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WATER IMMERSION TESTS OF UF_6 CYLINDERS WITH SIMULATED DAMAGE

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ABSTRACT

A series of water immersion tests of UF_6 cylinders with simulated damage was conducted to determine the extent of water inleakage. The uranium hexafluoride content was less than normal enrichment. Metal and glass cylinder units were used and metal-to-glass cylinder connections were also tested. In openings which occur where UF_6 has solidified on the metal container walls or across the valve, an insoluble plug of monohydrated UO_2F_2 and metallic products is formed. This results from the reaction of the UF_6 and water and the reaction of the resultant HF with the metal. Water will enter a void area if UF_6 is not immediately available for reaction. Similar plug formations do not occur in Laboratory tests using glass tubes.

Introduction

There has been a lack of valid data relating to the effects of small leaks in UF_6 cylinders when the cylinders are immersed in water. Some conclusions about such an occurrence have been based on a single laboratory test tube experiment. The effect of leaks occurring while a cylinder is exposed to air has been largely assessed through operational experience.

Because of this limitation in the knowledge of reactions resulting from accidental leakage, particularly that occurring while a cylinder is immersed in water, a series of both field and laboratory tests were conducted. This report describes the tests and presents a firmer base for definitive evaluation of the effects of UF_6 cylinder leakage.

Summary

The test data indicate that for metal cylinders in which UF_6 is condensed on the walls or across the valve, water inleakage will not occur. An insoluble plug of metal compounds and the monohydrate of UO_2F_2 forms immediately as a result of the UF_6-water-HF-metal reactions. However, inleakage of a sizable quantity of water into the void zone of a damaged UF_6 container is possible if the UF_6 is not in sufficient depth on the wall or valve surfaces.

The data also indicate that conclusions based on laboratory tests using glass containers of UF_6 are not applicable to metal containers.

FIELD AND LABORATORY TESTS

A. Field Immersion Tests of UF_6 Cylinders

The field tests were conducted with MD cylinders constructed of 99% nickel, each 12-in. diameter x 40-in. long, and filled with depleted UF_6 to the normal fill weight of 450 pounds.[1] Some of the tests were conducted at a pond location and the remainder in a tank containing about 800 gallons of water. The tank, 72 x 44 in. and 50 in. deep, was positioned near a water supply, and several hundred feet from the nearest personnel location.

Test I

A cylinder containing liquid UF_6 was cooled in a horizontal position until the UF_6 was solidified. The cylinder was then suspended by

[1] E. L. Keller, et al., Uranium Hexafluoride, Handling Procedures and Container Criteria, USAEC Report ORO-651, Rev. 1, 1967.

slings from a hoist, valve end downward. Normally, cooled cylinders are at approximately 2 psia, but, when the valve was opened, no inrush of air was noted, this indicating either the presence of solid UF_6 across the valve opening, a UO_2F_2 plug in the inlet line as a result of wet air inleakage, or that the cylinder had leaked to atmospheric pressure. The cylinder was lowered into the pond until half submerged. Small white bubbles of HF containing what appeared to be undissolved UO_2F_2 rose to the surface, decreasing in frequency and ceasing in about 20 minutes. After two hours, the cylinder was removed from the water, minor water drainage was noted, and weighed. The weight was essentially unchanged, indicating very little hydrolysis of the UF_6.

Since the valve appeared plugged with reacted material, the valve stem was removed and the cylinder again lowered into the water. The valve port opening was 3/8 inch. Only minor bubbling was noted. Several hours later, reweighing of the unit again indicated only a negligible loss of content. The valve was removed and a yellow-green plug was found directly below the valve connection. The plug appeared to be UO_2F_2 and some metallic compounds formed by HF reaction. Later analysis confirmed metallic contamination as shown in Table I; the UO_2F_2 was probably the relatively insoluble monohydrate.[2] With the valve removed, leaving an opening of 1 in., the cylinder was returned to the water and left suspended for about 60 hours. Examination on removal revealed that the plug was still in position, but a weight check indicated a slight gain because of water absorption.

Normally, MD cylinders are filled with liquid UF_6 in a vertical position and cooled in air in a horizontal position. The fill limit and the handling procedures probably caused some UF_6 condensation across the cylinder valve, thus providing an immediate reaction zone which prevented water entry.

Test II

Since there are occasions when cylinders are filled or heated and cooled in a vertical position, the above conditions were considered as possibly not applicable. Hence, one of the cylinders was filled and cooled in a vertical position, however, prior to use, it was found to be incompletely cooled. Since it was transferred in a horizontal position, the cylinder was immersed vertically in the tank of water at 74°F for overnight cooling. After cooling, it was lifted until the valve could be opened in air, and was then immediately submerged in the water. Bubbling occurred as in Test I, but stopped within 30 minutes. The valve stem was removed under water, and some additional bubbling was noted, probably as a result

[2] J. J. Katz and E. Rabinowitch, The Chemistry of Uranium, pp. 569-576, McGraw-Hill, New York.

of the cylinder being jarred in the process. The unit was then lifted from the tank and tilted for drainage but only a few cm^3 of solution were drained. On insertion of a thin steel rod through the valve opening, a hard formation was detected directly below the valve connection. Little penetration was achieved by tapping the rod.

The cylinder was placed upright in a metal frame storage cage and the valve removed, disclosing a yellow-green solid material directly below the valve opening. Because of the hardness of this material, a sample was taken by drilling. About 1/2 in. below the surface, the drilling exposed an orange band, probably UO_2F_2 formed at higher temperatures, about 250°C due to chemical reaction. UF_6 fumes were observed escaping from the cylinder after it had remained in the sun for about 1/2 hour, and the valve was replaced. After remaining in the sun for several hours the cylinder was stored vertically overnight in a cooler and resampled the following morning. The crust formation was readily broken with a glass thief which penetrated nearly to the bottom of the cylinder. The valve was replaced after the sampling operation. The analysis results from this composite sample are also shown in Table II for comparison with the plug analysis. The high concentration of tin shown is considered to result from chemical reaction of the HF with the valve threads which are tinned to provide a leak-tight connection.

The cylinder, at atmospheric pressure, was returned to the tank and positioned upright under water and the valve removed. Considerable bubbling occurred for a few seconds, mostly air displacement, followed by a gurgling roar as solution was blown from the cylinder to a height of about three feet. It is possible that the sampling hole did not refill with UF_6, but filled with water. Since UF_6 is a poor thermal conductor, and particularly so with a substantial bed width, the heat of chemical reaction was not readily removed. The reaction stopped almost immediately and only a few HF bubbles continued to rise. The cylinder was then inverted and left under water. Sixteen hours later the contents were completely dissolved.

Test III

An MD cylinder at about 2 psia pressure and with the UF_6 solidified in a horizontal position was submerged horizontally a few inches below the water level and the valve stem removed. At water contact with the UF_6, a few bubbles of HF were generated. The rate of bubble generation altered as the cylinder was lowered or raised in the tank; at about a 5-ft. depth, the water and gas pressures were equalized. The depth of the cylinder was changed several times so that bubble release rate could be observed at various levels. There appeared to be very little UO_2F_2 solution draining from the cylinder at any position.

Laboratory Report

Table I. Plug Analysis

Volatile				Nonvolatile			
Element	ppm	Element	ppm	Element	ppm	Element	ppm
Sb	30	Mn	200	Ag	5	Fe	~ 5000
B	< 1	P	800	Al	> 200	Mg	2000
Co	< 1	Si	< 2	Ba	< 10	Na	200
Cr	10			Be	< 1	Ni	200
				Bi	< 1	Pb	3000
				Cd	10	Sn	< 10000
				Cu	5000	Zn	700

Table II. Filled Cylinder Analysis

Volatile				Nonvolatile			
Element	ppm	Element	ppm	Element	ppm	Element	ppm
Sb	< 15	Mn	< 1	Ag	2	Fe	100
B	< 1	P	< 50	Al	20	Mg	100
Co	< 1	Si	3	Ba	< 10	Ni	100
Cr	5			Be	< 1	Pb	2000
				Bi	< 1	Sn	1000
				Cd	< 1	Zn	< 20
				Cu	2000		

After about 40 minutes, the cylinder was removed, tilted, (only a very small amount of water was drained) and placed in a cage in the sun. The valve was removed disclosing a plug below the valve opening. This material also had an orange band at a depth of about 1/2 inch. As soon as UF_6 fuming occurred, the valve was replaced.

Test IV

Since the UF_6 in the MD cylinders had been cooled in a manner that apparently aided in the formation of a plug at the valve connection, it was decided to make a comparison test with a small monel Harshaw cylinder, 3 1/2 in. diameter x 7 5/8 in. long, in which 4.8 lb. of UF_6 had been solidified in the lower section, opposite the valve. The cylinder, at a vacuum, was immersed in the tank vertically with the valve upward and the valve was opened under water. Numerous bubbles of HF escaped for several minutes, then formed only infrequently. The valve was removed and a quantity of solution, approximately half the cylinder volume, was emptied into a sample bottle. Analysis of this solution indicated a uranium concentration of 216 g/liter. The remaining content of the cylinder dissolved when the unit was immersed for a few hours in an inverted position.

Test V

The plugging effect when UF_6 was solidified across a cylinder valve was additionally investigated using two small Harshaw cylinders. The UF_6 was solidified in each case with the cylinder inverted, and was thus against the valve. One cylinder, with the valve end downward, was suspended over a 5 gallon bucket of water. The valve was opened in air, and the unit then 50% immersed. The second unit was similarly immersed in another bucket, and the valve opened under water. There was only minor chemical reaction in both instances. The cylinders were left immersed for two hours, then taken from the water and the valves removed. Both valves were plugged with the yellow-green material. Similar plugs were observed just under the valve connection, with an orange band about 3/8 in. below the surface.

With the valves removed, the cylinders were replaced in the water, valve end down. Twenty-four hours later no additional material had dissolved. The valve connection opening in a Harshaw cylinder is 5/8 in. diameter.

B. Laboratory Test - UF_6 Cylinders

Test I

To determine the extent of plug formation resistance against agitated water, a Harshaw cylinder filled as in A-V had the valve removed under water. Plug formation occurred shortly. The water was then agitated for 10 minutes by a motorized stirrer. Neither this method nor the action produced by a stream of air bubbles was able to dislodge the plug.

Test II

The field test of the Harshaw unit (A-IV) was repeated with a similarly filled and evacuated cylinder. It was immersed vertically in water, valve end up, and the valve fully opened. After several minutes, during which HF bubbles containing UO_2F_2 escaped at a rapid rate, small amounts of solution were observed being forced from the valve, which has a port of 1/8 inch. The cylinder was removed, the valve closed, and a weight gain of 389 g was measured.

In both cases, the Harshaw cylinder was first filled by the condensation of UF_6 vapor. The cylinder was warmed in a vertical position to liquify the UF_6 and then cooled so that the UF_6 was opposite the valve. Little or no UF_6 is condensed by this method across the valve opening or on the walls. Thus, water entry to the void space of the evacuated cylinder was readily made.

Following evacuation of the water, the cylinder wall temperature climbed rapidly due to the heat of reaction until the cylinder could not be held in the bare hand. The unit was placed in an ice bath, and for several days thereafter HF continued to accumulate as evidenced by gauge pressures prior to cylinder evacuation.

Test III

To simulate a small crack which might occur at the valve from an accident prior to entry into water, a Harshaw cylinder with a vacuum-pressure gauge connected to the valve was filled as above (B-II). After the unit was weighed, the cylinder valve was partially opened in air. Two minutes were required for a change in the gauge reading from 25 in. of vacuum to atmospheric pressure. The cylinder was then positioned vertically in a large jar of water (76°F) with the valve about 1 1/2 in. below the surface to determine whether the water would be able to enter and displace the air, or if HF formation would prevent significant water entry. Over a period of several minutes only a few bubbles were noted escaping; the gauge pressure fluctuated between 22 and 18 in. of vacuum, which is considered indicative of HF formation and its absorption in entry water. The valve was opened farther and the bubble rate accelerated to a timed rate of about 172/minute. The gauge indicated a pressure slightly above atmospheric at this time, dropping to atmospheric pressure as bubble formation decreased. The valve was then fully opened. The rate of bubble formation increased slowly until, in about 5 minutes, rapid gas evolution occurred, principally HF containing UO_2F_2 and probably some residual air.

To avoid a possible violent reaction such as that which occurred in the MD cylinder tank test, the Harshaw cylinder was removed from the water, packed in ice, and the valve removed. A plug found in the neck of the cylinder was readily broken free. Unlike the yellow-green UO_2F_2-metallic compound previously found, it was gray-white and appeared to be a mixture of UO_2F_2 and condensed UF_6. It is probable that, when small amounts of water entered, the heat of reaction may have caused adjacent UF_6 to vaporize and condense in the narrow water-cooled neck of the cylinder, thus producing an immediate reaction zone. A weight measurement indicated a gain of only 6.0 grams.

Test IV

The above test was repeated at a later date. With the valve opened slightly, pressure gradually increased from 25 in. of vacuum to 15 in. in about 15 seconds. The cylinder and valve were then immersed in water, valve upright. The pressure increased slowly to 13 in. of vacuum, where it remained stationary. No bubbles were emitted. The valve was additionally opened and the pressure went to zero, then to 2.5 psi. Air or HF bubbles were vented from the cylinder. In the next two minutes the pressure increased to 3.5 psi and an increase in bubbling was noted. The gas evolution slowly decreased and about five minutes later ceased; the gauge pressure was 1.5 psi. It appeared that a plug at the valve port had formed.

The valve was then fully opened, and the pressure decreased immediately to 15 in. of vacuum - then rapidly increased to zero. Apparently, the plug was removed on water entry. HF bubbles containing UO_2F_2 were released, but slowly, indicating water entry had resulted in solution formation, the water first dissolving the HF and causing a rapid pressure decrease. No change occurred on tapping the unit so the valve was closed, the cylinder removed and placed in dry ice. A decrease in pressure due to cooling occurred from 0 to 12 1/2 in. of vacuum within a 10-minute interval. The unit was reweighed and a weight gain of 467 g noted.

The valve assembly was removed and examined, but no plug was found in the gauge, the valve, or the neck of the cylinder.

The cylinder was placed in a jar of warm water for five minutes, after which, the solution was poured into a sample bottle. Volume obtained was 375 cm^3. The solution weighed 508 g, and the uranium concentration was 267 g/l. After removal of the solution, UF_6 fumes were emitted from the cylinder. The cylinder contents were removed on inverting the unit in a jar of water.

Test V

The previous tests have shown that for cylinders in which the UF_6 has been solidified at the valve end, an apparently insoluble plug is formed at this location on opening of the valve in either air or water. It was questioned as to whether the same condition would result if a crack were to occur in a cylinder wall at the solid UF_6 zone.

To simulate this accident, a 1/8 in. hole was drilled in the side of a filled Harshaw cylinder in the UF_6 zone. The hole was immediately covered on release of UF_6 and the cylinder promptly immersed in water. A few bubbles and some UO_2F_2 formed and dissolved, but the opening promptly plugged and the UF_6 was contained. Jarring dislodged some material but subsequent reaction was followed by additional plugging. Agitation of the water failed to alter this condition. A 1/4-in. hole was then drilled in the base of the cylinder and the test repeated. Similar results were obtained.

C. Laboratory Tests - Glass Tubes

Although it was considered unlikely that similar plugging conditions would occur with glass containers, such containers of UF_6 would enable reaction observations. Several 1.0 in. diameter, thick-walled glass tubes were therefore 2/3 filled with solid UF_6. One end of each tube was drawn to a length approximating that of a valve and sealed; each tube was at a vacuum. Four tubes were used in each test, with drawn ends having inner diameters of 1/16, 1/8, 1/4, and 1/2 inch. The sealed ends were broken under water in all cases.

Test I

The tubes were arranged in vertical positions, stem upward, and the stem tip broken. Water entered the tubes, forming yellow UO_2F_2 solution. Some HF evolved. Dissolving action continued until the solution was saturated; the rate of dissolution being commensurate with the tube openings. A layer of undissolved UO_2F_2 with an orange band about 1/4 in. below the surface then formed above the undissolved UF_6. This condition remained unchanged five days later. The concentrated solution in each tube remained static, and no change in the level of the material was evident. These results conform with that of a similar, single laboratory test made a number of years earlier.

Table III. Glass Tubes - Upright

Diameter Opening (in.)	Weight UF_6 (g.)	Height of UO_2F_2 (in.)	Height of UF_6 Remaining (in.)
.0625	100.7	1.06	3.25
.125	92.0	1.25	4.0
.25	74.0	.88	3.50
.50	98.8	.50	4.0

The tubes were removed and inverted in the jar of water. Over a long period (hours) the material slowly reacted and dissolved. Bubbles of HF which formed in the tubes dissolved or vented very slowly, delaying the action. The ends of the tubes were about 6 inches below the surface of the water.

Test II

One set of tubes was immersed with the stems downward. On water entry, the content of each tube reacted and the UO_2F_2 solution thus formed flowed by gravity from the tube and was replaced with fresh water in a continuous cycle until all the material had reacted. The displacement rate varied with the opening diameter. These results were also comparable to an earlier laboratory test.

Table IV. Glass Tubes - Inverted

Diameter Opening (in.)	Weight UF_6 (g.)	Time For Complete Reaction (min.)
.0625	92	120
.125	81	95
.25	— (tube broke)	—
.50	100.8	31

Test III

The third set of tubes was positioned horizontally on the bottom of a jar of water. UO_2F_2 solution was formed quickly, but considerably longer intervals than in Test II were required for complete dissolution. The tubes with small openings had undissolved material after several days. However, when the tubes were tilted slightly downward, the contents dissolved. Some solution was retained in the tubes and appeared quite concentrated, judging by the color.

In all the above tests, UO_2F_2 only was formed, no green-colored compounds were present as in the case of metal cylinders. In the upright and horizontal tests, where dissolving had ceased because of tube position, the orange band formation was visible below the yellow UO_2F_2 layer.

Test IV

Since the various metal cylinders containing UF_6 at the valve end had plugged immediately when the UF_6 came in contact with water, a similar test was performed with glass tubes in which the UF_6 was solidified at the drawn end. Two tubes were immersed in water, drawn end down, and the tip broken. No plugging occurred. The content of one tube dissolved rapidly, and that of the second dissolved very slowly because small bubbles of HF united to form one large bubble which tended to hold water out of the tube. Gradual moisture penetration along the wall of the tube occurred and the resultant HF formation finally produced sufficient pressure to permit the bubble to escape. After a few hours, the addition of more water within the tube, crumbling of the UO_2F_2 cake as a result of small HF bubble pressure, and solubility gradients caused all the material to dissolve.

D. Laboratory Tests - Glass-Metal Cylinders

To further validate the assumption that plugging action in metal cylinders is caused by a chemical reaction of the HF and metal whereby both insoluble metal products and the relatively insoluble monohydrate of UO_2F_2 are formed, metal valves were connected to 1.0 in. diameter glass tubes of UF_6, and the following tests were conducted:

Test I

A tube was drawn at one end to a 1/4 in. opening which was fitted with a brass coupling and valve; O-rings were used as seals. The UF_6 was solidified at the drawn end of the tube. With the valve opened under water, drawn end of tube in downward position, some dissolution of the UO_2F_2 formed by the reaction occurred, but ceased in about eight minutes. The tube was tilted and the open end moved near the surface of the water. No bubbles were noted, but the jarring of the tube during its return to the original position caused some dissolution and HF bubbles formed for about three minutes. No orange discoloration was observed in the glass tube. Stirring of the water mechanically produced no dissolving action. If any dissolution took place in a 40-minute period, it occurred within the metal coupling.

The tube was taken from the water, and the metal section was removed. In the process, the glass broke at about the O-ring level. Smoking of the UF_6 was noted, indicating that a negligible quantity of water had penetrated to the glass zone. In the removal of the solids from the metal inlet section of the valve, an orange band was found at a depth of about 1 1/2 inches. The glass tube was replaced in the water, drawn end downward. The contents dissolved in a few minutes.

Test II

A 1/4-in. diameter monel tube was fastened with a brass coupling to the 1/4-in. diameter opening of a 1.0-in. diameter glass tube of UF_6. The tube was placed in water, drawn end down, and the sealed end of the monel stem cut off. Some dissolving of the UF_6 occurred, but the reaction stopped in about 18 minutes.

The monel section was removed from the tube, leaving the brass coupling. The upper end of the monel tube, part of the brass coupling, and the adjacent section of the glass in the coupling section were plugged with yellow-green material. The glass tube with attached coupling was replaced in the water, narrow end down. Some slight dissolution occurred but a narrow orange band formed in the glass tube just above the coupling, and dissolution stopped.

Test III

As a final check on the effect of container materials and of the size of cylinder opening, a 1-in. diameter flanged monel sample tube was filled with UF_6 which was condensed at the flanged end. The flanged end was removed, and the cylinder immersed in water, open end down. Plugging occurred almost immediately with negligible loss of material. Both water agitation by stirring and a stream of air bubbles directed against the opening failed to alter this condition.

E. Special Test

This test was performed to determine, over a prolonged period of time the dispersal of UO_2F_2 in static water on immersion of a ruptured UF_6 cylinder. A 3 1/2-in. diameter glass bottle containing about 2.2 lb. of UF_6, with one end drawn to a 1/2 diameter sealed tube was positioned in a 6-in. diameter x 48-in. high lucite plastic tank filled with water. The bottle was at a vacuum. The top of the tube was broken and the bottle rapidly lowered to the bottom of the tank. A concentrated UO_2F_2 solution formed in the bottle. A yellow layer of undissolved UO_2F_2 containing an orange band of material formed above the unreacted UF_6. Molecular dispersion of the solution to the tank water was negligible after several days. After a three-week interval HF reaction with the glass near the top of the solid uranyl fluoride layer resulted in a leak. This enabled the solution to disperse throughout the lower part of the tank. No change in the UF_6 bed depth was noted.

Conclusions

Several conclusions may be drawn from these tests:

1. Accidents which result in UF_6 cylinder openings up to 1 in. diameter in areas of UF_6 deposition are unlikely to create a hazardous con-

dition either in air or water. The HF formed by chemical action of water and UF_6 will react with the metal of the cylinder valve or wall and produce compounds which, in conjunction with the monohydrated UO_2F_2, will cause the opening to plug and prevent further water entry.

2. Jarring of the cylinder may cause some dislodging of portions of the plug, but new, insoluble material is immediately formed.

3. Action of water currents, as in a brook or river, is unlikely to dislodge the plug.

4. Water entry into cylinders which are at atmospheric pressure prior to immersion as a result of valve leakage or valve damage will be negligible for small openings. Plug formation will occur in the valve or the cylinder neck zone.

5. Water entry into the void area of a damaged cylinder at vacuum pressure could occur in sizable quantity unless the leak is very small and at a location where plugging could readily occur, such as a valve leak. This assumes no UF_6 deposit on walls or valves.

6. Laboratory tests with UF_6 in glass containers provide data which are not applicable to metal containers of UF_6.

Acknowledgments

The field test operations were conducted by members of the Industrial Uranium Transactions Department, Production Division, Mr. E. J. Culbert, Supervisor. The laboratory tests were conducted by the Works Laboratory, Technical Division, Mr. R. J. Wertz, Supervisor. The Photography Department, Finance and Materials Division, Mr. J. Edwards, Supervisor, provided prompt and excellent service on short notification.

The efforts of these groups to successfully conduct the various tests in a limited time are appreciated. We are particularly obligated for the technical assistance provided by Mr. Wertz in test preparation and evaluation, and in review of this report.

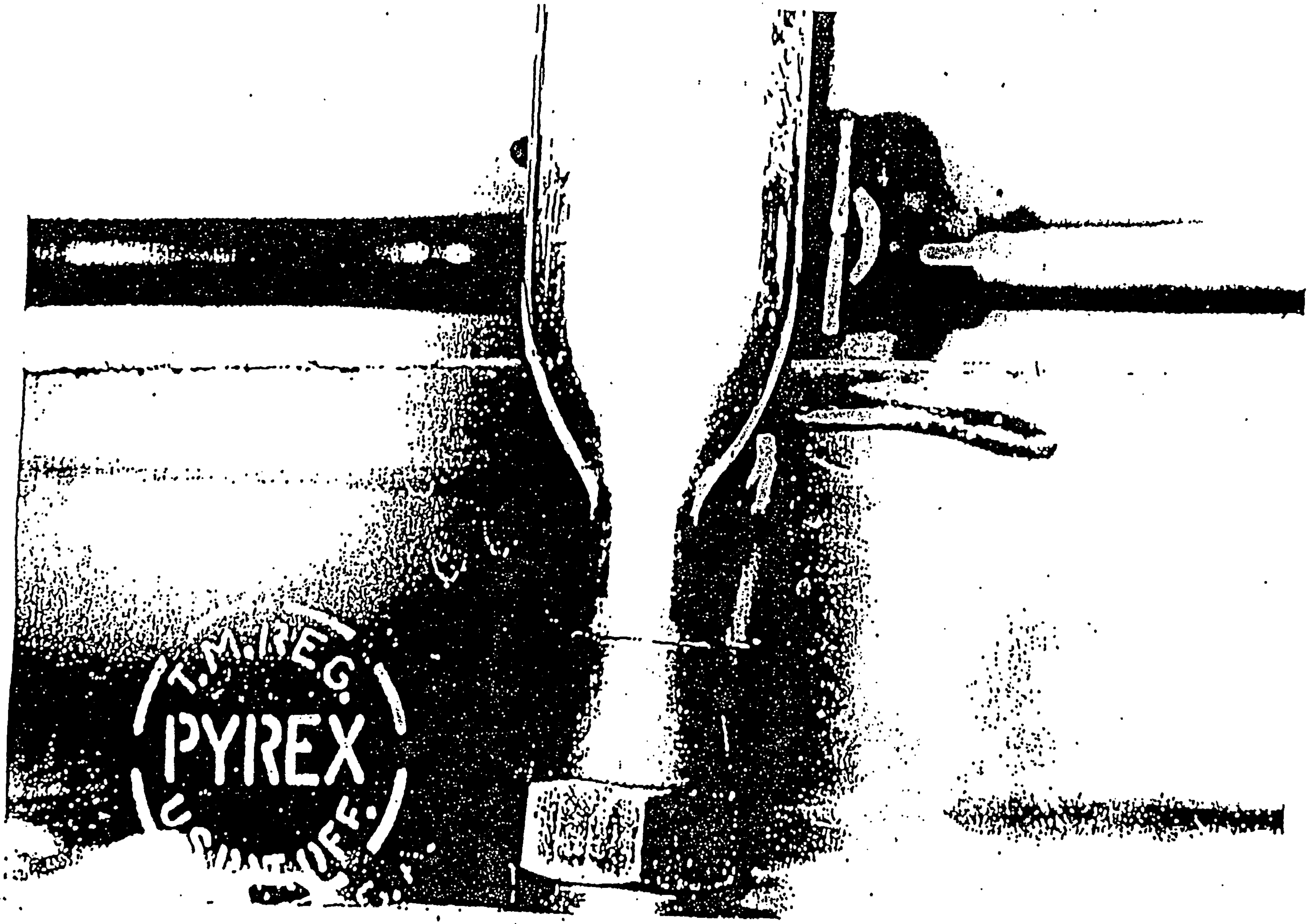
Appendix A

Water Immersion Tests of UF_6 Containers

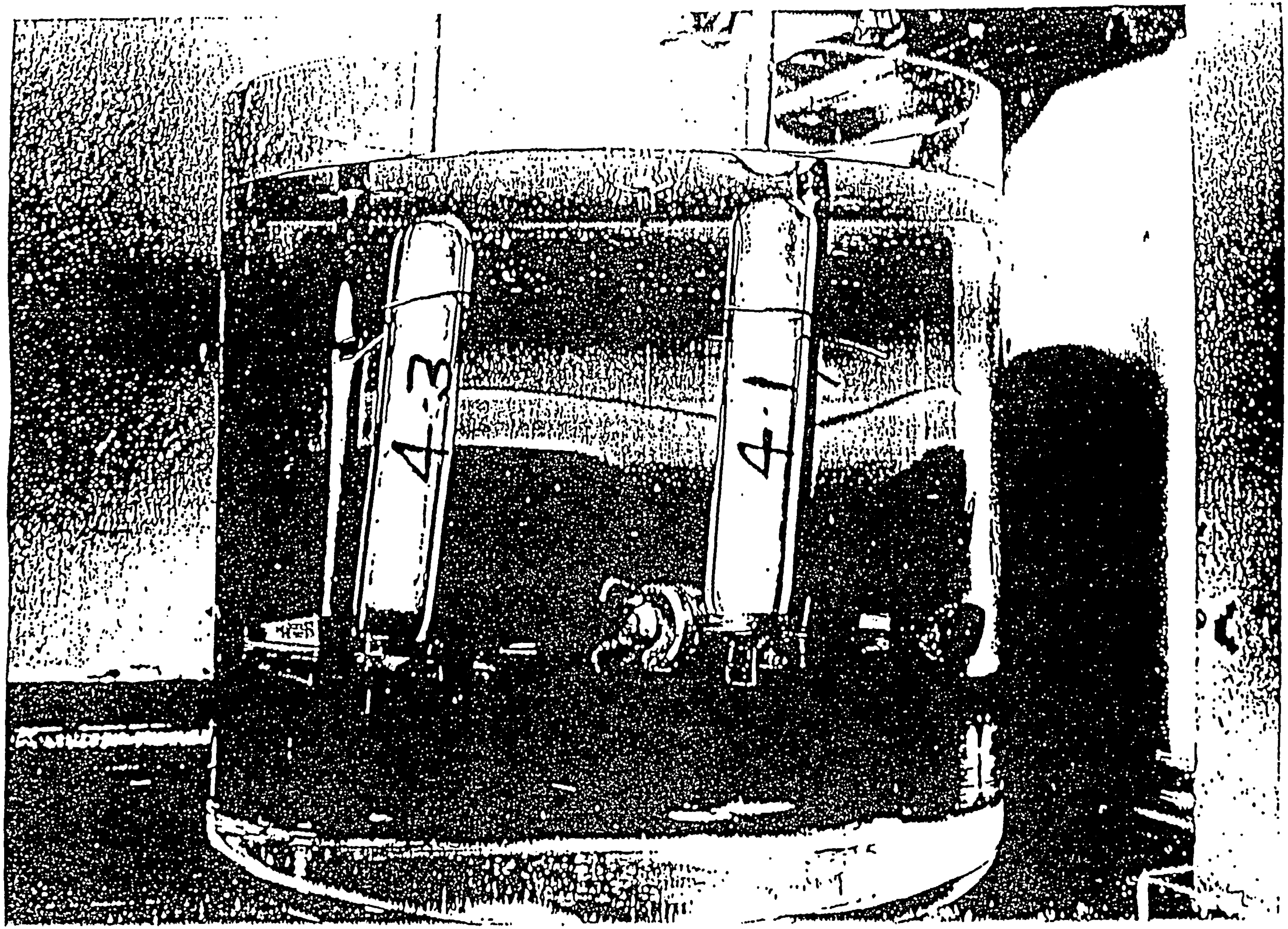
Photo Number	Title	Test
PE-67-825	Pond Test of MD Cylinder	A-I
PE-67-826	Tank Test of MD Cylinder	A-II
PE-67-827	MD Cylinder After Immersion	A-III
PE-67-841	Harshaw Cylinder - UF_6 at Valve End	B-I
PE-67-842	View of Plug Formation	B-I
PE-67-837	UF_6 Removal - Glass Cylinders	C-IV
PE-67-838	UF_6 Removal - Glass Cylinders	C-IV
PE-67-839	View of Orange Band Formation	D-II
PE-67-840	Special Test	E

SPECIAL TEST

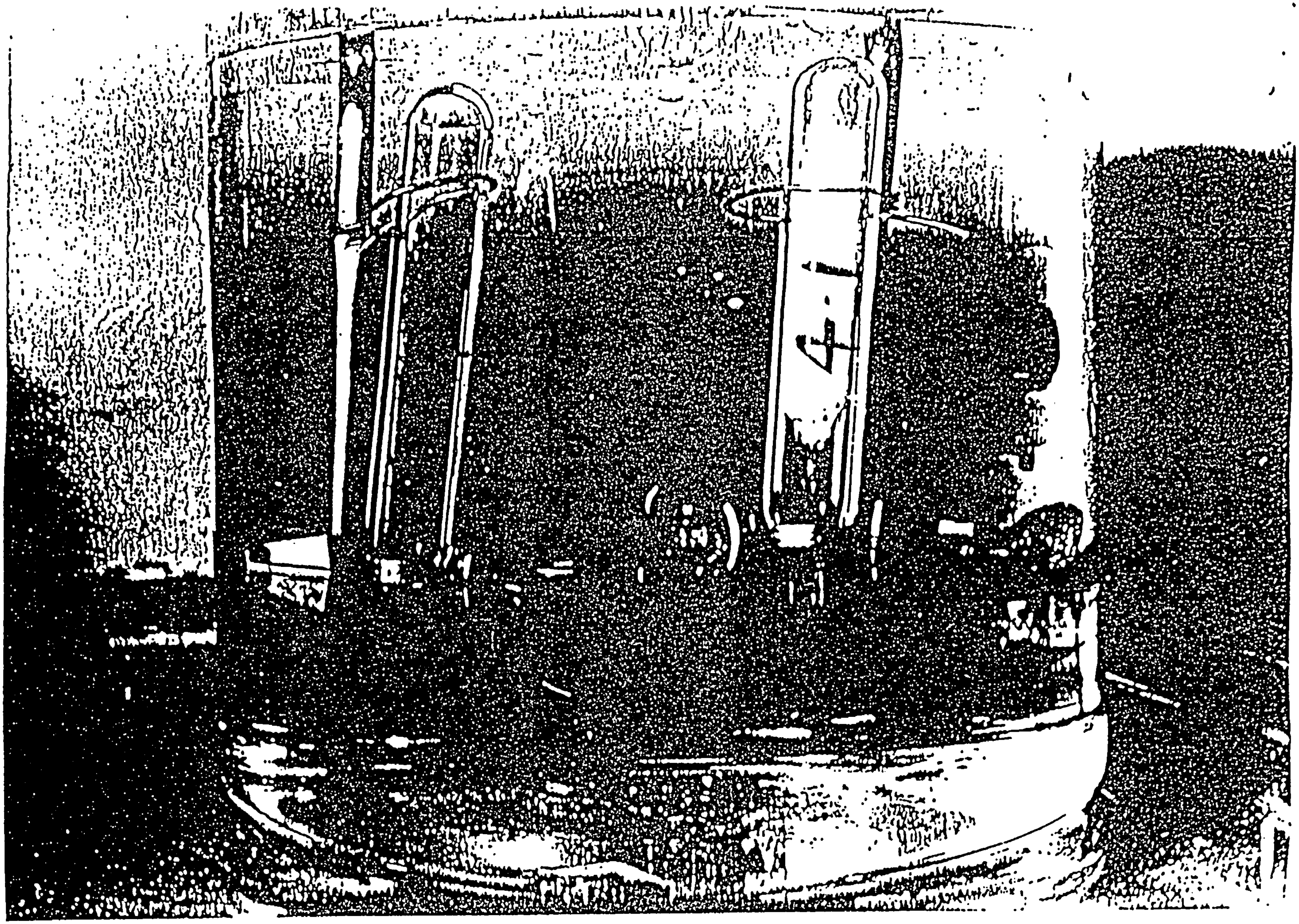
PHOTO NO. PH-67-839



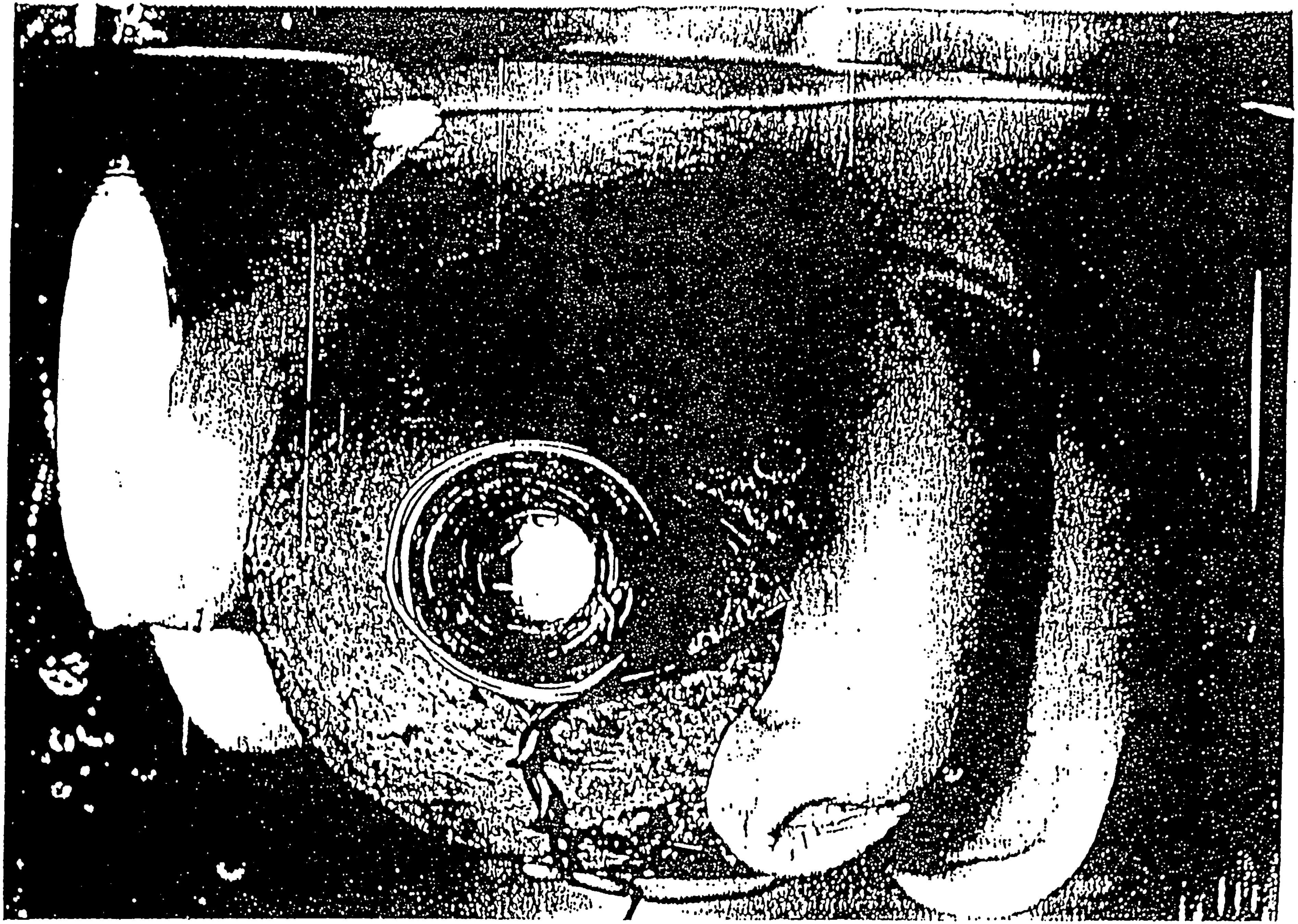
VIEW OF ORANGE BAND FORMATION



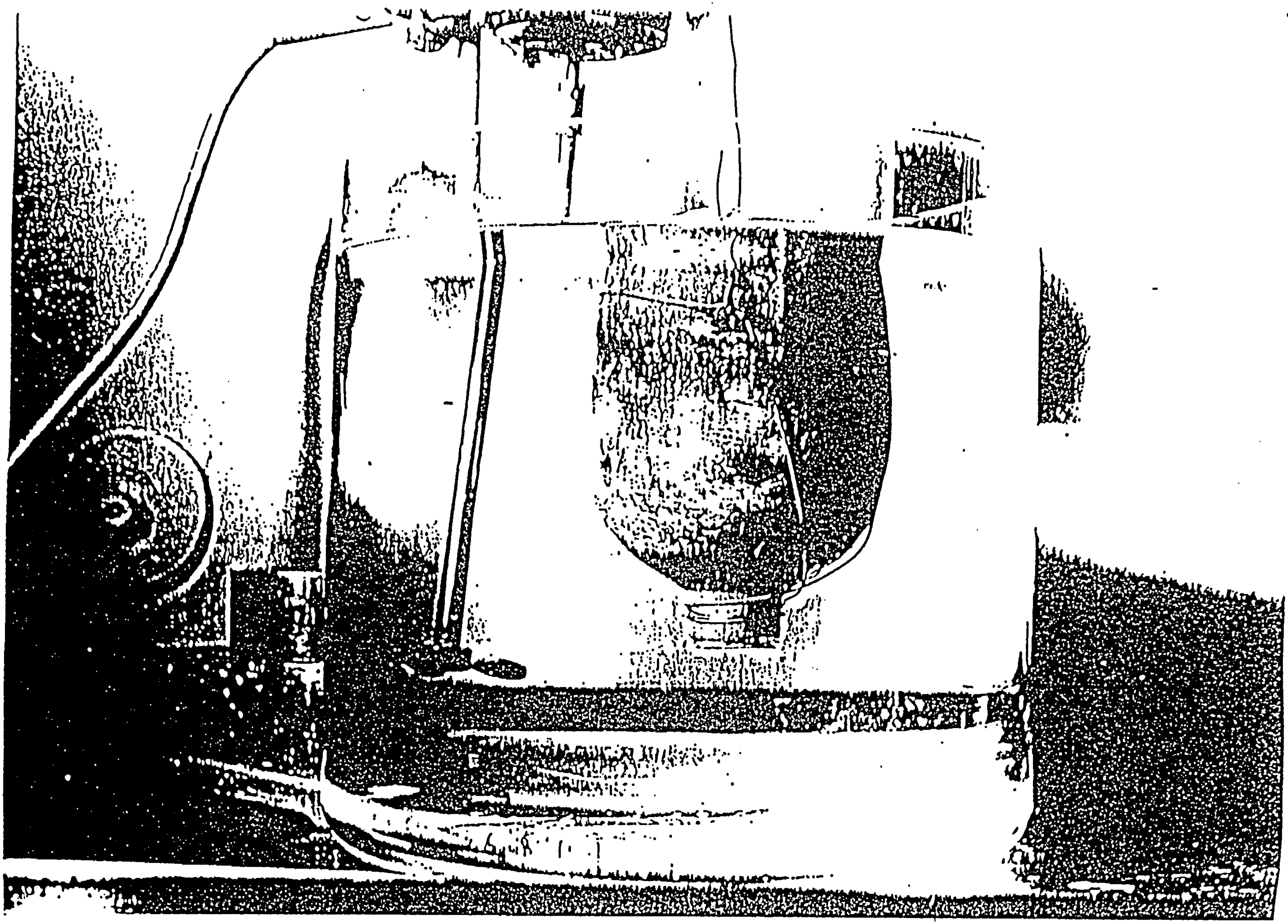
UF_6 REMOVAL - C [illegible] 5S CYLINDERS



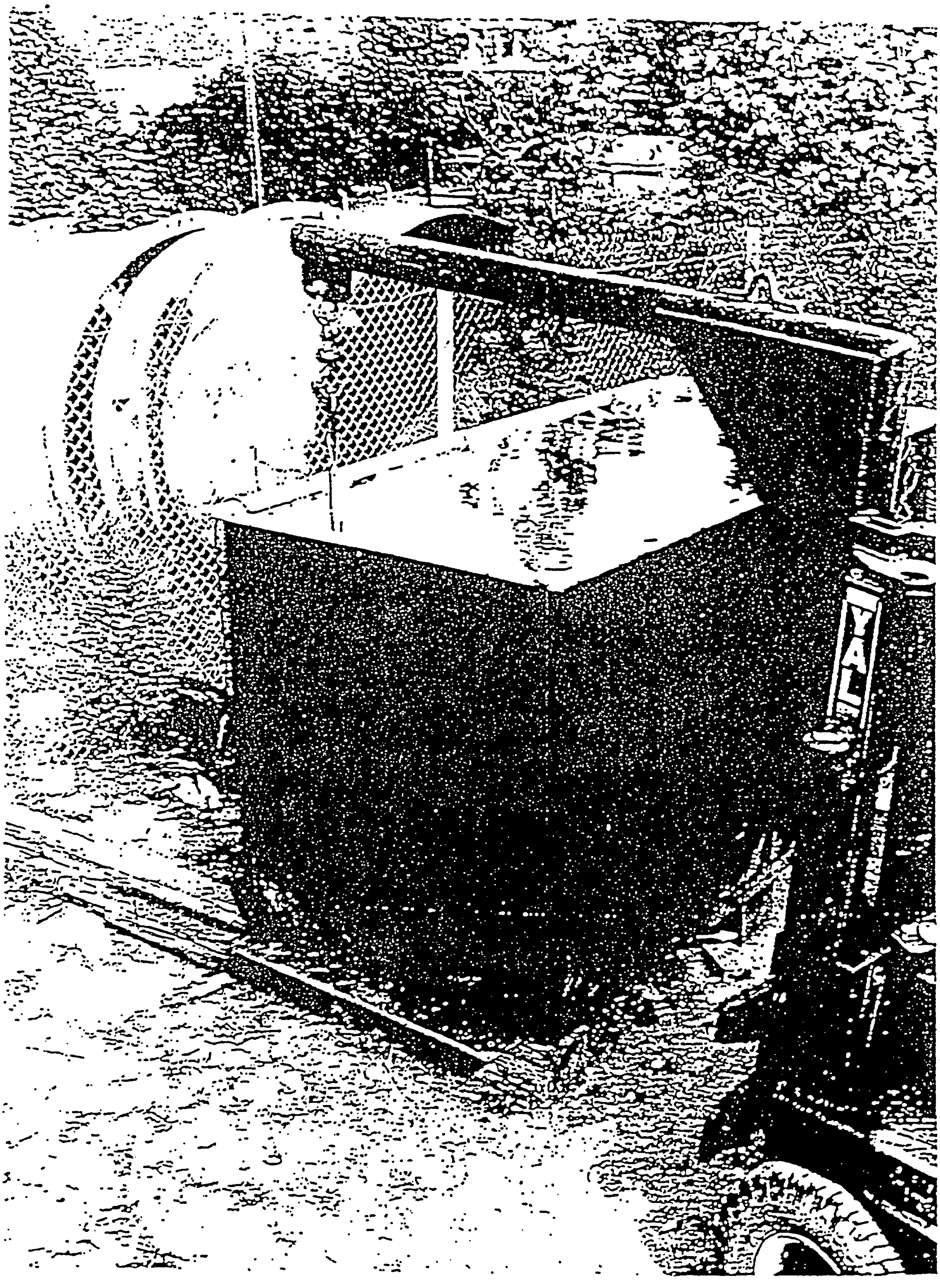
UF_6 REMOVAL - GLASS CYLINDERS



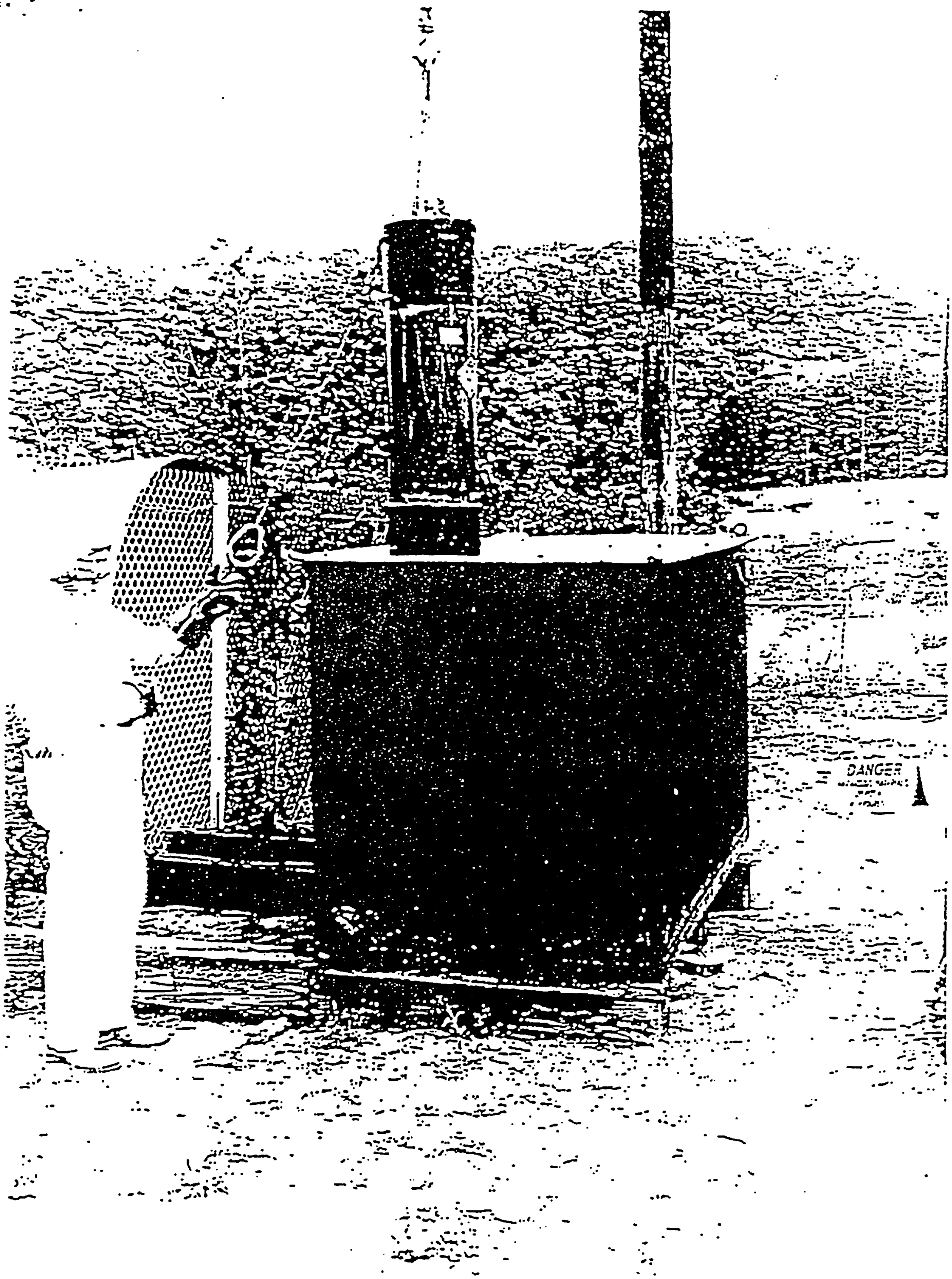
VIEW OF PLUG FORMATION



HARSHAW CYLINDER - UF_6 AT VALVE END



TANK TEST OF [illegible] CYLINDER



MD CYLINDER AFTER IMMERSION

POND TEST OF MD CYLINDER