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WADC TECHNICAL REPORT 54-527

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 Co. 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 (Formerly RDO 611-12, Organic Protective Coatings and Related Materials). Administration of the contract was under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Mr. Sam Collis acting as project engineer.

The contract period covered from May 15, 1952 to May 15, 1954. The initial problem concerned the development of a Nitric Acid resistant primer and coating of ATO Compartments employing natural or synthetic film forming resins with some emphasis on fluorocarbon compounds. As of April 22, 1954 per Change Order C-3(54-1525) the investigation was confined to the development of filleting and coating materials based specifically on fluorocarbon resins. After two years of work, including two extensions and presently proceeding into a third extension, the total expenditures are slightly over one-half the amount of the contract total.

The work of Mr. H. L. Cahn, Manager of Coatings Laboratory, terminated with the completion of this report and is gratefully acknowledged.

The assistance of the Management and Laboratory personnel of the M. W. Kellogg Co., particularly the services of Dr. Frank Honn, is sincerely appreciated and gratefully acknowledged. Acknowledgment is also to be made to the personnel of the Materials Laboratory of WADC, particularly to Mr. S. Collis and Major Michael for their patience and assistance in various phases of the project.

Many of the materials tested were not developed or intended by the manufacturer for the conditions to which they were subjected. Any failure or poor performance of a material is therefore not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

ABSTRACT

Numerous film-forming raw materials from solvent systems and non-volatile filleting compounds have been screened for their resistance to white fuming nitric acid. Results show Fluorocarbon Resin X-200 from M. W. Kellogg Company is the only available resin meeting this contract requirement for coating and filleting compounds. Air dry, force dry or low temperature bake coatings have been tentatively formulated to provide the required protection. Studies of physical and chemical properties in conjunction with the compounding of this resin indicate the need for additional work to provide a more practical drying cycle and work is being continued concerning this phase.

Filleting compounds have been developed and tested to meet basic requirements. Further studies are being made with regard to equipment and manufacturing methods. Work on all phases, including the refinement of coatings, production procedures on filleting compounds, and final cycling tests are to be carried out.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. R. WHITMORE
Technical Director
Materials Laboratory
Directorate of Research

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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 installations.

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) Specific Requirements of the Coating System

(1) The maximum allowable temperature for some aluminum aircraft components concerned is 300°F., therefore, any fusion or baking temperature required for curing shall be such that the temperature of the metal to which it is being bonded does not exceed 300°F. Air drying is preferable.

(2) Exposure for the minimum permissible time to red and white fuming nitric acid and selected hydrocarbons as liquids, fumes or vapors (both direct contact or occasional spillage).

(3) Exposure for the maximum possible time to red and white fuming nitric acid and selected hydrocarbons as liquids, fumes or vapors (both intermittent and prolonged direct contact).

Note: (This exposure to be subject to the test conditions detailed in Item (2).

(B) General Development of the Topcoat System.

At the outset of this program of investigation and development to produce a protective coating which would be adequately resistant to WFNA, it was essential that a background of pertinent information be acquired. By means of conferences with Air Force personnel at Wright Field, and with representatives of the M. W. Kellogg Company, and by means of reports on related projects, which were made available to us, a great deal of much needed data and information was acquired.

Inasmuch as the Kellogg Company's work with soluble derivatives of Kel-F was only in its initial stages, it was necessary to rely, temporarily at least, on existing resins in the search for something that might have potential use in this development program. With the aid of reports on related subjects, and calling on our own background of experience in specialty coatings, we either interviewed or contacted by correspondence representatives of producers of materials that might possibly fit into this program.

Having accummulated all data available at the time and with samples already on hand, laboratory work was begun, although further discussions and gathering of samples continued for many months as information on newer materials and the materials themselves became available. Until our first stock of WFNA was received, initial screening work was done with concentrated (70%) nitric acid almost exclusively in spot tests.

By the time we were ready for larger scale tests, our Testing Laboratory had constructed apparatus for continuous exposures to liquid and vapor phase WFNA. This consisted of (1) a large tank for use as a constant temperature bath, (2) glass jars for containing the acid, with bored rigid vinyl covers for the jars, and (3) slotted porcelain plates for supporting the panels and stirrers. The bored covers were fitted with water-cooled reflux condensers to confine the vapors (See Photo No. 2). The principle of this test apparatus was excellent but after some use, it was observed that we could not get reliable results. In order for this unit to function properly, and give a true picture of the degree of protection provided by the coating, it was necessary to have both sides and all edges of each panel completely coated. At best, it is difficult to insure total coverage of every spot on all edges. Furthermore, this allowed for no support of the panel during coating. Efforts were made to compensate for this by holding the panel on the partially dried coated side with a suction gripper, but this did not solve the entire problem. For this reason, this apparatus was abandoned in favor of the Pfaudler Test Unit (See Photo No. 1). This was far more successful in that it necessitated coating only one side of the panels and no edges, as the gasketing sealed off the coated faces of the panels from all other areas. These units were used henceforth throughout the testing. As time went on and progress toward our goal was made, the H-Cell (See Photo No. 7) was used as additional testing equipment in order to determine, in different terms, the degree of permeability of a free film, in contrast to the supported film as used in the Pfaudler Unit.

Before very much time had elapsed, however, it was determined conclusively that the use of 70% nitric acid for screening was without value, in that there was no correlation between resistance to either hot or cold conc. HNO_3 and WFNA. Failure to resist cold concentrated acid, of course, ruled a material out immediately. This very early work with 70% nitric acid, coupled with preliminary tests in WFNA, which soon became available, eliminated, very early, several resins from consideration. Among them were the epoxy resins, which have very limited acid resistance, and chlorinated rubber which, while somewhat acid resistant, is not tough enough for WFNA. Likewise, Parlon-Gilsonite-Aroclor combinations failed to make the grade. Synthetic cyclized rubber resin alone, with Aroclor and with 70% chlorinated paraffin were rapidly destroyed in contact with WFNA or were too brittle to receive further consideration.

A solution of Exon 400XR61, a partially fluorinated polyethylene showed some resistance to chemical attack by the acid but was very permeable to the vapor and liquid. Combinations of this resin with Aroclor and with chlorinated paraffin, respectively, were severely attacked by permeating the film.

Vinyl systems, utilizing a conventional type primer with topcoats based on the highest molecular weight copolymer, VYDR, failed due to permeability of the films. VYDR-Aroclor combinations also failed due to permeability. Stainless steel and high silicon iron pigmentations did not improve the over-all resistance to passage of the acid through the film.

Combinations of polycyclopentadiene and Aroclor looked good in preliminary testing with concentrated acid but they failed quickly in WFNA.

Other resins given preliminary testing in hot concentrated nitric acid and set aside because of poor resistance were Exon 402, a somewhat soluble PVC (polyvinyl chloride) Hypalon C-2, a chlorinated polyethylene and Vistanex B-100 (Polyisobutylene).
Much detailed work has been done, particularly on Vinylite VYDR-Aroclor coatings, wherein clear films and films with pigmentations of stainless steel, stainless steel and mica, and high silicon iron were evaluated at 77° and 125°F. These included exposures over various primers with and without previous alodizing of the aluminum, and over "Alodized" metal without additional priming. The final conclusion was that the relatively high permeability of all of these films to WFNA, both liquid and vapor, resulted in failure to provide the desired degree of protection. (See Photos No. 13, 14, 15, 16, 17). Other evaluations indicated that a VYDR-Chlorowax vehicle might be somewhat less permeable but the advent of Resin X-200 from the M. W.Kellogg Co. precluded any further exposures of these materials, inasmuch as it was the consensus of opinion that they could not approach the potential of a highly fluorinated resin such as X-200. During the seventh month of this project, the first very small sample of Resin X-200 was received from the Kellogg Co. With practically no information about this resin available from any source, a program of work was organized to learn something about its solution characteristics and solution viscosity. At first, only some very basic solution data was obtainable because the initial sample consisted of sixteen grams. It was sufficient to show that there was an appreciable quantity of undissolved matter which would be objectionable. During the next few months additional small quantities of resin were received and these enabled us to make an extensive evaluation of single solvents and solvent mixtures in an effort to evolve what we believed would be an optimum solvent (or blend) for this new resin. With few exceptions, our work centered about the solubility in various ketones since these exhibited considerable solvency very early in the investigation. Enough other types of solvents were evaluated, however, to observe a definite trend as to solvency properties. Among those which were not ketones was tetrahydrofurane, (THF) and this appeared to be the most promising constituent of any solvent blend. In fact, based on solution viscosity, general appearance of the

solution, and degree of insolubility, a tentative conclusion had been drawn that this solvent would be the principal constituent of any solvent mixture that was evolved. On the basis of that conclusion, a solvent mixture consisting of 70% THF, 20% methyl-ethyl ketone (MEK) and 10% ethyl butyl ketone (EBK) was decided upon as the blend with which to dissolve the resin and proceed with further tests.

The next step was to determine the degree of adhesion that might be expected over aluminum alloy panels. Alclad 24ST was the test panel material in all of this work. It was found that air dried films had poor adhesion to solvent cleaned, "Alodized" and "wash primed" panels. A single coat of this material, baked at 250°F. for thirty minutes, had good adhesion to bare and "Alodized" alloy, but excellent adhesion to "wash primed" metal.

As the development work in the production of Resin X-200 progressed from the laboratory to the semi-pilot plant stage, new problems arose. Our first sample of this type of production differed so radically from the laboratory resins that our carefully worked out solvent blend was essentially useless. Thus a whole new investigation of solvents was undertaken, including the work, at this point, on a study of the incorporation of hydrocarbon diluents.

Early in this review of solvent systems for semi-pilot plant batches of Resin X-200, THF was still the major constituent, blended with a minor portion of a mixture of ketones. Films cast from the clear supernatant portion of solution (in which was found 96-98% of the resin originally put into solution) air dried cloudy or hazy, but cleared upon the application of heat. This led to the conclusion that true solutions were not being obtained, and that force drying would be necessary to fuse the film.

Much time and effort was spent in trying to develop a coating and drying schedule which would yield multiple coat, uniform films of at least 10 mils thickness. Three coats could be applied but always on the fourth or fifth coat blistering occurred. If the drying temperature were held down near 200°F. several coats could be applied but these films were inferior in toughness and adhesion. Furthermore, additional heating at 250°F. - 300°F. blistered these films which up to approximately 200°F. were satisfactory from the point of view of smoothness and uniformity. Various other expedients were tried to gain the objective of a uniform thick film but to no avail. In comparing notes and test results with the Kellogg Co., and after having advised them of the difficulties being experienced, we were informed that they, too, were dissatisfied with the solution properties of the resin and were working on the development of a modification which was to be completely ketone soluble.

After having exhausted our supply of old resin, and while waiting for the new, revised material, attention was diverted back to Hypalon C-2, a 60-65% chlorinated polyethylene. After further evaluation of this resin, the following conclusions were drawn:

(1) It was unsuitable for the purpose at hand as an air dried film because of failure to resist permeation by WFNA.

(2) Baked films were far too brittle to be of value.

(3) No satisfactory acid resistant compatible plasticizer could be found.

With the arrival of the new ketone soluble Resin X-200, work was resumed on this material. Inasmuch as it represented something different from that with which we had been working, a fairly extensive study of solvents ensued, followed by tests on the incorporation of hydrocarbon diluents.

A solvent mixture consisting of a 3:1 by volume mixture of methyl isobutyl ketone (MIBK) and toluol was decided upon as being satisfactory, and a 15.45 percent solution of the resin in this blend was prepared. For spraying, this was further reduced to 3 parts base solution to 1 part thinner by volume. It soon became apparent, however, that we were having the same blistering problem as previously described. It was not possible to prepare panels of sufficient thickness and simultaneously of sufficient uniformity and smoothness to warrant exposure to WFNA.

It was found that the resin was compatible with low viscosity Halocarbon oils. Initially 1 part of oil to 3 parts of resin constituted the solids portion of the film. Clear films and graphite pigmented films were attempted but the same blistering trouble occurred as the thickness of the film began to approach 10-15 mils. Raising the Halocarbon oil content increased blistering, so work with the initial 1:3 ratio was resumed. A zirconium silicate pigmentation was prepared with this vehicle solution. A preliminary test of a two mil film on steel gave encouraging results. This was a five coat film, air dried 20-30 minutes and baked 30 minutes at 250°F. between coats. Additional work on this formulation indicated that we were still not free of blistering problems. On brushed films, unreduced, slight blistering occurred on the eighth coat. No further coats were applied in order to expose the films as thus far prepared. Further work was done baking these films at 250°F. and 300°F. but all blistered.

A new formulation was prepared in which the solids were composed of 75% Resin X-200 and 25% Kel-F 3-200 plasticizer which is a waxy low molecular weight polymonochlorotrifluoroethylene. The solvent included mixed ketones to balance the evaporation rate, and toluol as a diluent. Initially, this was used as vehicle for pigmentation with zirconium silicate. Pigmentations were prepared in which the ratio of pigment to vehicle solids was 1:1 and 0.5:1 by weight. The higher pigmentation received the first attention. Several baking schedules of each individual coat failed to produce satisfactory films because of blistering before many coats were applied. An entirely new approach was then investigated. Experience had shown that these films were dry to handle within fifteen minutes after they were applied. On this basis, multiple coats were applied with only fifteen minutes air drying between coats. Sufficient coats were applied to yield roughly 15 mils of coating thickness (before force drying). After all coats were applied, the panels were air dried for one to three days. If they were air dried one day, the initial force drying temperature was 100°F. If they air dried three days, the initial temperature was 125°F. This was the beginning of a gradually-increasing temperature force drying schedule. The cycle extended over a period of three days and concluded with four hours at 250°F. and one hour at 300°F. By this means a thick film was obtained, force dried at high temperature and free of blisters.

Concurrent with the preparation of these panels, others were prepared and air dried only, over a period of three weeks.

While it was shown that even force dried films would not resist the WFNA at 125°F. for very long, varying results were obtained with force dried and air dried films in WFNA at 77°F. Two tentative conclusions were drawn:

(1) A plasticizer content of 25% of the total solids was too high for elevated temperature exposure. It lowered the softening point of the film, making it more vulnerable to attack by the WFNA at elevated temperatures.

(2) The 1:1 ratio of pigmentation was too high, leaving the film too porous for resistance to WFNA. (See Photos No. 3,4,5,8,9,10).

The force drying schedules at gradually increasing temperatures, and the long air drying were repeated on the lower pigmentation formula. The air dried films were recognized as being of relatively poor quality and this was proven by their failure after only a few days exposure to WFNA. Sets of force dried panels went 23 and 32 days, respectively, before showing evidence of failure by the appearance of small blisters. While these results proved very encouraging, it was recognized that they would bear checking to determine whether or not they were reproducible. Sometime later a check of this exposure revealed satisfactory results for 38 days without any evidence of blistering. After that a few small blisters were noticed but the exposure continued for a total of 42 days, after which there was still no very serious damage to the films or metal substrate. (See Photos Nos. 11 and 12).

Similar films, force dried in the same manner were exposed to WFNA at 125°F. and did not show any signs of failure until the fourth day. They were left exposed for nine days, without very serious failures. This was by far the best resistance to any elevated temperature exposure yet obtained on a coating, but it was not as yet regarded as satisfactory. See Photo No. 6. The pattern established was definitely indicative that this could be improved. Evaluations of variations in type and quantity of plasticizer and of still lower pigmentations are indicated, although a certain minimum of pigment appears to be necessary.

In the meanwhile, the improved test results that were being obtained, indicated that H-cell testing would be in order. The H-cell is a method of measuring acid permeability through a membrane. See Photo No. 7.

Thick films, both clear and pigmented, were prepared. It was intended that sections of each be air dried different lengths of time and also force dried. The pigmented film was 15 mils thick and the clear film was 17 mils thick. They were prepared by applying multiple coats with only a fifteen minute air dry period between coats. Because of a two month delay in receiving the proper gaskets, these films had air aged all of that time. It was felt that force drying after all that time would be meaningless, and therefore, the H-cell tests were conducted on the two month air aged films.

The difference between these two H-cell exposures is quite remarkable. The pigmented film failed, as indicated by a drop from pH 5 to pH 3 on the water side of the cell in only two days. A previous test of this material, which was not regarded as being too reliable, showed five days resistance before the pH dropped to three or lower. The latest test, therefore, it is presumed, must have been on an inferior film. The contrast, however, was between the pigmented and the clear film. After a nine day exposure of the clear film the pH was 3.4. Because of an intervening weekend, the next reading was on the twelfth day, at which time the pH was 2.8. The film showed no ill effects as a result of this exposure. (See Photos Nos. 17, 18 and 19).

At this point it would be well to discuss the correlation between H-cell tests and Pfaudler unit exposures. In the latter, the first evidence of failure is a blister which results from the permeation of the film by a certain minimum volume of acid —

certainly much more than a single drop. It was determined, somewhat as a control, that a single drop of 70% nitric acid is sufficient to drop the pH of approximately a pint of water from 5 to 3. (The volume of water contained in the "H" cell is 300 c.c.'s.) Thus, it is to be expected that identical films will show failure in the H-cell much sooner than in the Pfaudler unit. On the other hand, while it requires considerably more acid penetration of the film to result in failure in the Pfaudler unit, it is not that any serious damage is done to the substrate before failure. Furthermore, the exposure in the Pfaudler unit, being total immersion in the liquid and exposure to undiluted fumes above the liquid, is much more severe than would be found in actual service.

Thus far it had been shown that a clear film (AF75) when air dried, has good resistance to WFNA as indicated by the H-cell exposure. Furthermore, a 19 day-air-dried film of AF75 lasted nearly four weeks in the Pfaudler unit. Similarly, a force-dried pigmented film (AF77) revealed good resistance, at 77°F. as indicated by the Pfaudler unit exposure. (See Photos Nos. 11, 12 and 20). It has not been possible to prepare similar clear films and put them through the same force drying schedule without the formation of blisters. In contrast to the air dried clear film, and force dried pigmented film, an air dried pigmented film has failed repeatedly in the Pfaudler unit. Following the same pattern, films consisting of alternate coats of clear AF75 and pigmented AF77 showed the properties of the clear film. Such a film could not be force dried without blistering, but when air dried for 19 days, it was exposed to WFNA at 77°F. for 40 days before showing blisters. (See Photo No. 11).

Certain of these tests will be repeated to test the uniformity of resin as received. A recently received batch shows the same general appearance in solution and same solution viscosity characteristics.

Referring to a previous statement that variations in type and quantity of plasticizer might have a significant effect on the resistance of the films, work was undertaken along these lines. A thorough evaluation of original viscosity and weight versus reduction viscosity and weight was made on 20% solutions of (1) straight resin, (2) 85:15 ratio of resin to plasticizer. The plasticizer in these cases was Kel-F 10-200 wax, chosen because it is a higher polymer, therefore, harder and higher melting than the "Plasticizer Grade" Kel-F 3-200. Attempts, thus far, to force dry multiple coat films of this material have all met with failure due to blistering. Nevertheless, in order to obtain some information on the resistance of the film with different amounts of 10-200 wax, these force dried films, even with some blisters, were exposed. These three formulations, identified as AF79A, AF79B and AF79C, had some blisters as a result of the application of twenty-seven coats, with a 15 minute air drying period between coats, and no application of heat. These were exposed to WFNA in the Pfaudler units at 77°F. and exhibited definite evidence of failure in 3, 17 and 10 days, respectively. (See Photos Nos. 21, 22, 23).

To accelerate the solvent release, panels were prepared using a 15 minute period between coats at 100°F. instead of 15 minutes at room temperature. This cycle offered no advantage.

While some work is being conducted on panels air dried for two to three weeks, emphasis is being placed on the force drying schedule, because a long air aging period before risking exposure of the coating to WFNA may in some cases be impractical. Pigmentation of the coating will be necessary to provide solvent release from the film, either through force drying or long term air drying. The degree of pigmentation required has not been determined.

(C) General Development of the Filleting Compound

In the normal engineering and design of aircraft structures, butt and overlap joints with and without riveting are encountered and it is required that such recesses, projections and depressions be properly filleted in order to provide a base over which the protective coating can be applied.

The filleting material should be formulated to be a putty-like consistency so that it can be applied to hairline or wide fissures by conventional methods. It is preferred that the filleting material be of an air dry type with ability to withstand force drying conditions that are required of the protective topcoating.

The first phase of the contract with respect to the filleting compound was spent in selecting the possible raw materials having chemical properties that would meet the requirements. In addition, special mixing equipment was necessary in order to produce the filleting materials. A number of conferences were held with representatives of various companies who manufactured resins having possible application. The result of these interviews, both oral and written, indicated that the following materials should be investigated as base resins for a filleting compound:

(1) A soluble fluorocarbon polymer, now known as X-200.
(2) Polyethylene.
(3) Chlorinated rubber (Parlon).
(4) Aroclors (Chlorinated biphenyls).
(5) Fluorinated waxes and oils.
(6) Chlorowaxes (chlorinated paraffins).
(7) Gilsonite compounds.

(8) Hypalon C-2 and S-2 (chlorinated and chlorsulfonated polyethylene).
(9) Polyisobutylenes and polybutenes. (Vistanex)
(10) Soluble fluorine polymer (Exon 400 XR61-Firestone).
(11) Miscellaneous materials from which no FNA resistance was expected but tried nevertheless for their physical properties.

After the initial research from the literature and from the various manufacturers, the above materials were formulated in different compounds and tested, irrespective of their filleting characteristics. Of the materials tried, the following looked most promising, at the outset:

(1) Polyethylene Vistanex mixtures.
(2) Vistanex-*Halocarbon oil mixtures.
(3) Vistanex-Chlorowax mixtures.
(4) Hypalon S-2 compounds.

(* low molecular weight polymonochlorotrifluoroethylene)

The above materials were screened in an exposure test involving immersion in 70% nitric acid at 160°F. followed by further immersion in 70% nitric acid at 250°F. These exposures involved immersion of a small piece of compounded material directly in the acid, unsupported. At this time, however, the Pfaudler columns were received and testing in WFNA began on supported panels, using fuming nitric acid in place of 70% nitric acid. Concurrently, the results of the work on polyethylene-Vistanex compounds by the Connecticut Hard Rubber Company were received and further work was done on these compounds to evaluate their ability to resist the acid, and to protect and adhere to the metal. From this work, it was decided that a blend of 30% Vistanex B-100 and 70% polyethylene PM-1 showed the best over-all resistance to WFNA and would be the most adaptable for application, particularly from a handling standpoint. It was anticipated that this mixture would have been in an extruded tape form to be softened in place by the use of heat. Extensive testing done on this material indicated that this compound gave the best resistance to be expected from any polymer other than a highly fluorinated polymer. It was later proven that this material gave erratic permeability tests on the basis of Pfaudler and H-cell exposures to the extent that further work was discontinued and attention directed to the formulation of X-200 polymer. (See Photo No. 24). To approach the formulation of X-200 and to produce a putty-like consistency, it was necessary to discover suitably resistant plasticizers for the X-200. During the search for plasticizers for the X-200, a new series of resin evaluations were undertaken. The best resistance to FNA exposure was achieved by the following:

(1) Parlon (chlorinated rubber).

(2) Hypalon C-2 (chlorinated polyethylene)

It was found that fluorocarbon oils and waxes were not compatible with resins other than the X-200, and that no resistant plasticizer could be found for Hypalon or Parlon.

At this time small quantities of the X-200 polymer produced in glass laboratory equipment were made available and the compounding development of the X-200 filleting material was begun. The initial work gave the following results:

(a) X-200 polymer is inherently resistant to WFNA.

(b) The X-200 polymer currently being used for the development of a protective coating is not suitable for a filleting compound due to the rapid rate of solvation and low softening point.

(c) Later samples obtained from M. W. Kellogg Co. which had higher molecular weights and/or a different polymer ratio of resin content revealed better possibilities as filleting compounds.

(d) Mixing of the compound resulted in the problem of removing occluded air. It was thought that a modification of the deairing type ceramic extruder would provide an answer to this problem.

Observations based on the results of testing:

> Even though a solid material in the form of a tape or a rod may be of use as a filleting bridge, such a material will depend in its function upon the adhesion to the metal to be protected and one point of leakage can ruin the entire fillet. In addition, the difficulty of applying a preformed tape or rod would necessitate a highly skilled application.

The use of volatile materials in the putty formulation usually results in blistering if the compound is subjected to elevated temperatures. The volatiles would have to be resistant to WFNA if no force drying or elevated fusion temperatures are used. In addition, the inclusion of volatile materials to give the desired viscosity would result in definite voids after a long air drying period.

For these basic reasons, therefore, it was decided that a putty (See Photo No. 25), containing no volatiles would be the best type of formula and this necessitated the retention of the liquid phase which must be WFNA resistant. Of the materials known, only the fluorinated oils and waxes would fulfill this requirement.

Removal of the air from the putty is absolutely essential for an impenetrable film. It has been noted that in spongy type putties where no air is removed, even though the WFNA has permeated, decomposition of the metal panel is not evident. This testing was done on steel panels, so that acid attack would be obvious. Immediately after removal from the acid, the fillet was cut and lifted from the panel. The panel was in its original condition even though acid had permeated into the fillet. After washing and permitting the acid saturated fillet to remain on the panel, in air, for one day, the attack on the panel was intense.

During the course of the investigation of the fluorinated resins, the difficulty in obtaining samples, both with respect to quality and quantity, led to evaluation of other type materials from which a putty might conceivably be developed but not necessarily having the inherent WFNA resistance. Numerous epoxy compounds were formulated and tested but failed due to the fact they did not cure sufficiently at 300°F. and would have a very limited pot life during the application. Parlon and Hypalon C-2 were resins showing a good degree of resistance to WFNA in the uncompounded state. They were, however, too brittle for coatings and needed a fluid media for putty use.

The plasticizer for these resins would have to be as resistant as the resins themselves to WFNA. The liquids to be used would be of highly halogenated nature, compatible with the resin and high boiling. The best possibility would be to use the Kel-F or Halocarbon oils and waxes. Unfortunately, these were not compatible. Chlorinated liquids were then checked (See Table I-51-63). Those with the highest degree of halogenation were most resistant to WFNA, highest boiling, and compatible. They were not, however, of a high enough boiling range.

After some search, Hooker Electrochemical supplied us with hexachlorobutadiene and hexachloropropane. The former was attacked by WFNA and was too volatile. The latter fulfilled all requirements except it was not a solvent for the Parlon or Hypalon C-2.

The hexachloropropane, having 75% of available hydrogens replaced by chlorine was as highly halogenated a liquid form as could be obtained. Therefore, this work was discontinued.

An increased supply of X-200 resin permitted us, during the latter part of the two year period, to concentrate fully on the development of a putty. The results of exposure to WFNA at 160°F. has shown that a fluorocarbon putty can be formulated that will meet the following specifications of a filleting compound:

1. The consistency is such that application of the putty by conventional means requires no special technique or equipment.

2. The adhesion to clean aluminum extrusions or sheet is considered satisfactory.

3. Resistance to WFNA degradation is excellent at 160°F. See Photo No. 25.

4. The physical properties of the applied putty meets all requirements from the standpoint of

   (a) the absence of volatile materials
   (b) no loss of adhesion due to elevated or low temperatures
   (c) no loss of adhesion when subjected to normal flight vibration.

The putty formulation which has evolved over this two year period is as follows:

Kellogg X-200 resin (Batch 4322)	100 parts
Kel-F 150 wax	60 "
Kel-F plasticizer grade oil	60 "
Ultrox filler	90 to 100 parts

The resin is ground in a hammermill. The above ingredients are mixed in a sigma blade mixer, under vacuum until homogeneous, for a period of approximately one-half hour. The mass is then fed into a ceramic extruder (See Photo No. 26) and extruded in the form of a rod of suitable diameter under high vacuum which seems to effectively remove the air. The advantage of this type of equipment is that the compound can be produced directly into the form of thin rods suitable for placing in a seam, or in a thick cylindrical rod suitable for inserting in a cartridge type filleting gun.

The effectiveness of this type of processing procedure with respect to the removal of the air will be checked as soon as sufficient quantity of materials are received. This testing, it is anticipated, will extend beyond the duration of the two year period and into the one year extension period.

(D) Possible Uses in Protection of Other Air Force Items

The prime objective of this contract has been the protection of fabricated aluminum sheet and extrusions in aircraft sections which will house or be exposed to spillage and fumes of WFNA and hydrocarbon jet fuel. Under these conditions, a limiting application temperature of 300°F. was required due to the adverse effect on some aluminum alloys when exposed to temperatures in excess of 300°F. This required, therefore, that the protective coating and the filleting compound be so formulated that it could be applied either as an air dry system or, if requiring force drying, that such temperatures and schedules of force drying not exceed 300°F. This limitation necessitated a special approach to the formulating problem. This would have been considerably eased if there had been no limit to the temperature of application. It was, and still is, conceivable that items, other than aluminum sections fabricated in aircraft, will be required to resist spillage and the fumes of WFNA. These items may be fabricated of ferrous or nonferrous metals, in the form of shipping drums, storage tanks, pipe and small bore tubing. In addition, solid, flexible forms of a material may be required to perform as sealants and gaskets to be used in conjunction with shipping containers and transmission devices. This coating may be applicable to safety clothing which is required to withstand spillage and fumes of WFNA.

As part of a program instigated several years ago in cooperation with the M. W. Kellogg Co., investigations have been continuing on the use of other Kel-F polymers and copolymers exclusive of their experimental resins X-200 and directed towards the protection of metal and cloth which will be exposed to highly corrosive conditions. Extruded tubing made from Kel-F, dispersion coatings of Kel-F and a new experimental, curable Kel-F rubber have been the subject of these investigations. These products plus the current status of their development are available, as a proprietary item, to the Air Force. At present, a coating, which is applied as a dispersion and fused at high temperatures, is designated by our product name of "Fluron". This product is applied by spray to metal surfaces which will withstand temperatures of 500°F. for a period of 12 to 16 hours, (e.g. steel drums). The proper application of Fluron dispersion results in an adherent, electrically resistant impenetrable film of 1 mil to 15 mils in thickness, depending upon the number of coating applications. Although this product is in the development stage, it has reached the point where it can be considered as a possible protective coating and lining for steel drums used in the shipping and handling of white and red fuming nitric acid.

Under certain conditions, the application of Kel-F polymers or modified Kel-F polymers to a cloth base, produces a resistant sheet which can be fabricated into safety gear suitable for use during the handling of WFNA. It is our belief, however, with regard to safety cloth, that a more sound development would involve the use of unsupported vinyl film. The vinyl film could be fabricated into suitable safety clothing which, although it will not withstand long time immersion in WFNA, will withstand intermittent exposure and would not be an expensive item such as those made from the Kel-F resin. In addition, the material and fabricating cost of vinyl sheeting would be such that in most instances it could be considered as expendable. In the matter of gaskets used to seal drums or pipe which will contain or conduct WFNA, the curable version of Kel-F, known as the Kel-F elastomer, is recommended. Although this material, when cured, exhibits rubberlike properties, it does not have the high degree of resistance to WFNA as shown by Kel-F resin or X-200. It does, however, have many times the resistance to acids when compared to other rubberlike materials and, when operating in the form of a gasket, can give excellent service. Its physical properties fulfill the conditions of a gasket with respect to high and low temperature exposures, fair compression set, good tensile and elongation properties. The Kel-F elastomer is an experimental product, at the present time, but indications are that the initial price of the Kel-F elastomer should drop as soon as production instead of pilot equipment is installed. It is the only material of a rubber-like nature that can give long service in contact with WFNA but it requires additional study from the standpoint of compounding and testing before it can be relied upon as a substitute for standard rubber formulations.

As mentioned before, all of the above materials are in various stages of development in our laboratories and are available to the U. S. Air Force in the form of preliminary samples. It is to be understood that this work did not, in any form, interfere with the normal progress of the subject matter of this contract.

III CONCLUSIONS

I. Preliminary Raw Material Testing in WFNA.

A. Resins

All halogenated vinyl polymers tested showed either high permeability or excessive degradation. Excessive degradation was experienced with epoxy resins, terpene or unsaturated polymers, chlorosulfonated polyethylene or chlorinated paraffins. Resins found resistant to WFNA in varying degrees were chlorinated polyethylene (Hypalon C-2) chlorinated rubber (Parlon), Polyisobutylene (Vistanex), Polyethylene, solution-type fluorocarbon polymers such as Kellogg X-200. Of these only Kellogg X-200 resin could be considered sufficiently impermeable, flexible and resistant to WFNA.

B. Pigments

The pigments found sufficiently resistant to WFNA were:

Aluminum Oxide
Zirconium Silicate
Chrome Oxide

C. Solvents:

The only volatile liquids found resistant to any degree to WFNA were highly halogenated materials such as trichlorethylene, perchloroethylene, tetrachloroethane and hexachloropropane.

D. Plasticizers

The only non-volatile liquids found resistant to WFNA were low molecular weight polymonochlorotrifluoroethylene polymers such as Kel-F or Halocarbon oils and waxes.

The later stages of coatings and filleting compounding were therefore based upon:

(a) Kellogg Resin X-200
(b) Kel-F or Halocarbon Oils and Waxes.
(c) Aluminum Oxide, Zirconium Silicate or Chrome Oxide pigmentation.

II. Compounding

A. Coatings

1. Processing and Application:

   The best solvents for resin X-200 are Tetrahydrofurane, Dimethyl formamide, Methyl ethyl ketone and Methyl isobutyl ketone. The method of processing the compound is either by a high shear mixer or simple jar rolling. Depending upon the lot of resin used, a viscosity suitable for spraying could be obtained with a 15-20% by weight solution of X-200 resin in MEK, MIBK and Toluol. Usage of lower boiling solvents resulted in excessive feathering during spraying of the coating. Ordinary spray equipment is satisfactory, using suction cup and atomizing pressures of approximately 45 psi. Higher pigmentation of the compound results in thicker sprayed coatings and inclusion of a plasticizer provides greater spraying ease.

2. Drying of the coating.

   Force drying seems necessary to provide an impermeable coating. At this stage of the investigation prolonged air drying (15-20 days) was necessary to provide any degree of resistance to WFNA. At present, air drying does not favorably compare to force drying. A balanced blend of solvents is necessary to provide complete solvent release without blistering during force drying. Pigmentation facilitates solvent release during drying. An optimum drying cycle has not been determined.

3. WFNA Resistance of coatings.

   The best resistance is obtained with a coating of the following type.

   a. Unpigmented or low pigmentation (less than 50% by volume of the resin.)

   b. Unplasticized or plasticized to a degree less than 20% by weight of the resin.

   c. Complete solvent removal, or removal to a degree afforded by a 15-20 day air drying or a 30 min. force drying at 300°F.

4. The maximum resistance to WFNA coupled with good compounding and processing characteristics has been obtained with the following formulation:

AF-77

Resin X-200	137
Kel-F 3-200 oil	45.7
MEK	274
MIBK	274
Toluol	182
Ultrox	87.3
	1000.0

Resistance to WFNA - maximum times -

44 days at 77°F.
10 days at 125°F.
1 day at 160°F.

The failure encountered has been blistering due to penetration and not degradation of the coating itself.

B. Filleting

1. Processing - as a putty, by either caulking or putty knife.

   a. Selection of the proper type X-200 resin was necessary:

      (1) The solution grade X-200 resins could not be reduced to small agglomerate size to produce a smooth putty. They produced a high viscosity putty that tended to flow upon application of moderate heat.

      (2) Many "off-grade" X-200 resins were checked for this work so as to pinpoint a putty grade resin.

   b. Solvents could not be used in a heavy filleted section, to do so would result in solvent entrapment, blistering and acid penetration.

   c. The putty would need deaeration during processing to produce a void free fillet.

2. Application of the putty.

   The putty is applied to the joints or seams and sprayed with the coating. It is then either air dried or baked. Upon baking, no solvents, air or low boiling plasticizers can be tolerated as resultant blistering occurs. Pigment concentration by volume needs be one-half that of the X-200 resin to prevent vertical sag upon baking.

3. Resistance of the filleting compound.

Baked putties from various types of X-200 resins resisted WFNA at 160°F. as follows:

Insoluble resins permitted penetration due to incomplete fusion. Soluble resins permitted excessive distortion due to low softening point. Moderately soluble resins would withstand WFNA for four days.

Unbaked putties would not withstand WFNA at 160°F. for even one day.

Zirconium silicate (Ultrox) was found to be the pigment most resistant to penetration by WFNA.

4. The maximum resistance thus far attained in a filleting compound is -

Compound 59-2

Kellogg "off spec" X-200 resin Batch J-4322	100
Kel-F 3-200 Oil	120
Ultrox	100

When deaerated and baked at 290°F. for one hour, this formulation will withstand WFNA at 4 days at 160°F.

III. Requirements satisfied.

The following requirements of this project have been satisfied:

A. The X-200 coating and filleting material provides in itself sufficient adhesion to aluminum to eliminate the use of an adhering media.

B. The X-200 resin is extremely resistant to degradation by WFNA.

C. The coating and fillet will resist normal foot traffic and flight vibrations.

D. The X-200 compounds can be applied by simple, conventional and practical methods.

IV RECOMMENDATIONS

I. The best coating formulation developed thus far has been: AF-77. This compares to other promising systems as follows:

WFNA RESISTANCE

	77°F.	125°F.	160°F.
AF-77 with X-200 resin	44 days	10 days	1 day
AF-43 with Vinyl:Aroclor	5 days	—	—
AF-19 Vinylidene Fluoride type	3 days	—	—

For filleting compounds, formulation 59-2 has shown the best resistance to WFNA. It lasted 4 days at 160°F. with no sign of failure. The closest competition is a Vistanex:Polyethylene tape - Compound 34 which showed penetration and some decomposition after 3 days at 120°F.

In the light of these results and the great number of other basic resins checked, the only known solution to the problem lies in Kellogg X-200 resin.

II. Through close work with Kellogg, we are endeavoring to establish specifications for both the coatings resin and filleting type material. These specifications will have to be established before any definite compounds can be recommended.

III. Our present plan of attack is as follows:

A. To establish the composition of the coating and filleting compound on the basis of new X-200 resin developments.

B. To modify and simplify processing and application of the protective coating.

C. To assume that X-200 resin is the only present hope of fulfilling the contract requirements:

Thus it is obvious that the only recommendation which can be made is to facilitate further development work on X-200 resin both as a raw material and as a compound.

V FUTURE WORK

I. Future Modifications

As a protective coating system reaches a final stage of development and final testing is considered, it will be necessary to vary the exposure testing procedures mainly to accommodate a complex metal test specimen. Simulated sections of aircraft will be made up in miniature in which loose and tight riveted butt and overlap joints are used as well as sharp corners and edges. Portions of this complex specimen will be filleted where necessary and over the entire area will be applied the protective coating. This entire test assembly then will be subjected to WFNA at elevated temperatures and under conditions which will simulate actual contact of the specimen with WFNA as well as 100% contact with the fumes of WFNA. This type of test specimen will require a heated, corrosion resistant chamber into which the mock-up section can be inserted.

II. Future Evaluation

To evaluate all lots of X-200 resin, preparatory to setting up a resin specification.

To develop a completely satisfactory drying cycle for the coating.

To remove air from the putty during the mixing cycle.

To test all resulting coating and filleting compounds in the following manner:

(1) Plain panel tests in which the protective coating is applied over the metal and filleting compound in place.

(2) Exposure of a simulated aircraft section to spillage and fumes of WFNA at elevated temperatures, the metal surfaces being protected by the coating applied over the filleting compound in butt and overlap joints.

(3) Subjecting identical sections as tested in (2), to the cycling tests which will involve alternate heating and cooling, followed by examination of the condition of the specimen to gain an idea of the amount of protection that the coating and filleting compound affords. This cycling test will consist only of the two temperature extremes and will not be subjected to vibration tests.

(4) Upon the successful conclusion of the above testing programs, specifications and materials will be prepared and submitted to WADC. It is then anticipated that field testing of the protective coating and filleting system will be conducted jointly between WADC and U. S. Stoneware Co.

(5) Manufacture of sufficient quantity of coating and filleting materials to be submitted to Materials Laboratory, WADC.

APPENDIX I

Test equipment, Design, Construction and Operation

Test equipment, material and supplies.

Several sheets of aluminum .080" thick and .25" thick were purchased and cut into suitable panels 2" x 6". Sheet of steel .25" were also purchased and prepared in like manner. All panels to be subsequently used for checking of coatings were reworked by deburring the edges and corners, degreasing and storing under solvent to prevent rust and oxidation.

A series of glass cylindrical testing tanks were prepared. During the construction of the testing facilities, 3/4" unplasticized, polyvinyl chloride sheets were purchased and machined to produce covers fitting over the testing tanks and so designed as to receive a glass ground reflux condenser. Stirring rods and motors were then vertically mounted on the cover to produce internal agitation and splashing.

Photograph No. 2 shows the testing vessel, the ceramic panel holder, the cover with reflux condenser and stirring rod in place within the large constant temperature tank which is internally heated to maintain the elevated temperatures. A large constant temperature tank was constructed to house the 7 testing units in the manner described above, and contents of the tank being heated by thermostatically controlled electric immersion heaters. By means of this tank, multiple test exposures can be conducted at any given temperature up to 175°F.

Since the handling of WFNA is hazardous, both to personnel, equipment and building, a separate building constructed of weatherproof masonite was erected at a distance from the laboratory to house and protect the corrosion testing equipment. This building was constructed to minimize any possibility of contamination or splash of WFNA. The floor was built of acid-proof brick directly on the ground. This building is approximately 5' x 8' x 7' high and is serviced with air, water, steam and electricity and ventilated at the top by a corrosion-proof fan. Equipment includes the following:

(a) A large heating tank, (Photograph No. 2), thermostatically controlled and containing water as a heat transfer medium.

(b) Circulation of the water by two electric stirrers to insure uniform distribution of the heat.

(c) An electric timing device included near the tank to indicate the number of hours the test has progressed.

(d) Each glass testing vessel has its separate motor and speed rheostat to create mixing and splashing within the jar.

(e) A glass reflux condenser is fitted into the top cover plate and flexible tubing conducts fumes from the top of the condenser to the outside of the building, there it is bubbled under a slight head of water pressure to absorb the fumes.

Safety equipment was purchased, including safety clothing, gloves and head protection. This equipment is used whenever it is necessary to add WFNA, to remove panels from the testing vessels which are in operation, or to remove panels for final examination. Due to the hazardous nature of WFNA, these precautions were taken in order to minimize accidents, burns, or toxicological reactions.

An adequate supply of WFNA was purchased and stored on the outside of the building. This material was removed by air pressure in a closed system. In addition, jet fuel was purchased for later tests.

A low temperature cabinet manufactured by the American Instrument Company, Catalogue No. 4-3352, was purchased and installed for future cycling tests on fabricated panels sections. This cabinet has not, at present, been used for it is impractical to run cycling tests on coatings or filleting materials which previously have not shown resistance to WFNA. Following the final development of both the coating and the filleting compound, this instrument will be used in the cycling tests, exposing the test section to alternate low and high temperatures, followed by examination of the film and fillet.

An adhesion tester was designed to evaluate the adhesion of the protective coating material to aluminum and steel. It later developed that this tester was impractical for use due to the thinness of the protective coatings developed and the soft putty-like nature of the filleting materials. For this reason, this equipment was not used in connection with this project.

Photographic equipment and accessories were purchased and set up in the laboratory dark room for use by members of the laboratory staff to record the results of the panel exposures, adhesion tests, condition of the films before and after exposure to WFNA and to make permanent record of other activities concerning the project.

During the early months of the project, some difficulty was encountered in procuring WFNA from the General Chemical Co. It had been our intention to purchase this material in small lots as required but they indicated they were no longer in a position to supply us. Testing of panels was considerably delayed in the beginning until a full drum of WFNA was received from WADC. This acid is still being used for the testing of coatings and filleting materials.

During a conference held at Dayton between the representatives of WADC, M. W. Kellogg Co. and U. S. Stoneware Co., various testing methods to evaluate the resistance of WFNA were discussed. Of those mentioned, the Pfaudler unit seemed to be most applicable to our work. As a result, two complete units were purchased and put into use. It was found, however, that the metal parts of these units will not withstand the corrosive conditions in the exposure apparatus due to the unavoidable fumes that are always present. In order to correct this at the time, additional units were purchased, we redesigned the Pfaudler unit, using stainless steel for the metal parts. Sufficient stainless steel, glass and Teflon gaskets were purchased to make up 6 complete units as shown in Photograph No. 1 and 32. The cost of these units, modified to withstand the corrosion, was practically the same as the original units and they are still in use.

Four "H" cells have been constructed for determining the rate of penetration of WFNA through unsupported films. These cells are the modified versions of the cells developed at the U. S. Naval Air Rocket Test Station. The joint between which the sample is clamped is made from two male ball and socket joints, Edges were lapped with carborundum so a perfect fit was obtained. This type of joint gives an area of 9.72 sq. in. for exposure of the acid and water. However, the H-cells made in this way have small area of gasket face and must be drawn up quite tight. This damages some of the softer specimens being tested. Some failures were caused by actually squeezing through the specimen as a result of clamping too tight. The H-cells were then remodeled and Corning Glass pipe was used for the joints to increase the area of contact between the cell halves. Teflon envelope gaskets were used between the joints and gaskets and in this manner a tight cell was obtained without damaging the specimen. The remodeled H-cells are shown in Photograph No. 7. Testing of unsupported thin films will continue when required, though it is found that more practical and direct results are obtained by applying the coating in question over a steel panel and subjecting the panel to WFNA noting the amount of corrosion and the condition of the film.

In order to evaluate the workability of the filleting compound, aluminum panels 24 ST3 were prepared to form a typical butt joint and an overlap joint, simulating conditions that would be met in aircraft construction. These panels had been used to evaluate the adhesion, lack of shrinkage, and amount of flow at elevated temperatures when the filleting compound was hand-pressed into place. These panels are also to be used in future exposure tests by applying the filleting compound followed by a suitable protective coating over the bare metal and the filleted area.

Test Methods

Exposure tests, Coatings

As explained previously, it had been planned to expose coated specimens in a glass tank at various temperature ranges. The problem of suitably protecting corners, edges and attachment holes in practically every case nullified the results of the test. As is usual in exposure tests, the amount of attack or the degradation of the coating adjacent to a corner or edge is disregarded, but in exposure tests involving WFNA, the underfilm corrosion is so great and so fast that other methods which would eliminate the edge and corner effect had to be resorted to. It is in this connection that the Pfaudler units proved their worth. Practically all of the exposure tests for the past year have been conducted using Pfaudler units as evidenced by the photographs of various test specimens. This type of test is strictly empirical and is difficult to duplicate as well as describe without the benefit of the actual sample or photographs. At best, photographs can only tell one part of the story and it would be helpful if some reasonably accurate method of measurement based on the results of exposure tests could be developed. Specific details as to the results of exposure tests are described under Protective Coatings Development.

Exposure tests, Filleting compounds

In testing a soft, putty-like material, the usual test panels cannot be relied upon. It is difficult to apply a soft, putty material uniformly over a panel area without mechanical discontinuity. It is for this reason that exposure tests on putties were conducted in the Pfaudler unit, realizing that some leakage would take place since it is impossible to draw up the unit tightly without completely destroying the filleting film. More practical tests will be used involving butt and lap joints when the coating is applied over the metal as well as the filleted area. It is to be noted that we are not relying on 100% resistance of the filleting material to WFNA. It is more important that the exposure tests indicate the proper functioning of the filleting compound as a fillet, permitting the applied topcoat to provide the necessary protection. We have, however, exposed filleting compounds to various temperatures and found that when the filleting compound itself is properly mixed and dispersed, and is free from minute air bubbles, it affords a high order of resistance to penetration by both liquid and fumes of WFNA.

TABLE I

GENERAL RESUME OF MATERIALS TESTED FOR HNO_3 RESISTANCE

	Material	Test	Time	Results
1.	Hypalon S-2 compound	Conc. HNO_3 260°F.	30 min.	Disintegrated
2.	Vistanex:Al_2O_3	" " "	"	Swelling
3.	50:50 Comb. of 1 and 2	" " "	"	Swelling, blistered
4.	Vistanex:Aroclor:Carbon black	" " "	"	Hard, brittle, otherwise good
5.	Vistanex:Polyethylene:Al_2O_3	" " "	"	Slight swelling
6.	Vistanex:Aroclor:Al_2O_3	" " "	"	Great swelling, Complete failure
7.	Vistanex:Polyethylene:Ultrox	" " "	"	Slight swelling
		FNA 77°F.	52 hrs.	O.K.
8.	Vistanex:Ultrox	Conc. HNO_3 260°F.	30 min.	Swelled, spongy
9.	Vistanex:Aroclor:Ultrox	" " "	"	Great swelling, Complete failure
10.	Vistanex:Halocarbon oil: Ultrox	" " 160°F.	"	Swelled, spongy
		FNA 77°F.	52 hrs.	Softened, swelled-failure
11.	Vistanex:Halocarbon wax: Ultrox	Conc. HNO_3 160°F.	30 min.	Swelled slightly
		FNA 77°F.	52 hrs.	Penetration-otherwise O.K.
12.	Vistanex:Epoxy:Ultrox	Conc. HNO_3 160°F.	30 min.	Crumbled
13.	Vistanex:Parlon:Ultrox	" " "	"	Weak - crumbly
14.	Vistanex:Advagum 1098:Ultrox	" " "	"	Spongy, blisters
15.	Vistanex:Advawax 2575:Ultrox	" " "	"	Spongy, blistered
16.	Vistanex:Advawax 2080:Ultrox	" " "	"	Spongy
17.	Vistanex:Chlorowax 70:Ultrox	" " "	"	Slight surface attack
		FNA 77°F.	30-1/2 hrs.	O.K.
18.	Hypalon S-2 compound	Conc. HNO_3 160°F.	30 min.	Very sl. surface attack
		FNA 77°F.	3-1/2 hrs.	Blistered
		" 125°F.	1 hr.	Dissolved

TABLE I (Cont'd.)

	Material	Test			Time	Results
19.	Vistanex:Exon 400XR61	Conc.	HNO_3	250°F.	30 Min.	Spongy
		"		160°F.	30 min.	Hardening
20.	Vistanex:Chlorowax 70:Ultrox	"	"	250°F.	"	Spongy
		"	"	160°F.	"	"
21.	Vistanex:Chlorowax 40:Ultrox	"	"	"	"	"
22.	Vistanex:Chlorowax 40	"	"	250°F.	"	Sl. swelling
		"	"	160°F.	"	No change
23.	Vistanex:Polybutene H-35	"	"	250°F.	15 min.	Swelling, loss of shape
		"	"	160°F.	30 min.	Little change
24.	Vistanex:Polybutene H-200	"	"	250°F.	20 min.	Swelling
		"	"	160°F.	30 min.	Little change
25.	Vistanex:Exon 400XR61:Chlorowax 40	"	"	250°F.	20 min.	Sl. attack Mod. distortion
		"	"	160°F.	30 min.	Little change
26.	Vistanex:Exon 400XR61:Halocarbon	"	"	250°F.	30 min.	Blisters,porous
27.	Vistanex:Exon 400XR61:Halocarbon Oil	"	"	250°F.	"	Blisters
28.	Vistanex:Carbon black	WFNA at 25°C.				Penetration at 45 hrs.
29.	Vistanex:Heat treated Carbon black	"	"	"		Penetration at 72 hrs.
30.	Vistanex:HiSil	"	"	"		Degraded at 72 hrs.
31.	Vistanex:Chlorowax 40	"	"	"		Degraded at 195 hrs.
32.	Vistanex	"	"	"		Degraded at 195 hrs.
33.	Vistanex:Polyethylene	"	"	"		No sign of failure at 96 hrs.
	Vistanex:Polyethylene	"	"	120°F.		White and blistered at 48 hrs. (See Photo No. 30)
	Vistanex:Polyethylene	Navy "H" Cell				0.2 g. WFNA/sq. meter in 140 hrs.

TABLE I (Cont'd.)

Material	Test	Results
34. Vistanex	WFNA at 125°F.	Swelling - 6 days (See Photo No. 29)
35. Polyethylene	" " "	Cracked, brittle- 6 days (See Photo No. 29)
36. Vinyl chloride	" " "	Cracked, brittle- 6 days (See Photo No. 29)
37. Hypalon C-2	" " "	Surface attack- 6 days (See Photo No. 29)
38. Hypalon S-2 compound	" " "	Complete failure- 2 hrs. (See Photo No. 28)
39. Vinsol	" " "	Complete failure 5 days
40. Exon 400 XR61	" " "	Blistered, brittle- 2 days
41. Marbon 9200	" " "	Dissolved - 4 days
42. Advawax 2080	" " "	Dissolved - 4 days (See Photo No. 28)
43. Chlor-isopol	" " "	Dissolved - 4 days (See Photo No. 28)
44. Kellogg X-200	" " "	No change - 6 days
45. Parlon	" " "	Surface attack - 5 days (See Photo No. 27)
46. Saran F-120	" " "	Complete failure- 5 days
47. Paraffin wax	" " "	Melted
48. Epoxy AN-501	" " "	Complete failure- 1 day

TABLE I (Cont'd.)

	Material	Test	Results
49.	Velsicol AB-11-2	WFNA at 125°F.	Complete failure
50.	Indopol H-300	" " "	Complete failure 1 day
51.	Halocarbon oil	" " "	O.K. - 5 days
52.	Chlorowax 40	" " "	Complete failure- 4 days
53.	Chlorowax 70	" " "	Decomposed - 4 days
54.	Vistac 1	" " "	Decomposed - 2 days
55.	Aroclor 5460	" " "	Decomposed - 2 days
56.	Aroclor 1260	" " "	Decomposed - 2 days
57.	Aroclor 1254	" " "	Decomposed - 2 days
58.	Chloroparaffin 40-4004	" " "	Decomposed - 2 days
59.	Methyl Pentachlor Stearate	" " "	Decomposed - 1 day
60.	o-dichlorobenzene	" " "	Decomposed - 1 day
61.	Perchloroethylene	" " "	V. Sl. residual matter - 3 days
62.	Trichloroethylene	" " "	V. Sl. residual matter - 3 days
63.	Dichloropentanes	" " "	Some residual matter - 3 days
64.	Nujol	" " "	Decomposed - 1 day
65.	Hexachlorobutadiene	" " "	Decomposed - 5 hrs.

TABLE I (Cont'd.)

Material	Test	Results
66. Saran A	WFNA at 125°F.	Decomposed - 1 day
67. Hexachloropropane	" " "	V. Sl. residue - 5 days
68. Kel-F 1-3 oil	" " "	O.K. - 6 days
69. Kel-F 150 wax	" " "	O.K. - 6 days
70. Kel-F Plast. grade oil	" " "	O.K. - 6 days

TABLE II
SOLVENT SYSTEM CHARACTERISTICS OF EXON 402, HYPALON C-2 AND S-2

MATERIALS	AF-20	AF-21	AF-22	AF-32
Exon 402	5	5	5	10
THF	95		47.5	45
MEK		47.5		
Cyclohexanone		47.5	47.5	45
Viscosity Gardner	<A		<A	D
Clarity	Clear	Hazy	Clear	Clear

MATERIALS	AF-23
Hypalon S-2	20
Toluene	80
Viscosity Gardner	V
Clarity	Clear

MATERIALS	AF-24	AF-61A	AF-61B	AF-61C	AF-61D	AF-61E	AF-61F	AF-62A	AF-62B	AF-62C
Hypalon C-2	20	20	20	20	20	20	20	20	20	20
Toluene	80							28	40	28
DiMethyl Formamide		80		24	32			24		24
THF			80		48					
Xylol				56				28		
MEK						80				
MIBK							80		40	28
Solvesso 100										
Viscosity Gardner	W	S	M	T-U	S-T	S	M	T	W	T-U
Clarity	Brown	Clear	Sl.Hazy	Clear	Clear	Hazy	Hazy			

TABLE II (Cont'd.)

SOLVENT SYSTEM CHARACTERISTICS OF EXON 402, HYPALON C-2 and S-2

MATERIALS	AF-62D	AF-62E	AF-62F	AF-63A	AF-63B
Hypalon C-2	20	20	20	20	20
Toluene			40		
DiMethyl Formamide	24				
THF					
Xylol	28	80	40		40
MEK					
MIBK	28				40
Solvesso 100				80	
Viscosity Gardner	T	X	X-Y	Z	V
Clarity					

TABLE III

SOLVENT SYSTEM CHARACTERISTICS OF VISTANEX, VYDR AND HYPALON C-1

	AF-25	AF-26	AF-27	AF-28	AF-30
Vistanex B-100	5	5	5	5	5
Dipentene	95		47.5	24	
Toluene		95	47.5	71	71
Solvesso 10C					24
Viscosity-Gardner	N	E	I	G	—

	AF-29
Hypalon C-1	20
Toluene	80

Remarks: Viscous, very cloudy

	AF-41A	AF-41B	AF-41C	AF-41D	AF-42E	AF-42F	AF-42G	AF-42H
VYDR	7.5	7.5	7.5	9.75	7.5	7.5	9	9.75
Chlorowax 40	7.5		3.75	5.25	5	2.5	4.5	3.75
Chlorowax 70		7.5	3.75		2.5	5	1.5	1.5
MEK	42.5	42.5	42.5	42.5	42.5	42.5	42.5	42.5
Cyclohexanone	42.5	42.5	42.5	42.5	42.5	42.5	42.5	42.5

TABLE IV
SOLUBILITY OF KELLOGG X-200 RESIN IN VARIOUS SOLVENTS

Materials	AF-34	AF-35	AF-36A	AF-36B	AF-36C	AF-36D	AF-36E	AF-37A	AF-37B
Resin X-200									
Batch 911	20	20							
Batch (912									
(913									
Blend (914			15	15	15	15	15	15	15
(915									
(919									
(920									
Batch 1019									
Batch 979									
" J-4006									
MEK	40		85						
Cyclohexanone	40	28						85	
THF		52							
Nitromethane				85	40				
Nitropropane					45	85			
Butyl Acetate							85		
Isophorone									85
Ethyl Butyl Ketone									
Toluol									
Xylol									
Cellosolve Acetate									
Isopropyl Benzoate									
Dipentene									
Acetophenone									
Ethyl Acetate									
Dimethyl Formamide									
HiFlash Naphtha									
Tetralin									
Butanol									
Viscosity-Gardner	Q-R	U	A	Ins.	Ins.	Ins.	G	P	Ins.
Solubility	Hazy Lg. Particles	Hazy Lg. Part.	Hazy Lg. Part.				V.Hazy Lg. Part.	Hazy Lg. Part.	
Color	W.White	Straw	W.White						

TABLE IV (Cont'd.)

SOLUBILITY OF KELLOGG X-200 RESIN IN VARIOUS SOLVENTS

Materials	AF-37C	AF-37D	AF-37E	AF-37F	AF-38A	AF-38B	AF-38C	AF-38D	AF-38E
Resin X-200									
Batch 911									
" (912									
(913									
Blend (914	15	15	15	15	15	15	15	15	
(915									
(919									
(920									
Batch 1019									15
Batch 979									
" J-4006									
MEK									
Cyclohexanone									
THF									
Nitromethane									85
Nitropropane									
Butyl Acetate									
Isophorone									
Ethyl Butyl Ketone	85								
Toluol									
Xylol		85							
Cellosolve Acetate			85						
Isopropyl Benzoate				85					
Dipentene					85				
Acetophenone						85			
Ethyl Acetate								85	
Dimethyl Formamide									
HiFlash Naphtha									
Tetraline							51		
Butanol							34		
Viscosity-Gardner	E	Ins.	Ins.	Ins.	Ins.	Ins.	Ins.	A	A
Solubility	Hazy							Hazy	Clear Sl.insol. part.
Color								W.White	Amber

TABLE IV (Cont'd.)
SOLUBILITY OF KELLOGG X-200 RESIN IN VARIOUS SOLVENTS

Materials	AF-38F	AF-39A	AF-44A	AF-44B	AF-44C	AF-44D	AF-46A	AF-46B	AF-46C
Resin X-200									
Batch 911									
" (912									
(913									
Blend (914									
(915									
(919									
(920									
Batch 1019	20	20							
" 979			20	20	20	20			
" J-4006							20	20	20
MEK		80		8	16				
Cyclohexanone									
THF	80		64	56	56	56	64	72	
Nitromethane									
Nitropropane									
Butyl Acetate									
Isophorone									
Ethyl Butyl Ketone			16	16	8	24			
Toluol									
Xylol									
Cellosolve Acetate									
Isopropyl Benzoate									
Dipentene									
Acetophenone									
Ethyl Acetate									
Dimethyl Formamide							16	8	80
HiFlash Naphtha									
Tetralin									
Butanol									
Viscosity-Gardner	A		B	C-D	B	C-D			
Solubility	Clear Sl.Insol. Part.		Clear	Sl.haze	Clear	V.Sl.haze			
Color	Amber								

TABLE IV (Cont'd.)
SOLUBILITY OF KELLOGG X-200 RESIN IN VARIOUS SOLVENTS

Materials	AF-46D	AF-47A	AF-47B	AF-47C	AF-47D	AF-47E	AF-48A	AF-48B
Resin X-200								
Batch 9o1								
" (912								
(913								
Blend (914								
(915								
(919								
(920								
Batch 1019								
" 979								
" J-4006	20	20	20	20	20	20	20	20
MEK								
Cyclohexanone								
THF	80	56	60	60	60	40	64	60
Nitromethane								
Nitropropane								
Butyl Acetate								
Isophorone								
Ethyl Butyl Ketone								
Toluol				20		10		
Xylol			20				16	12
Cellosolve Acetate								
Isopropyl Benzoate								
Dipentene								
Acetophenone								
Ethyl Acetate								
Dimethyl Formamide		24						8
HiFlash Naphtha					20	30		
Tetralin								
Butanol								
Viscosity-Gardner								
Solubility	Clear Sl.Gel Sl.Insol.	Sl.Cloudy	Clear	Clear Some insol.	Clear Some Insol.	Swollen but undis-solved resin	Clear	Clear Some In-Sol.
Color								

TABLE IV (Cont'd.)
SOLUBILITY OF KELLOGG X-200 RESIN IN VARIOUS SOLVENTS

Materials	AF-48C	AF-48D	AF-48E
Resin X-200			
Batch 911			
" (912			
(913			
Blend (914			
(915			
(919			
(920			
Batch 1019			
" 979			
" J-4006	20	20	20
MEK			
Cyclohexanone		8	
THF	60	60	52
Nitromethane			
Nitropropane			
Butyl Acetate			
Isophorone			
Ethyl Butyl Ketone	8		
Toluol			
Xylol	12	12	20
Cellosolve Acetate			
Isopropyl Benzoate			
Dipentene			
Acetophenone			
Ethyl Acetate			
Dimethyl Formamide			8
HiFlash Naphtha			
Tetralin			
Butanol			
Viscosity-Gardner			
Solubility	Hazy	Clear Some Insol.	Clear but undissolved particles
Color			

TABLE IV (Cont'd.)

SOLUBILITY OF KELLOGG X-200 RESINS IN VARIOUS SOLVENTS

Materials:	AF-49A	AF-49B	AF-49C	AF-49D	AF-49E	AF-50A	AF-50B	AF-50C	AF-50D	AF-51A	AF-51B
Resin X-200:											
Batch J-4006	20	20	20	20	20	20	20	20	20	20	20
" J-4587											
Cyclohexanone	8	8				12				8	12
THF	68	64	64	68	64	64	64	64	68	68	64
Xylol	4	8	8	4	4	4	8	4	4		
Ethyl Butyl Ketone			8	8	12						
DiMethyl Formamide							8	12	8		
Toluol										4	4
Dioxane											
Cellosolve Solvent											
Diethoxytetrahydrofuran											
Methyl cellosolve											
Butyl cellosolve											
MIBK											
MEK											
Viscosity Gardner											
Solubility	Clear V.Good	Clear V.Good	Clear V.Good	Clear V.Good	Clear V.Good	Clear Sl.insol.	Clear V.Good	Clear	Clear	Clear Sl. insol.	Clear Sl. insol.

TABLE IV (Cont'd.)

SOLUBILITY OF KELIOGG X-200 RESINS IN VARIOUS SOLVENTS

Materials:	AF-51C	AF-51D	AF-51E	AF-52A	AF-52B	AF-52C	AF-53A	AF-53B	AF-53C	AF-54A	AF-53D
Resin X-200:											
Batch J-4006	20	20	20	20	5	20	20	20	20	20	20
Batch J-4587											
Cyclohexanone											1
THF		64				68	68	68		68	68
Xylol											
Ethyl Butyl Ketone											
DiMethyl Formamide											
Toluol											
Dioxane	80										
Cellosolve Solvent		16	80								
Diethoxytetrahydrofuran				80	95	12					
Methyl cellosolve							12				
Butyl cellosolve								12			
MIBK									80	11	12
MEK											
Viscosity Gardner											
Solubility	Ins.	Ins.	Ins.	Ins.	Ins.	Sl.Ins.	Ins.	Ins.	Ins.	Ins.	Sl.Ins.

TABLE IV (Cont'd.)

SOLUBILITY OF KELLOGG X-200 RESINS IN VARIOUS SOLVENTS

Materials:	AF-54B	AF-54C	AF-55A	AF-55B	AF-55C	AF-55D	AF-55E	AF-64A	AF-64B	AF-64C
Resin X-200:										
Batch J-4006	20	20	20	20	20	20	20			
Batch J-4587								20	20	20
Cyclohexanone										
THF	68	64	68	64	68	68	64			
Xylol										
Ethyl Butyl Ketone		4	8	12	8	4	8			
DiMethyl Formamide										
Toluol		4	4	4		4				
Dioxane										
Cellosolve Solvent										
Diethoxytetrahydrofuran	1									
Methyl Cellosolve										
Butyl Cellosolve										
MIBK	11	4			4	4	4		40	20
MEK		4					4	80	40	60
Viscosity Garnder								U-V	U-V	U-V
Solubility	Sl.Ins.	Clear	Sl.Ins.	Sl.Ins.	Ins.	Sl.Ins.	Sl.Ins.	Clear	Clear	Clear

TABLE IV (Cont'd.)

SOLUBILITY OF KELLOGG X-200 RESINS IN VARIOUS SOLVENTS

Materials:	AF-64D	AF-64E	AF-65
Resin X-200:			
Batch J-4006			
Batch J-4587	20	20	20
Cyclohexanone			
THF			
Xylol			
Ethyl Butyl Ketone			
DiMethyl Formamide			
Toluol			29
Dioxane			
Cellosolve Solvent			
Diethyoxytretrahydrofuran			
Methyl Cellosolve			
Butyl Cellosolve			
'IBK	60	80	80
EK	20		
Viscosity Gardner	R-S	Q	H
Solbility	Clear	Clear	Clear

TABLE V
COATING SYSTEMS

	FORMULATION			COATING					EXPOSURE		
Form No.			Method	No. Coats	Drying	°F. Temp.	Hrs. Time	Nitric Acid	°F. Temp.	Hrs. Time	Type Failure
AF-1	Parlon (10cps)	99	Slush	1	Force	200	2-1/2	70%	77	50-120	Wrinkled
	Parlon(1000 cps)	1									
	Aroclor 5460	40									
	Aroclor 1254	60									
	Xylol	200	Dip	4	Force	200	2-1/2	70%	175	1/2	Brittle
	Ferro 900	5									
			Note:	Not resistant with Aroclors							
AF-2	Pylene PC-7	50									
	MIBK	37.5	Dip	10	Force	350	2	70%	77	96	Decomposed
	Xylol	37.5									
AF-3P	Parlon(1000 cps)	60									
	Aroclor 1254	60									
	Ferro 900	3									
	Milled Zircon	108									
	Ultrox	12									
	Xylol	200									
			Note:	Not resistant							
AF-4P	Gilsonite	37.5									
	Parlon (1000cps)	37.5	Spray	6	Force	250	1				
	Ferro 900	1.8									
	Aroclor 1254	75									
	Milled Zircon	135									
	Ultrox	15									
	Xylol	150									
	Toluene	220									
			Note:	Not resistant							
AF-5	RBH Resin 569	42									
	Aroclor 1254	28	Spray	Multiple	Force	325	1/2	70%	77	17	Darkened
	Xylol	105									

TABLE V (Cont'd.)
COATING SYSTEMS

	FORMULATION		COATING						EXPOSURE		
Form No.			Method	No. Coats	Drying	°F. Temp.	Hrs. Time	Nitric Acid	°F. Temp.	Hrs. Time	Type Failure
AF-6B	Marbon 9200	36									
	Aroclor 1254	24									
	Xylol	70	Spray	Multiple	Force	200	1/2	Fuming	77	1/2	Wrinkled
	Toluol	70						Fuming	77	3	Consumed
AF-7	Marbon 9200	15-45	Spray	Multiple	Force	200	1/2	70%	77	24	Too brittle
	Chlorowax 70	45-15									No decomp.
	Xylol	70									
	Toluol	70									
AF-8	Exon 400XR61	108									
	Xylol	276	Spray	Multiple	Force	325	1	Fuming	77	1-1/2	No effect
	Dryolene	162						Fuming	77	27	Penetration
	Ethyl acetate	42									
	Butyl alcohol	12									
			Note:	Best of resins so far							
AF-9A	RBH Resin 569	85									
	Aroclor 1254	57									
	Milled Zircon	135	Spray	5	Force	325	1-1/2	70%	77	96	No effect
	Ultrox	15									Discoloration
	Xylol	165									
AF-11 Primer	VYHH	11									
	VYDR	53									
	VMCH	67.2									
	Blue Lead	57.5									
	Kronitex AA	24									
	Cyclohexanone	238									
	DOP	5.2									
	MEK	238									
	MIBK	170.4									
	Toluene	134.4									

TABLE V (Cont'd.)
COATING SYSTEMS

	FORMULATION		COATING						EXPOSURE		
Form No.			Method	No. Coats	Drying	°F. Temp.	Hrs. Time	Nitric Acid	°F. Temp.	Hrs. Time	Type Failure
AF-12	VYDR MEK Cyclohexanone Dyphos	22.4 151 151 2.5	Spray on AF-11 Base	16	Force	210	1/2	Fuming	125	28	Cracked Brittle
			Note: Too brittle-needs plasticizer								
AF-13	VYDR Milled Zircon Ultrox TiO_2 Dyphos MEK Cyclohexanone	80 64 8 8 4 388 388	Spray on AF-11 base	6	Force between coats	200	1/2	Fuming	125	28	Cracked brittle
			Note: Too brittle-needs plasticizer								
AF-14	Parlon (20 cp) Primer RN-34 (Epon) Red Lead Rezyl 869 5X asbestine Xylol	133 6.3 1420 267 80 800									
AF-15	Parlon (20 cp) RN-34 Chlorowax 70 Aroclor 1254 Xylol	100 5 30 40 440	Spray on AF-14 base	5	Force between coats	300	1/4	Fuming	125	28	All consumed
			Note: RN-34 epoxy decomposes								
AF-16A Primer	RBH Resin 569 Aroclor 1254 Milled Zircon Ultrox Powdered Graphite Xylol	180 120 200 25 39 600	Spray	15	Force	250-300	1/4	WFNA	125	28	Consumed
			Note: RBH resin not resistant								

TABLE V (Cont'd.)
COATING SYSTEMS

Form No.	FORMULATION		COATING Method	COATING No. Coats	COATING Drying	°F. Temp.	Hrs. Time	Nitric Acid	EXPOSURE °F. Temp.	EXPOSURE Hrs. Time	Type Failure
AF-17	Exon 400XR61 Aroclor 5460 Aroclor 1254 Xylol Dryolene Ethyl acetate Butyl alcohol Stainless steel flake	75 37.5 37.5 251 148 40 11 146	Spray on AF-33 primer	9	Force between coats	200	1	WFNA	125	24	Decomposition
			Note:	Aroclors not too resistant							
AF-18B	VYDR Aroclor 1254 Stainless steel flake MEK Cyclohexanone	31.2 31.2 115 207 207	Spray on AF-16 primer	7	Force between coats	200	1/2	WFNA	77	44	No decomposition Permeable Destroyed primer
			AF-33 primer	7	Force between coats	200	1/2	WFNA	77	44	Destroyed primer Brittle
AF-19C	Exon 400XR61 Chlorowax 70 Chlorafin 40 Xylol Dryolene Ethyl acetate Butanol	75 50 25 251 148 40 11	Spray on AF-33 Primer	10	Force between coats	200-300	1	WFNA	125	24	Blistered Permeable
			Note:	Chlorowaxes more resistant than Aroclors							
AF-18A	VYDR Aroclor 1254 MEK Cyclohexanone	75 75 425 425	Spray over Tygonite primer Alodized	24	Force between coats	250	1/2	WFNA	77	70	Permeable Destroyed primer

TABLE V (Cont'd.)
COATING SYSTEMS

	FORMULATION			COATING					EXPOSURE		
Form No.			Method	No. Coats	Drying	°F. Temp.	Hrs. Time	Nitric Acid	°F. Temp.	Hrs. Time	Type Failure
AF-18C	VYDR Aroclor 1254 Stainless steel flake C-3000 MICA MEK Cyclohexanone	31.2 31.2 115 57 207 207	Spray on AF-16A 2 coats and AF-18B 1 coat	7	Force between coats	200	1/2	WFNA	77	44	Penetration
AF-16B Primer	RBH Resin 569 Aroclor 1254 Milled Zircon Ultrox 620 Powd.graphite Xylol	225 75 200 25 39 550									
			Note: Good adhesion to aluminum								
AF-17 BP	Exon 400 XR-61 Aroclor 5460 Aroclor 1254 Stainless St.Fl. Xylol Dryolene Ethyl acetate Butyl alcohol	75 37.5 37.5 146 251 148 40 11	Spray on AF-33 primer	9	Force between coats	Up to 300°F.	3-1/2	WFNA	77	44	Permeable Decomposed
AF-19 CP	Exon 400XR61 Chlorowax 70 Chlorafin 40 Xylol Dryolene Ethyl acetate Butanol Stainless St.Fl.	75 50 25 251 148 40 11 146	Spray on AF-33 primer	10	Force between coats	Up to 300°F.	3-1/2	WFNA	77	44	Permeable Blistered
			Note: Better than 17 BP								

TABLE V (Cont'd.)
COATING SYSTEMS

	FORMULATIONS			COATING					EXPOSURE		
Form No.			Method	No. Coats	Drying	°F. Temp.	Hrs. Time	Nitric Acid	°F. Temp.	Hrs. Time	Type Failure
AF-40	VYDR	31	Spray	10	Force between coats	250	1/2	WFNA	77	70	Permeable Embrittled
	Aroclor 1254	31									
	MEK	206									
	Cyclohexanone	206									
	Duriron	115									
			Note: Duriron better than stainless steel flake								
AF-43	VYDR	65.6	Spray	10	Force between coats	250	1/2	WFNA	77	70	Permeable Embrittled
	Aroclor 1254	65.6									
	MEK	439.5									
	Cyclohexanone	439.5									
	Duriron	250									
AF-55E	Resin X200 (J-4006)	20	Spray	10	Force between coats	300	1/2	WFNA	77	168	Penetration No attack
	THF	64									
	MEK	4									
	MIBK	4									
	EBK	8									
AF-66	Hypalon C-2	20	Spray	6	Force between coats	250	1/2	WFNA	77	2	OK
	MIBK	56.5									
	Toluol	56.5	Spray	15	Air dry			WFNA	125	3	Destroyed
AF-68	Hypalon C-2	73.7	Spray	8	Air dry			WFNA	77	3	Penetration
	MIBK	208									
	Toluol	208	Spray	12	Air dry		1/4 hr.per coat	WFNA	77	5	Penetration
	8485 Graphite	28									
AF-70	Resin X-200 (J-4387)	40	Spray	3	Force	175	1/2	Blistered on baking			
	MIBK	160	Spray	4-8	Force	200	1/2	Blistered on baking			
	Halocarbon oil 11-14	10.3									

TABLE V (Cont'd.)
COATINGS SYSTEMS

			COATING						EXPOSURE		
Form No.	FORMULATIONS		Method	No. Coats	Drying	°F. Temp.	Hrs. Time	Nitric Acid	°F. Temp.	Hrs. Time	Type Failure
AF-73	Resin X-200 (J-4387) MIBK Bassichi Pwdr. glass Halocarbon oil 11-14	100 400 3.5 5.15	Spray	7	Force between coats	300	1/2	WFNA	77	4	Penetration
AF-74	Resin X-200 (J-4387) Halocarbon oil 11-14 MIBK Ultrox	100 5.15 400 3.5	Spray	20	Force between coats	215	1/2	WFNA	77	48	Blistered
AF-76	Resin X-200 (J-4545 Plasticizer oil MEK MIBK Toluol Ultrox	266 88.9 533 533 355 341									
AF-76A	Same as AF-76		Spray	6	Force between coats	250 (Gradually increase bake to 300°F. in 6 days)	1/2		(Blisters)		
AF-76B	Same as AF-76		Spray	18	Force between coats		2/3	WFNA	125	67	Water leakage Cracking
AF-76C	Same as AF-76		Spray	3	Force between coats	250	2/3	(Blisters)			
AF-76D	Same as AF-76		Spray	4	Force between coats	250	5/6	(Blisters)			

TABLE V (Cont'd.)
COATING SYSTEMS

	FORMULATION		COATING						EXPOSURE		
Form No.			Method	No. Coats	Drying	°F. Temp.	Hrs. Time	Nitric Acid	°F. Temp.	Hrs. Time	Type Failure
AF-76E	Same as AF-76		Spray	20	Air dry between coats	Increase temp. to 300°F. over 5 days		WFNA	77	288	Penetration
AF-76F	Same as AF-76		Spray	20	Air	77	504	WFNA	77	696	Seems OK
			Note:	Breaking at compression points							
AF-77	Resin X-200 (J-4613) Plasticizer oil MEK MIBK Toluol Ultrox	266 88.9 533 533 355 170	Spray	20	Air dry-10th coat Force dry up to 300°F. over 2-1/2 days			WFNA	125	96	Blisters
AF-77G	Same as AF-77		Spray	18	Increase temp. to 300°F. over 2-1/2 days			WFNA	77	552	Blisters
			Note:	Long gradual bake cycle - reduces permeability							
AF-77H	Same as AF-77		Spray	20	Increase temp. to 300°F. over 2-1/2 days			WFNA	77	768	Looks OK
AF-77I	Same as AF-77 with reduced Ultrox		Spray	20	Overnight air dry on 7th to 15th coats			WFNA	77	144	Small blisters
AF-75	Resin X-200 (J-4613 Plasticizer oil MEK MIBK Toluene	15 5 30 30 20	Spray	19	90 hrs. air dry 70 hrs.-increase temp. to 300°F.			(Blistered)			

TABLE V (Cont'd.)
COATING SYSTEMS

				COATING					EXPOSURE		
Form No.	FORMULATION		Method	No. Coats	Drying	°F. Temp.	Hrs. Time	Nitric Acid	°F. Temp.	Hrs. Time	Type Failure
AF-75AA	Same as AF-75		Spray	27	Air dry - 19 days			WFNA	77	672	Vapor-complete failure Liquid-small blisters
AF-75BB	Alternate coats AF-77 - AF-75		Spray	24	Air dry - 19 days			WFNA	77	1060	Small blisters
			Note:	No apparent penetration							
AF-77K	Same as AF-77		Spray	20	Air dry - 9th coat force dry up to 300°F. over 2-1/2 days			WFNA	160	16	Large blisters
AF-77 Set 1	Same as AF-77		Spray	20	Air dry - 3 weks.			WFNA	125	168	Large blisters
AF-77 Set 2	Same as AF-77		Spray	20	Air dry - 2 weks.			WFNA	125	240	Large blisters
AF-77 Set 3	Same as AF-77		Spray	20	Force dry - up to 300°F. over 2-1/2 days			WFNA	125	96	Large blisters
AF-77 Set 4	Same as AF-77		Spray	20	Force dry - up to 300°F. over 2-1/2 days			WFNA	77	984	Blisters
AF-79A	Resin X-200 (J-4652	40	Spray	27	Air	77°		1/4hr.WFNA between coats	77	72	Blisters
	MEK	60	Note:	No apparent penetration.							
	MIBK	60									
	Toluene	40	Spray	27	Force	Up to 300°F.		85 hrs.WFNA	77	1032	Blisters
			Note:	No apparent penetration.							

TABLE V (Cont'd.)
COATING SYSTEMS

	FORMULATION		COATING						EXPOSURES		
Form No.			Method	No. Coats	Drying	°F. Temp.	Hrs. Time	Nitric Acid	°F. Temp.	Hrs. Time	Type Failure
AF-79B	Resin X-200 (J-4652) TR wax MEK MIBK Toluene	34 6 60 60 40	Spray	27	Air	77	1/4 between coats	WFNA	77	240	Blisters
			Spray	27	Force	Up to 300°F.	85	WFNA	77	2520	Blisters
			Notes:	No apparent penetration							
AF-79C	Resin X-200 (J-4652) TR Wax MEK MIBK Toluene	32 8 60 60 40	Spray	27	Air	77	1/4 between coats	WFNA	77	96	Blisters
			Spray	27	Force	Up to 300°F.	85	WFNA	77	2520	Blisters
			Notes:	No apparent penetration							
AF-33 Primer	G.E. R-108 Milled Zircon Ultrox Titanox R-610 Butvar Butahol	434 240 30 30 20 407									

TABLE VI
FILLETING SYSTEMS

COMPD. NO.			METHOD	°F. TEMP.	CURING	Min. TIME	°F. TEMP.	PSI PRESS.	NITRIC ACID	°F. TEMP.	HRS. TIME	TYPE FAILURE
1	Hypalon S-2 Al_2O_3(325 mesh) Trimal Wood Rosin WG Captax	100 100 37.5 10 3	Milled	Cold	Press	30	320	1000	70%	260	1/2	Destroyed
2	Vistanex B-120 Al_2O_3	100 200	Milled	Cold					70%	260	1/2	Sl.Swelling Turned white
2A	50/50 Mix of 1A and 1B				Press	30	320	1000	70%	260	1/2	Swelled Blistered
3	Vistanex B-120 Aroclor 1260 Micronex W-6	100 50 200	Milled	Cold	Press	5	320	1000	70%	250	1/2	V. Sticky Nonporous No swelling
4	Vistanex B-120 Polyethylene DYNJ Al_2O_3	100 100 400	Milled	250	Press	5	320	1000	70%	250	1/2	Spongy
5	Vistanex B-120 Aroclor 1260 Al_2O_3	100 50 300	Milled	Cold	Press	5	320	1000	70%	250	1/2	High porous Bad swelling and deformation
6	Vistanex B-120 Polyethylene DYNJ Ultrox	100 100 400	Milled	250	Press	5	320	1000	70%	250	1/2	Spongy-Sl. blisters-pockmarks
7	Vistanex B-120 Ultrox	100 200	Milled	Cold	Press	5	320	1000	70%	250	1/2	Pockmarks Sl. spongy
8	Vistanex B-120 Aroclor 1250 Ultrox	100 50 300	Milled	Cold	Press	5	320	1000	70%	250	1/2	Like No. 5 Failure

TABLE VI (Cont'd.)
FILLETING SYSTEMS

COMPD. NO.			METHOD	°F. TEMP.	CURING	Min. TIME	°F. TEMP.	PSI PRESS.	NITRIC ACID	°F. TEMP.	HRS. TIME	TYPE FAILURE
9	Vistanex B-120	100	Milled	Cold	Press	10	350	1000	70%	160	1/2	V.Spongy
	Halocarbon Oil 11-14	50										
	Ultrox	300							FNA	77	52.6	Decomposed Deformation
			Note:	Putty-like material								
10	Vistanex B-120	100										
	Halocarbon oil 14-25	50	Milled	Cold	Press	10	350	1000	70%	160	1/2	Spongy-better than No. 8
	Ultrox	300										
11	Vistanex B-120	100	Milled	Cold	Press	10	350	1000	70%	160	1/2	Sl.spongy
	Halocarbon Wax 8-00	50										
	Ultrox	300	Milled	Cold	Press	10	350	1000	FNA	77	53	Penetration
12	Vistanex B-120	100	Milled	Cold	Oven	30	350	—	70%	160	1/2	Weak-crumbly High Absorption
	Pylene PC-11	50										
	Ultrox	300										
13	Vistanex B-120	50	Milled	Cold	Press	10	350	1000	70%	160	1/2	Weak-crumbly Incompatible
	Parlon (1000 cp)	50										
	Ultrox	200										
14	Vistanex B-120	50	Milled	Cold	Press	10	350	1000	70%	160	1/2	V.Spongy Blistered
	Advagum 1098	50										
	Ultrox	200										
15	Vistanex B-120	50	Milled	Cold	Press	10	350	1000	70%	160	1/2	Sl.weaker tensile Not as good as No. 6.
	Advance 2575	50										
	Ultrox	200										
16	Vistanex B-120	50	Milled	Cold	Press	10	350	1000	70%	160	1/2	Slightly spongy
	Advawax 2080	50										
	Ultrox	200										

TABLE VI (Cont'd.)
FILLETING SYSTEMS

COMPD. NO.			METHOD	°F. TEMP.	CURING	MIN. TIME	°F. TEMP.	PSI PRESS.	NITRIC ACID	°F. TEMP.	HRS. TIME	TYPE FAILURE
17	Vistanex B-120 Chlorowax 70 Ultrox	100 50 300	Milled	Cold	Mill	20	Cold	—	70% FNA	160 77	1/2 30-1/2	Sl.surface attack No apparent effect
18	Hypalon S-2 MG Oxide Stabelite Resin Tetrone A Asbestine	100 20 2.5 1 25	Milled	Cold	Press	30	320	1000	70% FNA FNA	160 125 77	1/2 1/2 3.6	No apparent effect Color change Dissolved Lg. blister
			Note: Recommended by Du Pont									
19	Vistanex B-120 Veloform	100 200	Milled	Cold	Mill	20	Cold	—	70% 70%	160 250	1/2 1/2	Sl.hardening Sl.hardening
20	Vistanex B-100 Chlorowax 70 Ultrox	100 50 300	Milled	Cold	Mill	20	Cold	—	70% 70%	160 250	1/2 1/2	Spongy-swollen Bad sponging and swelling
21	Vistanex B-100 Chlorowax 40 Ultrox	100 50 300	Mill	Cold	Mill	20	Cold	—	70%	160	1/2	Swelled-porous
22	Vistanex B-100 Chlorowax 40	100 50	Mill	Cold	Mill	20	Cold	—	70% 70%	160 250	1/2 1/2	OK-Sl.bleaching Swelled-spongy
23	Vistanex B-100 Indoil 35	100 50	Mill	Cold	Mill	20	Cold	—	70% 70%	160 250	1/2 1/4	OK Melted
24	Vistanex B-100 Indoil H-300	100 50	Mill	Cold	Mill	20	Cold	—	70% 70%	160 250	1/2 1/2	OK Alm. melted

TABLE VI (Cont'd.)
FILLETING SYSTEMS

COMPD. NO'			METHOD	°F. TEMP.	CURING	MIN. TIME	°F. TEMP.	PSI PRESS.	NITRIC ACID	°F. TEMP.	HRS. TIME	TYPE FAILURE
25	Vistanex B-100 Veloform Chlorowax 40	50 100 50	Mill	Cold	Mill	20	Cold	—	70% 70%	160 250	1/2 1/2	OK Lost shape Swollen
26	Vistanex B-100 Veloform Chlorowax 40	100 50 50	Mill	Cold	Mill	20	Cold	—	70% 70%	160 250	1/2 1/2	OK Lost shape Swellen
27	Vistanex B-100 Veloform Halocarbon wax 8-00	100 200 75	Mill	Cold	Mill	20	Cold	—	70%	250	1/2	Blistered Sl.swollen
28	Vistanex B-100 Veloform Halocarbon oil 14-25	100 200 75	Mill	Cold	Mill	20	Cold	—	70%	250	1/2	Blistered
29	Vistanex B-100 Micronex W-6 Philblack A	400 50 50	Mill	Cold	Mill	20	Cold	—	FNA FNA	77 77	24 45	Sl.blistering Sagging
30	Same as 29		Mill (Carbon black preheated at 350°F. for 2 hrs.)	Cold	Mill	20	Cold	—	FNA	77	72	Blistering
31	Vistanex B-100 Treated Hi-Sil	400 100	Mill	Cold	Mill	20	Cold	—	FNA	77	72	Severe attack
32	Vistanex B-100 Chlorowax 40	400 100	Mill	Cold	Mill	20	Cold	—	FNA	77	195	Mod. attack weakened
33	Vistanex B-100	100	Mill	Cold	Mill	20	Cold	—	FNA	77	195	Penetration

TABLE VI (Cont'd.)
FILLETING SYSTEMS

COMPD. NO.			METHOD	°F. TEMP.	CURING	MIN. TIME	°F. TEMP.	PSI PRESS.	NITRIC ACID	°F. TEMP.	HRS. TIME	TYPE FAILURE
34	Vistanex B-100 Polyethylene PM-1	30 70	Mill	250	Mill	20	Hot-warm	—	FNA	120	48	Penetration Whitening
35	Vistanex B-100 Polyethylene PM-1	80 20	Mill	250	Mill	20	Hot-warm	—	FNA	120	48	Bad blistering
36	Vistanex B-100 Polyethylene DYGT	80 20	Mill	250	Mill	20	Hot-warm	—				
37	Vistanex B-100 Polyethylene DYNJ	80 20	Mill	250	Mill	20	Hot-warm	—				

TABLE VII
KELLOGG X-200 FILLETING COMPOUNDS

Batch No.	55-1	55-2	55-3	55-4	55-5	55-6	56-1	56-3	56-2	57-1	57-2
J-1019	100	100	100	100	100	100					
J-4322							100	100	100		
J-4613										100	100
J-4559											
J-4611											
J-4547											
J-4646											
KelF-1-3 oil	150	100	100	100	66.7	66.7	129				
Kel-F Plast. oil								116.5	148	100	300
Kel-F 150 Wax											
Ground Glass	200		100			66.7					
Ultrox				200	66.7		129	90.3	90.3	100	273.4
Ease of grind-resin	Poor	Poor	Poor	poor	poor	Poor	Good	Good	Good	Poor	Poor
Putty Viscosity	OK	Stiff	OK	Stiff	Rubbery	Stiff	Dry stiff	Too stiff	OK	Too stiff	Rubbery stiff
300°F. Vertical flow	OK	Flows	OK	OK	OK	Flows	OK	OK	OK	Sl.	OK
Degree of fusion 1 Hr. at 300°F.	Too hi filled	Good	Good	Too hi filled	Good	Good	Poor	Poor	Poor	Good	Good
WFNA resistance 160°F.			Sl. pock-marks (6 days)				Cracked blistered (1 day) Volatility of oil?		Cracked (1 day)		

TABLE VII (cont'd.)
KELLOGG X-200 FILLETING COMPOUNDS

	57-3	57-4	57-5	57-6	57-7	57-8	57-9	58-1	58-2	59-1	59-2
Batch No.											
J-1019	100										
J-4322		100							50		100
J-4613			100								
J-4559				100						40.5	
J-4611					100					5.5	
J-4547						100			50	36.3	
J-4646							100	100		17.7	
Kel-F 1-3 Oil											
Kel-F Plast. Oil	100	100	100	100	100	100	100	112.5	130	100	50
Kel-F 150 Wax										50	100
Ground Glass								100			
Ultrox	100	100	100	100	100	100	100	100	100	100	90
Ease of grind-resin	Poor	Good	Poor	Fair	Good	Poor	Poor	Poor			
Putty viscosity	OK	OK	OK	Dry	Dry	Sl.stiff	Sl.stiff	Heats up Too stiff	Heats up Too stiff	Heats up Too stiff	OK
300°F. Vertical flow	OK	OK	Sl.	OK		OK	OK			Sl.	OK
Degree of fusion 1 hr. at 300°F.	Good	Sl.stiff	Good	Poor		Fair	Good				Fair
WFNA Resistance 160°F.										Excess flow with pene. (4 days)	Sl. cracking (4 days)

Semi-production runs in ceramic extruder

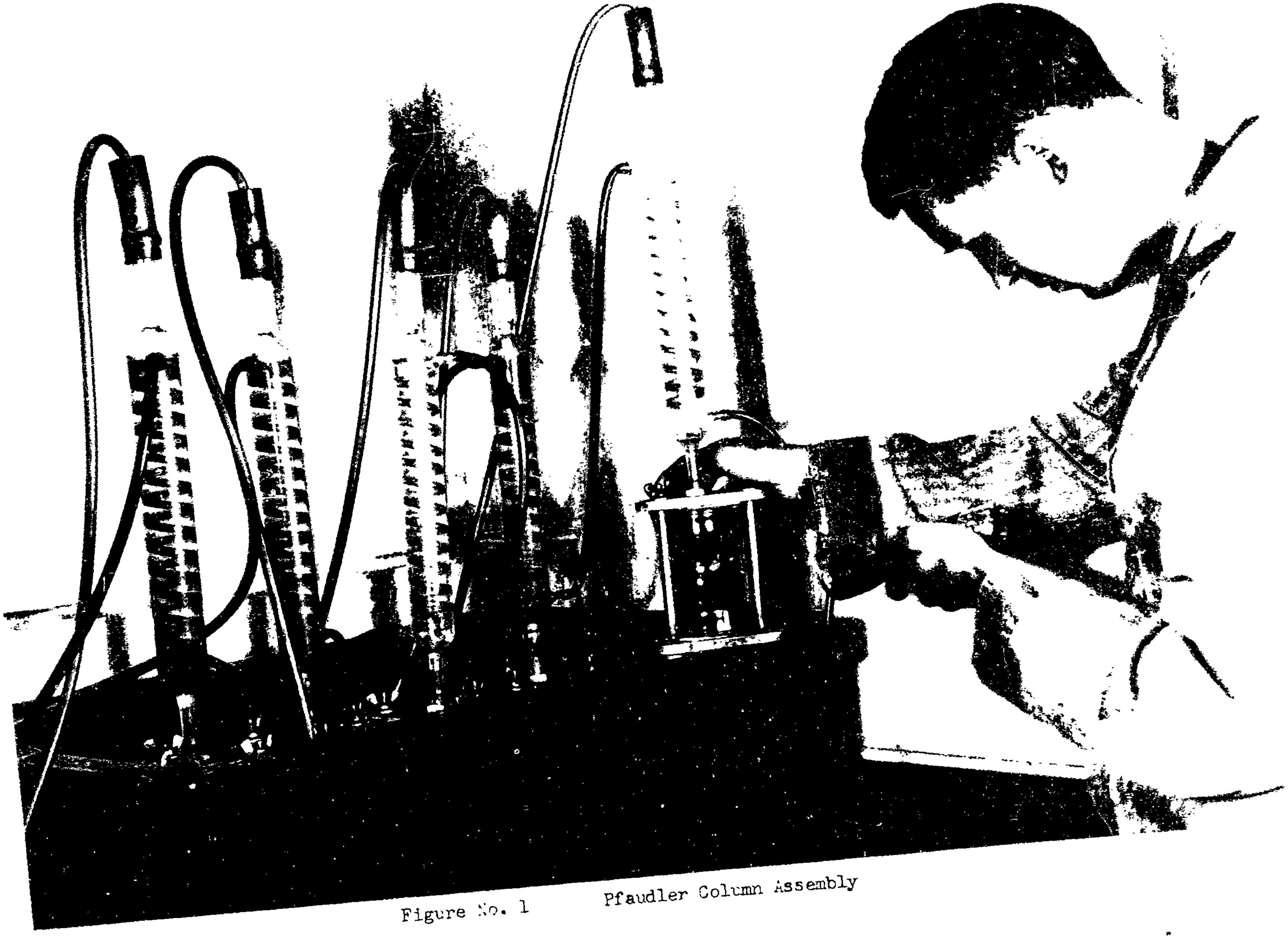


Figure No. 1 Pfaudler Column Assembly

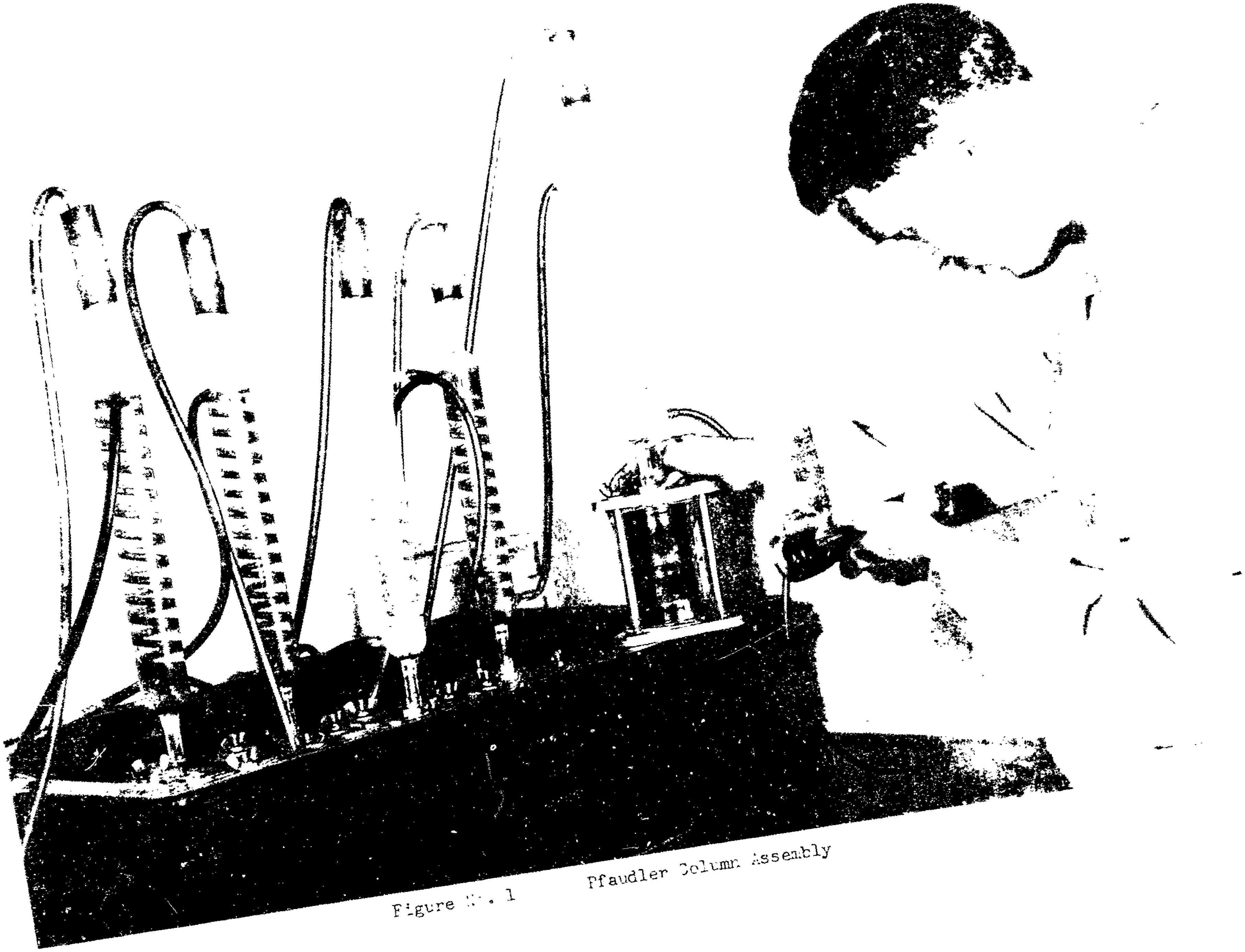
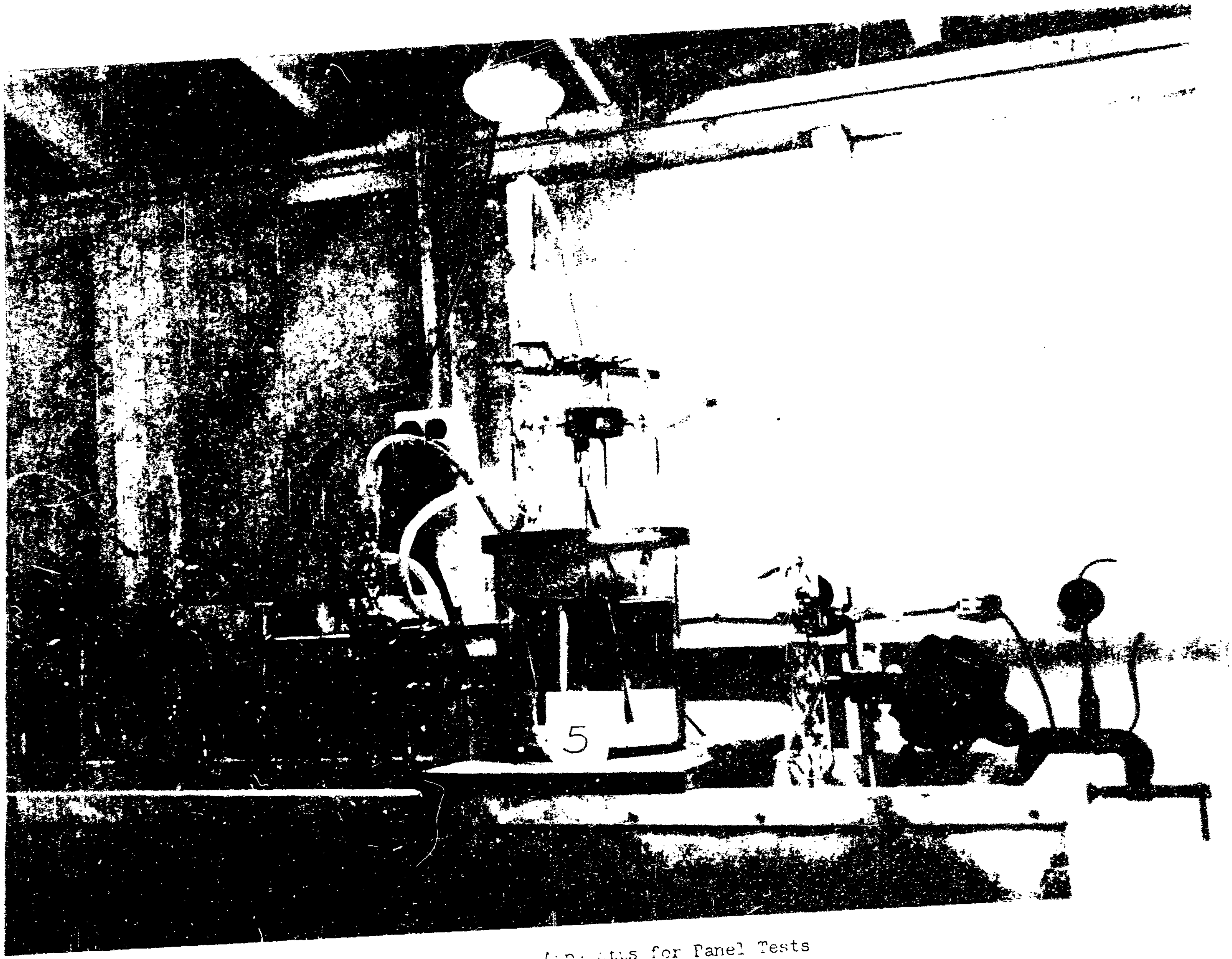


Figure No. 1 Pfaudler Column Assembly



[illegible] Apparatus for Panel Tests

Figure No. 3 Pigmented, Plasticized Kellogg X-200 Film exposure to WFNA-AF-76B

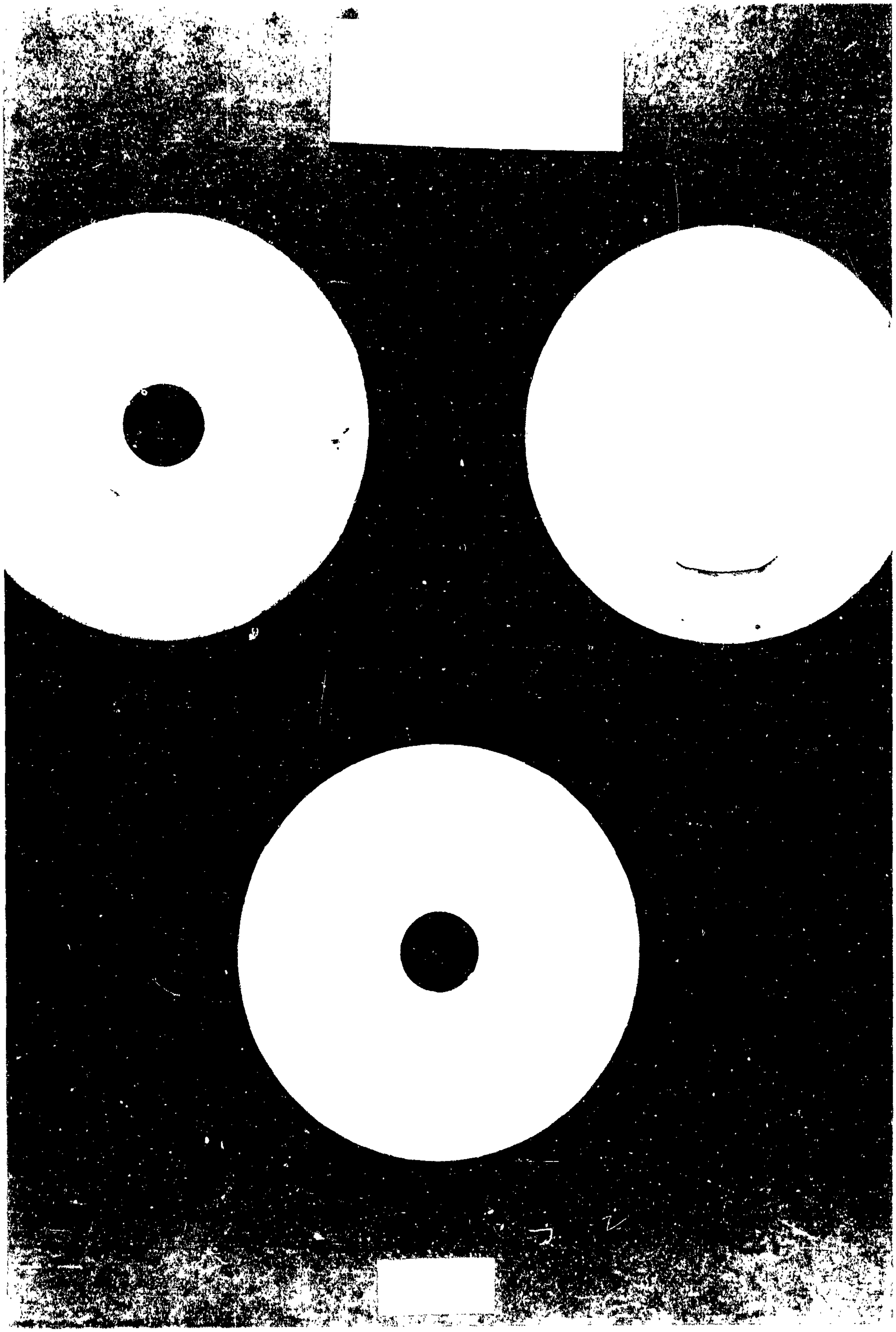


Figure No. 4 Pigmented, Plasticized Kellogg X-200 Film exposure to WFNA-AF-76E

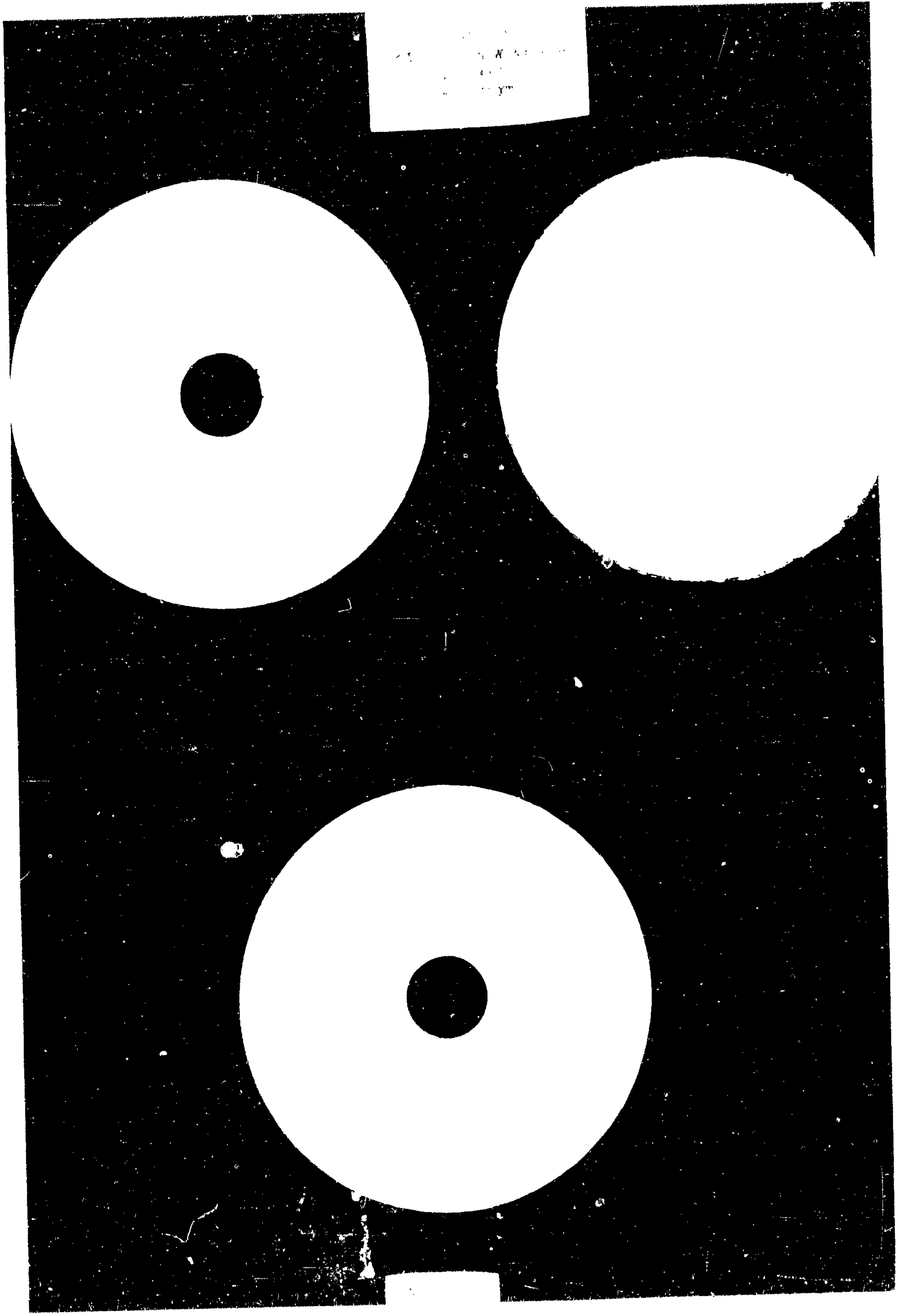


Figure No. 5 Pigmented, Plasticized Kellogg X-200 Film exposure to WFNA-AF-76F

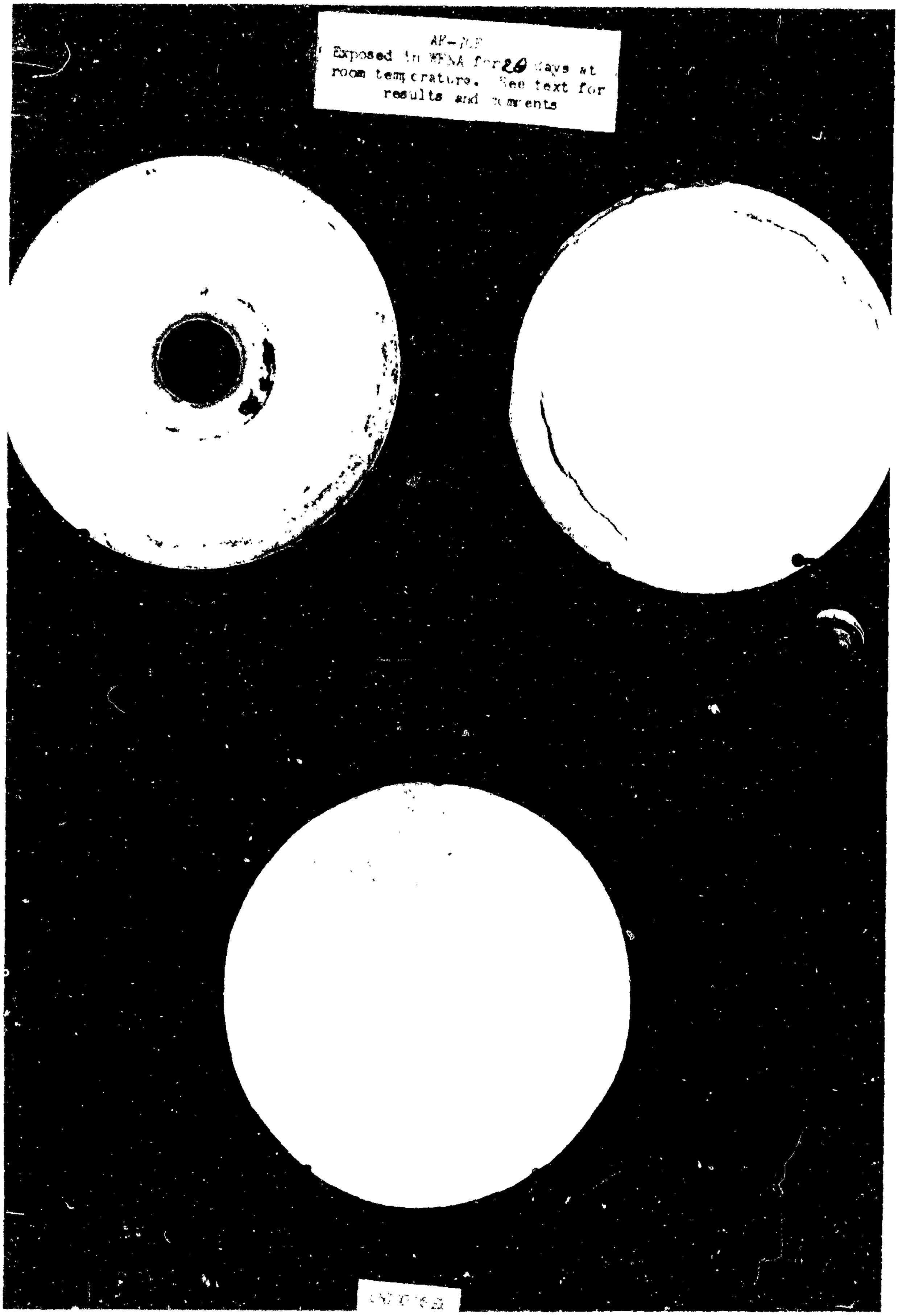
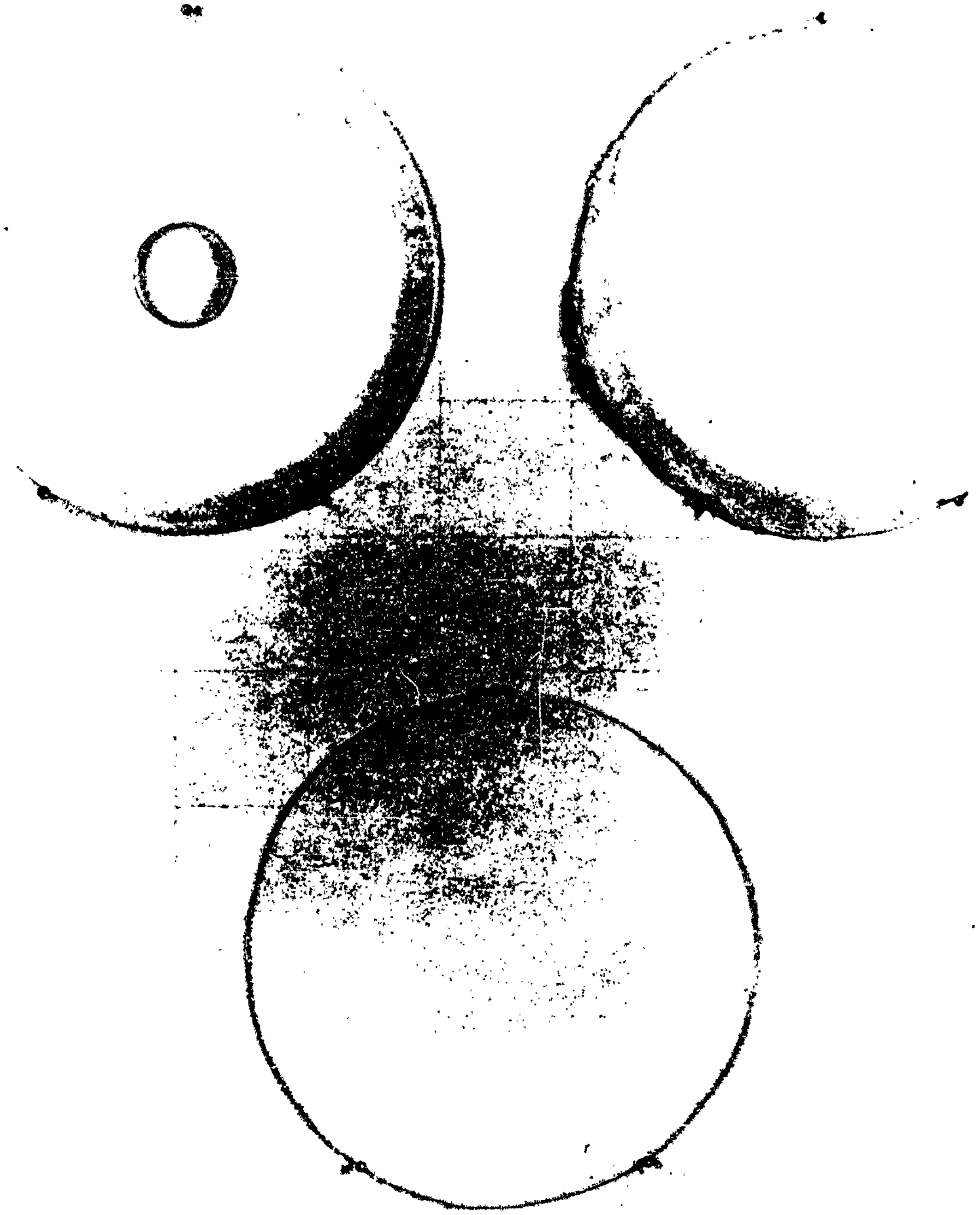


Figure No. 6 Pigmented, Plasticized Kellogg X-200 Film exposure to WFNA-AF-77



AF77
EXPOSED TO WFNA AT 125°F.
9 DAYS
(BLISTER APPEARED AFTER 4 DAYS -
SEE TEXT)

UNEXPOSED

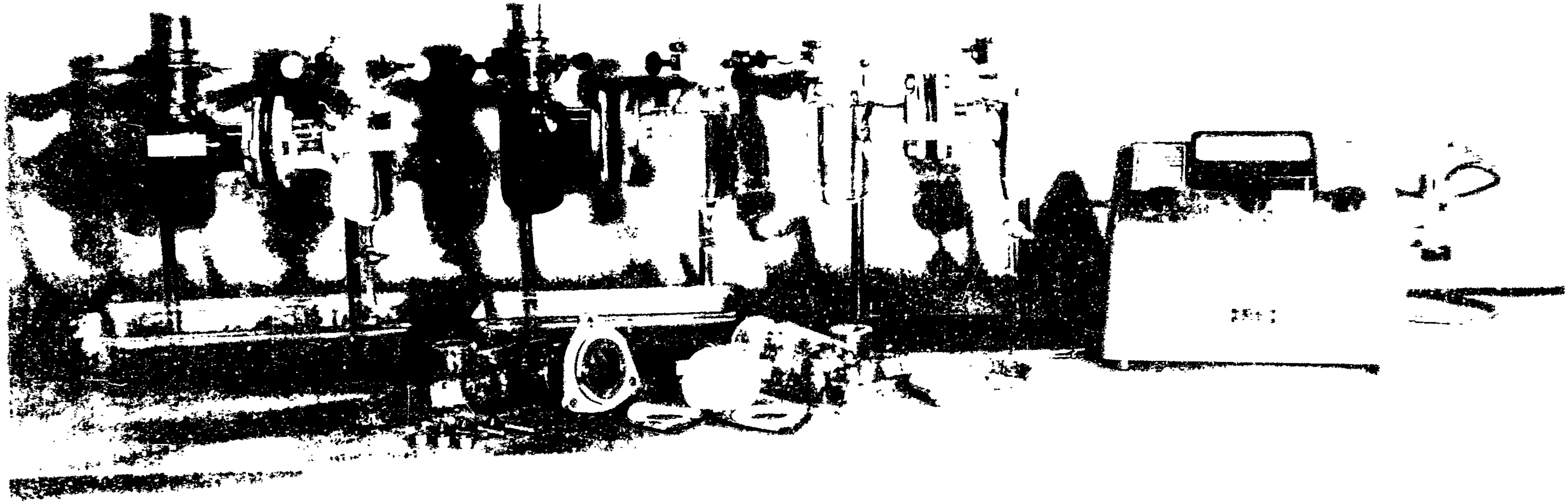


Figure No. 7 Navy "M" Cell

Figure No. 8 Pigmented, Plasticized Kellogg X-200 Film exposure to WFNA-AF-77G

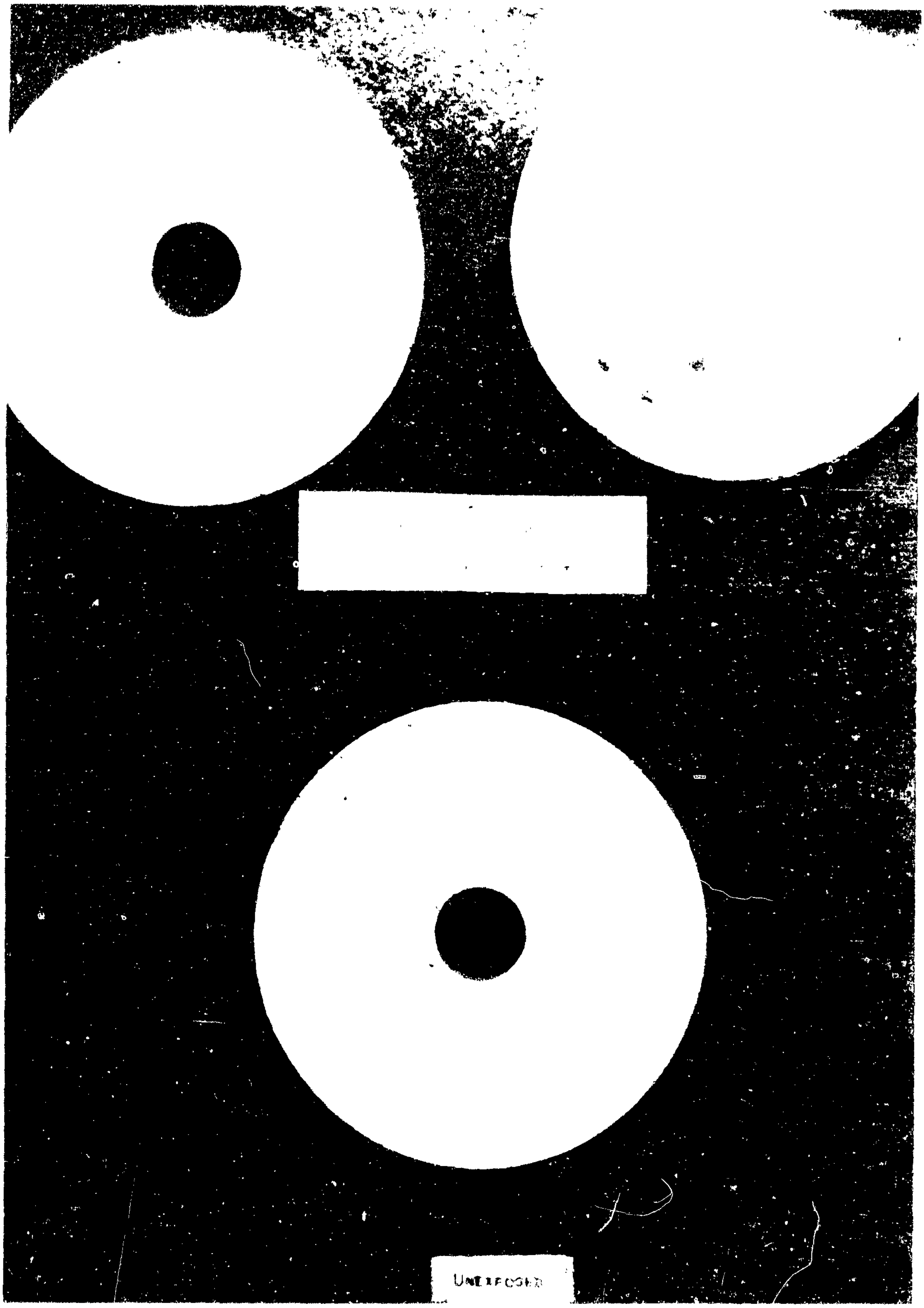


Figure No. 9 Pigmented, Plasticized Kellogg X-200 Film exposure to WFNA-AF-77H

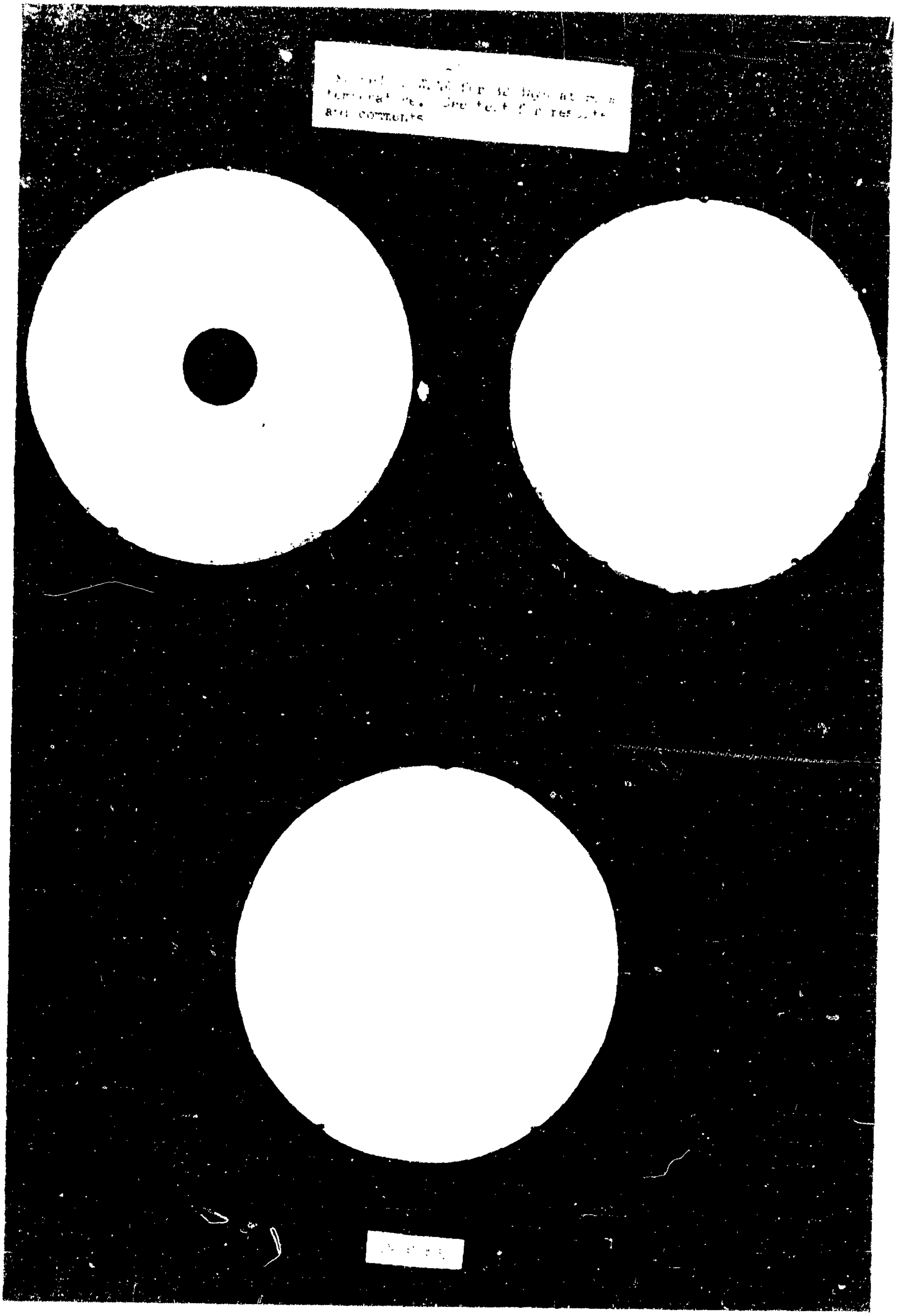


Figure No. 10 Pigmented, Plasticized Kellogg X-200 Film exposure to WFNA-AF-77I;

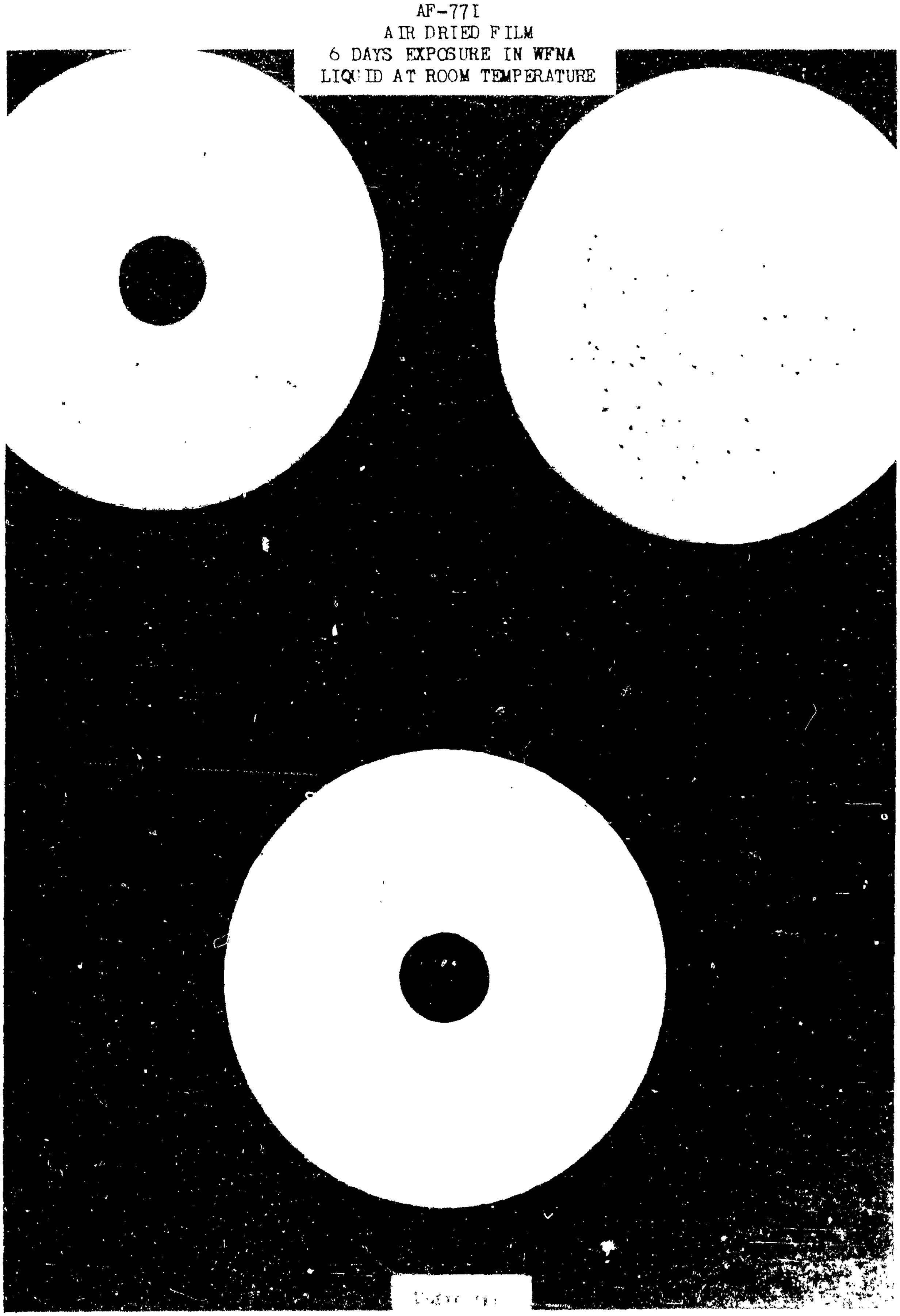


Figure No.11 Pigmented, Plasticized Kellogg X-200 Film exposure to WFNA-AF-77:AF-75BB

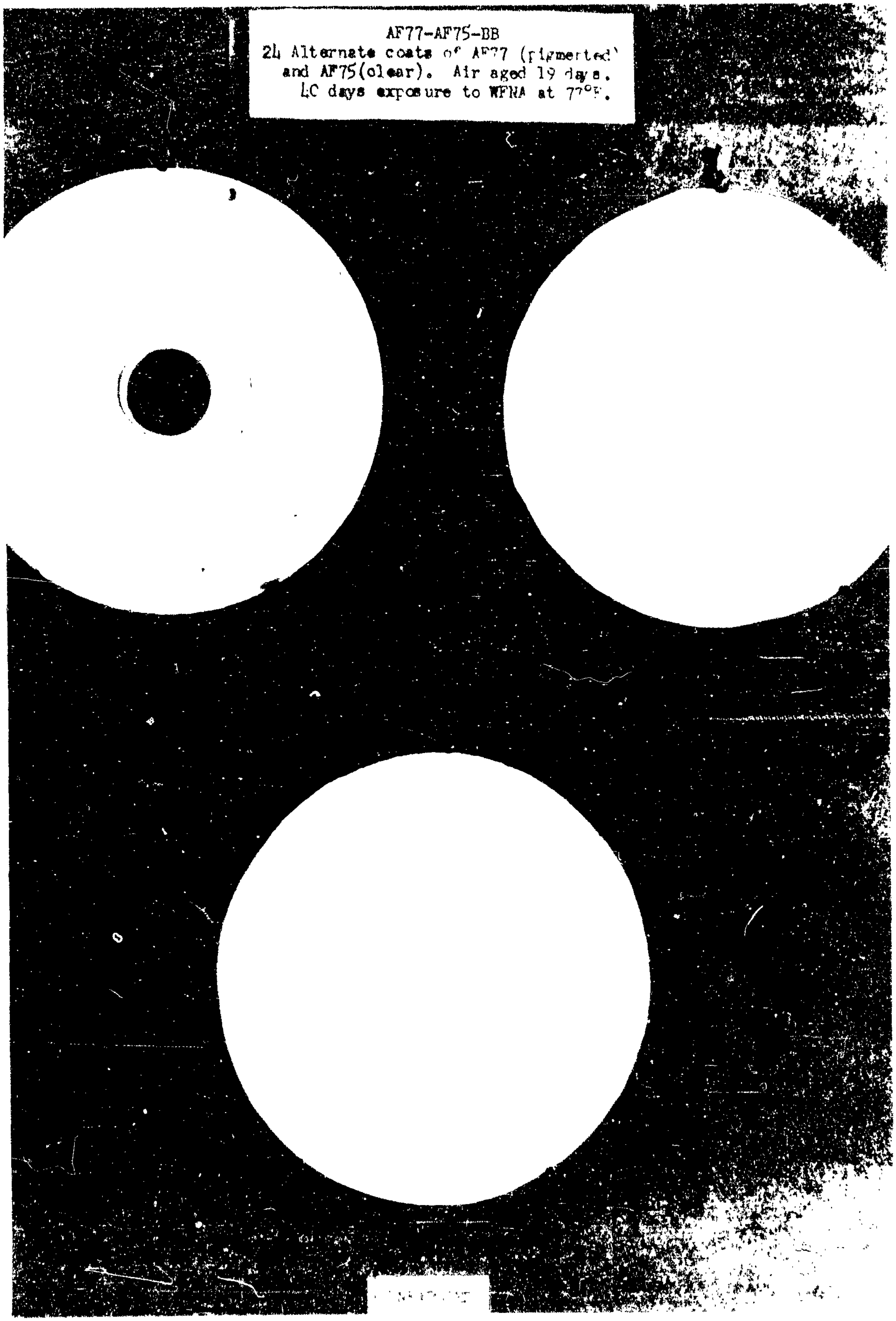
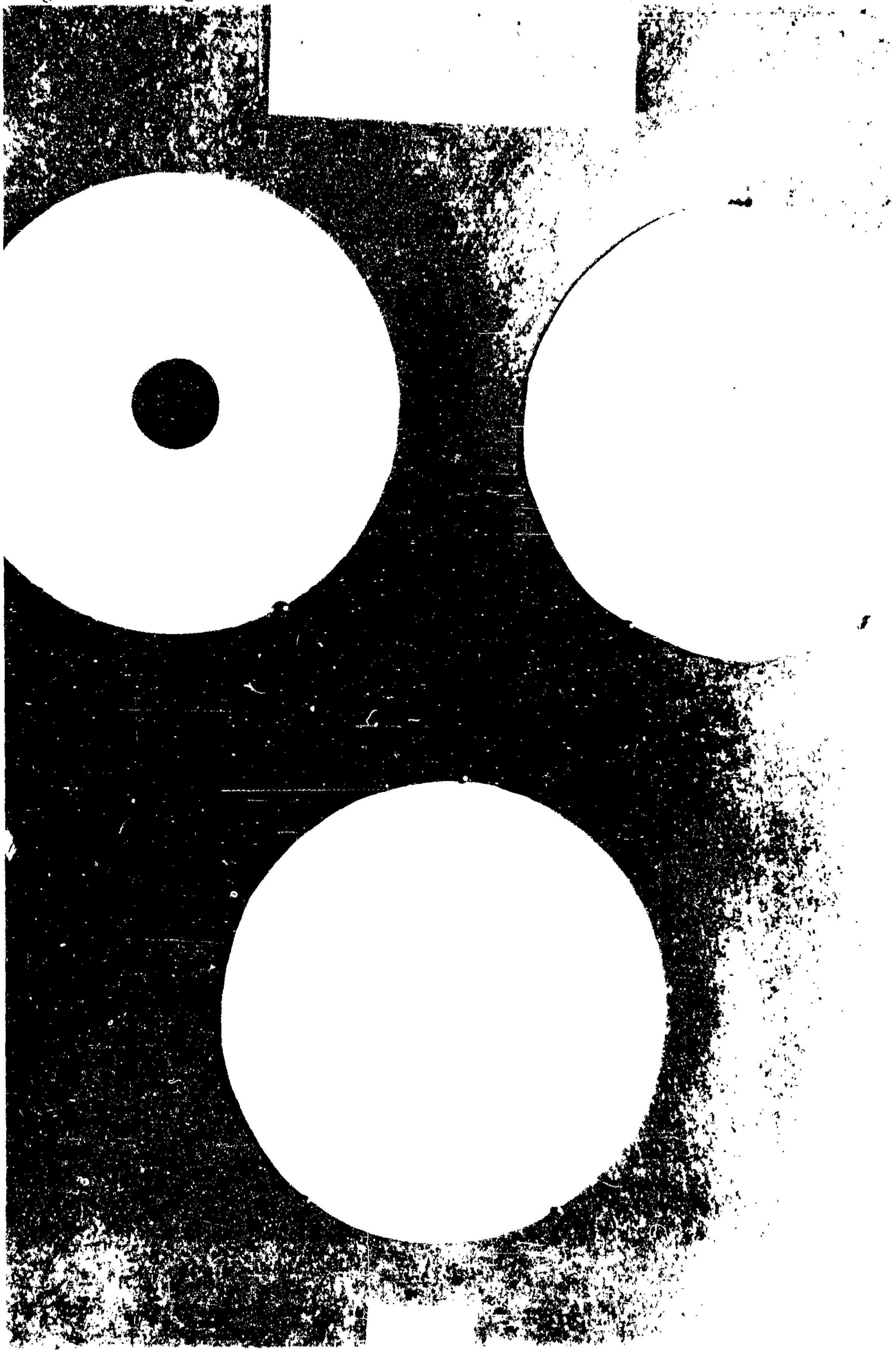


Figure No.12 Pigmented, Plasticized Kellogg X-200 Film exposure to WFNA-AF-77. Set K



AF-43
Figure No.13 Vinyl:Chloride Aroclor Film-pigmented with Diriron-exposure to WFNA-AF40:

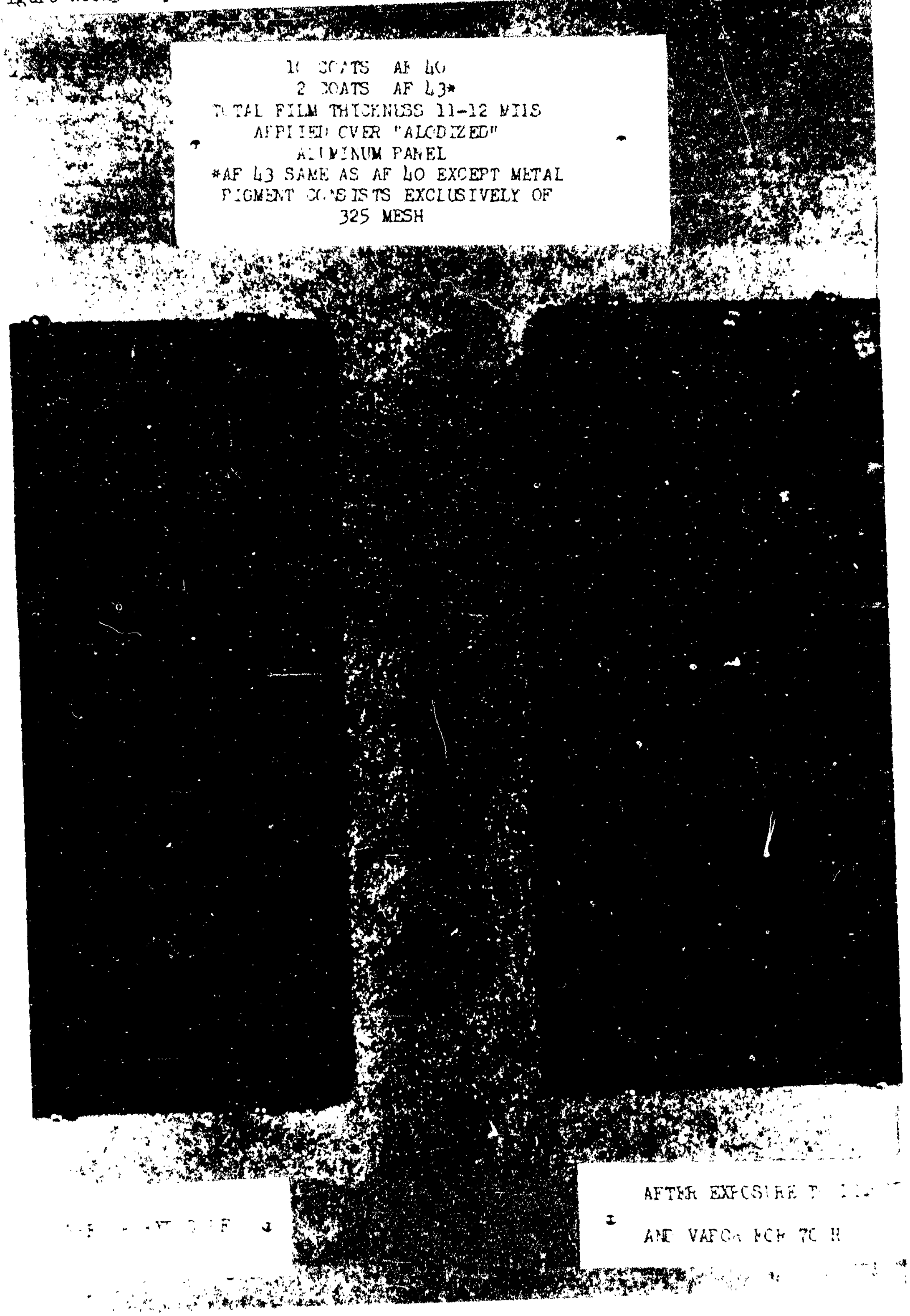


Figure No.14 Vinyl Chloride Arcclor Film-unpigmented-with primer-exposure to WFNA-AF18A

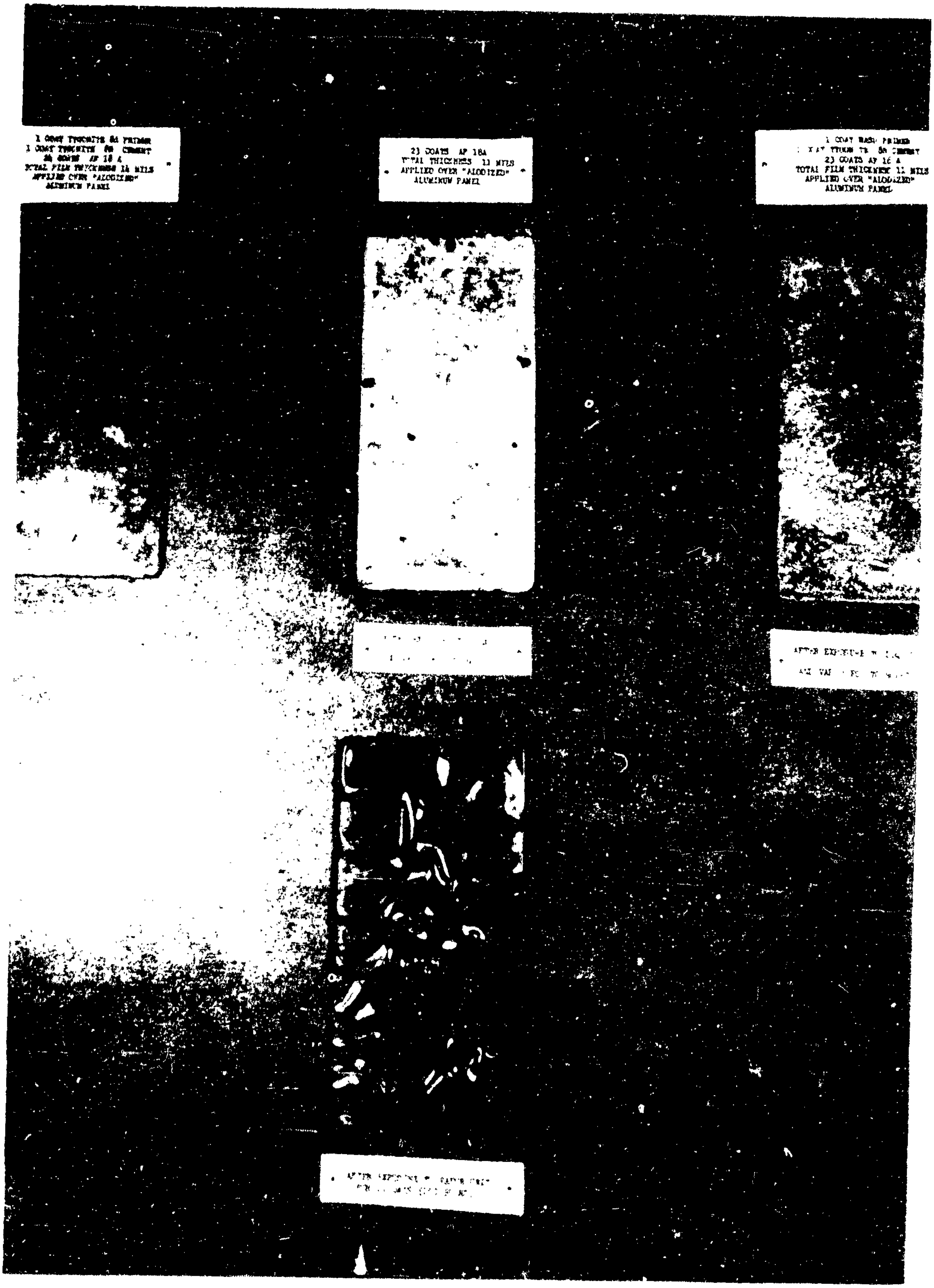


Figure No.15 Exon 400XR-61 and vinyl Chloride Films-Plasticized-pigmented and unpigmented Exposure to WFNA-AF-19CP; AF-17BP-AF-18B

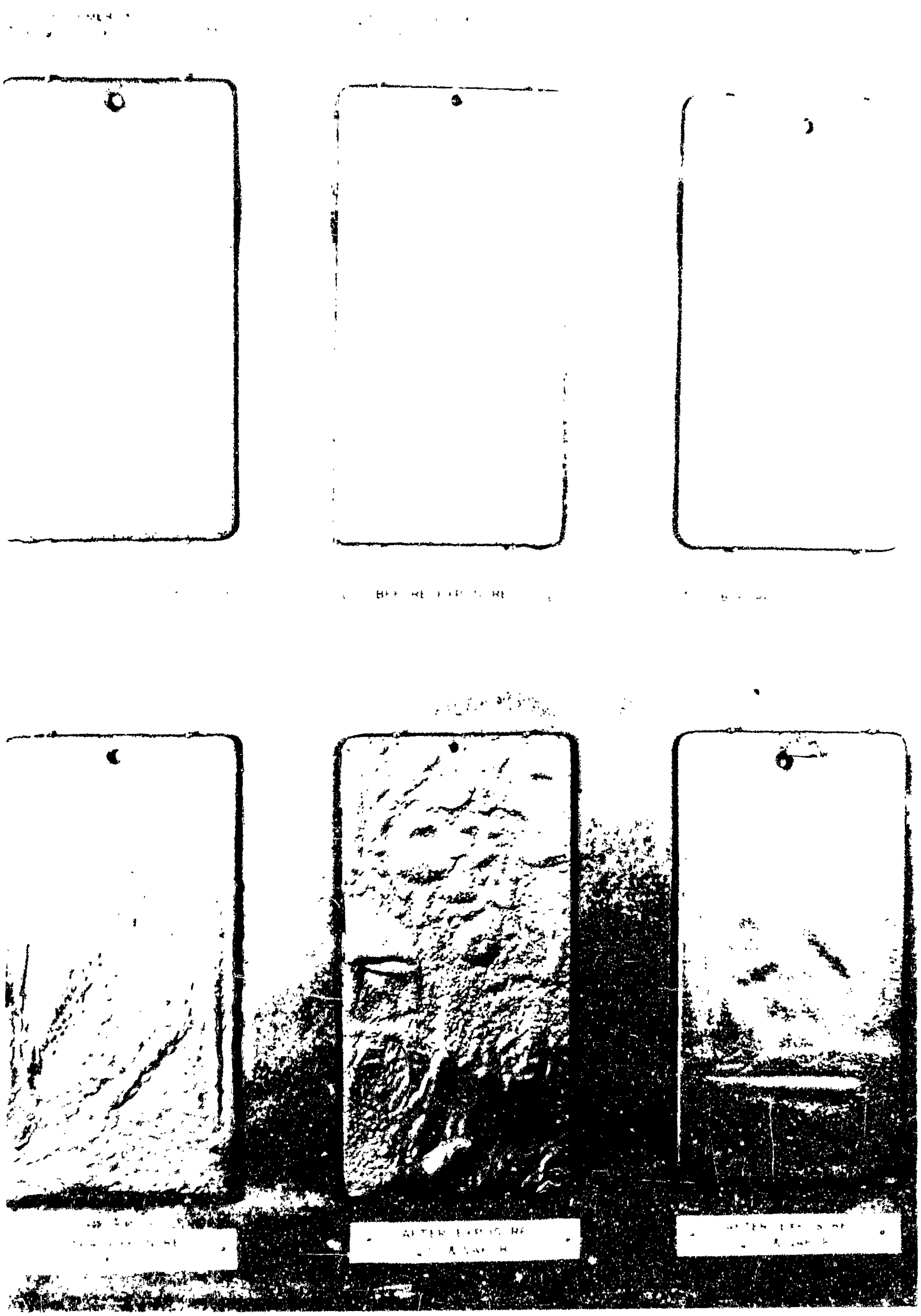


Figure No.16 Vinyl Chloride: Aroclor Film-RBH Resin 569 Primer-Exposure to WFNA
AF-16A; AF-16-B; AF-18B; AF-18C

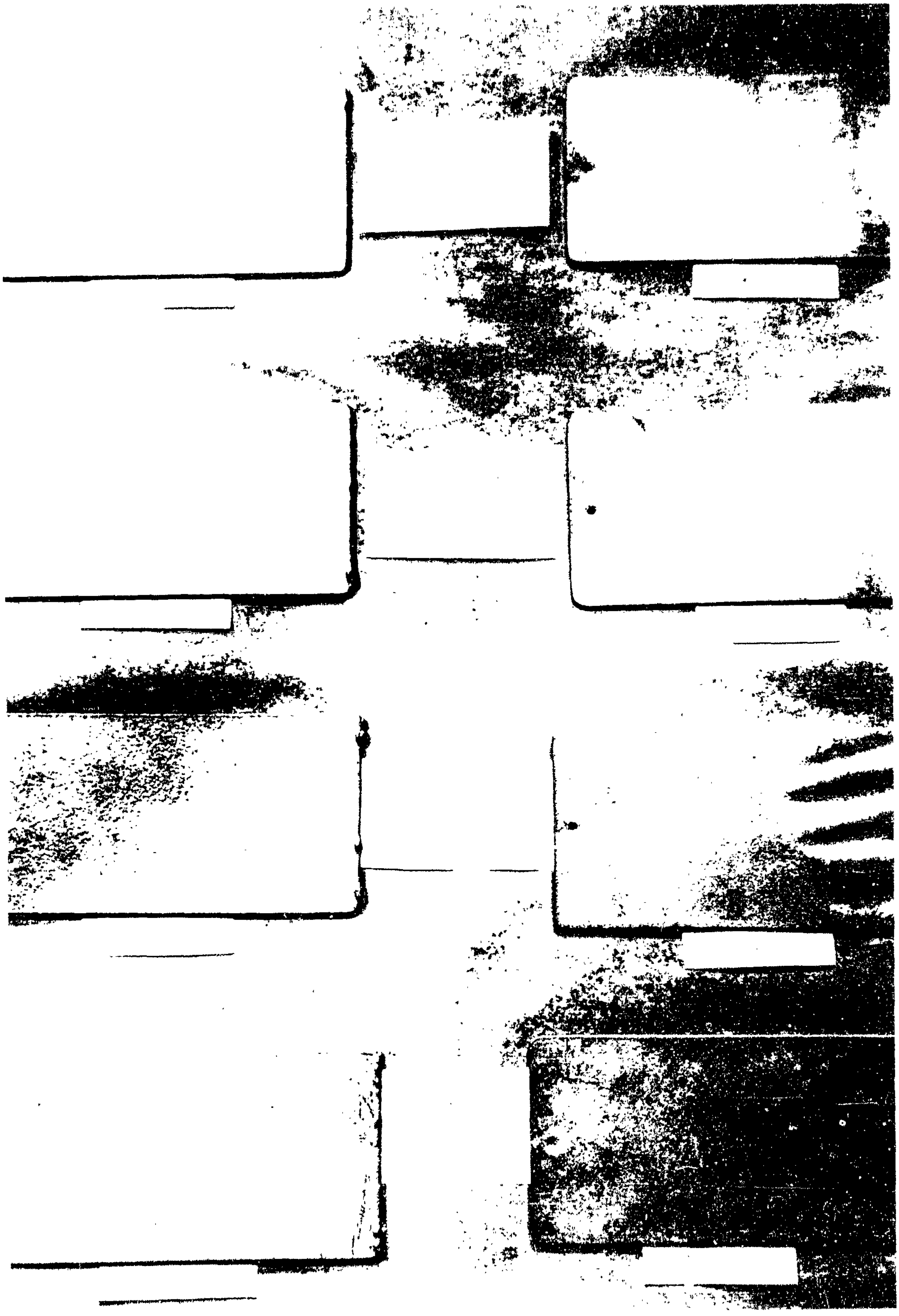
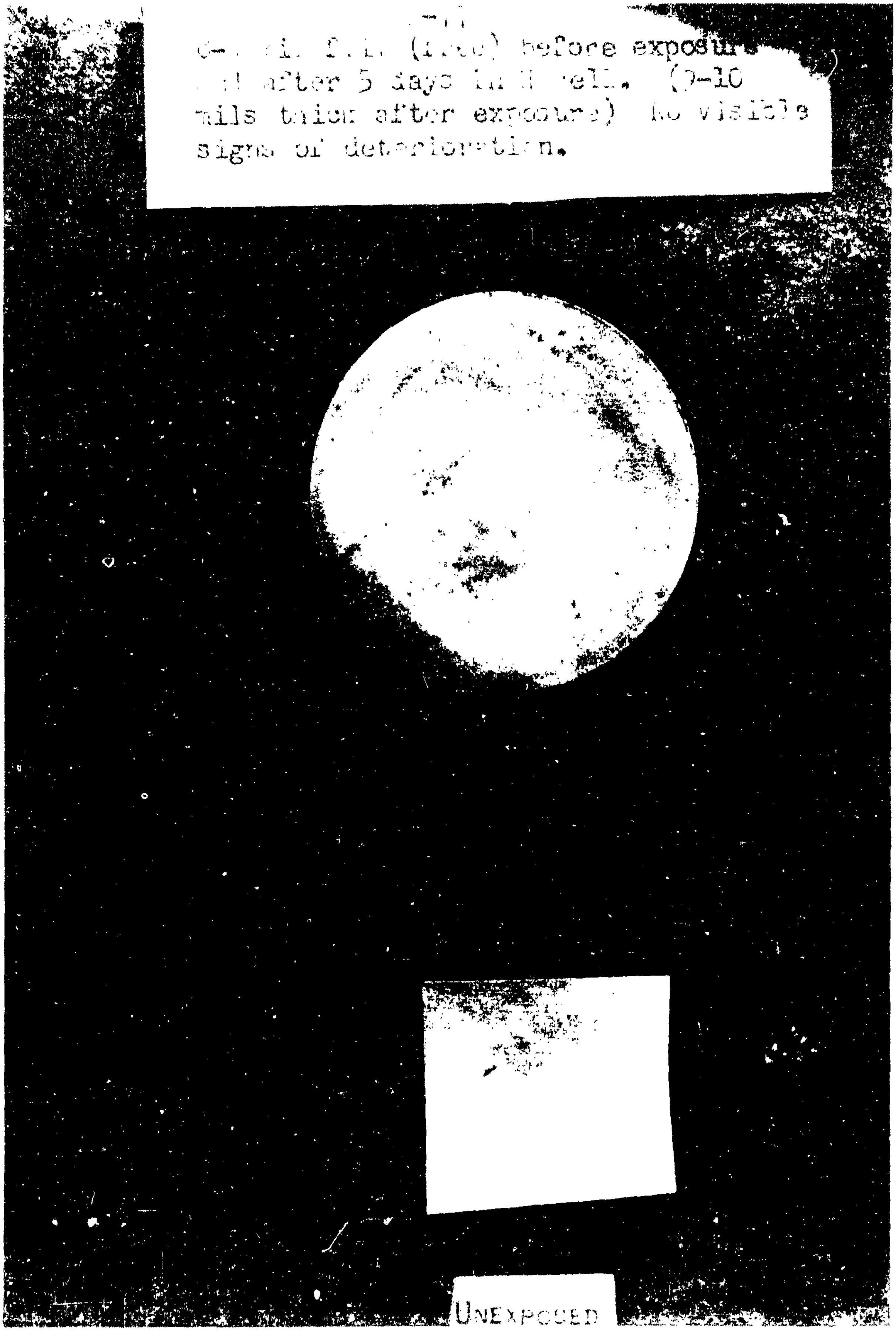


Figure No. 17 Pigmented Plasticized Kellogg X-200 Film Navy "H" Cell Exposure AF-77



6-[illegible] mil film ([illegible]) before exposure and after 5 days in H cell. (9-10 mils thick after exposure) No visible signs of deterioration.

Figure No.16 Pigmented Plasticized Kellogg X-200 Film Navy "H" Cell Exposure-AF-77

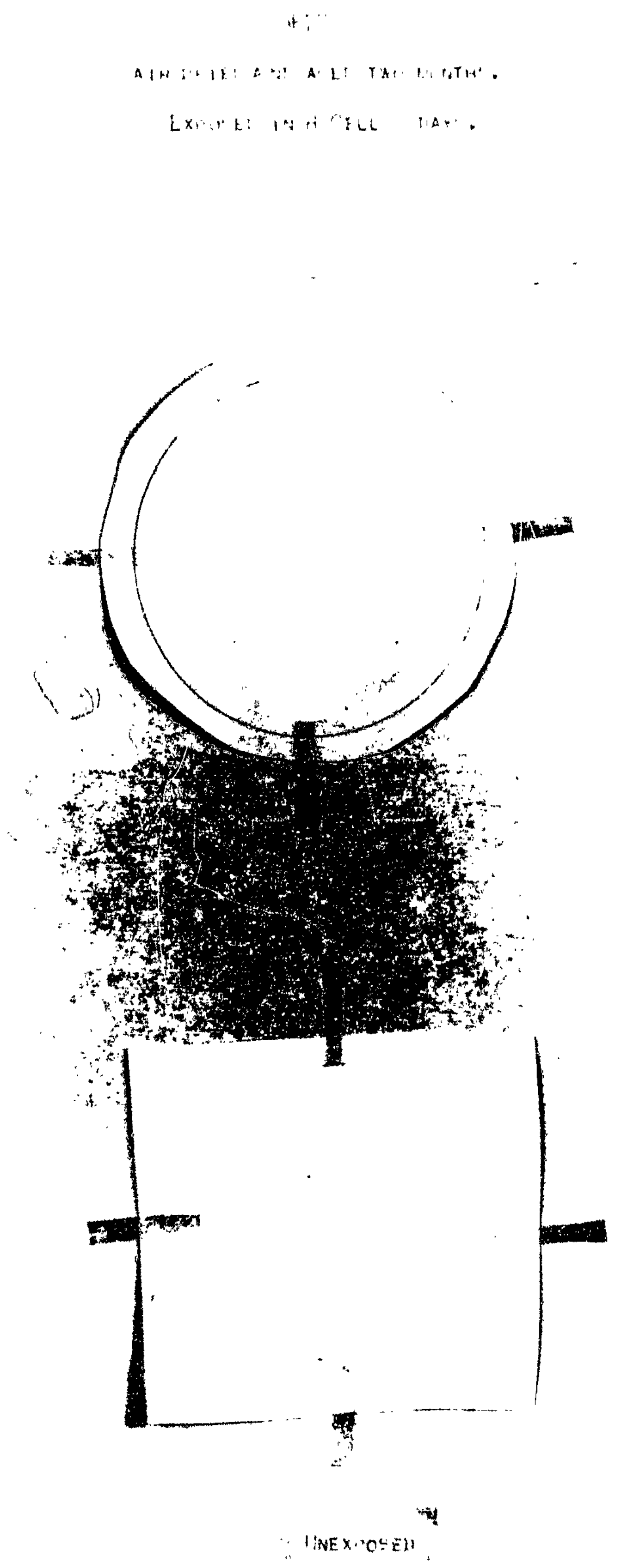


Figure No. 19 Kellogg X-200 Film Navy H-Cell Exposure-AF-75

Figure No. 20 Clear Kellogg X-200 Film Exposure to WFNA-AF-75AA

Figure No. 21 Clear Kellogg X-200 Film Exposure to WFNA-AF-79A

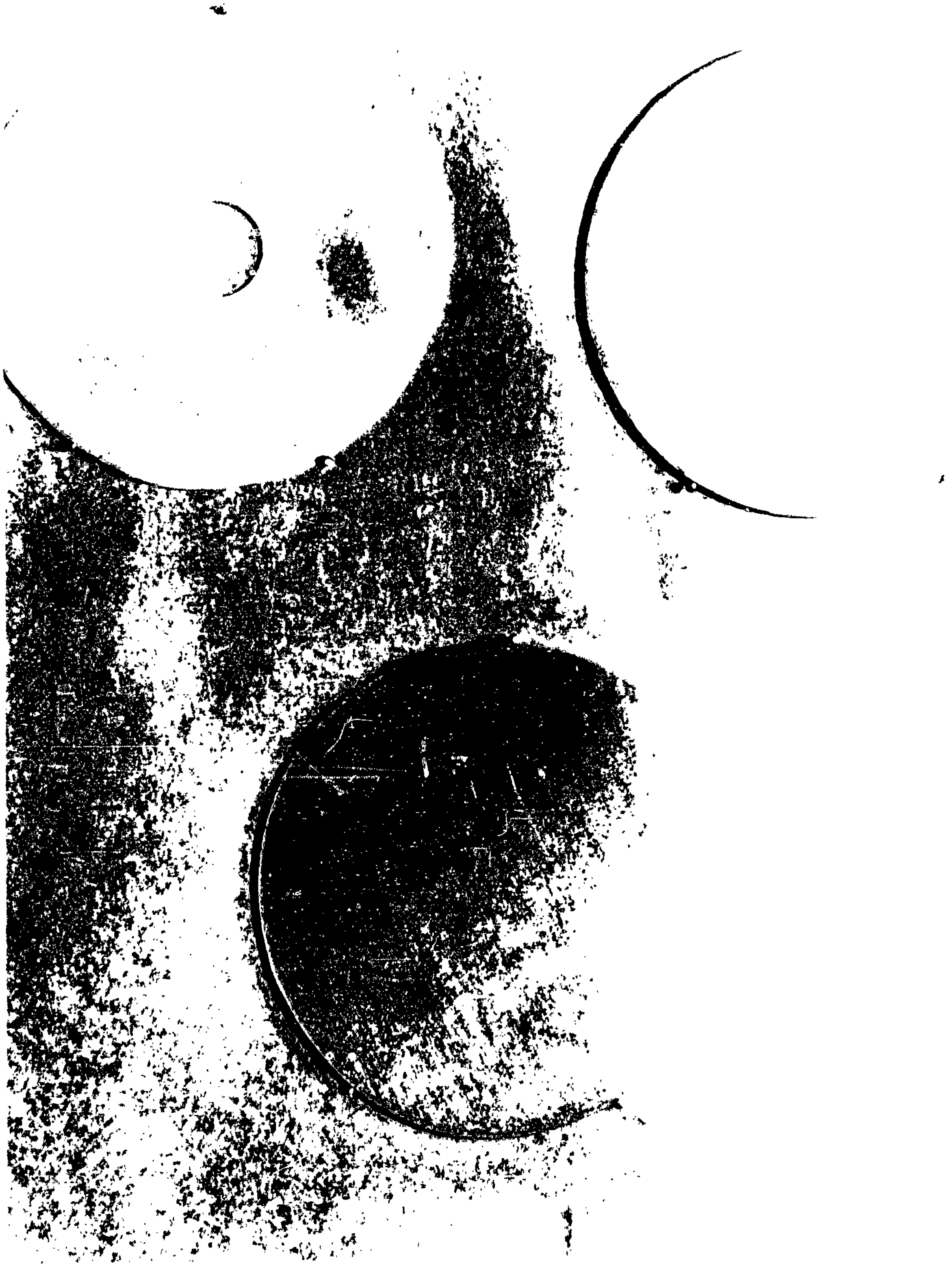


Figure No. 22 Clear Kellogg X-200 Film Exposure to WFNA-AF-79B

Figure No. 23 Clear Kellogg X-200 Film Exposure to WFNA-AF-79C

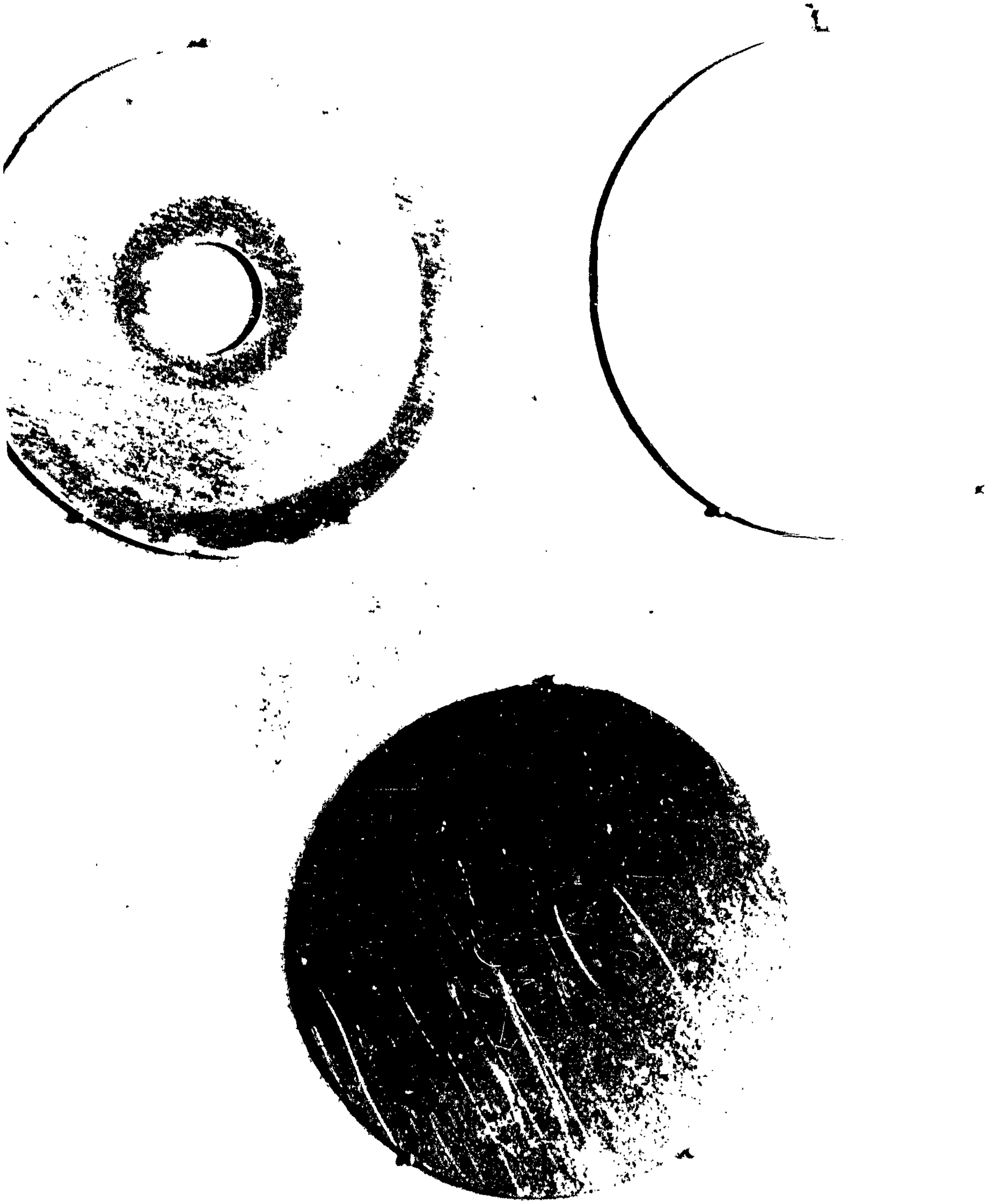


Figure No. 24 Kellogg X-200 Resin

Figure No. [illegible] Putty made from "off grade" Kellogg X-200 Resin-59-2

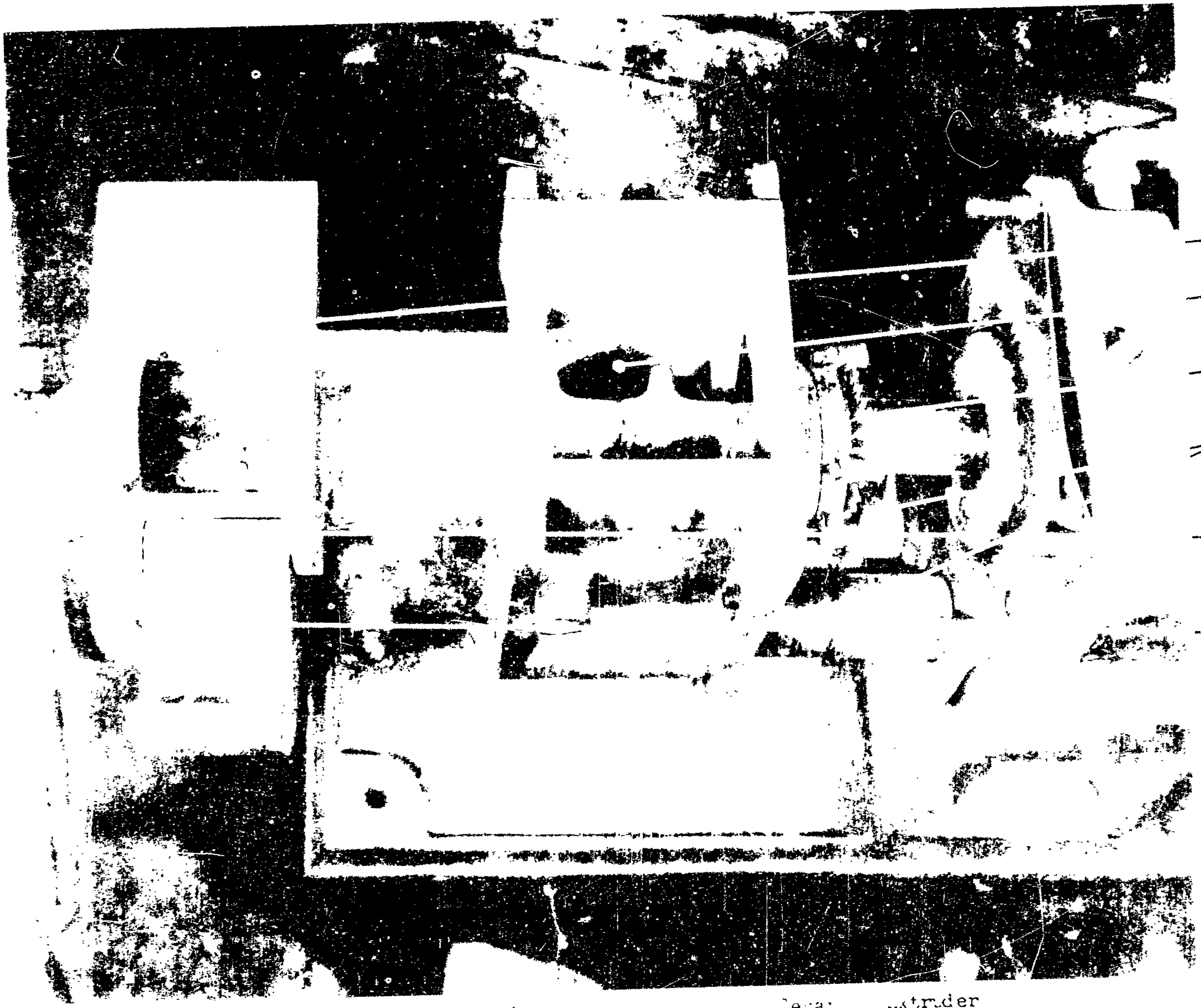


Figure No. 26 Ceramic Extruder

Figure No. 27 Base Resin Exposures to WFNA Parlon and Hypalon S-2

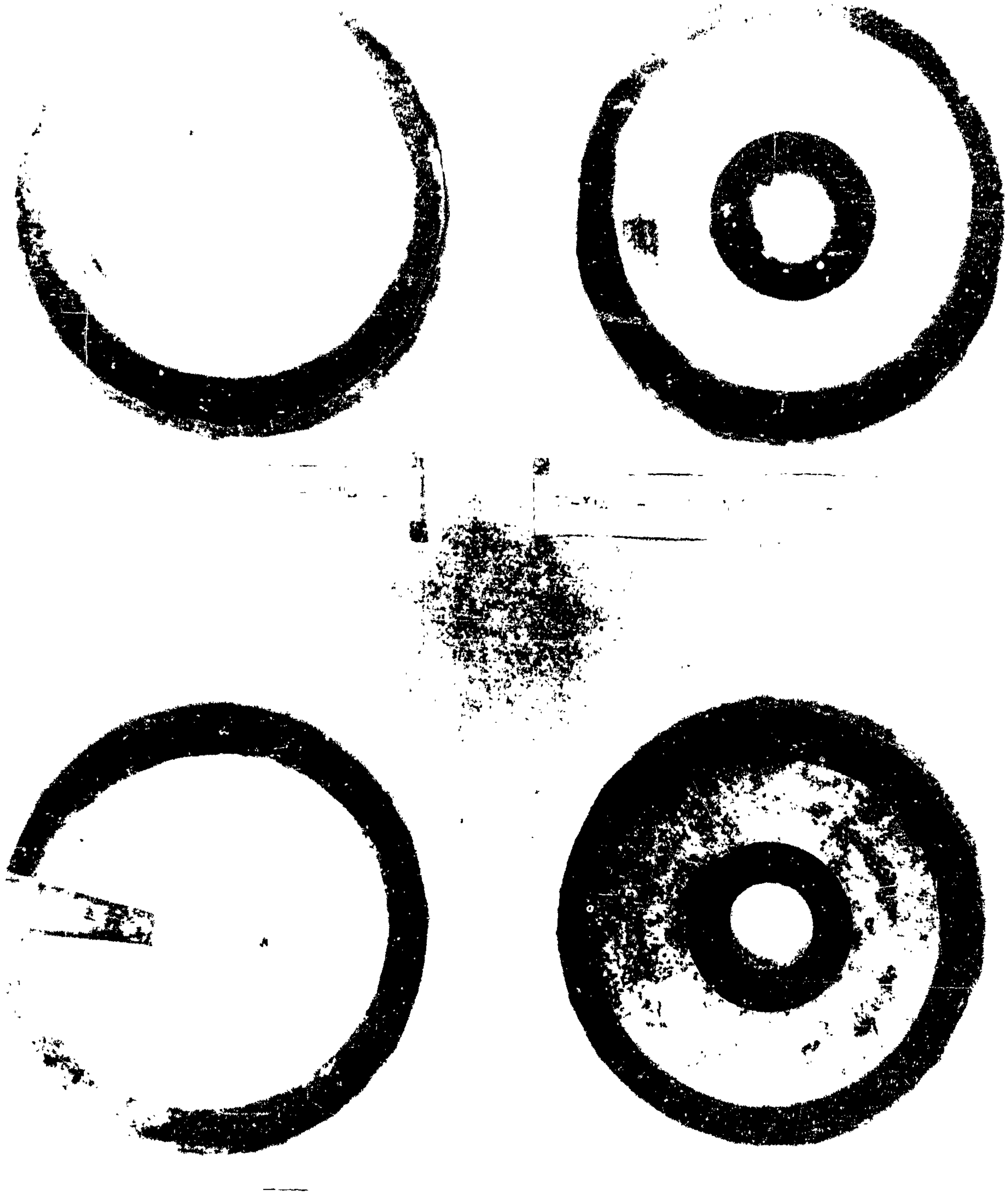
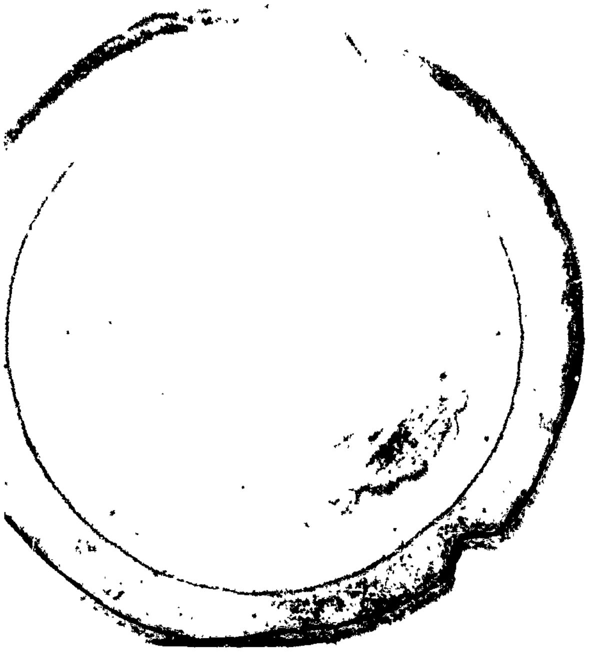


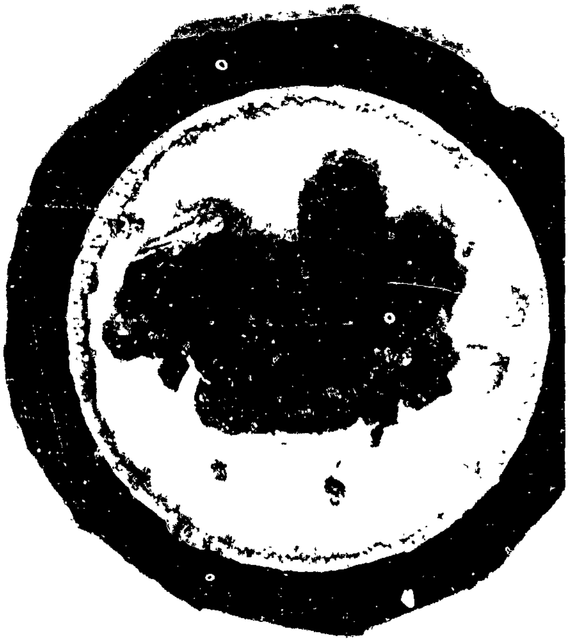
Figure No. 28 Base Resin Exposures to WFNA Hypalon S-2, Exon 400XR61, Advawax 2000 Chlor Isopol



[illegible]-VIII - CHLOROSULFONATED POLYETHYLENE
HYPALON S-2
2 HOURS EXPOSURE

50-XIV - EXON XR-61 - 2 [illegible] EXPOSURE

[illegible]

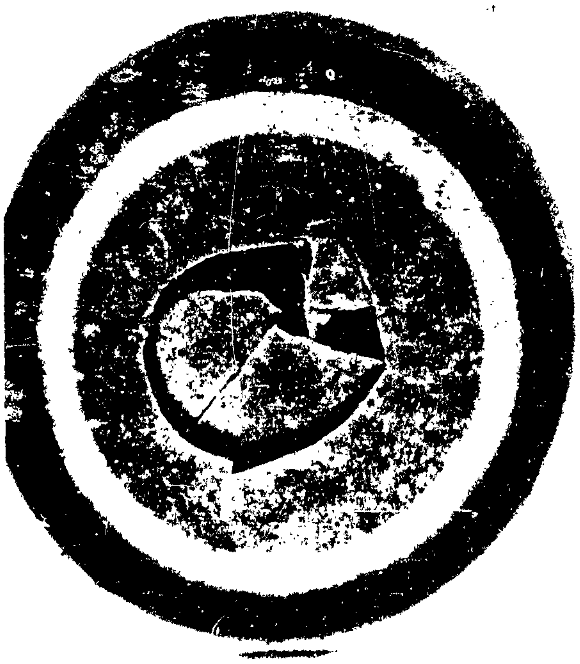


[illegible]

Figure No.29 Base Resin Exposures to WFNA Polyisobutylene, Polyethylene, Vinyl Chloride. Hypalon S-2

50-I- POLYISOBUTYLENE - VISTANEX B-120
6 DAYS EXPOSURE

50-II- POLYETHYLENE - DYNJ
6 DAYS EXPOSURE



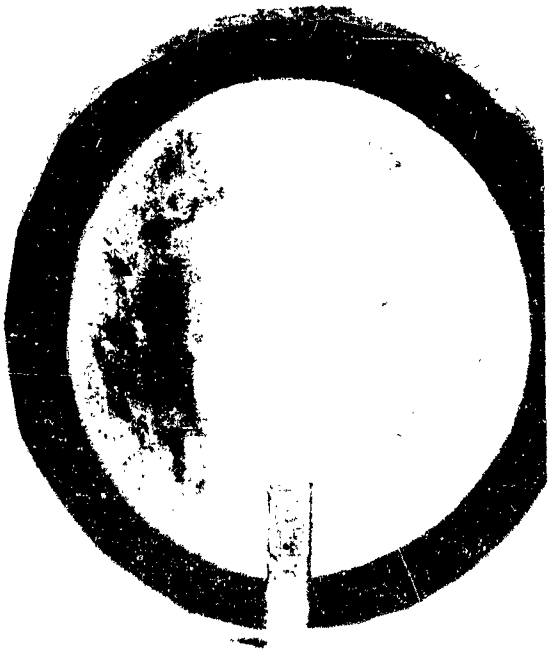


Figure No. 30 Vistanex: Polyethylene Compound-Compound 34

Figure No. 31 Vistanex: Polyethylene Compound- Compound 35

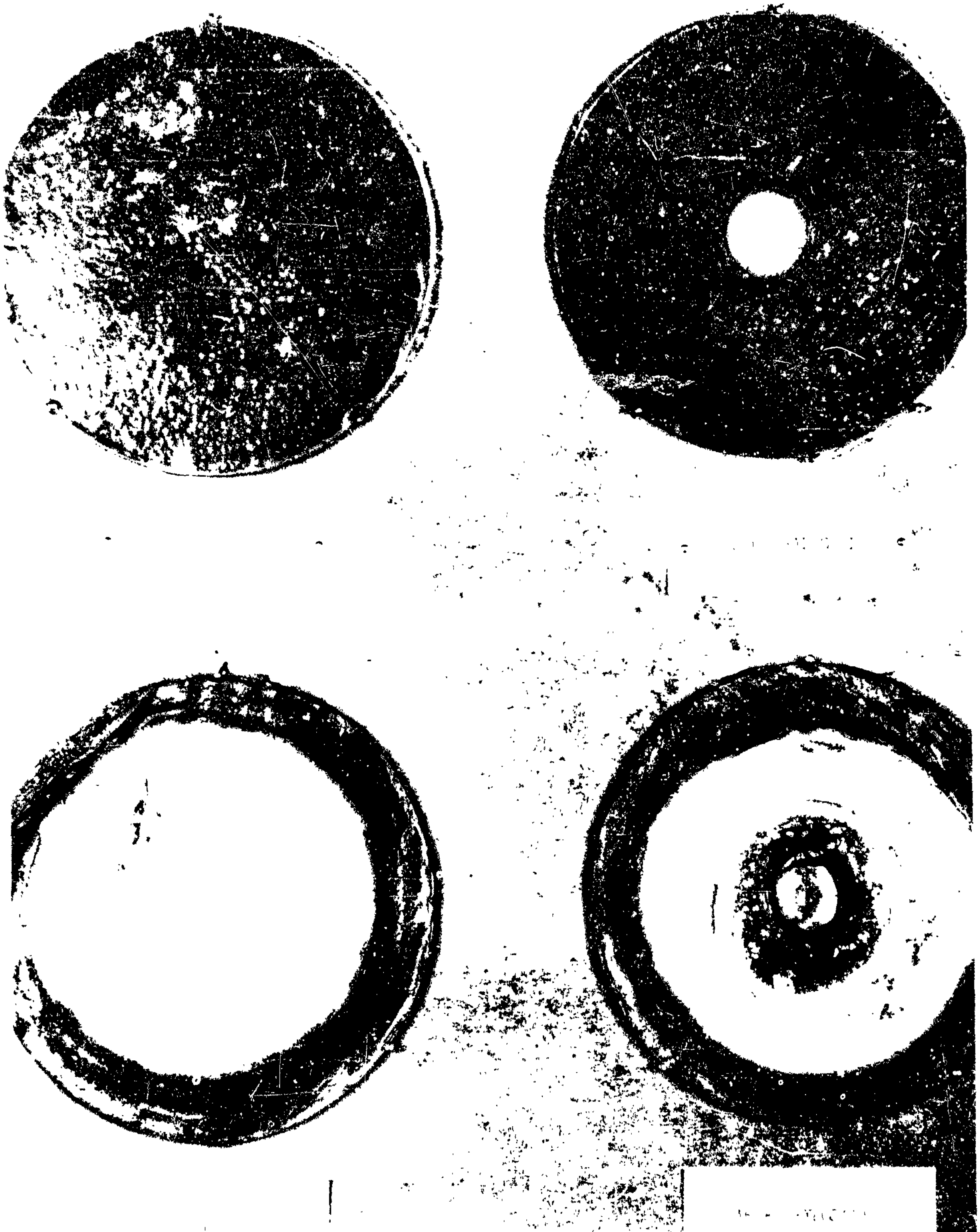


Figure No. 32 Pfaudler Column Disassembled