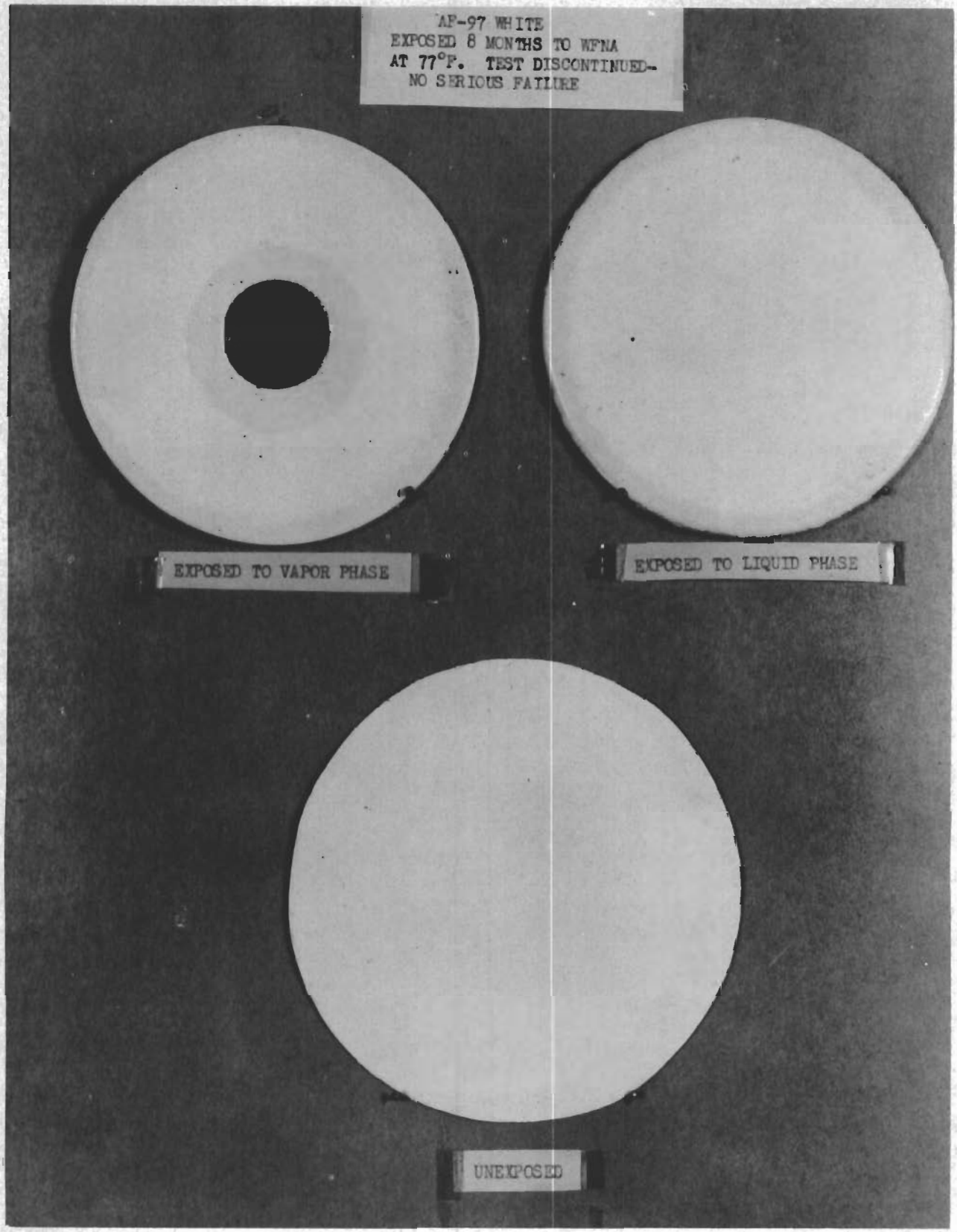

Figure No. 10 AF-97 (pigmented) Exposed in Pfaudler Unit at 77°F. to WFNA.

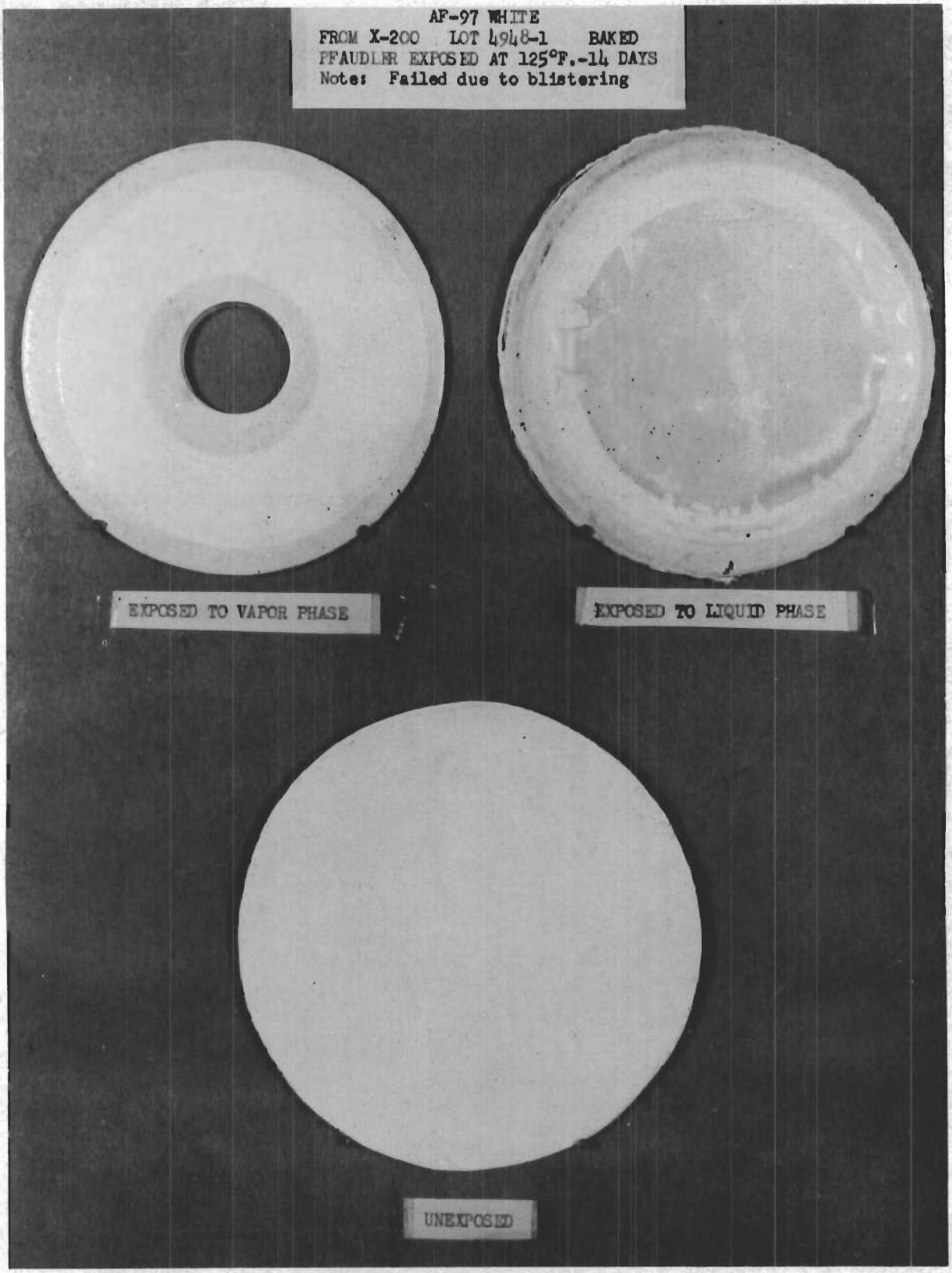

Figure No. 11 AF-97 (Pigmented)

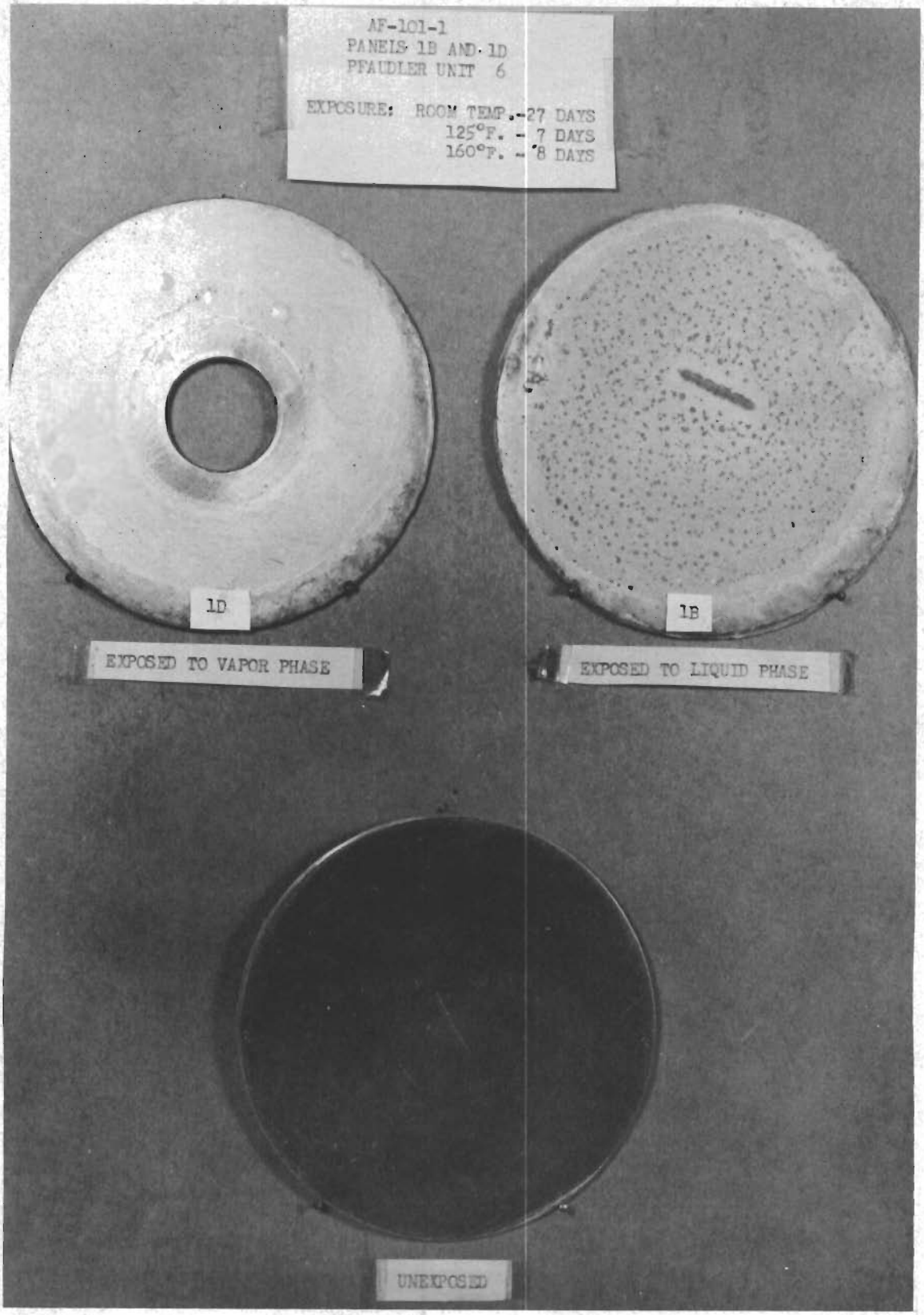

Figure No. 12 AF-101-1 Exposed in Pfaudler Unit at 77° F.

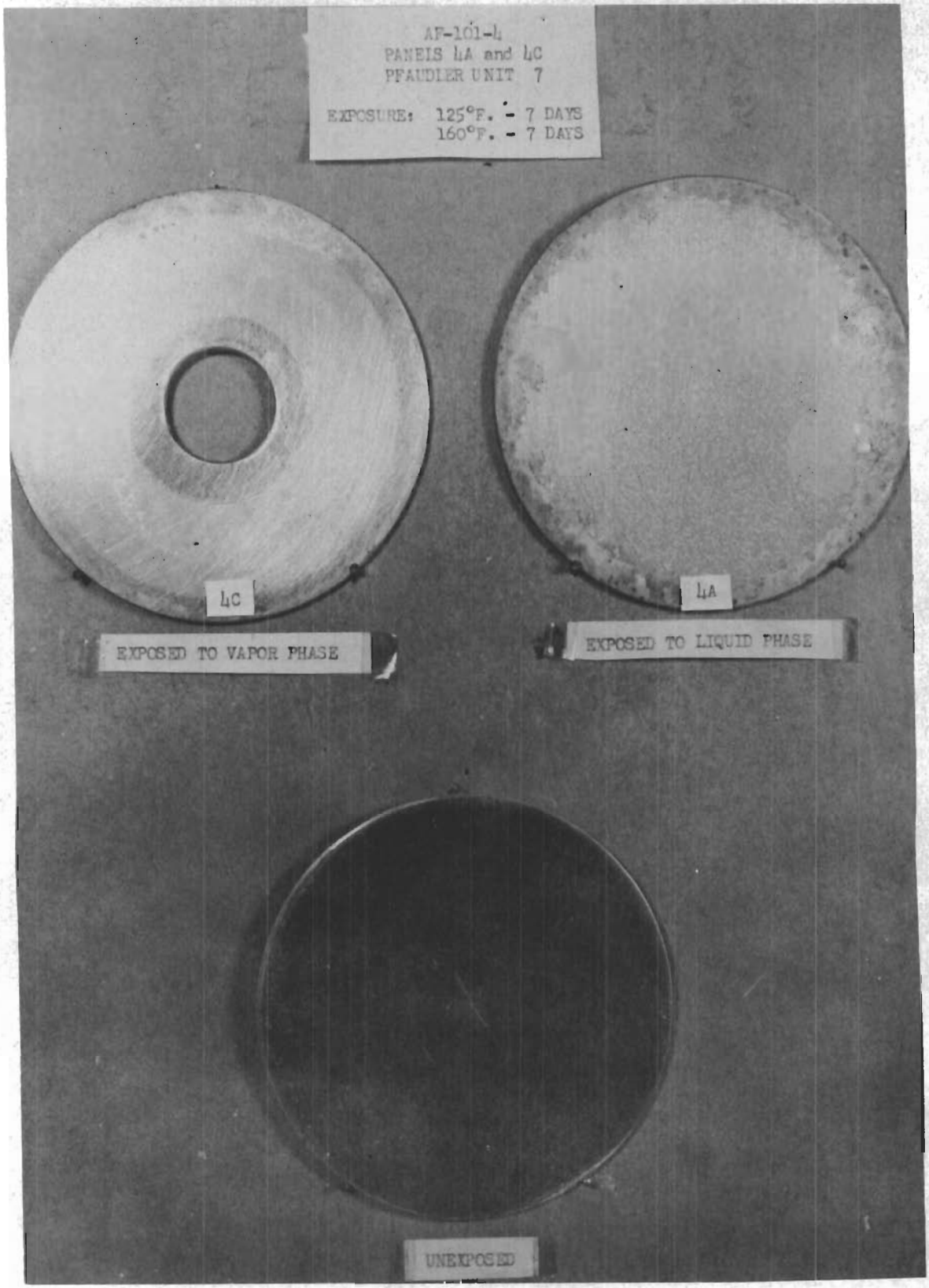

Figure No. 13 AF-101-4 Exposed in Pfaudler Unit at 77°F.

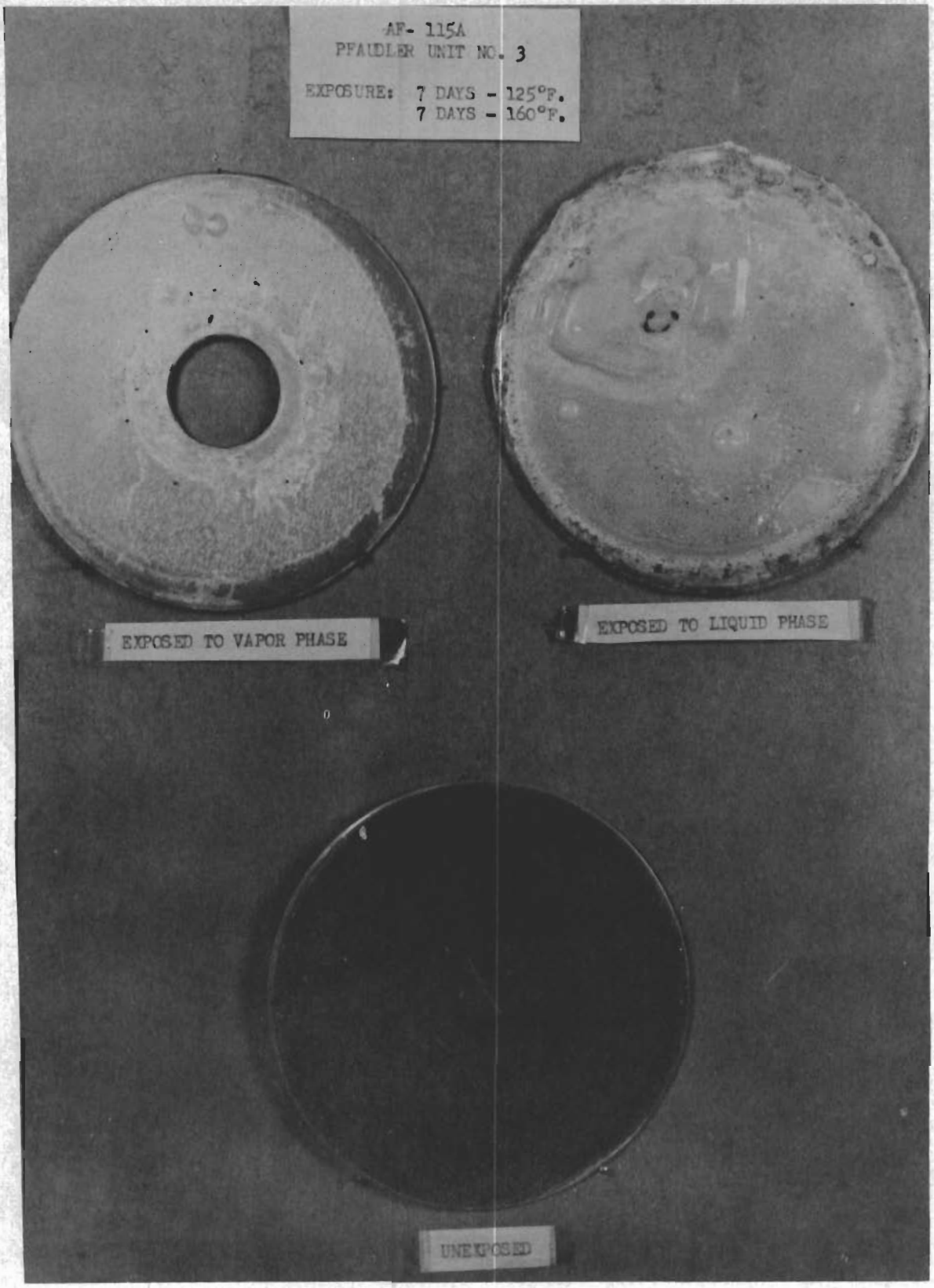

Figure No. 14 AF-115 A Exposed in Pfaudler Unit at 77° F.

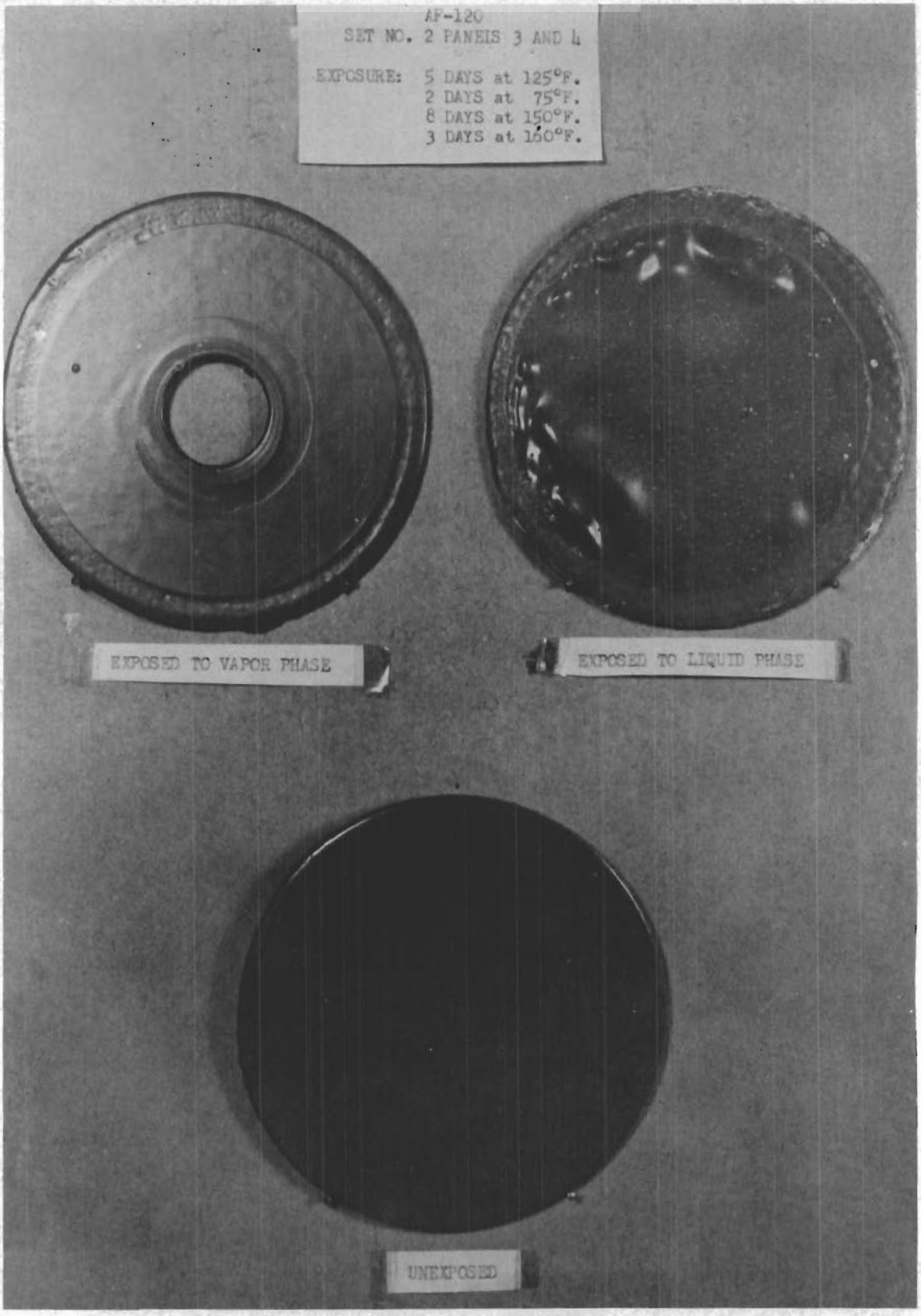

Figure No. 15 AF-120

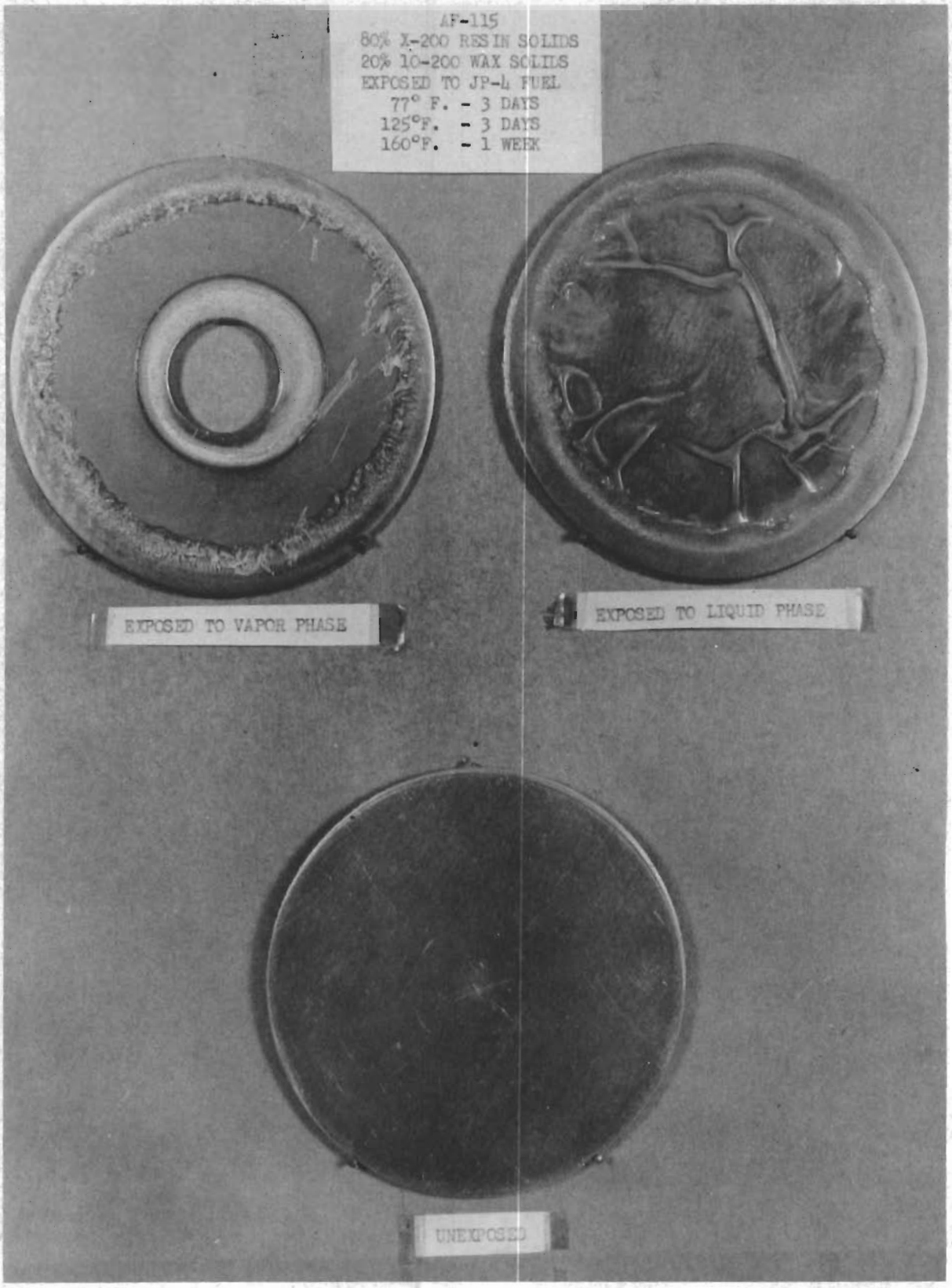

Figure No. 16 AF-115 Exposed in Pfaudler Unit to JP-4 Fuel.

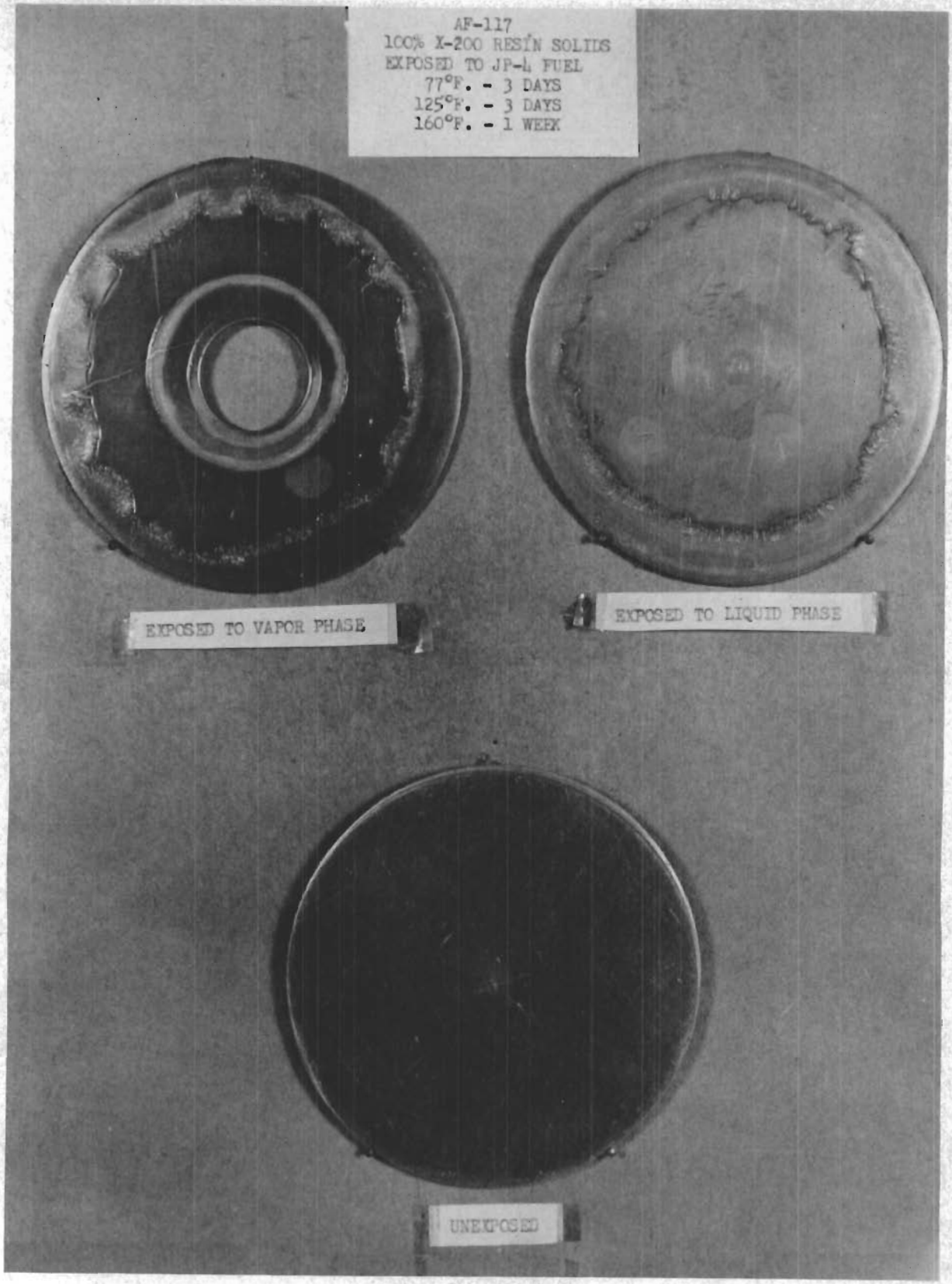

Figure No. 17 AF-117 Exposed in Pfaudler Unit to JP-4 Fuel.

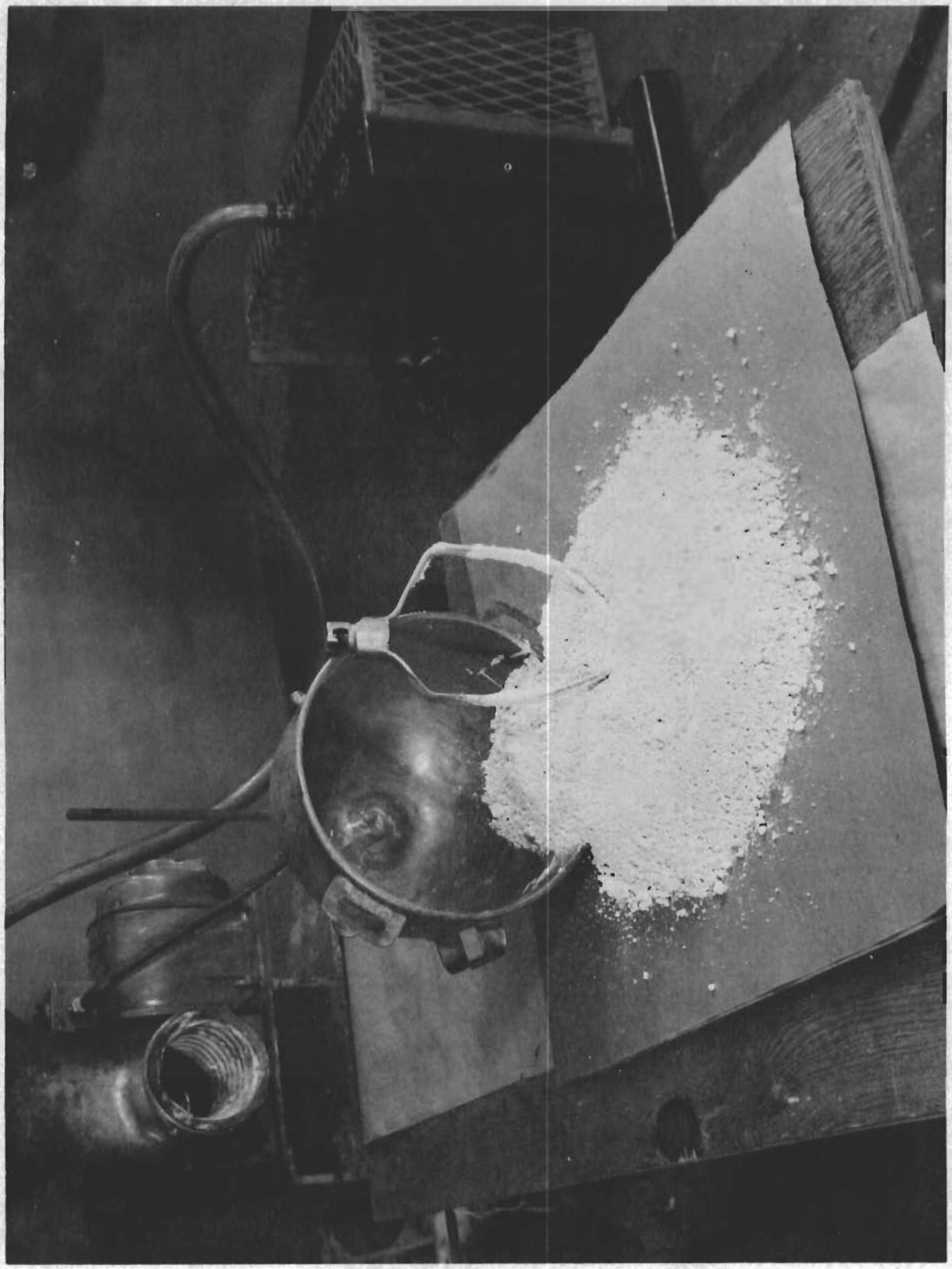

Figure No. 18 Kel-F-Putty

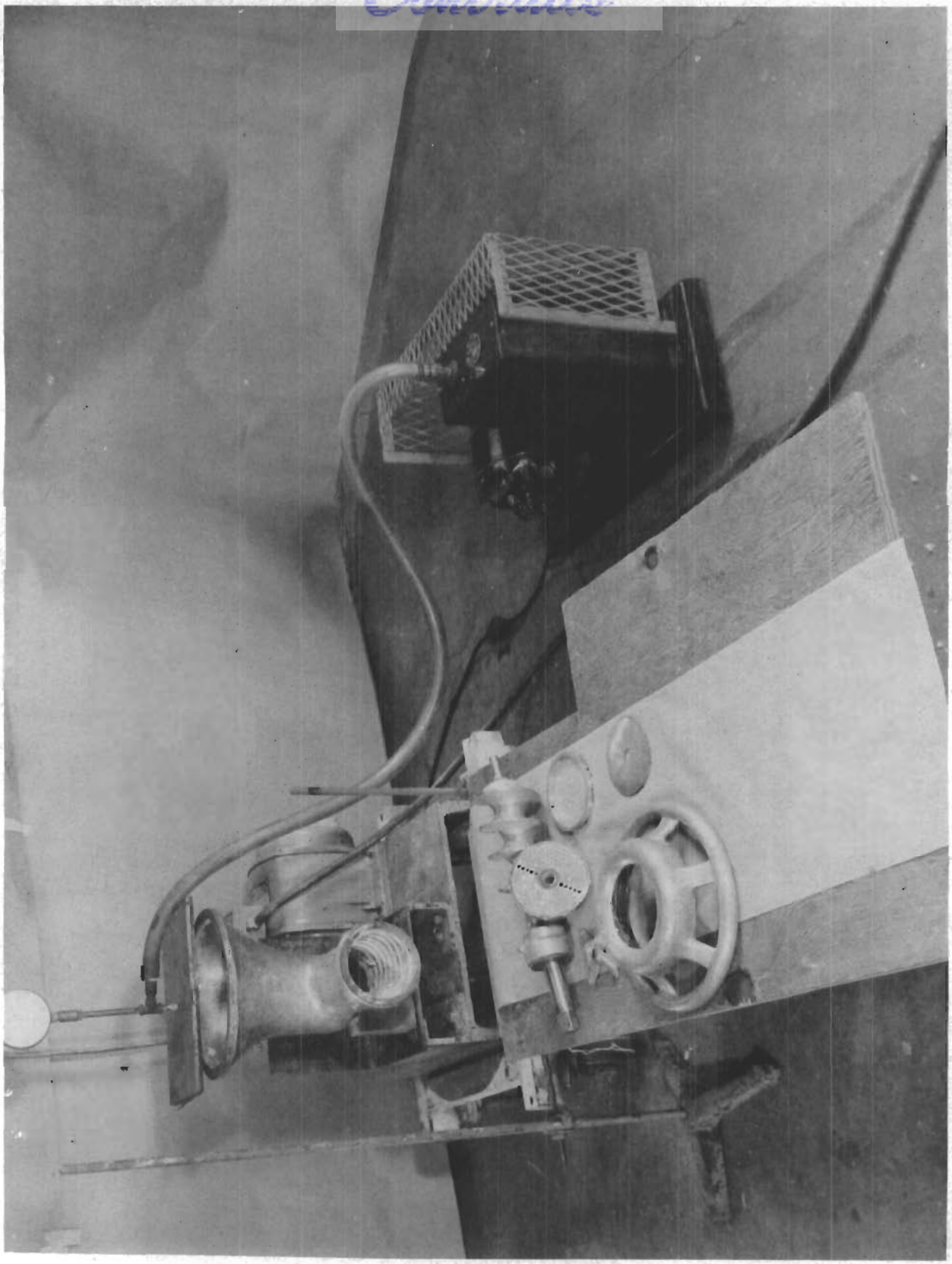

Figure No. 19 Extrusion Set-up for Kel-F Putty

Figure No. 20 Uncoated Test Compartment

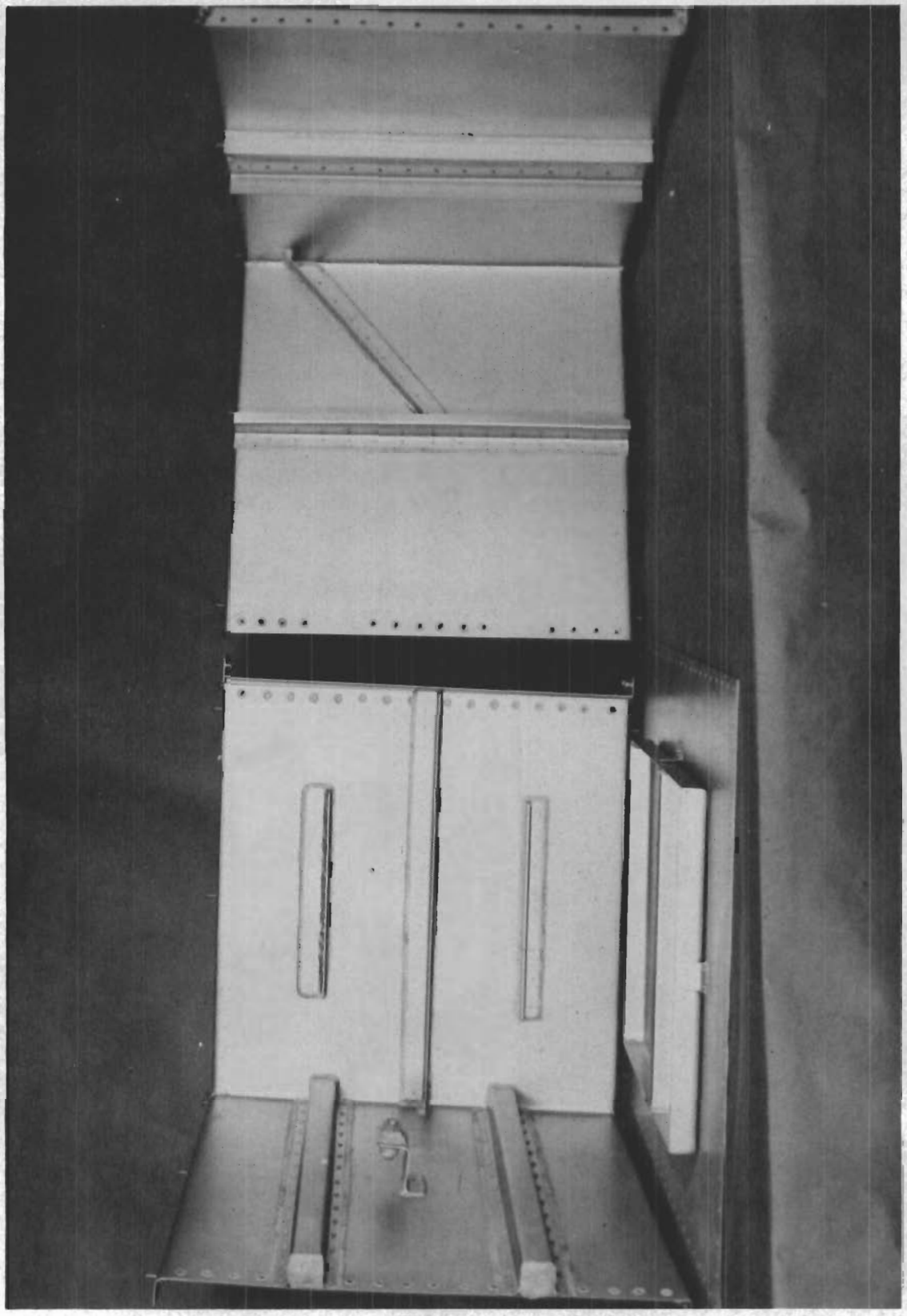

Figure No. 21 Filleted and Primed Test Compartment

Figure No. 22 Acid and Fuel Tray

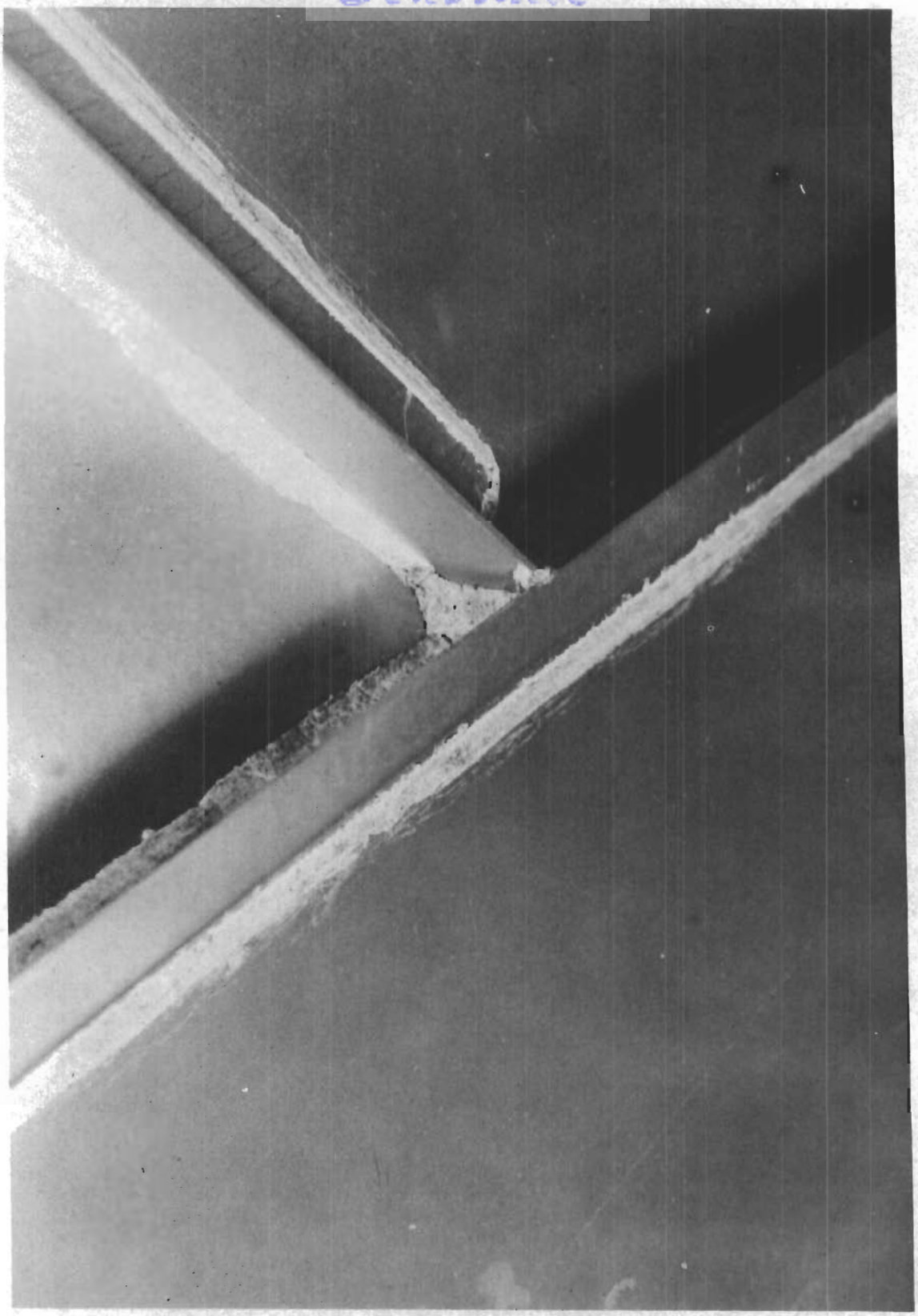

Figure No. 23 Representative Filleting of Crossbraced Structure

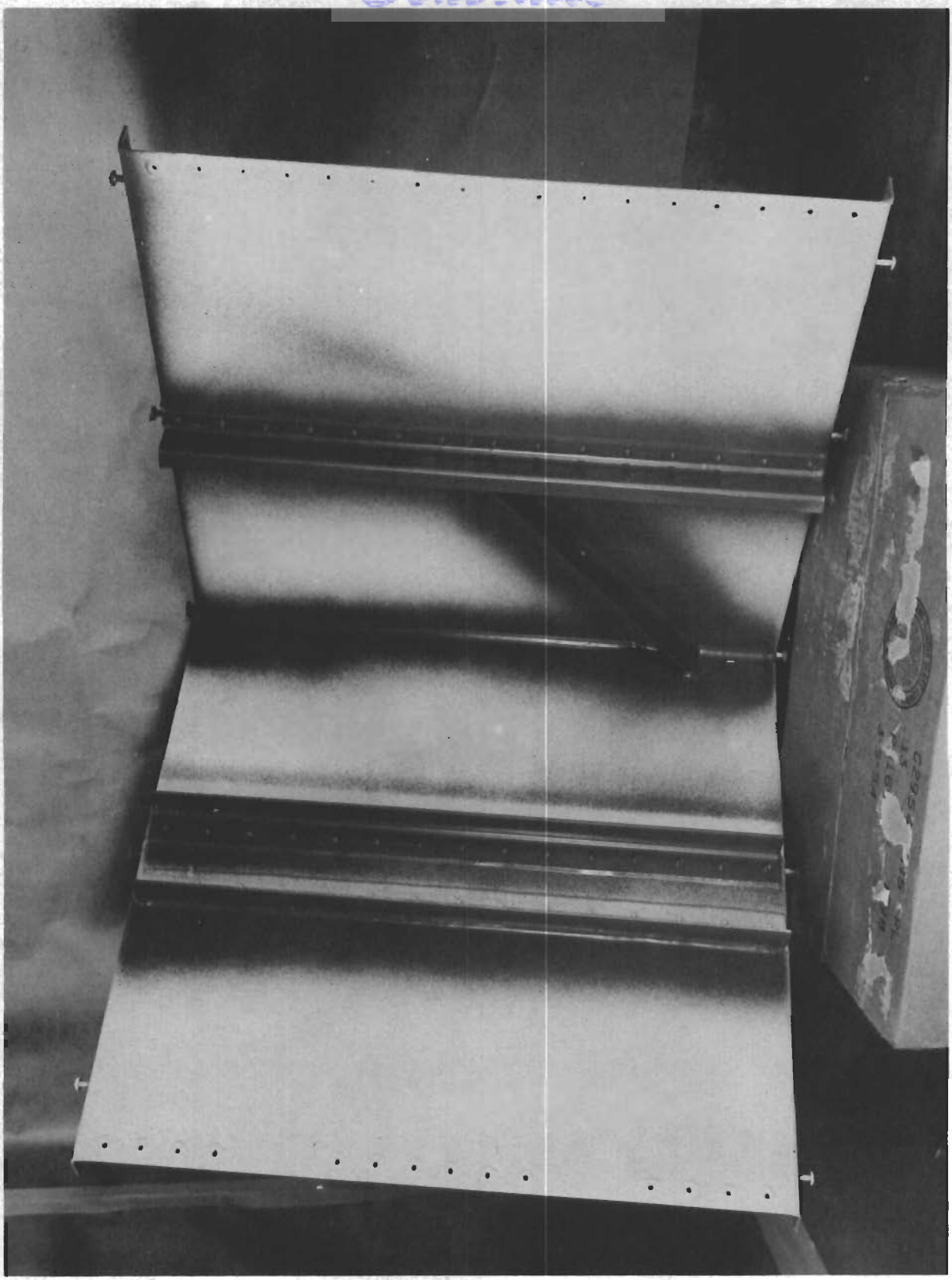

Figure No. 24 Alternating Coats of AF-140B Green, and AF-140C White

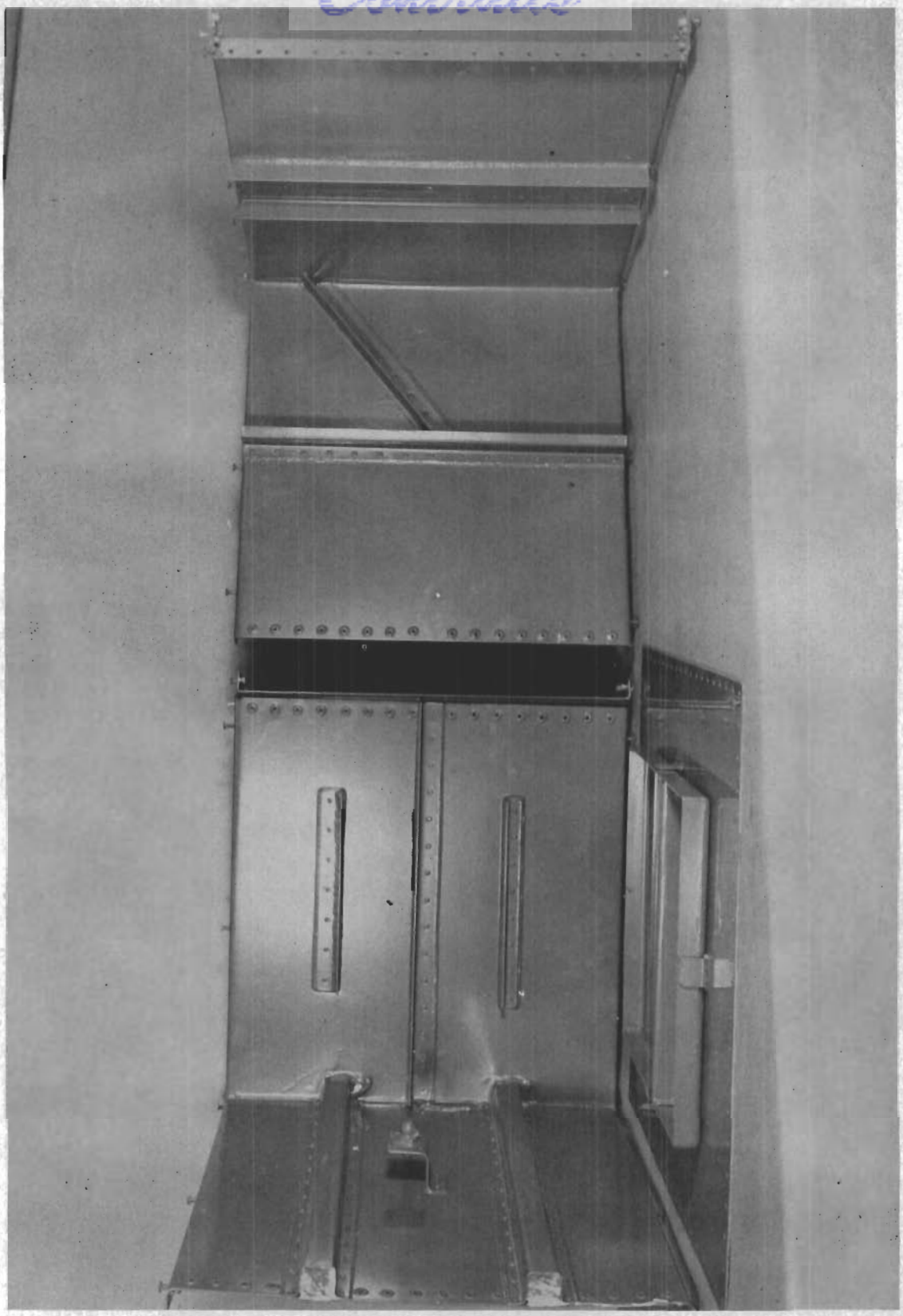

Figure No. 25 Cell A - Coated, Baked and Ready for Assembly

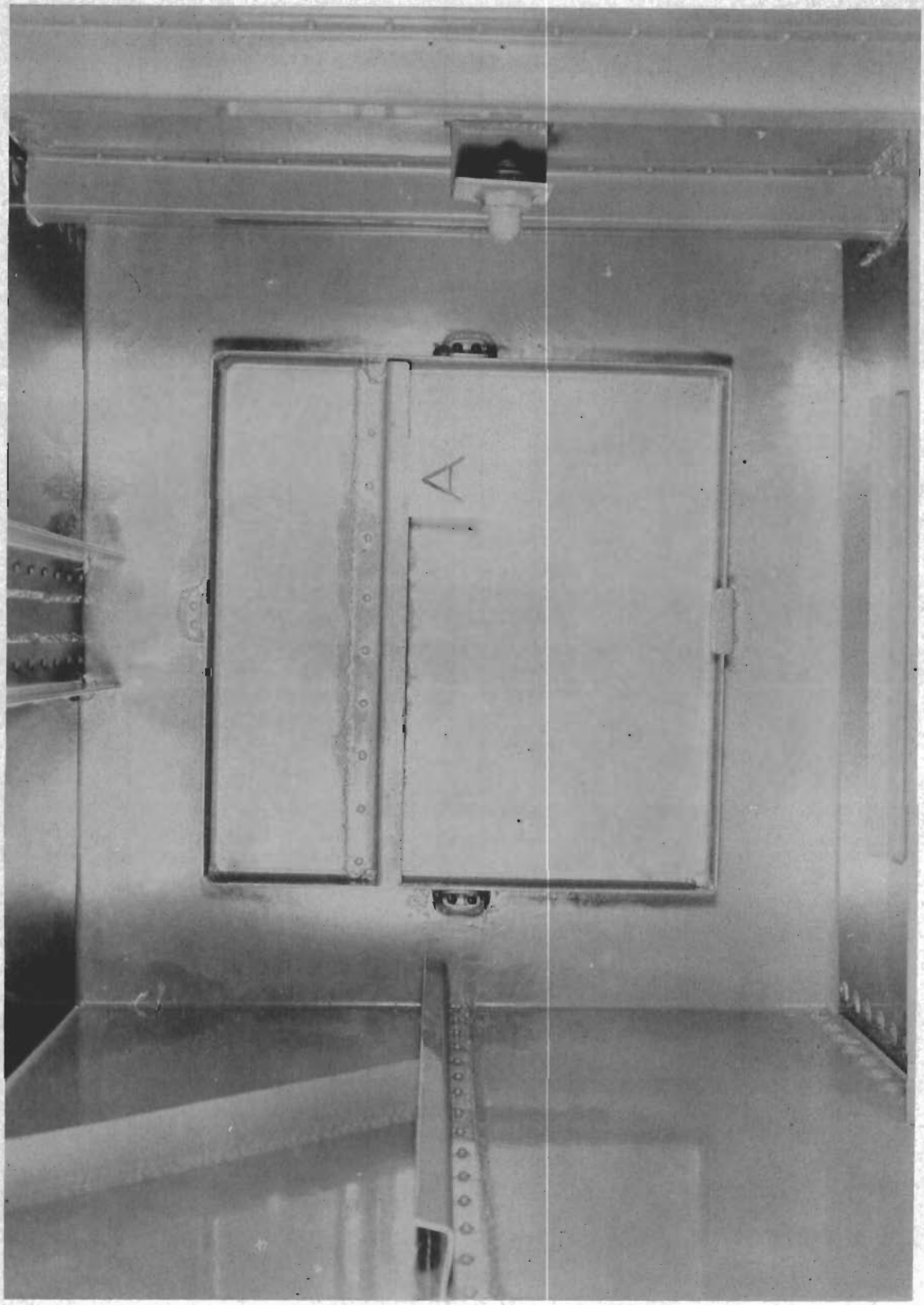

Figure No. 26 Cell A - Cycled and Exposed to JP-4 Fuel for 10 days at 160° F.

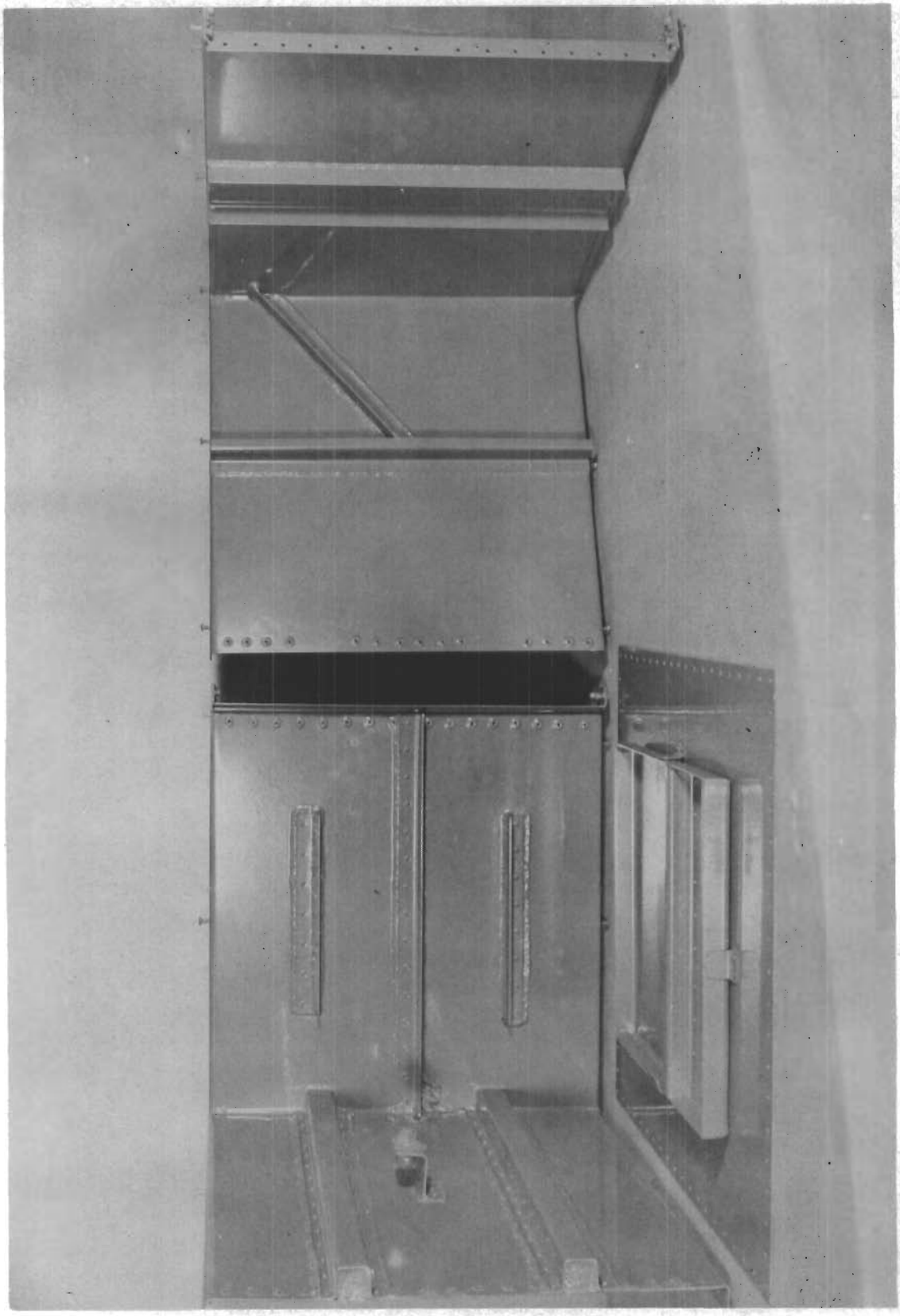

Figure No. 27 Cell B - Coated, Baked and Ready for Assembly

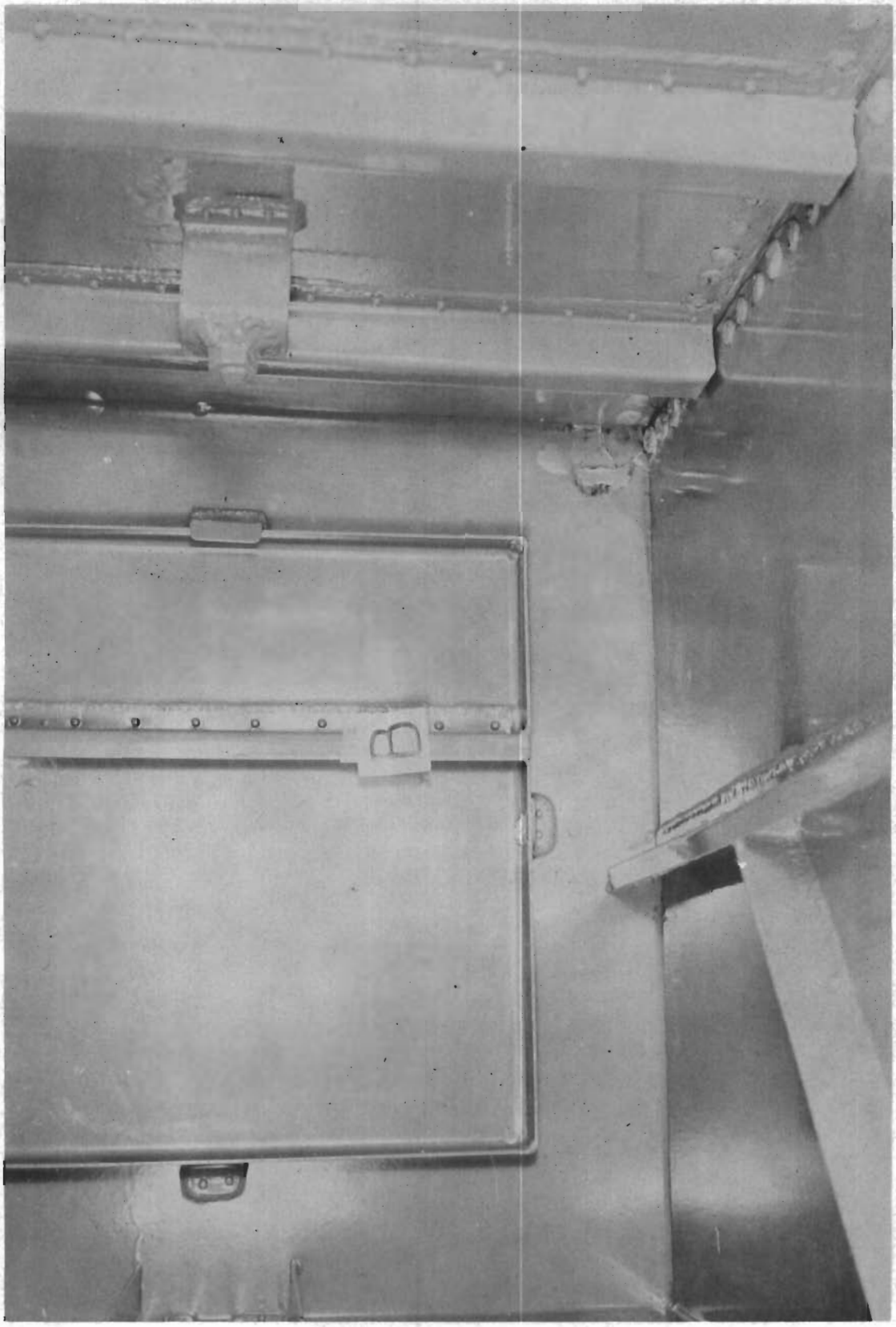

Figure No. 28 Cell B - Not Cycled - Exposed 5 days to WFNA at 160°F.

Figure No. 29 Cell B - Close-up of Severe Breakdown at one Corner

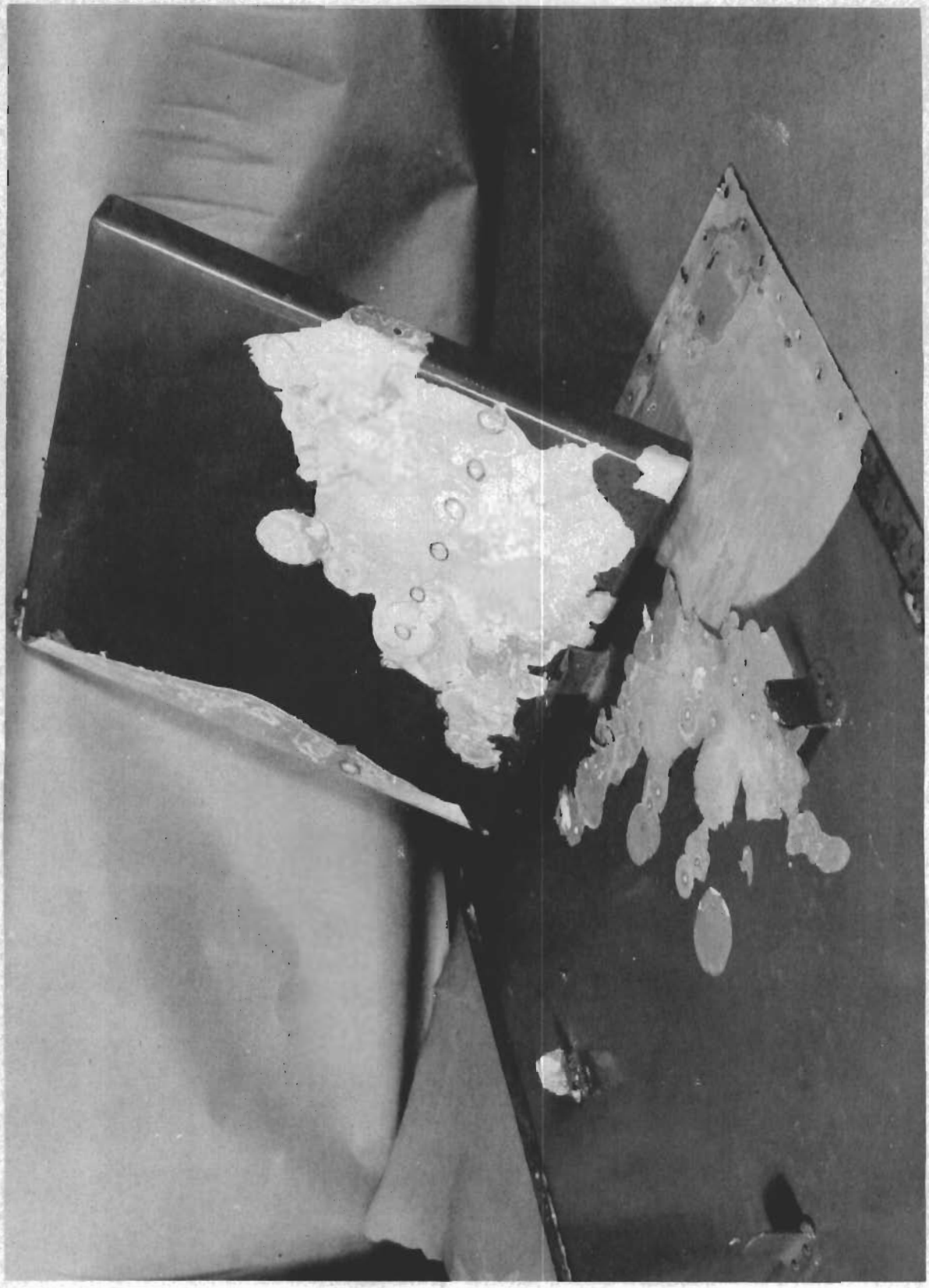

Figure No. 30 Cell B - Bottom of Acid Container Tray
Penetration Through Rivets in Z Section

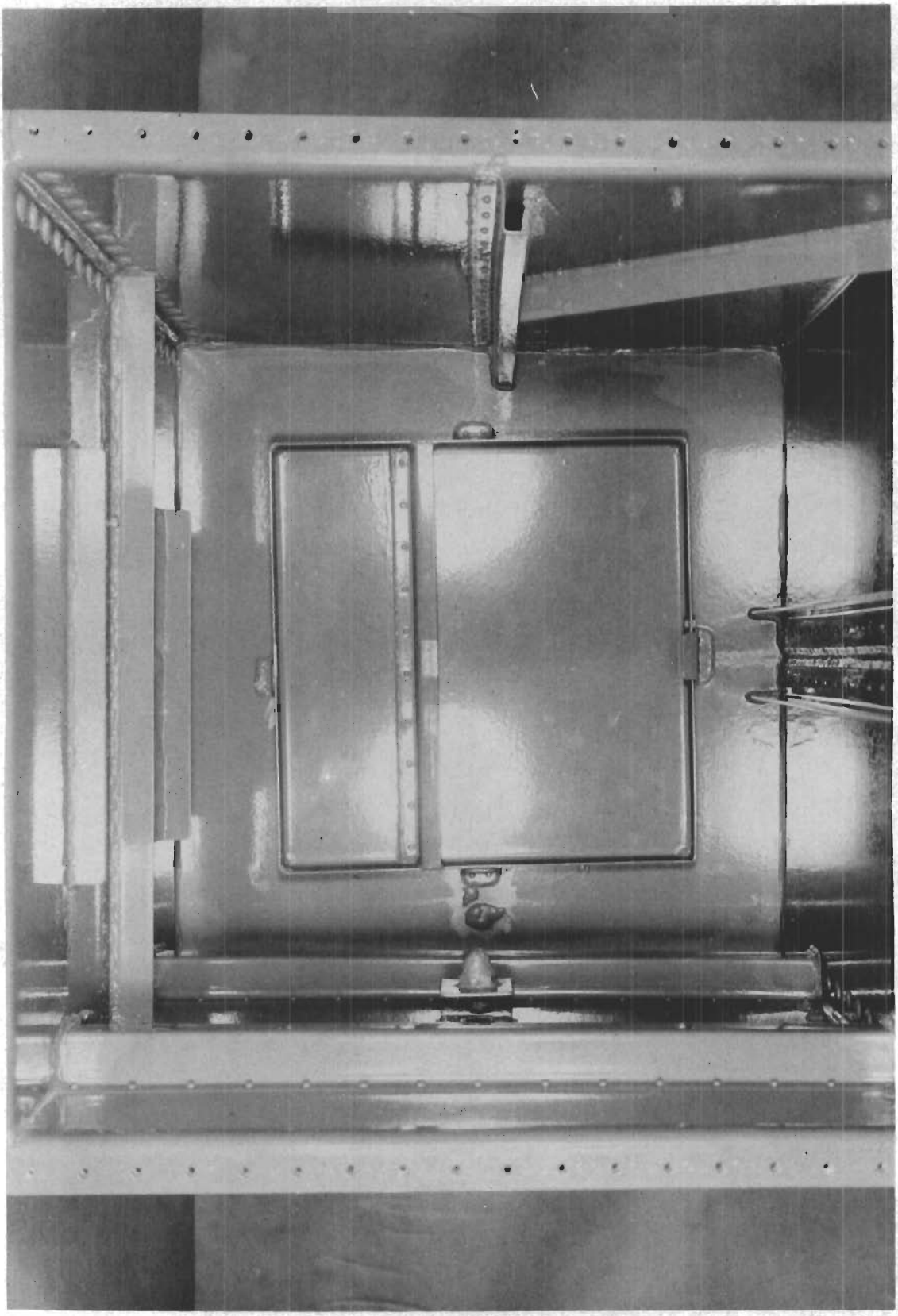

Figure No. 31 Cell C - Coated, Assembled, Post Filleted and Touch-up Coated, Baked, Cycled, and Ready for WFNA Test.

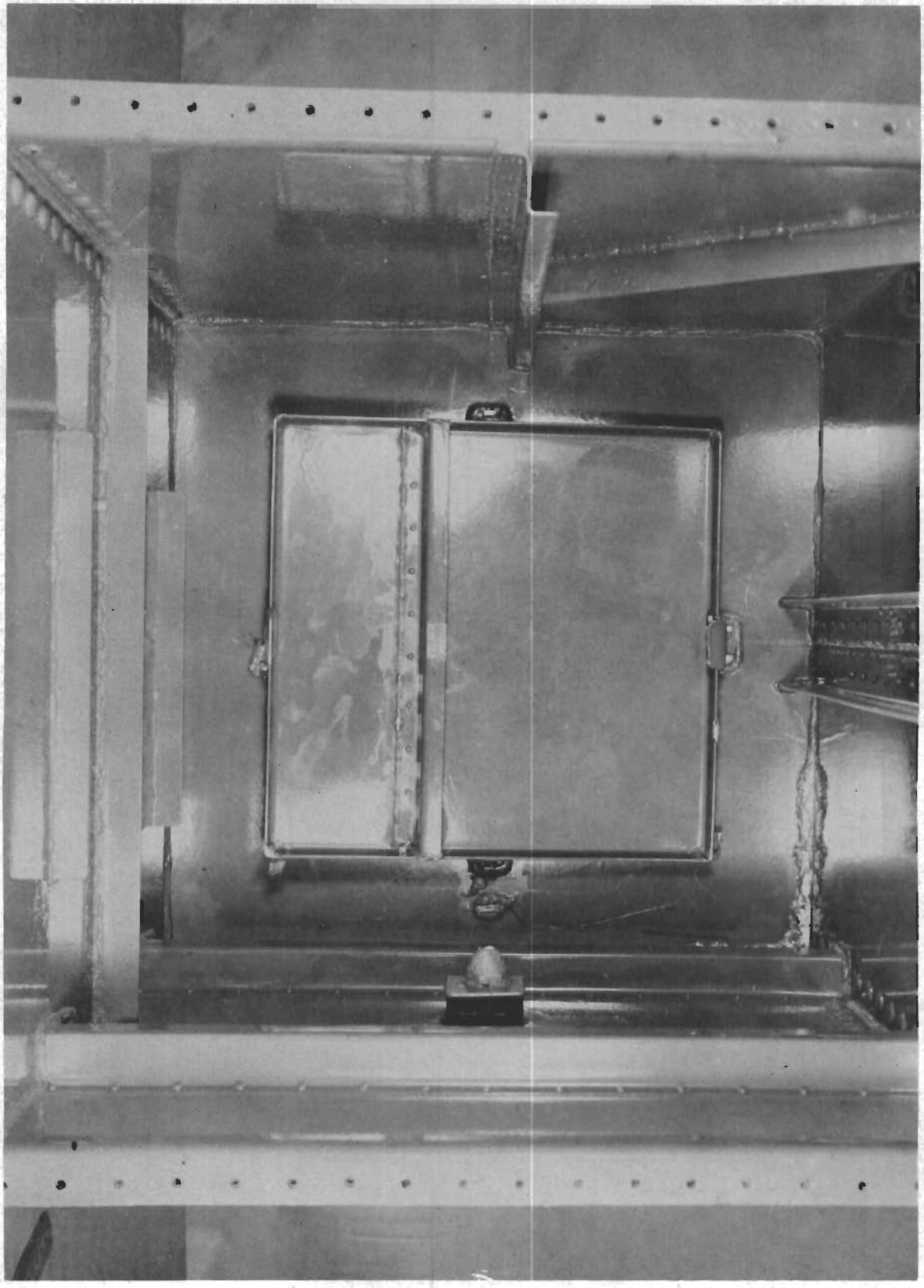

Figure No. 32 Cell C - Following 5 days Exposure to WFNA at 160°F.

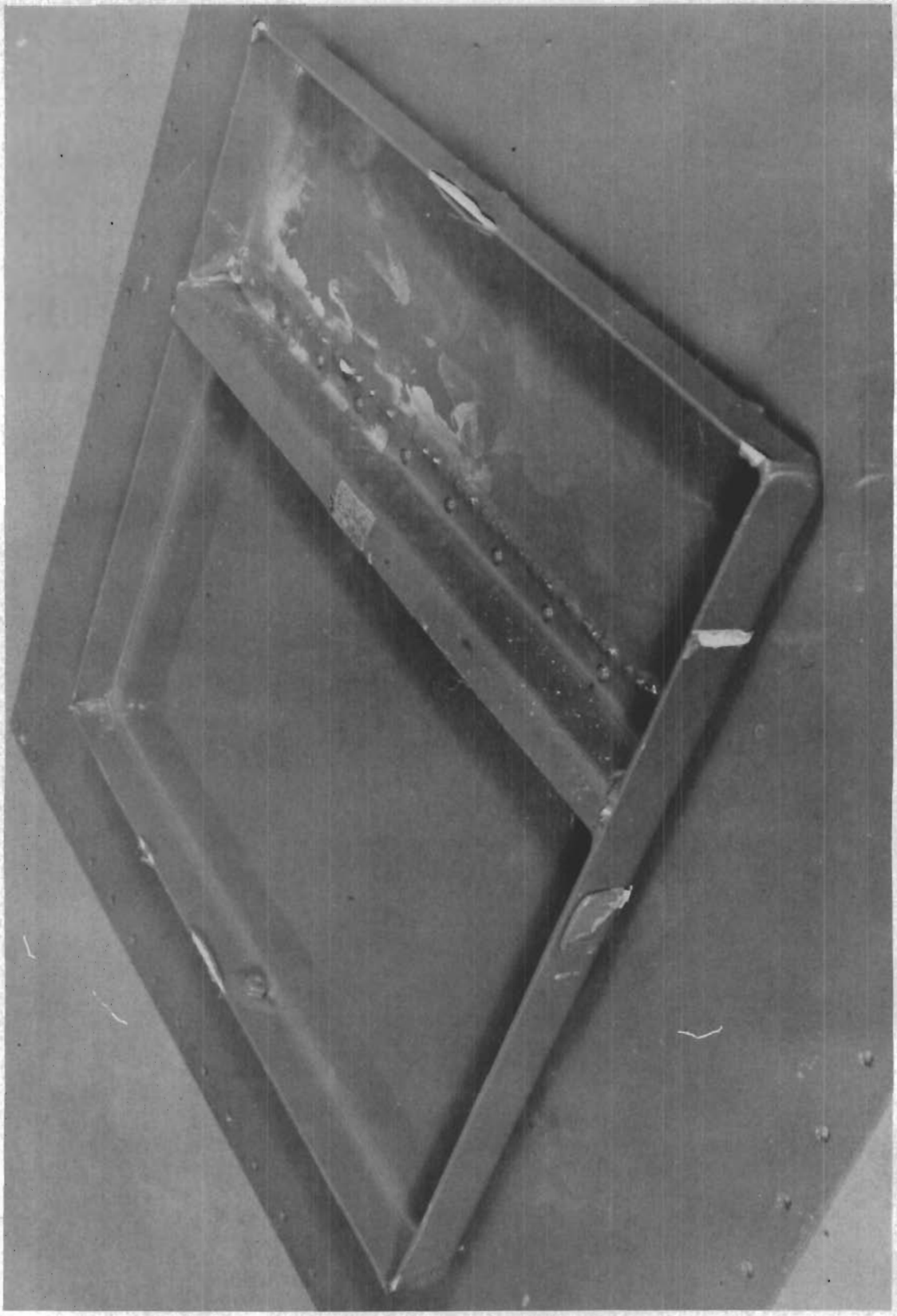

Figure No. 33 Cell C - Acid Container Tray, 5 days Exposure to WFNA at 160°F.

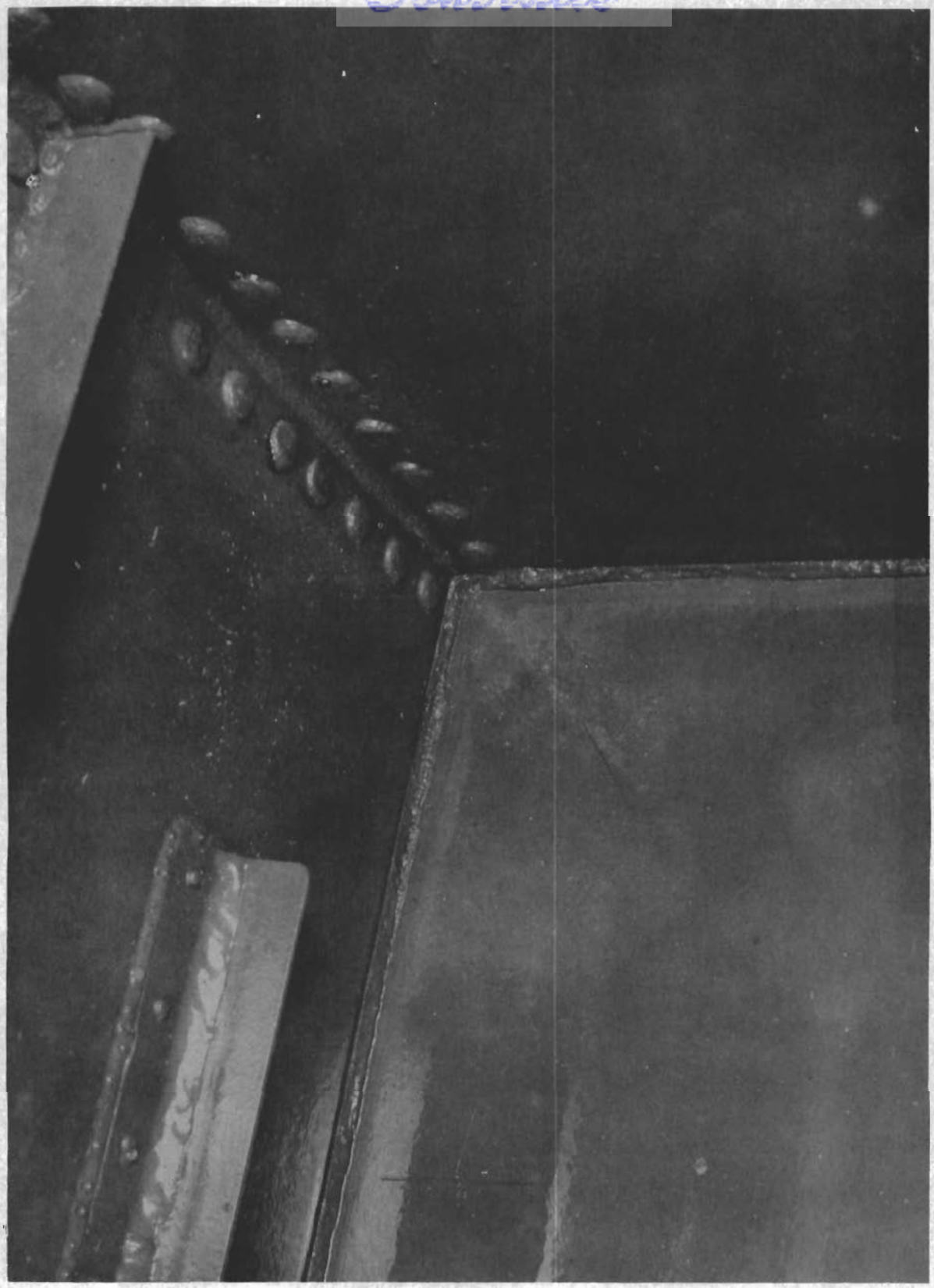

Figure No. 34 Cell C - Close-up - 5 days Exposure WFNA at 160°F. Illustrates Post-Filleting Protection

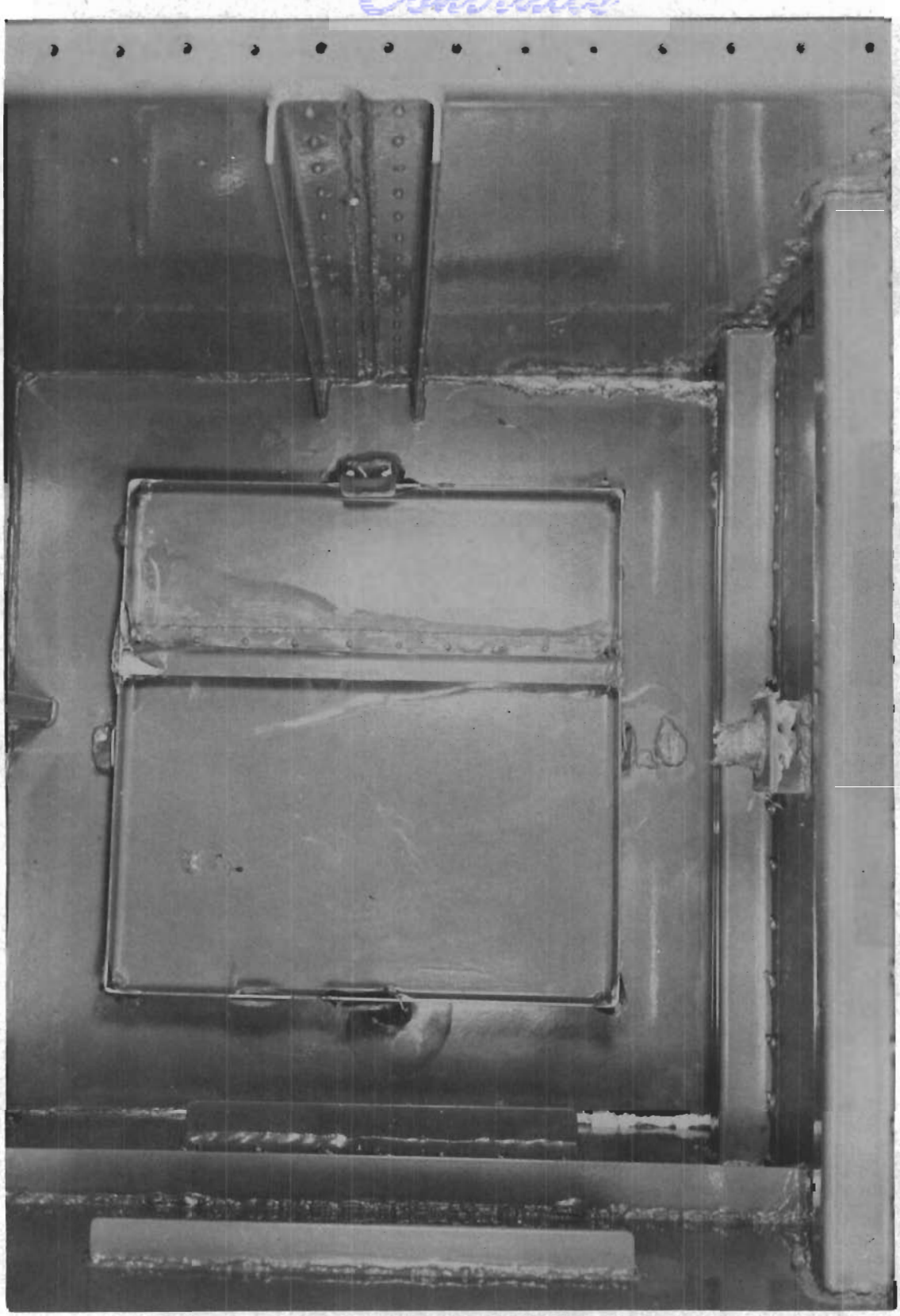

Figure No. 35 Cell C - 10 Days WFNA Exposure at 160°F.

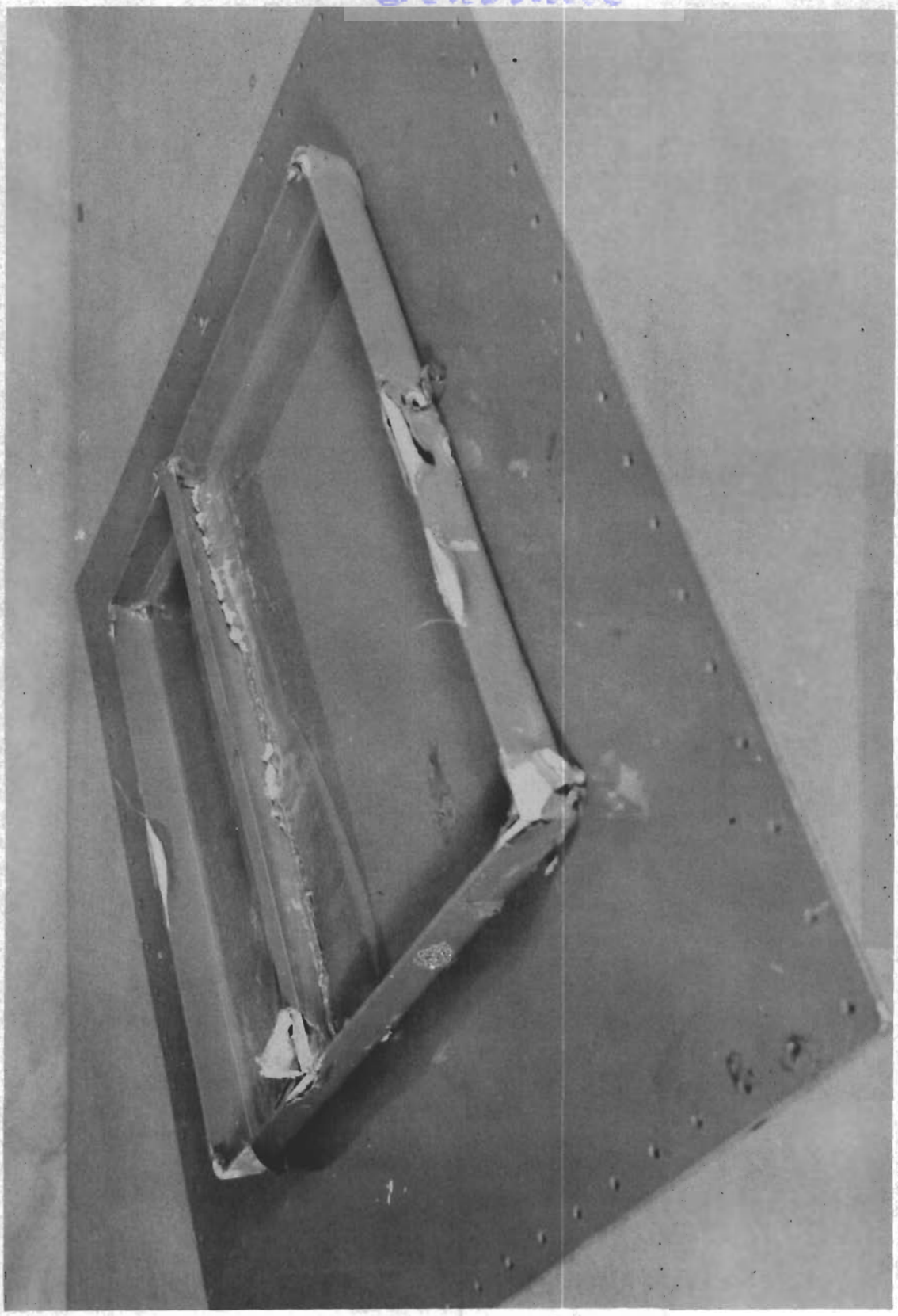

Figure No. 36 Cell C - Acid Container Tray and Base
After 10 Days Exposure to WFNA at 160°F.

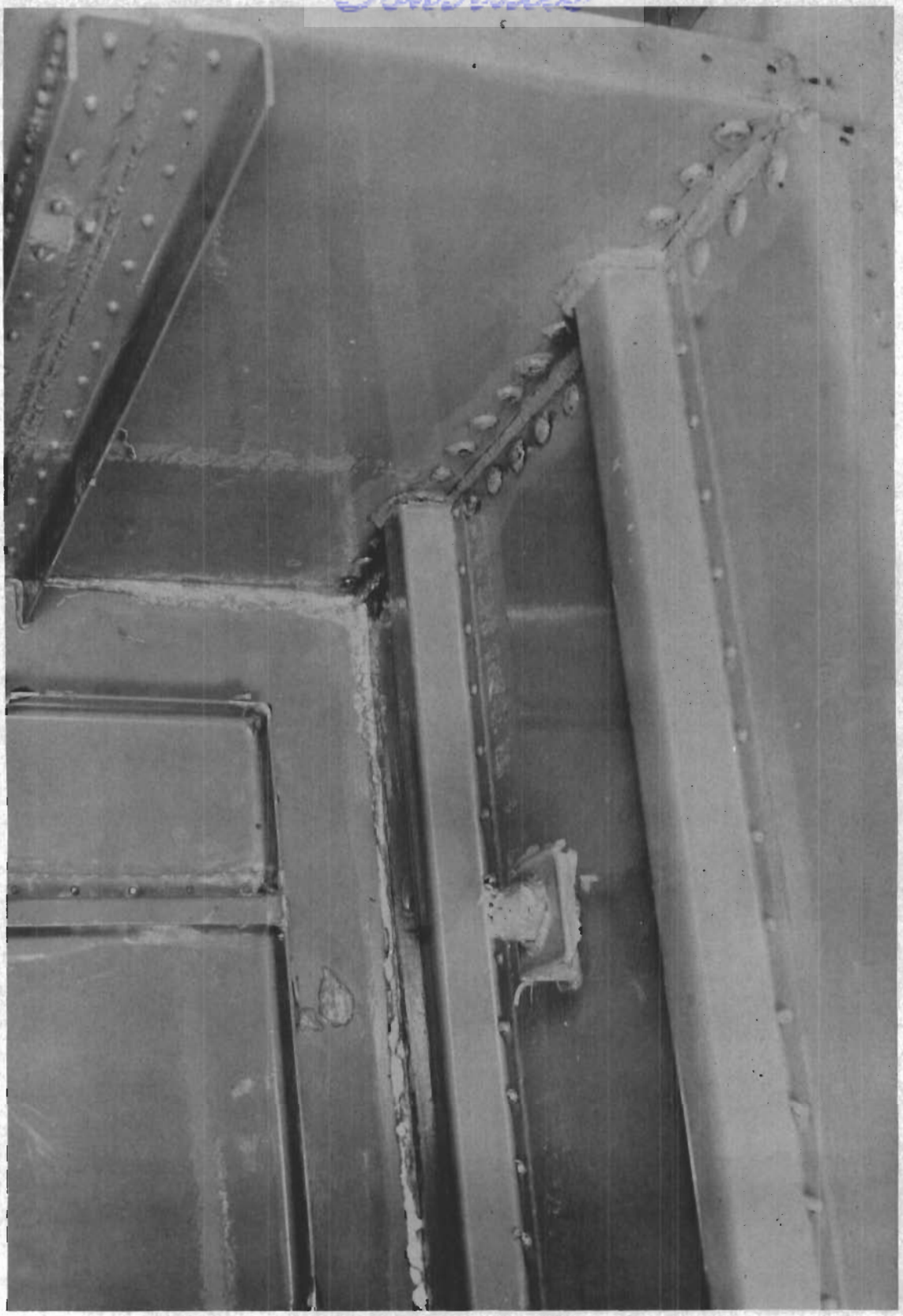

Figure No. 37 Cell C - Illustrates WFNA Breakdown of Improperly Compressed Filleting

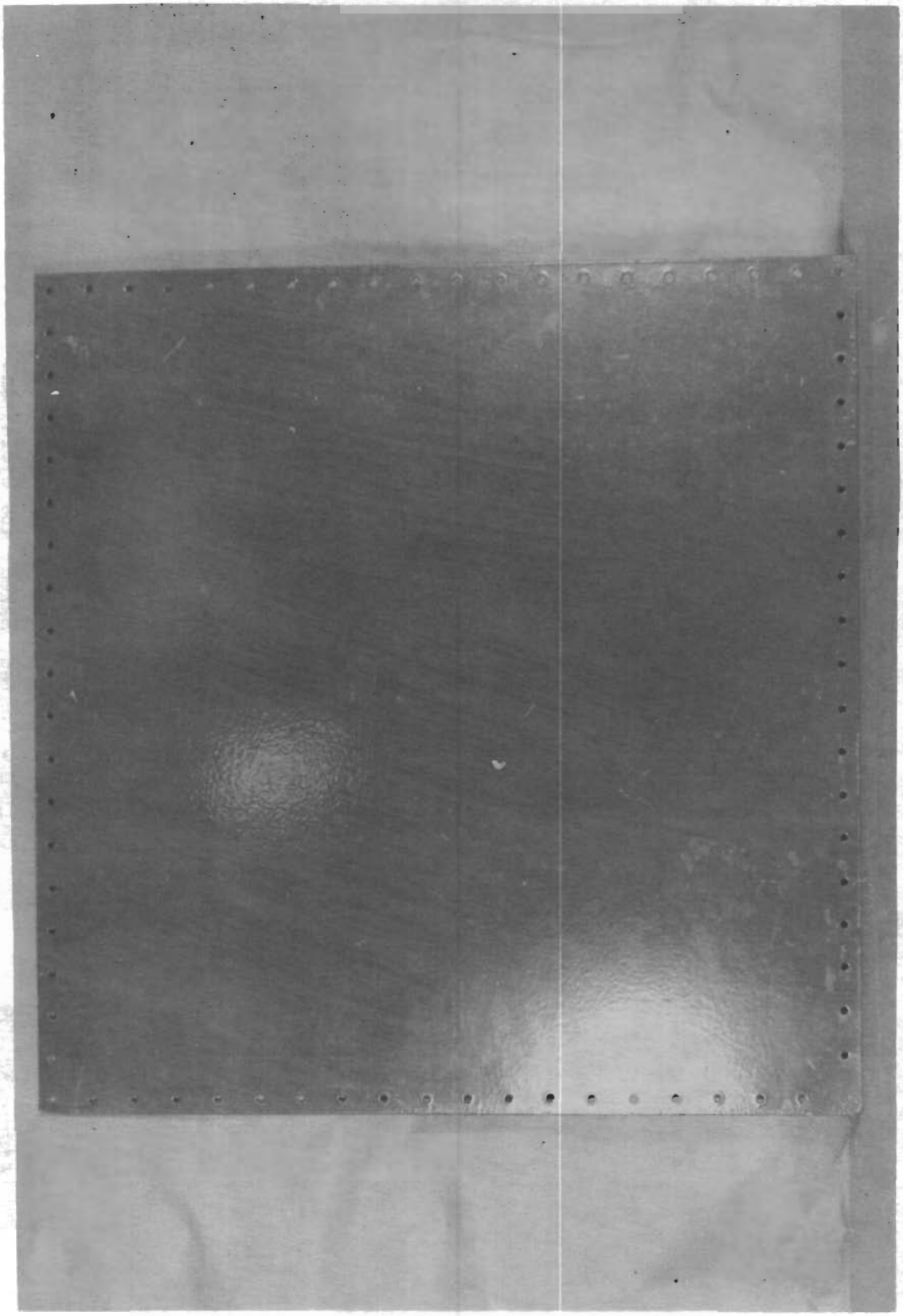

Figure No. 38 Cell C - Closeup of Compartment Cover After 10 Days Exposure to WFNA at 160°F.

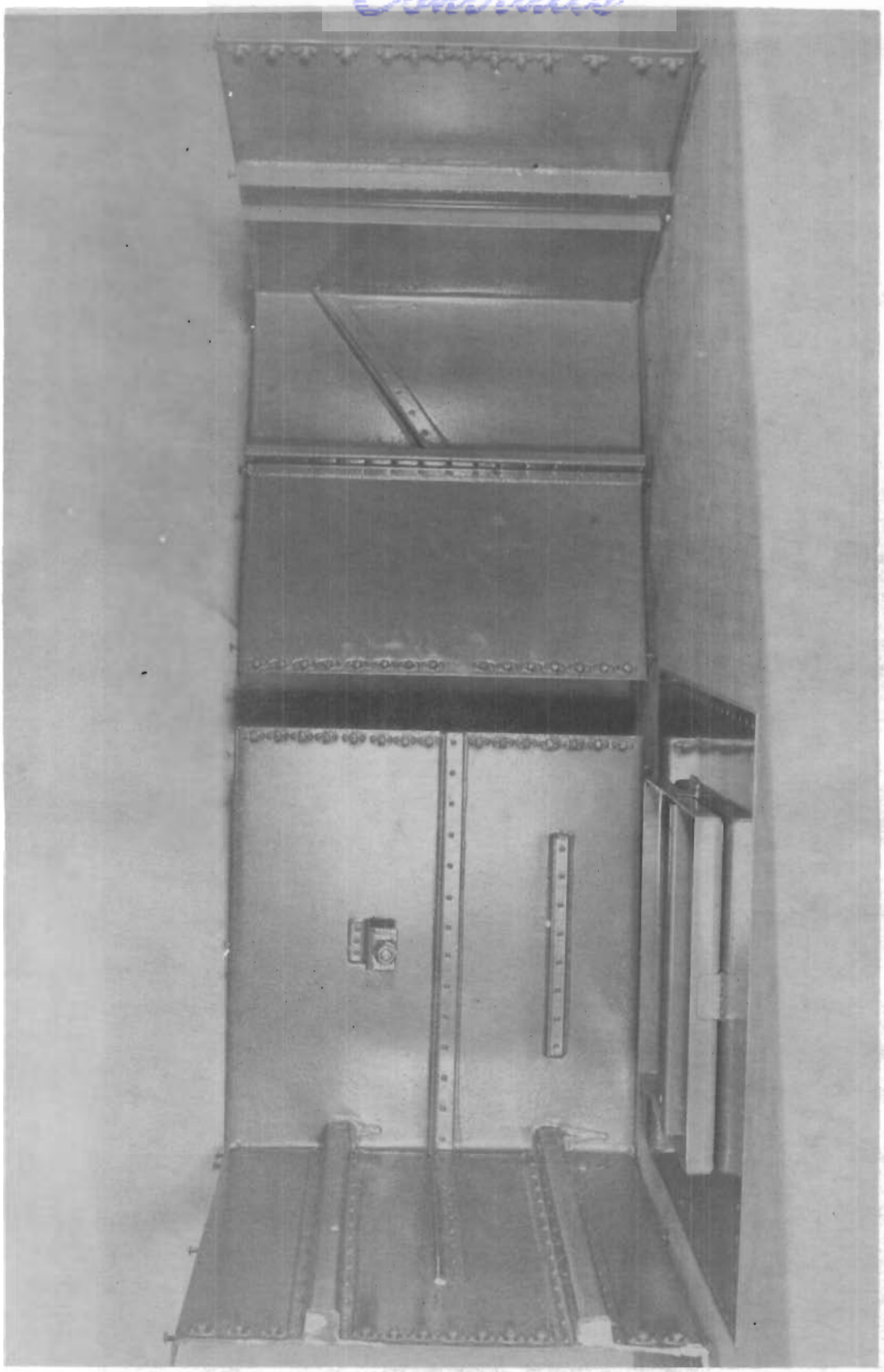

Figure No. 39 Cell D - Coated, Baked, and Cycled and Ready for Test in JP-4 Fuel at 160°F.

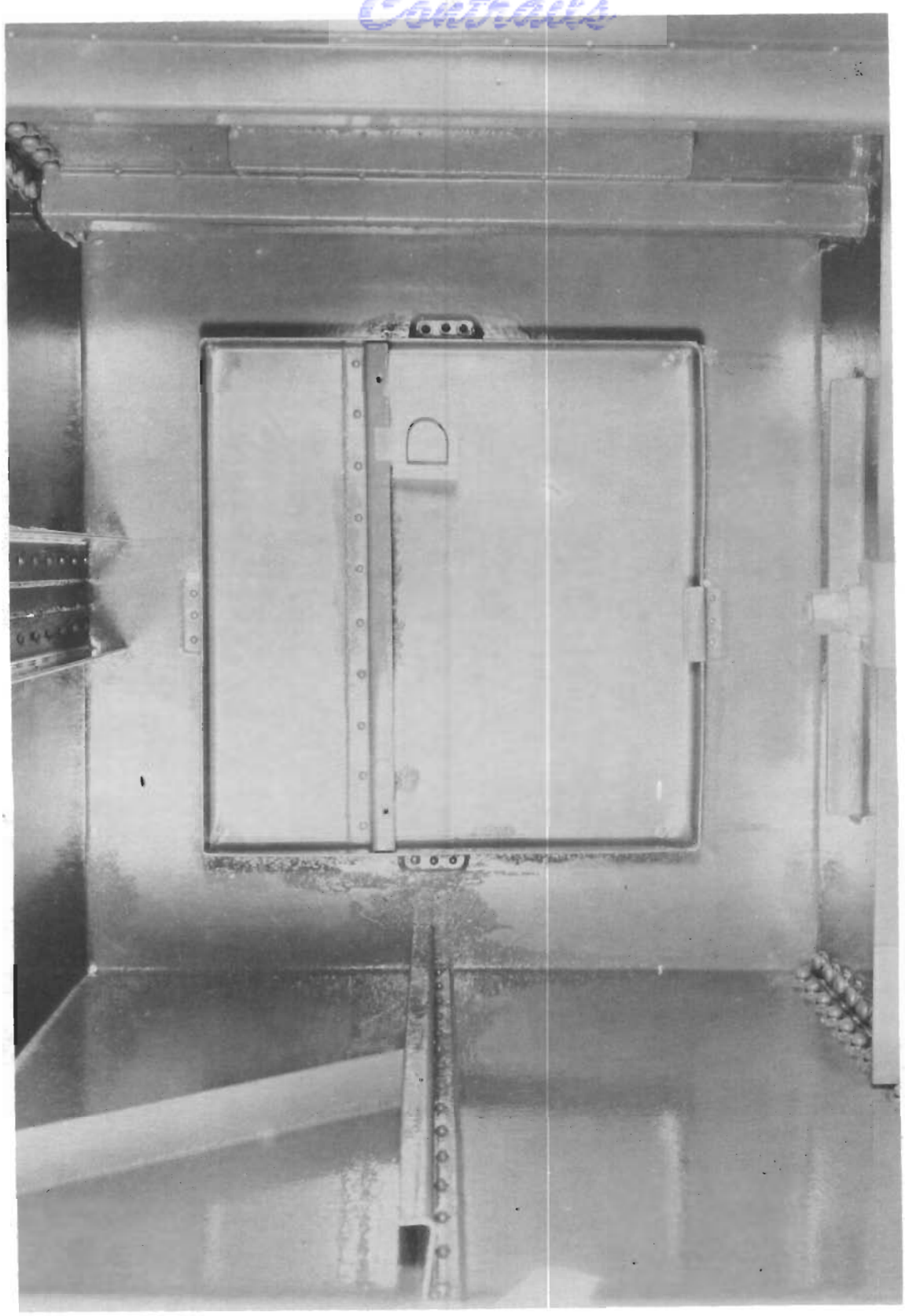

Figure No. 40 Cell D - Exposed 10 Days to JP-4 Fuel at 160° F.