

ACCELERATED CORROSION TEST FOR
METAL DRAINAGE PIPES

Final Report

by

Dr. James D. Garber, P.E.
Professor and Head of Chemical Engineering

and

Mr. Jong Her Lin, Instructor

Research Report No. 206

Research Project No. 86-1P(B)

Conducted by

DEPARTMENT OF CHEMICAL ENGINEERING
UNIVERSITY OF SOUTHWESTERN LOUISIANA
LAFAYETTE, LOUISIANA

"The contents of this report reflect the views of the authors who are responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official views or policies of the State or the Federal Highway Administration. This report does not constitute a standard, specification, or regulation. The Louisiana Department of Transportation and Development does not endorse products, equipment, processes or manufacturers. Trademarks or manufacturers' names appear herein only because they are considered essential to the object of this report."

June 30, 1987

SUMMARY

The Louisiana Department of Transportation in 1985 reported on a ten-year study which evaluated the corrosion resistance of 16 different types of drainage pipes at 11 different field locations in the state. With new culvert/coating combinations being produced frequently, it is essential that a short-term accelerated test be developed which will predict the corrosion resistance of various coatings.

This project chose to use electrochemical techniques to develop a laboratory test method. The electrical potential of the culvert was raised above its natural potential, and it was placed in a corrosive solution containing hydrogen ions, chlorides, and sulfate ions. Since it was found that the galvanized steel culverts contained a variable amount of zinc/in², and that some of the coatings can dissolve slightly into the test solution or can crumble off when the coupon is being handled, it was decided to use atomic absorption (AA) to measure the % zinc remaining on the coupon instead of simply weighing the coupon. Another technique which proved valuable in evaluating coupons exposed to the tests was the area method of measuring the % zinc remaining by using a planimeter. Both methods gave approximately the same results.

The best accelerated test method developed in this study was a high-pressure oxygen test (500 psi oxygen) at ambient temperature. The coupon was placed inside a 316SS Baroid cell, and every 24 hours the test solution was changed. After three days the coupon was removed. The % zinc remaining on the coupon after testing was found to be the basis of rating the coating. The coupon must be in electrical contact with the cell since there is a galvanic effect which occurs.

An alternative method which was developed involved the use of a potentiostat to maintain a 1-volt potential difference between two test coupons in the standard test solution. The method requires that the % zinc be determined from an area analysis since about 2,000 ml of test solution are used, and it is difficult to get good AA results when only a portion of the sample is digested.

Both methods were able to produce results which correlated with field results.

METRIC CONVERSION FACTORS*

<u>To Convert From</u>	<u>To</u>	<u>Multiply by</u>
<u>Length</u>		
foot	meter (m)	0.3048
inch	millimeter (mm)	25.4
yard	meter (m)	0.9144
mile (statute)	kilometer (km)	1.609
<u>Area</u>		
square foot	square meter (m ²)	0.0929
square inch	square centimeter (cm ²)	6.451
square yard	square meter (m ²)	0.8361
<u>Volume (Capacity)</u>		
cubic foot	cubic meter (m ³)	0.02832
gallon (U.S. liquid)**	cubic meter (m ³)	0.003785
gallon (Can. liquid)**	cubic meter (m ³)	0.004546
ounce (U.S. liquid)	cubic centimeter (cm ³)	29.57
<u>Mass</u>		
ounce-mass (avdp)	gram (g)	28.35
pound-mass (avdp)	kilogram (kg)	0.4536
ton (metric)	kilogram (kg)	1000.
ton (short, 2000 lbs.)	kilogram (kg)	907.2
<u>Mass per Volume</u>		
pound-mass/cubic foot	kilogram/cubic meter (kg/m ³)	16.02
pound mass/cubic yard	kilogram/cubic meter (kg/m ³)	0.5933
pound-mass/gallon (U.S.)**	kilogram/cubic meter (kg/m ³)	119.8
pound-mass/gallon (Can.)**	kilogram/cubic meter (kg/m ³)	99.78
<u>Temperature</u>		
deg Celsius (C)	kelvin (K)	$t_K = (t_C + 273.15)$
deg Fahrenheit (F)	kelvin (K)	$t_K = (t_F + 459.67) / 1.8$
deg Fahrenheit (F)	deg Celsius (C)	$t_C = (t_F - 32) / 1.8$

*The reference source for information on SI units and more exact conversion factors is "Metric Practice Guide" ASTM E 380.

**One U.S. gallon equals 0.8327 Canadian gallon.

TABLE OF CONTENTS

SUMMARY-----	ii
METRIC CONVERSION FACTORS-----	iii
LIST OF TABLES-----	vi
LIST OF FIGURES-----	vii
INTRODUCTION-----	1
SCOPE-----	2
LITERATURE SURVEY-----	3
General-----	3
Evaluation of In-Service Pipe-----	8
Accelerated Tests-----	11
EXPERIMENTAL RESULTS-----	15
Materials and Equipment Used in Testing-----	15
Potentiostat Test-----	16
High-Pressure Oxygen Test-----	27
Anodic Undercutting of Coupons-----	51
Examination of Culvert Metal and Coating-----	54
DISCUSSION OF RESULTS-----	59
Potentiostat Test-----	59
High-Pressure Oxygen Test-----	61
Comparison of Weighing, AA, and Area Analysis-----	65

CONCLUSIONS-----	69
RECOMMENDATIONS-----	72
BIBLIOGRAPHY-----	73
APPENDIX A -	
Photographs of Coupons After Exposure to 1-Volt Potentiostat Test-----	75
APPENDIX B -	
Photographs of Coupons After Exposure to High-Pressure Oxygen Test No. 2-----	82
APPENDIX C -	
Atomic Absorption Determination of Zinc and Iron in Solution-----	100

LIST OF TABLES

Table No.	Title	Page No.
1	1-Volt Potentiostat Test-----	20
2	2-Volt Potentiostat Test-----	26
3	High-Pressure Oxygen Test No. 1-----	30
4	High-Pressure Oxygen Test No. 1 (pH)-----	33
5	High-Pressure Oxygen Test No. 2-----	35
6	High-Pressure Oxygen Test No. 3-----	40
7	High-Pressure Oxygen Test at Elevated Temperature (130° F)-----	47
8	% Zinc Remaining on Coupons After Potentiostat Test-----	52
9	% Zinc Remaining on Coupons After High-Pressure Oxygen Test No. 2-----	54
10	% Zinc Remaining on Coupons After High-Pressure Oxygen Test No. 3-----	55
11	% Zinc Remaining on Coupons After High-Pressure Oxygen Test No. 4-----	56
12	Zinc Content of a 2" X 2" Coupon from Each Culvert Material-----	57
13	Comparison of % Zinc Remaining on a Coupon Using Atomic Absorption (AA) and Area-----	67

LIST OF FIGURES

Figure No.	Title	Page No.
1	Potential - pH Equilibrium Diagram for the Fe, Fe(OH) ₂ , and Fe(OH) ₃ System at 25° C-----	5
2	Potential - pH Equilibrium Diagram for the Fe, Fe ₃ O ₄ , Fe ₂ O ₃ System at 25° C-----	6
3	Potential - pH Equilibrium Diagram for the Aluminum - Water System at 25° C-----	7
4	Model 363 Potentiostat Used in the Testing-----	17
5	1-Volt Constant Potential Accelerated Corrosion Test-----	21
6	Complete Set-Up Required to Run the Elevated Potentiostat Test-----	22
7	1-Volt Constant Potential Accelerated Corrosion Test at 125° F-----	23
8	0.5 Volt Constant Potential Accelerated Corrosion Test in 5% HCl-----	25
9	Two of the Baroid Cells Used in the High-Pressure Oxygen Test-----	28
10	Cumulative Weight Loss in High Pressure Oxygen Test No. 1-----	31
11	Solution pH of High Pressure Oxygen Test No. 1-----	32
12	Total Weight Loss in the High-Pressure Oxygen Test No. 2-----	34
13	Cumulative Zinc Weight Loss in High Pressure Oxygen Test No. 2-----	37
14	Cumulative Iron Weight Loss in High Pressure Oxygen Test No. 2-----	38
15	Cumulative Zinc and Iron Loss in High Pressure Oxygen Test No. 2-----	39
16	Cumulative Weight Loss in High Pressure Oxygen Test No. 3-----	41

Figure No.	Title	Page No.
17	Cumulative Zinc Weight Loss in High-Pressure Oxygen Test No. 3-----	42
18	Cumulative Iron Weight Loss in High-Pressure Oxygen Test No. 3-----	43
19	Cumulative Zinc and Iron Loss in High- Pressure Oxygen Test No. 3-----	44
20	Solution pH of High-Pressure Oxygen Test No. 3-----	45
21	Cumulative Weight Loss in High-Pressure Oxygen Test No. 4-----	48
22	Solution pH of High-Pressure Oxygen Test No. 4-----	49
23	Galvanic Effects of Coupons in 316SS Baroid Cells--	50

INTRODUCTION

The Louisiana Department of Transportation recognizes that there is a need for metal pipe to provide drainage in this state. Concrete can normally serve effectively for the life of a highway provided the foundation bedding is stable. In many locations in this state the bedding is not supportive and metal pipe is necessary. The major problem with metal pipe is that it deteriorates due to the corrosion process. These culverts are placed in environments containing oxygen and water, which provide the ingredients by which corrosion can occur. The presence of galvanizing (zinc) on the steel provides some cathodic protection, and various coatings assist in slowing down the corrosion process by providing resistance to current flow.

In March, 1985, the Louisiana Department of Transportation reported on a ten-year study which evaluated the corrosion resistance of 16 different types of metal drainage pipes at 11 different representative field installations in the state. (1)* There are obviously many more pipes available for selection than there is manpower or time available to evaluate them. To assist in the decision making process, the study included two accelerated laboratory tests: (1) a one-month test in the Salt-Fog Chamber, and (2) a 1500-hr. test in a Weather-Ometer. Although the results were somewhat subjective, these results provided some hope that an accelerated test could be developed.

The purpose of this study was to develop an accelerated test to be performed under controlled environmental conditions which will be able to evaluate a number of culverts and culvert/coating combinations in a relatively short period of time. The test should produce results which will correlate with field results.

*Underlined numbers in parentheses refer to Bibliography.

SCOPE

The objective of this project was to develop an accelerated test which would assist the highway engineer in evaluating the usefulness of a new type of coated steel culvert. The test method was to be short in duration (in the order of days), and the results must correlate with field results.

Since the corrosion rate of a metal can be accelerated by elevating its potential, the test methods investigated involved utilizing this approach. The environmental species that cause corrosion in the field, such as various salts and oxygen, were used in each of the designed tests.

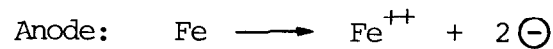
A total of six different types of culverts were used in the tests. They were the following: galvanized steel, asbestos-bonded galvanized steel, asphalt-coated galvanized steel, asbestos-bonded asphalt-coated galvanized steel, fiber-bonded bituminous-coated galvanized steel, and polyethylene galvanized steel (blackclad). The galvanized steel provided a convenient baseline since it has no coating other than zinc.

Two test methods were designed based on conditions found in the field. These methods were (1) the High-Pressure Oxygen Test, and (2) the Potentiostat Test. Several tests using each of these methods were run on representative culvert coupons until the results obtained correlated with field data.

LITERATURE SURVEY

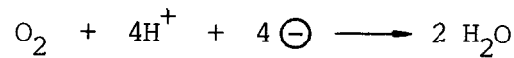
General

Corrosion is defined as the deterioration of a metal because of a reaction with its environment. The corrosion reaction is normally an electrochemical reaction. For the corrosion reaction to proceed, three basic elements are required: an anode, a cathode, and an electrolyte. The corrosion reaction of steel in an acid environment is shown as follows:

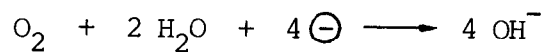


The existence of oxygen can accelerate the reaction whether it occurs in an acid or basic solution. (2)

Acid solution:



Neutral or basic solution:



A pipe or culvert under a pavement usually has less access to oxygen than the parts under the unpaved shoulder. Thus, cathode and anode sites form and cause corrosion on the pipe under the pavement edge. (3) This phenomenon is called an oxygen concentration cell, and it is responsible for many culvert failures.

The parameters which affect underground pipe corrosion were studied widely. (3) (4) They can be summarized as follows:

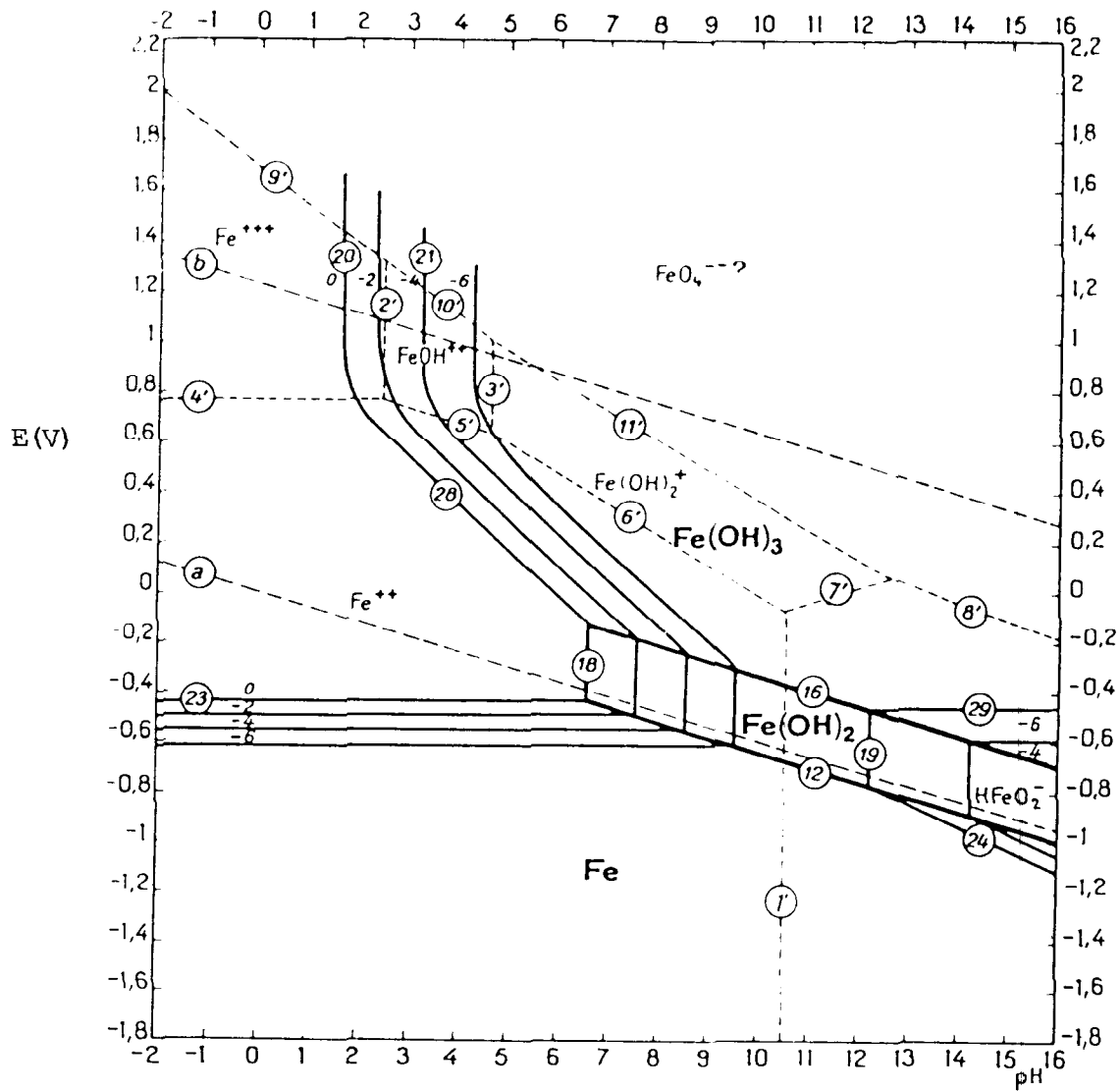
- (1) Soil characteristics, including type of soil, moisture content, pH, degree of aeration, soil resistivity, soil conductivity, redox potential, salt content, micro-organisms, etc.

- (2) Pipe materials, including steel, copper, cast iron, concrete, aluminum, stainless steel, plastics, etc.
- (3) Coating and coating material. Underground corrosion is affected by bare pipe or coated pipe, and affected by coating materials and coating methods. (5) (6)
- (4) Cathodic protection. Underground corrosion is significantly different between cathodically protected and non-protected pipe.
- (5) Others, such as stray current, abrasive flows, etc.

One very simple way to determine if a metal will corrode in an aqueous environment is to examine its Pourbaix diagram. (7) The diagrams use pH and oxidation potential to show the region of corrosive attack. Figure 1 shows the diagram for iron at 25°C. pH measurements in the soil are made quite readily and the pipe-to-soil potential is usually measured using a copper-copper sulfate half cell. (8) This cell reading is then converted to the standard hydrogen electrode scale so that the diagrams can be used. The region in which Fe^{++} is stable is the corrosion region. The figure reveals that iron will corrode at pH values as high as 8.5, but that the corrosive region is larger at lower pH values. This diagram would represent a soil system in which there is very poor aeration. This lack of oxygen would tend to cause the hydroxide scales to form. These are soluble scales which can dissolve in the environment and so are not protective.

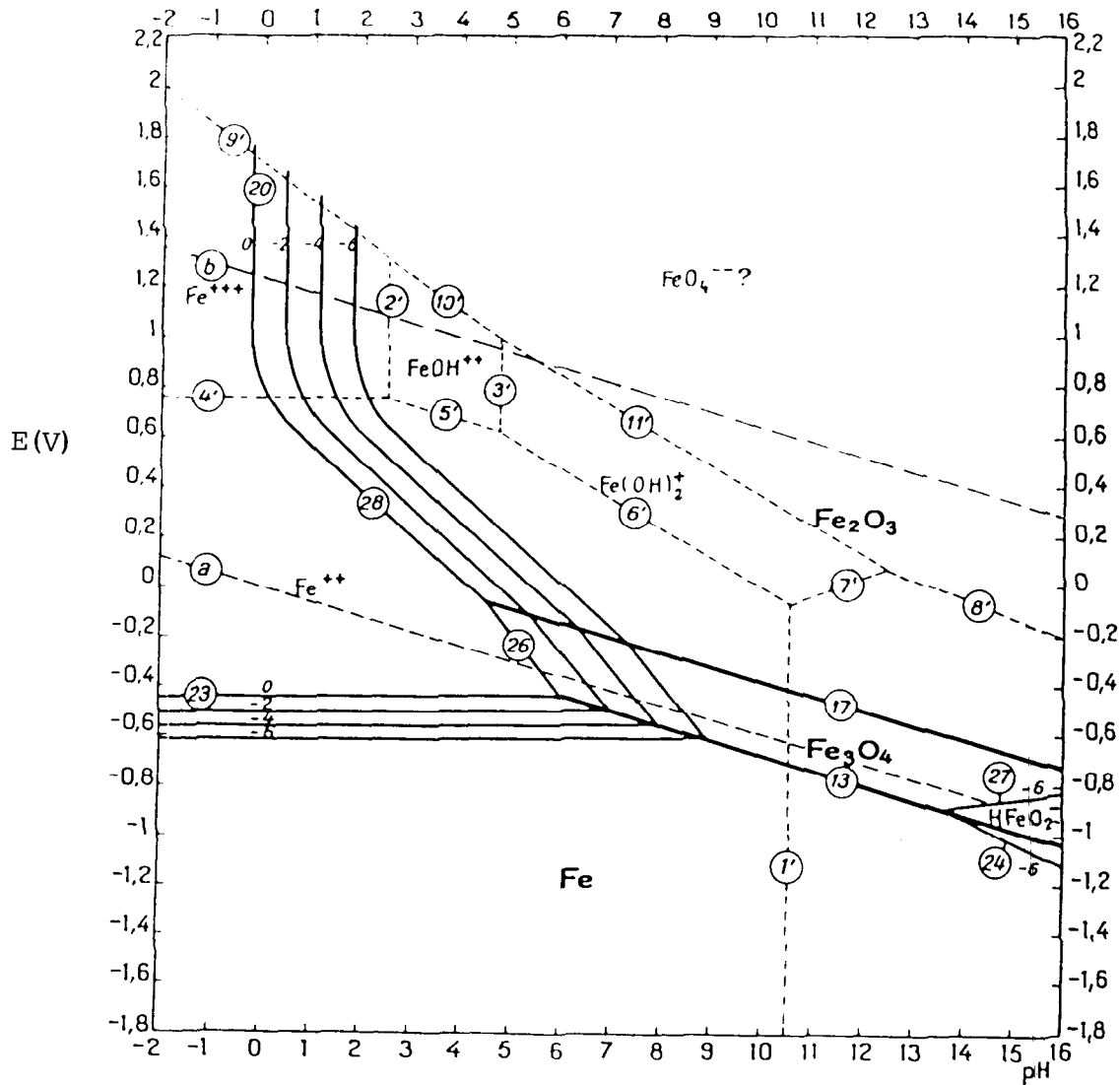
Figure 2 shows the Pourbaix diagram for iron in a well aerated environment. The corrosive region is again described by Fe^{++} and can be affected by pH values as high as 8.5. The difference is that the oxide scales are insoluble in this environment, and they tend to form a protective layer on the metal pipe.

The same diagram for aluminum is shown in Figure 3. Again, the corrosive region is designated as Al^{+++} while the passive region is where $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ (hydrargillite) forms. The diagram shows that for a



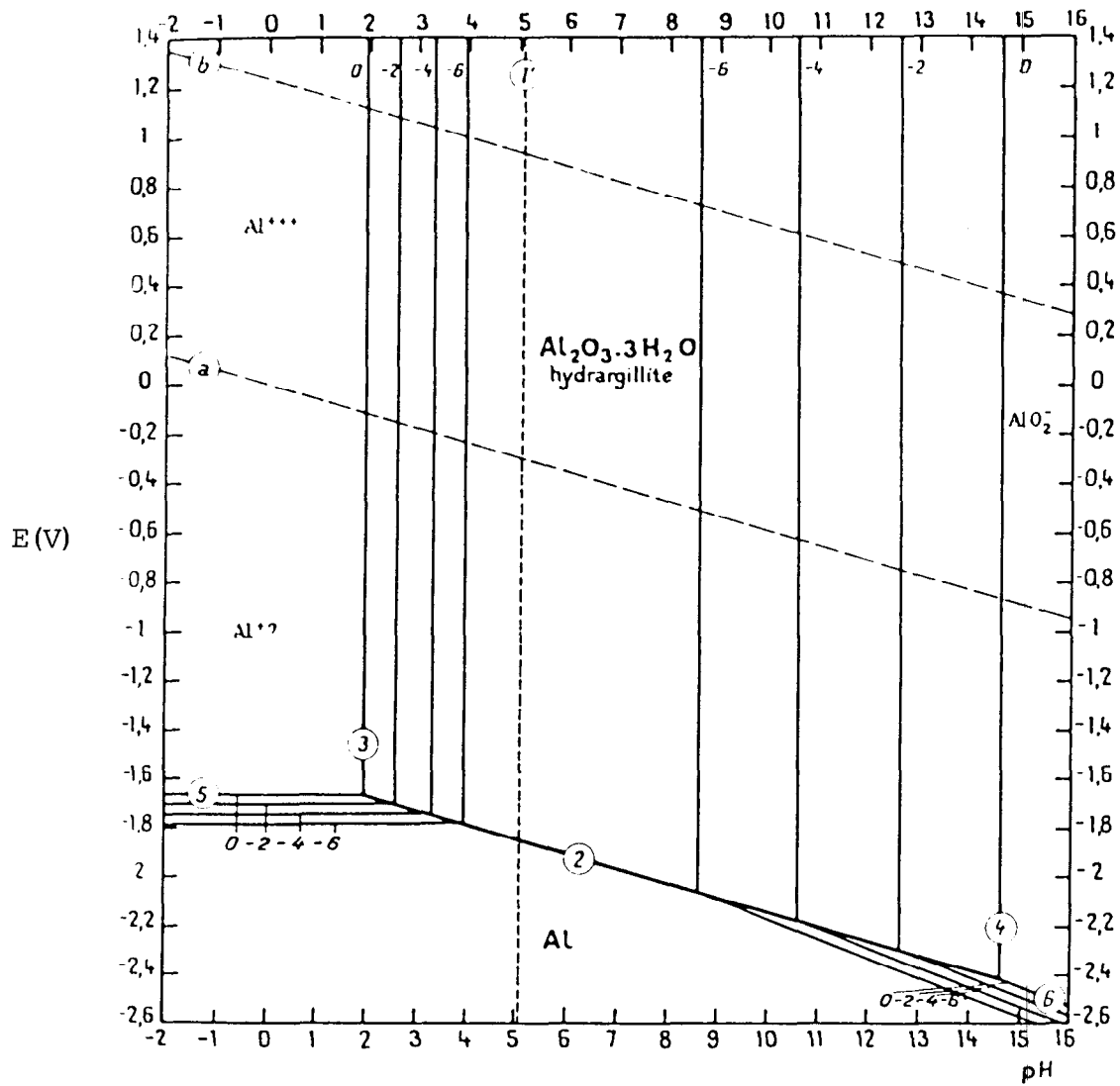
Potential - pH Equilibrium Diagram for the Fe, Fe(OH)₂, and Fe(OH)₃ System at 25°C.

FIGURE 1



Potential - pH Equilibrium Diagram for the
 Fe, Fe_3O_4, Fe_2O_3 System at 25°C.

FIGURE 2



Potential - pH Equilibrium Diagram for the Aluminum - Water System at 25°C.

FIGURE 3

pH value greater than 4, the material should be passive. It can be shown experimentally that the aluminum is corrosive in strong acids or caustic solutions, which verifies the Pourbaix diagram. Since the protective scale which forms requires oxygen, the aluminum functions best in aerated environments.

Since the metals which are being used for culvert pipe are iron and aluminum, it is important to know as much as possible about the environment. In particular, the pH and potential of the soil should be measured since the Pourbaix diagrams can predict if corrosion is expected to occur. The diagrams also indicate how the presence of oxygen in the soil affects the corrosion rate. It suggests that sediment inside a pipe could lead to a lack of aeration and produce an oxygen concentration cell. Likewise, on the outside of a culvert, it is likely that the top of the culvert has more oxygen than the bottom portion. These facts suggest that the bottom of a culvert would be more susceptible to attack, and this fact has been observed in the field.

Evaluation of In-Service Pipe

In the past decades, the highway departments of most states, including Louisiana, have undertaken studies of highway drainage performance. (1) (9) The method mainly consists of burying different types of pipe in the field, then periodically unearthing and inspecting them for up to 10 years. This method provides good data to the field engineer, but takes too long to evaluate each pipe in each available environment.

In California, a method for estimating service life of steel culvert was developed after 7000 corrugated steel culverts were studied. (10) By combining pH and electrical resistivity of the soil, together with field test data, a method was developed for estimating years to perforation of metal culverts. It is easy to measure pH and soil resistivity and check the correlation. Since the correlation was developed in California, the ability to apply it to other states is somewhat questionable. However, a number of states, including Louisiana, use the correlation to help in the design of culverts.

In Utah (11) the independent variables included chemical composition of water and soil, pH, total soluble salt content, resistivity at naturally

occurring soil moisture, etc. A mathematical model was formulated and charts were constructed as pipe material selection criteria. Because of the field test data, this criteria may not correlate with field experience when:

- (1) resistivity is less than 150 ohm-cm
- (2) soil pH < 7.0 or > 9.6
- (3) soluble salts are $< 0.8\%$ or $> 10\%$
- (4) sulfate contents $> 0.5\%$
- (5) there is abrasive flow

In Maine (12) a study in 1982 revealed that the variables affecting corrosion were water velocities, O₂ concentration, water temperature, pH of soil and water, electrical resistivity of soil and water, and bedload. It used a standard backfill with a resistivity of 10,000 ohm-cm. Due to the relatively flat grade, abrasion was not a problem.

The Florida study, (13) which was issued in 1976, revealed a very close similarity to Louisiana since the soil pH range was 5.2 to 8.4 and its resistivity range was 107 to 82,500 ohm-cm. The location of corrosion was primarily on the inside of the culvert. The study emphasized that fully bituminous coated steel culverts add 10 years of life while if the same piece were fully paved, 30 years would be added.

The study in Georgia (14) was performed in 1977, and concluded that field tests of less than 10 years are suspect. It lists the California Test, AISI Test, and New York Test as its primary methods of predicting pipe life. The location of corrosion was primarily within 24" of the end and occurred on the interior side of the culvert. The soil side showed no cases of aggressive corrosion of bare galvanized steel nor any breakdown of coating. Again, the paved bituminous coated culverts added about 35 years, while the unpaved bituminous coated steel added only 10 years.

The New York study (15) on uncoated steel and aluminum culverts

showed an interesting approach by dividing the state into 2 regions: Zone 1, which was less aggressive, and Zone 2, which was most aggressive. In Zone 1, 90% of the culverts had corrosion rates of < 2 mils/year, (mpy) while in Zone 2, 90% have corrosion rates of < 4 mpy. It developed a technique for estimating the life of culverts based on 190 galvanized steel and 35 aluminum culverts.

The American Iron and Steel Institute (16) has developed a procedure for estimating the service life of corrugated steel culvert pipe. The design life is taken as the average number of years before perforation could be expected. The method is divided into 2 ranges, one above pH 5.8 and one below 5.8. Above pH 5.8, the Institute recommends using a pitting rate of 1.3 mpy in ordinary installations and 3.0 mpy in unusually soft water installation. Below pH 5.8, the California chart is used. It also estimates the extra years that are derived from coatings. Asphalt coating and paving is recommended unless the pipe is in a very severe erosion situation.

A paper by Con. Edison Co. (17) on fundamental parameters in underground pipe corrosion revealed that non-aerated soils are the worst variety since protective scales cannot form. It pointed out that breaks in the coating could expose the metal to two types of soil where a 0.5V potential could exist. The corrosion would be worst for a small anode-large cathode situation. It presented a formula for predicting culvert life for both steel and aluminum:

$$\text{Steel} \quad \text{mpy} = \frac{230 \Delta E}{\rho r}$$

$$\text{Aluminum} \quad \text{mpy} = \frac{585 \Delta E}{\rho r}$$

where ΔE is the potential difference, mv

ρ is the soil resistivity, ohm-cm

r is the radius of the uncoated area, inches

It found that potential differences of up to 800 mv can occur from one spot on a culvert to another. However, a difference of 0.25V is far more common. This study suggests that it is possible to estimate the corrosion rate from soil redox potentials, resistivity measurements, and the average

size of the coating break.

In March, 1984, Mississippi issued a report (18) on the use of aluminum culverts along the Gulf Coast. This report was generated by Kaiser Aluminum, which identified its primary product as a 3004 type of core with an external and internal coating of 7072 aluminum. The coating represented about 10% of the total material. The 7072 aluminum contains about 1% zinc, which makes it anodic to the core metal. This tends to stop pits from propagating into the base metal. The report recommends this material be used within the pH range of 4 to 9 for soil and water. It is also necessary that the resistivity be kept above 500 ohm-cm except in the case of seawater, provided sediment does not form inside the pipe.

The U.S. Department of Transportation issued a report (3) in 1978 dealing with the ability of drainage pipe to endure the processes of corrosion and abrasion. This provided a summary of the findings of a large number of state highway and transportation departments with respect to culvert corrosion. It found that the simplest criterion for estimating the corrosivity of a given soil to metal is its resistivity. It was found that oxygen concentration cells constitute a major type of corrosion mechanism. It was stated that bituminous coated pipe had a tendency to lack adhesion. Several states recommended this type of pipe if the soil resistivity is greater than 2,400 ohm-cm. Bituminous-coated pipe will add 10 to 15 years to the life of the galvanized material and this time can be increased to 25 years by paving the lower quadrant of the pipe. Asbestos-impregnated, bituminous-coated galvanized pipe is superior since the asbestos fibers help hold the coating to the pipe.

Accelerated Tests

The number of types of culverts available for use as drainage pipe has grown in recent years. Field tests of culverts should be evaluated for a period of at least 10 years. This has prompted the various departments of transportation to develop accelerated testing methods which will allow for rapid discrimination between the various culverts. The following is a literature review of the various laboratory methods that have been attempted.

The salt spray test was developed in 1939 for testing coated surfaces and became an ASTM specification. There are some other variations of the salt spray for special purposes. (19) This method sprays salt solution with compressed air onto a coated surface for periods from 500 to 4000 hrs. The tested surface is then checked and rated for blistering, rusting, and undercutting. There have been some questions as to the reliability of this test method. (20) The Louisiana Department of Transportation has found this test is subjective when used to evaluate coated culvert materials.

The Louisiana Department of Transportation used a Weather-Ometer test of 1500 hours. This test consists of a carbon arc Weather-Ometer with automatic humidity control. The method was run on various coated steel culverts and the results were again subjective.

Pesigan and Singly (21) attempted to determine whether the Langelier index is a reliable indicator of a water's corrosive potential. They used laboratory data to establish an empirical model which relates corrosion to the variables which affect it. Using a regression analysis, they were able to establish that chemical species such as chlorides, sulfates, and dissolved oxygen accelerate corrosion, while calcium reduces the corrosion rate. The developed model is as follows:

$$CR = \frac{(Cl)^{.509} (SO_4)^{.0249} (Alk)^{0.423} (DO)^{0.780}}{(Ca)^{.676} (\beta)^{0.030} (10^{SI})^{.107} (Day)^{0.381}}$$

This information can be helpful in understanding how water chemistry affects corrosion rate. Examining the power of each of the species reveals that dissolved oxygen has the largest effect on corrosion rate while chlorides are next in their pronounced effect on corrosion.

Electrochemical polarization methods measure the current and potential of a corrosion cell. The methods give a numerical value of corrosion rate, thus predicting the performance time of an underground metal pipe without unearthing it. (22) By using Petrolite instruments, Martin (23) fully demonstrated the application of electrochemical polarization to corrosion problems such as corrosion rate, pitting, and inhibitor effects, etc. Scully and Bundy (24) performed electrochemical polarization tests in soil boxes on coated and bare pipe steel with 3 soils of varying resistivity. They concluded that electrochemical techniques can identify the differences in

corrosion rate more clearly than pipe-to-soil potential measurement. In another paper, Scully and Bundy (25) ran some scan rates using a potentiodyne on polyethylene coated pipe. The 40 mil coated pipe showed a very low current density which was below the microampere level.

A more advanced method called AC impedance (26) has been developed which used very small excitation amplitudes in measurement. It can provide valuable kinetic and mechanistic information about scales of coatings. This makes it an ideal testing method for underground pipe corrosion analysis. Padgett and Moreland (27) used AC impedance to study anti-corrosive properties of coating supplemented by salt spray and outdoor exposure. They commented that AC impedance gave a much clearer quantitative picture of the corrosion processes that are occurring.

Work on developing an accelerated test was done by Welch of the Utah Department of Transportation. (11) He found that the salt chamber and the ozone electrolytic cell test did not provide any results that correlated with field data. He did develop a Mudpack test which was 8 weeks in duration. This test used the following procedure:

- (a) The material was immersed into 2 mudpack containers at $100^{\circ} \pm 2^{\circ}\text{F}$. The mud was then saturated with distilled water; and,
- (b) The pH level of the mud packs were 7.2 and 9.6. The resistivities were 150 ohm-cm and 200 ohm-cm.

The test results indicated that there was some deterioration of the coupons. The results listed below describe the deterioration.

<u>Coupon</u>	<u>Description of Coupon</u>
Galvanized pipe	Flakes of zinc coating and pitting
Aluminum	Became dark after 7 days. Formed uniform rough oxide coating after 3 weeks, with no change thereafter.
Bituminous-Coated	Showed no corrosion beneath coating. Lost adherence near the edges where the core metal had been exposed.
Asbestos-Bonded Bituminous-Coated	Remained in good condition with slight adhesion loss near the sample edge.

Welch concluded that the mud pack test is considered one of the most potentially promising laboratory accelerated test methods currently available to determine the relative durability characteristics of culvert materials. He suggested more extensive testing to quantify the relative effects of temperature, pH, water, salt, etc.

The National Research Council's Report 50 on the durability of drainage pipe (9) described two laboratory tests to check coating effectiveness. Test Number 1 immersed the samples in several common corrosive solutions: seawater, 5 to 18 percent NaCl, 1 to 10 percent H_2SO_4 , 2 to 5 percent Na_2CO_3 , 2 to 5 percent Na_2SO_4 , and 2 to 5 percent $(NH_4)_2CO_3$. These solutions caused delamination to occur on the steel. Test Number 2 is a "rocker" abrasion testing method which uses a sand and gravel bedload. The plain bituminous coatings showed better resistance than the asbestos-impregnated bituminous-coated specimens.

In 1982, the Indiana Highway Department developed an accelerated test (28) which involved the exposure of pipe samples to low pH fluid for a period of up to 2 years. The fluid is a sulfuric acid solution with an acidity of pH 2. The pH was readjusted on a regular basis. A total of 29 pipe samples was tested. Several pipes that performed well in this environment were (1) Epoxy-coated, (2) Polymeric-Bituminous-coated, and (3) Asbestos-bonded Bituminous-coated.

EXPERIMENTAL RESULTS

Based on the literature survey of long-term and accelerated testing, it is clear that there is a definite need for a test method which can evaluate a variety of culvert coatings in a relatively short period of time. The test method results must correlate with results found in the field. Using this criteria, it was decided to use two test methods which accelerate the corrosion process by elevating the potential of the culvert metal in a corrosive environment. This was achieved by using the potentiostat test, and the high pressure oxygen test. In both tests, a 2" X 2" test coupon was cut from actual culvert material obtained from the Louisiana Department of Transportation.

A test solution similar to the standard NACE (National Association of Corrosion Engineers) solution was used in most of the tests. The solution consists of acetic acid and sodium chloride in distilled water. According to various literature sources, besides the hydrogen and chloride ions which are contained in the NACE solution, sulfate ions and dissolved oxygen are factors which increase the corrosivity of the corrosion media. (21) The initial pH of this standard test solution was 2.7.

As mentioned, two test methods were examined in an attempt to develop an accelerated test method. A description of the equipment used, the two test methods, and a method of interpreting the results follows.

Materials and Equipment Used in Testing

The following materials and equipment were used in the development of a test method:

1. Six (6) test culverts
 - a. Galvanized steel (GS)
 - b. Asbestos-bonded galvanized steel (without asphalt coating) (ABGS)
 - c. Asphalt-coated galvanized steel (AGS)
 - d. Asbestos-bonded asphalt-coated galvanized steel (ABAGS)
 - e. Fiber-bonded bituminous-coated galvanized steel (FBBGS)
 - f. Polyethylene galvanized steel (Blackclad), 10 mils coating (PGS)

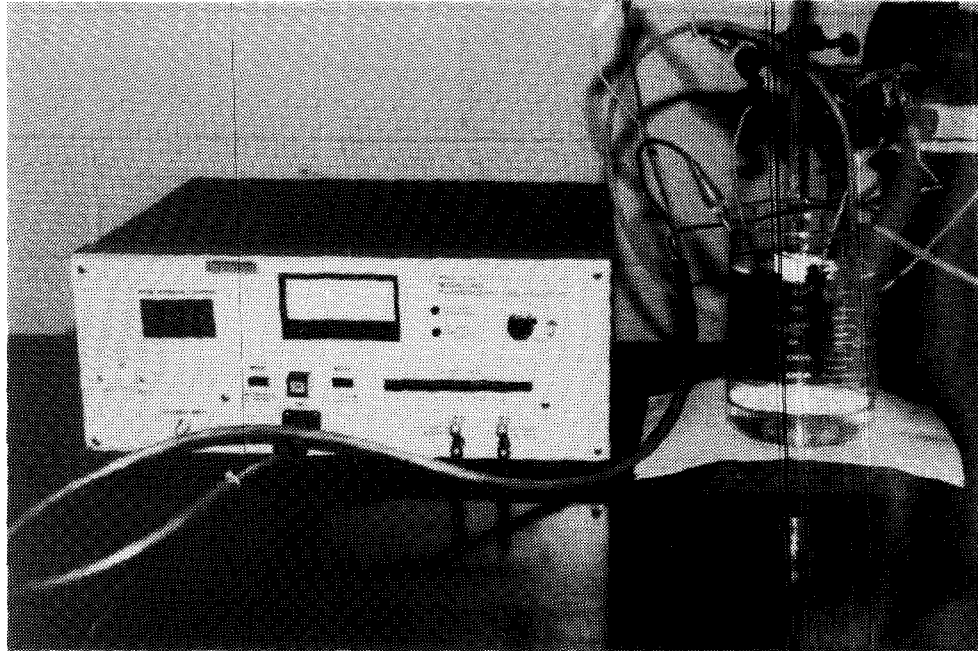
2. A Scanning Electron Microscope was used for microstructure and elemental analysis.
3. A Perken-Elmer Atomic Absorption Unit was used to measure the amount of zinc and iron contained in the test solution.
4. An MP4 Polaroid camera was used for taking macro photographs of coupons at 1X.
5. An EG & G Model 363 Potentiostat/Galvanostat was used to maintain a potential difference between the two electrodes.
6. Four (4) 316 stainless steel Baroid cells were used for high pressure oxygen testing.
7. One liter of the standard test solution consists of the following ingredients: 10g glacial acetic acid, 50g sodium chloride, 20g sodium sulfate, and 954g distilled water.

Potentiostat Test

A metal that is corroding in a given environment has a natural corrosion potential. If this potential is increased to a higher value, most metals will corrode at much higher rates. In this experiment, two 2" X 2" coupons from the same culvert were placed in the previously described solution and a 1-volt potential difference was maintained between them. The Model 363 Potentiostat, which is shown in Figure 4, was able to maintain that potential difference by automatically varying the current to or from the coupons.

The general procedure that was developed in this test is listed below:

1. Prepare 2 volumes of the standard test solution.
2. Prepare the coupon by drilling a hole approximately $\frac{1}{4}$ " from the top of the coupon and approximately centered lengthwise. The hole should be just large enough to allow a plastic bolt to be inserted through the hole.
3. When coated coupons are used, a conducting surface for the connections must be made. Remove the coating on one side of the coupon approximately $\frac{1}{4}$ " radius around the hole. Make certain this area is clean and free from anything that would inhibit current flow.



Model 363 Potentiostat Used in the Testing

FIGURE 4

4. Next, for coated coupons, make certain that the edges are clean and free from coatings.
5. Weigh the samples and measure the exact dimensions.
6. Attach copper wire leads to the coupons by stripping a $\frac{1}{2}$ " section of the wire, then bend a loop with the stripped wire. Insert the plastic bolt through the loop and the hole in the coupon so that the copper wire makes contact with the conducting surface. (see step 3)
7. Pour the solution (prepared in step 1) into the beaker. Place the beaker onto the ring stand. Obtain a sample and determine the pH.
8. Attach the copper wire to the clamps so that the coupons are extending downward approximately 3-4". Additionally, approximately 3" of wire (the end without the coupon) should be extended upward. Strip a 1" section of this part of the wire.
9. Lower the clamp until the coupons are completely immersed in the solution. (Rule-of-thumb: cover with at least 1" of solution.) The coupons should be spaced roughly 3 inches apart. It is important that once this distance is set, it be maintained throughout all tests.
10. Make sure the potentiostat is turned off. Then connect the lead to the 1" stripped section (step 8) as follows:
working electrode (green) - cathode
counter electrode (red) - anode
reference electrode (white) - anode
11. Set the mode switch to the "Control E" setting. Set the potential difference to -1.00 volts.
12. Purge the beaker with pure oxygen continuously by running a tube from the cylinder of compressed oxygen into the beaker. Bubble oxygen slowly into the solution.
13. Turn the potentiostat on and press the cell button so that it reads "ON."

14. Check the condition of the coupon every 24 hours and replace the solution. Thoroughly dry the coupons for four hours at 130° F, then record the weight loss of the coupons and the pH change of the solution.
15. Place the coupons back into the new solution and continue the test.

The results of this 1-volt test, using the described procedure, are presented in tabular form in Table I and graphically in Figure 5. These weight loss results are very interesting since there was a good spread between several of the six different types of drainage pipe. The pipes that were in the ten-year study conducted by the Louisiana Department of Transportation (galvanized, asphalt-coated and asbestos-bonded asphalt-coated) showed the same trend as in the field. During the first three days, the asbestos bonded-asphalt coated pipe showed the least weight loss, the asphalt-coated next, and the uncoated-galvanized steel showed the greatest weight loss. Figure 5 shows that this method was unable to distinguish significantly between the blackclad, the asbestos-bonded asphalt-coated, the fiber-bonded bituminous-coated, and the asbestos-bonded materials.

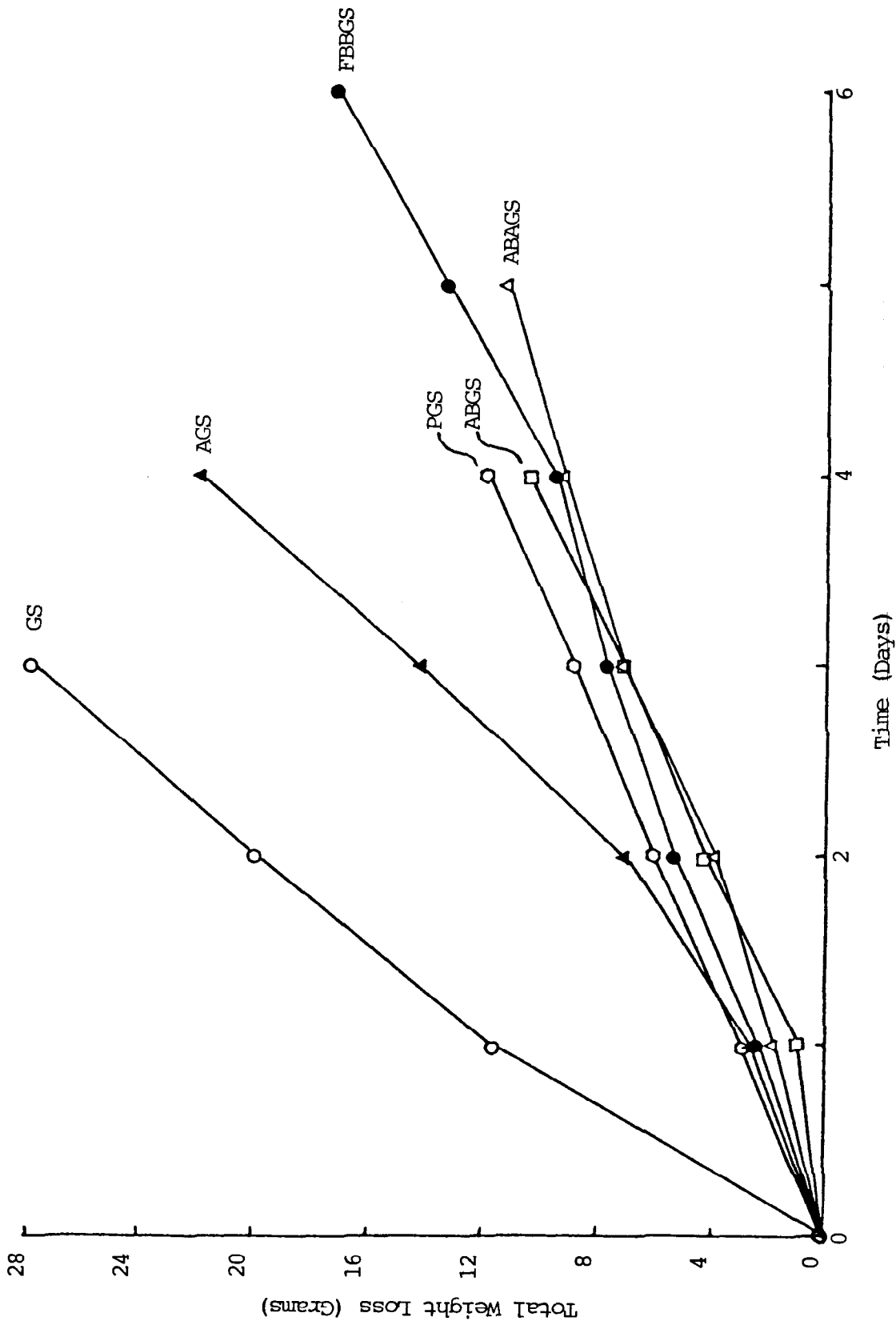
In an attempt to further differentiate between these lower curves in Figure 5, it was decided to increase the temperature of the test from ambient to 125° F. The higher temperature test required that the present method of potentiostat testing be modified slightly. The major changes were: (1) a one-liter volume of test solution was used, (2) the copper wire on the anode was replaced with a Hastelloy C276 wire, and (3) the test solution was heated to 125° F. The complete set-up, which includes the potentiostat and high temperature bath, is shown in Figure 6. Figure 7 gives a summary of the test results, which produced a significant spread in the data. The test results after two days revealed that the asbestos-bonded asphalt-coated material was best, with the asbestos being the worst. The blackclad material performed better in this test than it did at room temperature. There was some deterioration of the asphalt and bituminous coatings at the higher temperature. A large amount of sediment formed at the bottom of the test cell.

Table 1

1-VOLT POTENTIOSTAT TEST

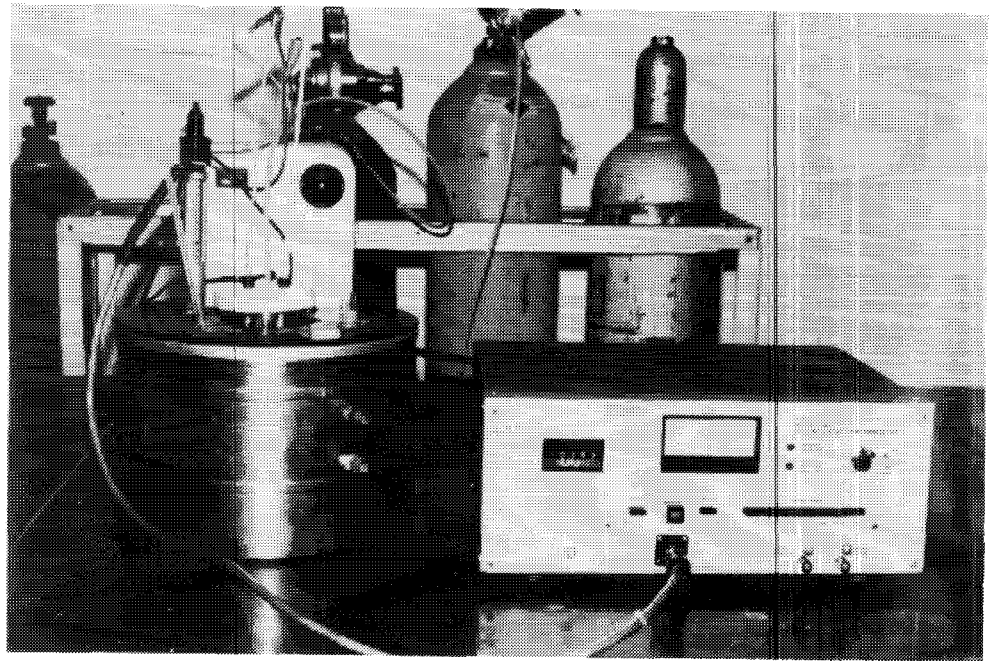
Day	Total Weight Loss of Various Coupons, g					
	<u>Galvanized Steel</u>	<u>Blackclad</u>	<u>Asbestos Bonded</u>	<u>Asphalt Coated</u>	<u>Fiber-Bonded Bituminous-Coated</u>	<u>Asbestos-Bonded Asphalt-Coated</u>
1	11.756	2.874	0.928	2.590	2.404	1.844
2	20.015	5.990	4.272	7.053	5.330	3.980
3	27.859	8.878	6.974	14.144	7.757	7.100
4	---- *	11.954	10.402	21.892	9.533	9.354
5		---- *	---- *	---- *	13.253	11.27
6					17.206	---- *

*Coupon connection was destroyed and test was terminated at this point.



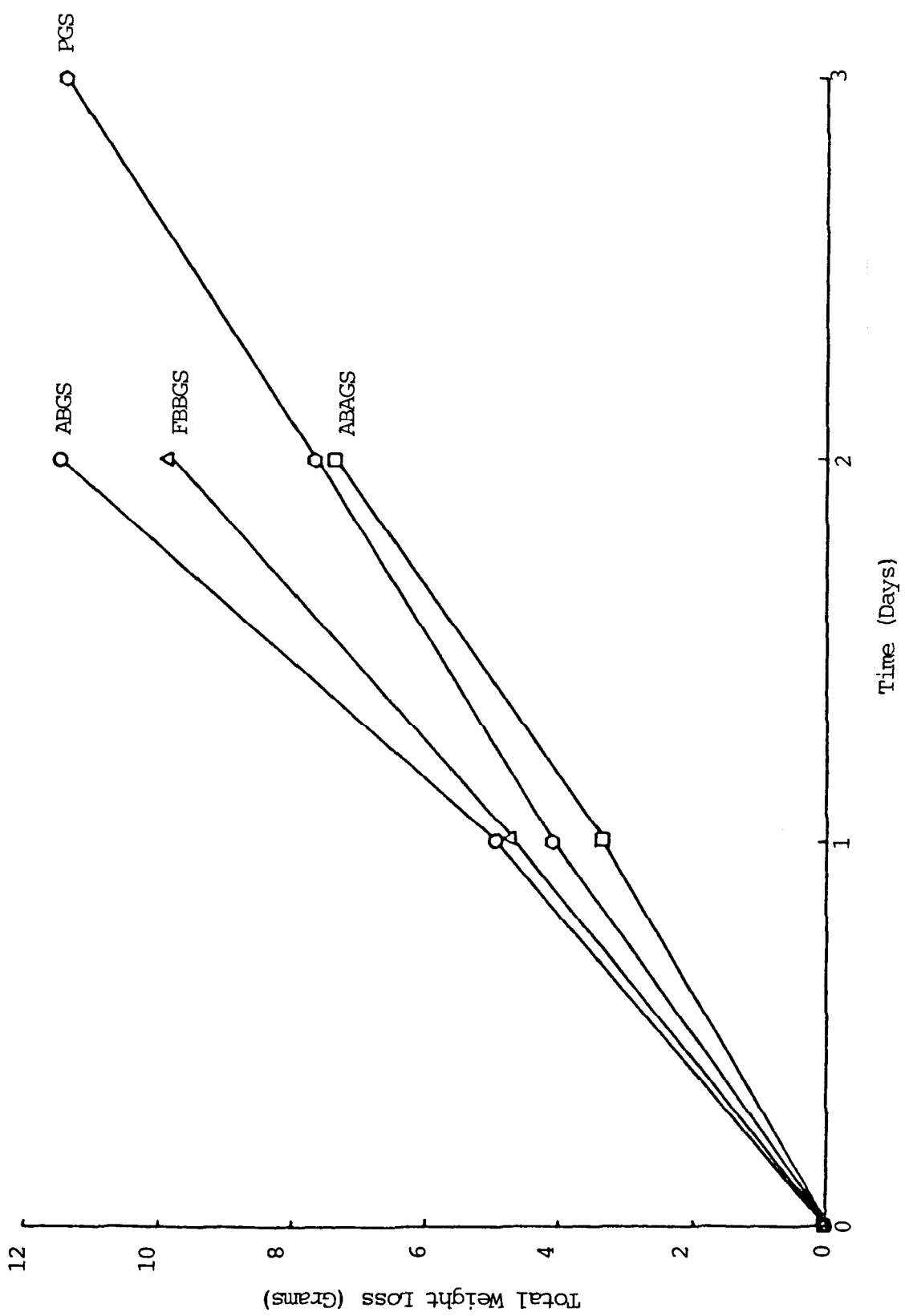
1-Volt Constant Potential Accelerated Corrosion Test

FIGURE 5



Complete Set-Up Required to Run the
Elevated Potentiostat Test

FIGURE 6



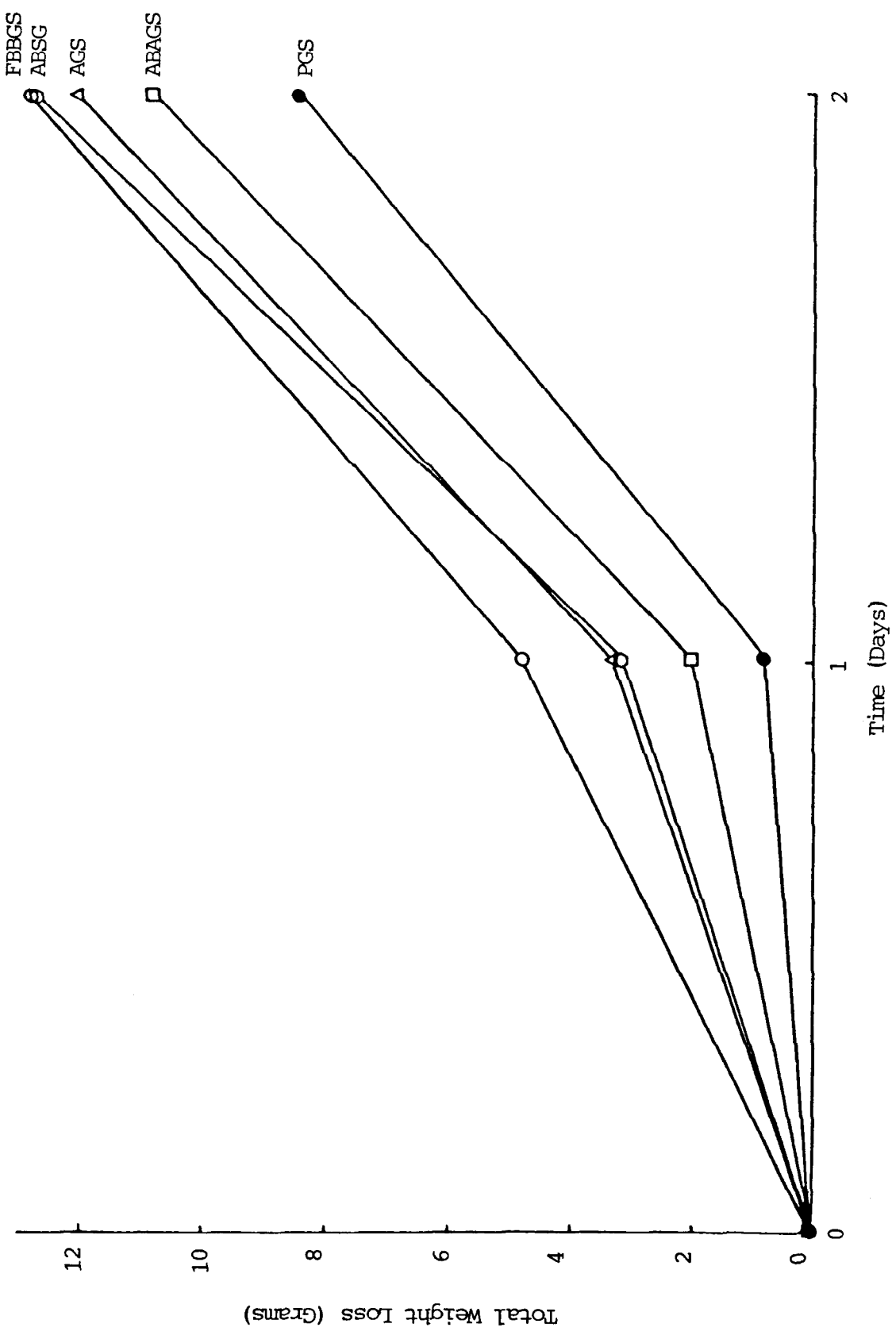
1-Volt Constant Potential Accelerated Corrosion Test at 125°F

FIGURE 7

In an attempt to produce an even greater differentiation among the test culverts using the potentiostat, it was decided to attempt to run the various culvert materials at extreme conditions of environment and potential. These two test methods and their results are as follows.

1. 5% HCl Acid. In this room temperature test, coupons from each culvert were connected using a Hastelloy C276 wire. The area around the connection was covered with bee's wax before placing the coupon in the one liter 5% HCl solution. The counter electrode was a 10 gauge Hastelloy C276 wire, and a standard calomel reference electrode was used to maintain the potential of the coupon 0.5 volts above its natural potential. The coupons were removed after 24 hours, dried for two hours at 130° F, and run for a second 24-hour period. The results of the tests for asphalt, asbestos, blackclad, asbestos-asphalt, and fiber-bituminous are given in Figure 8. In this environment, the blackclad performed best while the fiber-bituminous performed worst. It should be noted that the strong acid produced some obvious deterioration of the bituminous and asphalt coatings.
2. 2-Volt Potential Difference. This test method used a similar procedure for testing as the one-volt differential method. A few changes have been made in the procedure which include (1) a 1.5-liter volume of test solution, and (2) Hastelloy wire for the anode and cathode connections. Bee's wax was placed on the anode and cathode connections so that there would be zero potential difference between the two coupons when starting the process.

The tests were run for a period of three days with the 1.5 liter solution being changed every 24 hours. The test coupon was removed after the third day and checked for weight loss after drying for four hours at 130° F. Since the coupon is only weighed after the test is completed, there is only one data point to examine. Table 2 lists the results of the two tests which were performed on each coated culvert material. The asbestos-asphalt material showed the smallest loss, while the asbestos and asphalt materials showed the greatest loss. All of the coupons experienced severe corrosion attack and weight loss.



0.5-Volt Constant Potential Accelerated Corrosion Test in 5% HCl

FIGURE 8

Table 2

2-VOLT POTENTIOSTAT TEST

Total Weight loss, g

<u>Coupon</u>	<u>Test 1</u>	<u>Test 2</u>	<u>Average</u>
Asbestos	19.94	18.17	19.1
Asphalt	21.59	15.88	18.7
Fiber-Bituminous	16.87	17.47	17.2
Blackclad	14.55	13.27	14.0
Asbestos-Asphalt	11.93	12.1	12.0

High-Pressure Oxygen Test

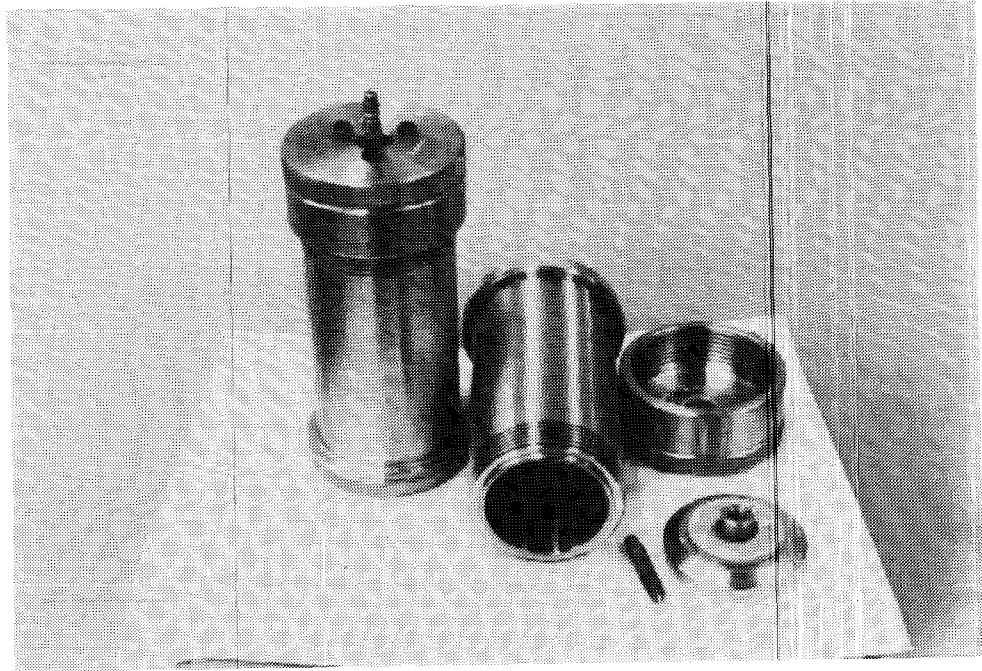
Since in most cases the cause of underground corrosion is dissolved oxygen in a conductive fluid, it was decided to increase the concentration of oxygen in the environment by increasing its partial pressure. Pure oxygen was used instead of air, which contains only 21% oxygen. Baroid cells made of 316 stainless steel and having a total volume of 500 ml (see Figure 9) were pressurized to 500 psi. The object was to see if the corrosion rate could be accelerated enough to provide a means of evaluating the effectiveness of the various coatings. Several tests were run using these cells and their conditions are summarized below.

<u>Test</u>	<u>Description</u>
No. 1	Room temperature, solution pH = 2.7, heating and weighing coupon after each day.
No. 2	Room temperature, solution pH = 2.7, remove solution each day, heat and weigh coupon after third day.
No. 3	Room temperature, solution pH = 5.7, remove solution each day, heat and weigh coupon after third day.
No. 4	High temperature (130° F), solution pH = 2.7 heating and weighing coupon after each day.
No. 5	Evaluation of galvanic effect between the test coupon and the 316SS Baroid cell.

The various tests were designed to examine the effect that pH and temperature have on the coupons. Test No. 2 examines whether or not the coupon should be heated every 24 hours during the testing period. A description of each of the tests on the various 2" x 2" culvert coupons follows.

1. Test No. 1. In this room temperature test, the standard test solution was used. The cells were pressurized with pure oxygen to 500 psi. The exact procedure used in this test follows:

Test coupons were carefully cut from the samples and weighed. The standard corrosive solution was prepared. The aging cells were filled with 200 ml solution and the coupons were inserted. The cell was pressurized to 500 psi with oxygen at room temperature. After 24 hours the coupons were removed, heated at 130° F for



Two of the Baroid Cells Used in the
High-Pressure Oxygen Test

FIGURE 9

two hours, then re-weighed. The difference in weight of each coupon was recorded at the end of each day. Using the same coupon but a fresh test solution, the procedure was repeated for a second and third day. The pH of the solution was measured at the end of each day.

The results of this test are presented in tabular form in Table 3 and are shown graphically in Figure 10. These weight loss results are interesting since they show essentially the same order as the potentiostat test results presented earlier in Figure 5. However, the results are actually somewhat better since this test produces a spread between the asbestos-bonded, fiber-bituminous, and asbestos-asphalt coupons. The asbestos-asphalt coupon showed the best results as expected from the field studies.

Figure 11 shows the change in the solution pH from the beginning to the end of each run. Note that the starting solutions all had a pH of 2.7 units. The pH difference is dramatic after the first day and much less significant after that. The results are presented in tabulated form in Table 4.

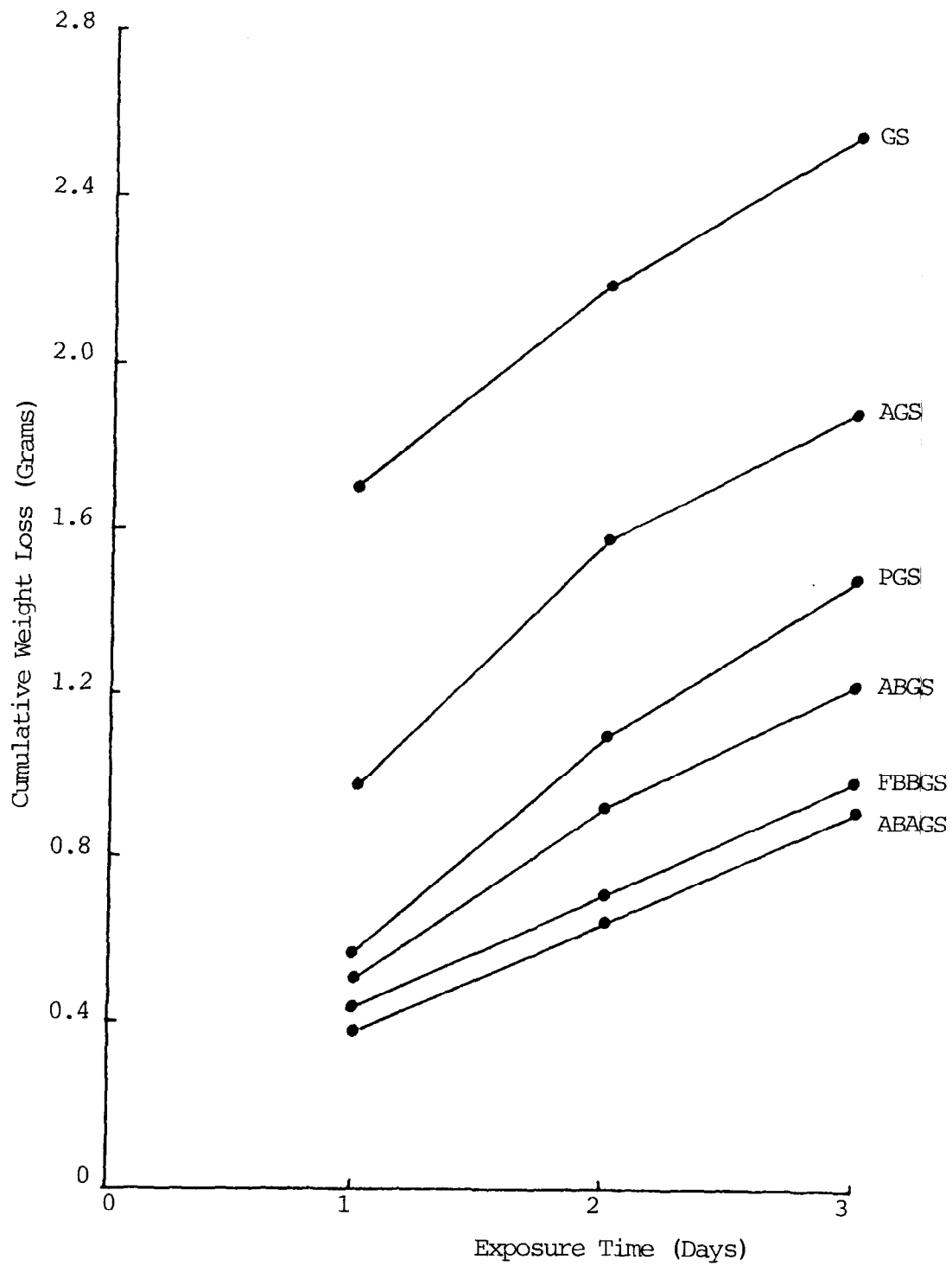
2. Test No. 2. A less labor-intensive test procedure than Test No. 1 was developed in which the coupon is only weighed at the end of the three-day test. In this procedure, every 24 hours the cell contents were emptied, and the cell and coupon were flushed with 50 ml of distilled water. The cell was again filled with 200 ml of solution and pressurized to 500 psi with oxygen. The coupon was not removed until day three. After removal, the coupon was heated for six hours at 130° F before weighing. It is believed that not touching the coupon between 24-hour testing periods would greatly reduce coating loss from handling and eliminate the effect of heating. Table 5 presents the results of several tests which were performed on each of the six types of culverts under examination. The average values are presented graphically in Figure 12. Also listed in Table 5 are the corresponding weight losses based on the amount of zinc and iron in the solution as determined by atomic absorption (AA).

Table 3

HIGH-PRESSURE OXYGEN TEST NO. 1

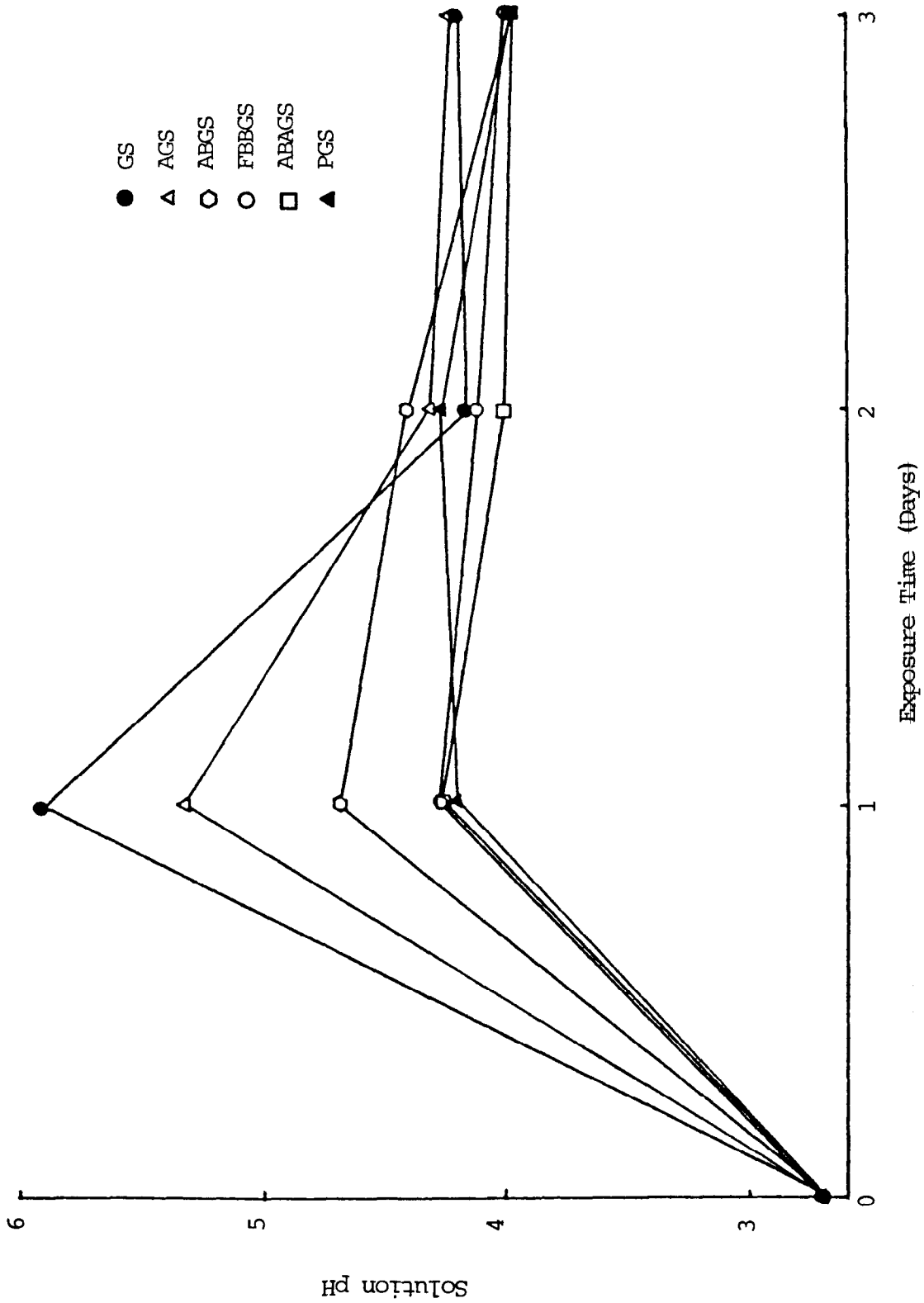
Cumulative Weight Loss of Various Coupons, g

DAY	<u>Galvanized Steel</u>	<u>Blackclad</u>	<u>Asbestos Bonded</u>	<u>Asphalt Coated</u>	<u>Fiber-Bonded Bituminous Coated</u>	<u>Asbestos-Bonded Asphalt-Coated</u>
1	1.702	0.578	0.510	0.974	0.450	0.383
2	2.175	1.111	0.934	1.584	0.712	0.657
3	2.543	1.485	1.225	1.873	1.002	0.930



Cumulative Weight Loss in High-Pressure Oxygen Test No. 1

FIGURE 10



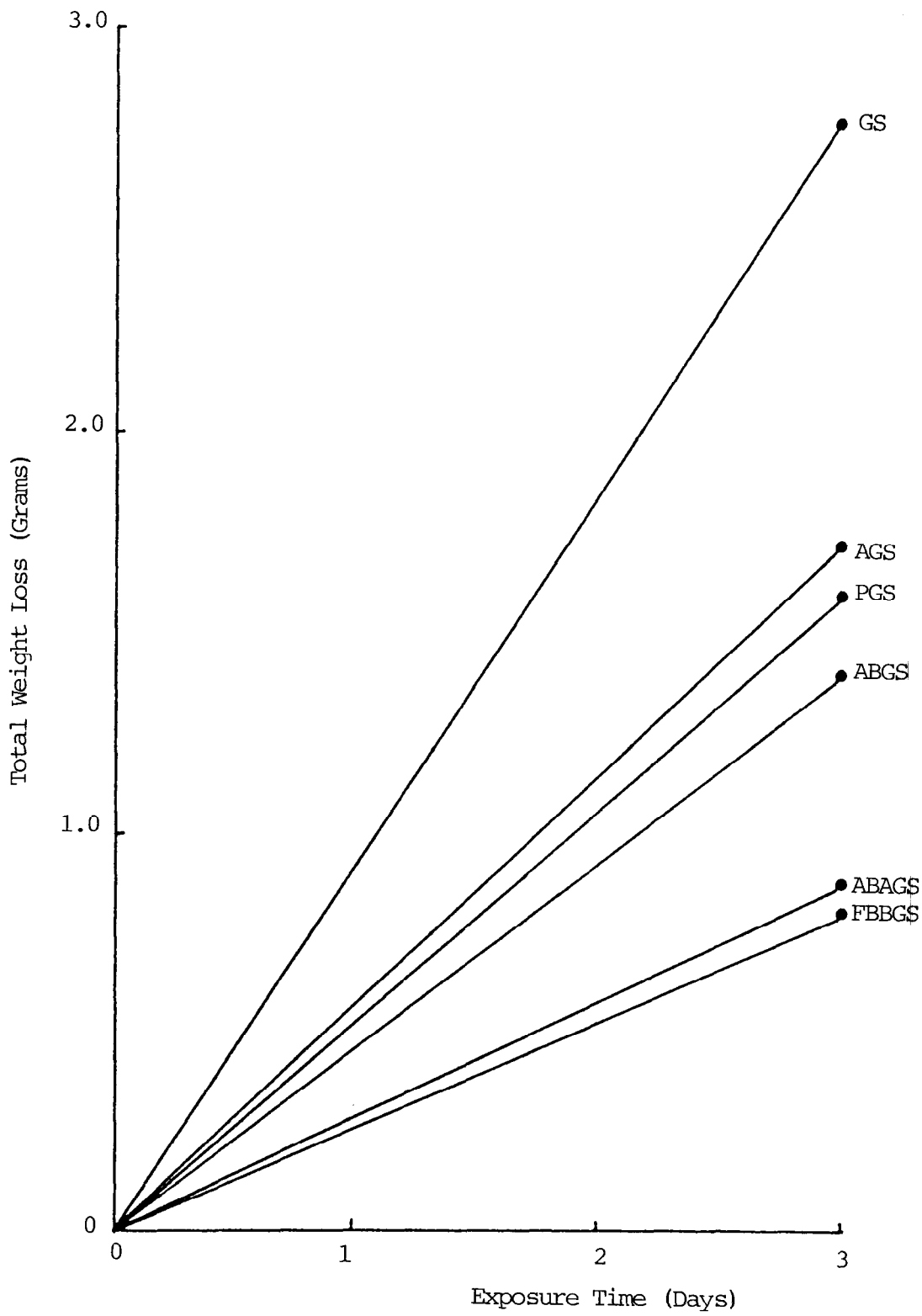
Solution pH of High-Pressure Oxygen Test No. 1

FIGURE 11

Table 4

HIGH-PRESSURE OXYGEN TEST No.1 (pH)

Day	<u>Galvanized Steel</u>	<u>Blackclad</u>	<u>Asbestos Bonded</u>	<u>Asphalt Coated</u>	<u>Fiber-Bonded Bituminous-Coated</u>	<u>Asbestos-Bonded Asphalt-Coated</u>
1	5.90	4.21	4.69	5.33	4.27	4.26
2	4.16	4.26	4.40	4.31	4.12	4.01
3	4.21	4.01	4.04	4.23	4.04	4.01



Total Weight Loss in the High-Pressure Oxygen Test No. 2

FIGURE 12

Table 5

HIGH-PRESSURE OXYGEN TEST NO. 2

Weight Loss from Weighing and Atomic Absorption (AA)

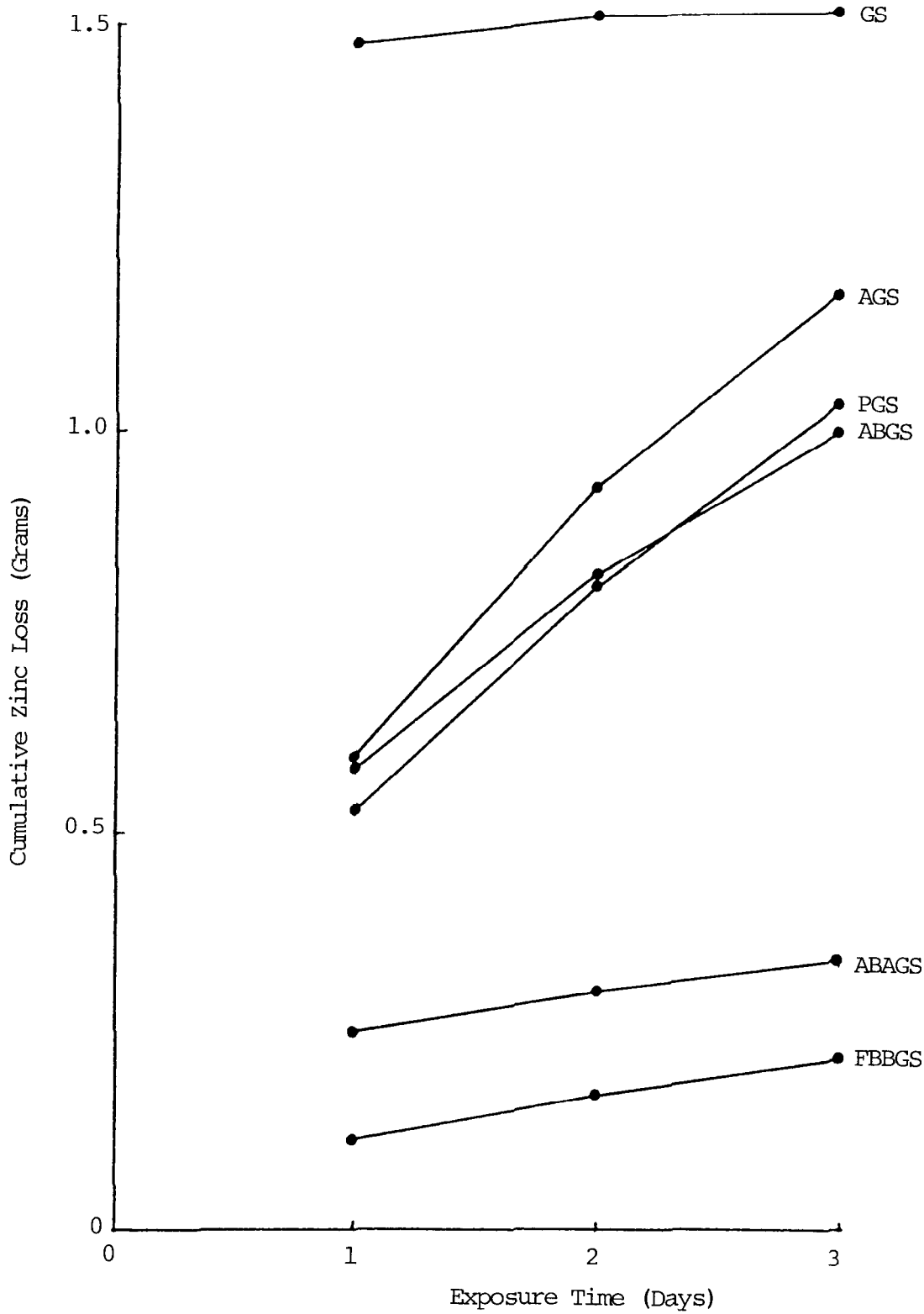
<u>Coupon</u>	<u>Weighing, g</u>	<u>AA, g</u>	<u>% difference*</u>
Galvanized	2.46	2.48	8.2
	2.91	2.33	
	2.73	2.43	
	<u>2.89</u>	<u>2.85</u>	
	2.75	2.52	
Asphalt	1.48	2.02	1.7
	1.96	1.62	
	<u>1.70</u>	<u>1.58</u>	
	1.71	1.74	
Blackclad	1.48	1.53	7.0
	1.58	1.40	
	<u>1.70</u>	<u>2.21</u>	
	1.59	1.71	
Asbestos	1.49	1.55	4.1
	1.28	1.43	
	<u>1.39</u>	<u>1.37</u>	
	1.39	1.45	
Asbestos-Asphalt	0.853	0.843	1.0
	0.888	0.962	
	<u>0.848</u>	<u>0.812</u>	
	0.863	0.872	
Fiber-Bituminous	0.815	0.753	5.6
	0.802	0.752	
	<u>0.790</u>	<u>0.765</u>	
	0.802	0.757	

* % difference is defined as the difference between the two methods divided by the larger number.

Since the metal is composed of zinc and iron, these AA numbers should theoretically correspond to the weight loss values. In the AA analysis, the 250 ml of solution from each test day are completely digested and then run for zinc and iron using AA. Plots of the cumulative zinc and iron are presented in Figures 13 and 14, respectively. These data allow a plot to be constructed of the cumulative weight of zinc and iron for each day. Figure 15 shows a typical weight loss plot which can be constructed using daily AA data. The average AA results in Table 5 are within $\pm 8\%$ of the weight loss data from weighing.

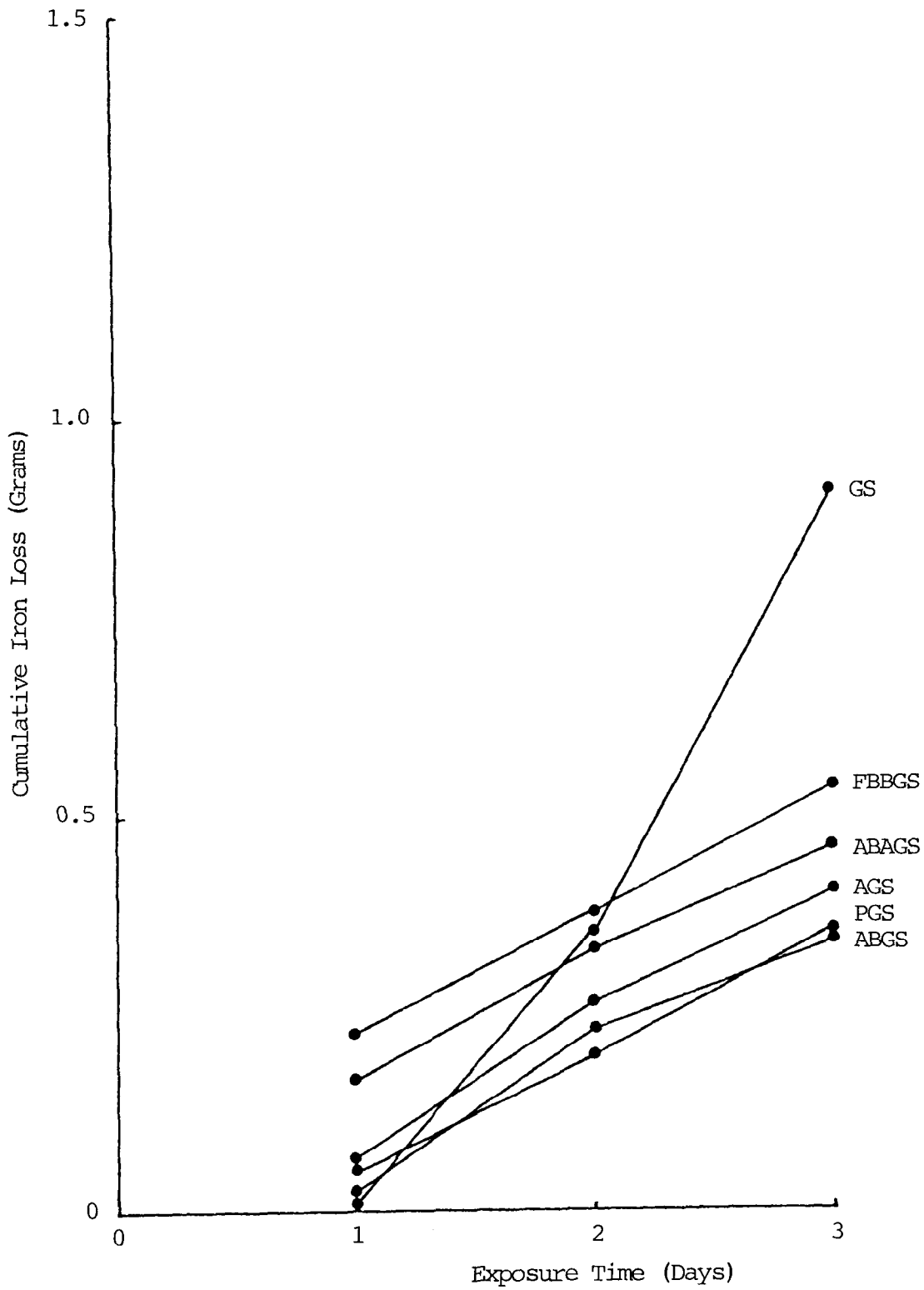
3. Test No. 3. In an attempt to evaluate the effect of pH on the experiments performed in the Baroid cells, a set of tests were performed using the standard solution but excluding acetic acid. The resultant test solution had an initial pH of 5.7. The procedure followed in this test is exactly the same as in Test No. 2. The test solutions were changed every 24 hours, but the coupons were left in the cell until the end of the third day. They were then heated at 130° F for six hours and the weights recorded. The results for a set of runs of the six culverts under examination are presented in Table 6. The order of the culverts is the same as found in Test No. 1. These results are presented graphically in Figure 16. The AA test was run on the test solution that was removed each day. The daily zinc and iron results of this test are shown graphically in Figures 17 and 18, and the cumulative results are presented in Table 6 and shown in Figure 19. pH measurements made on the test solution revealed that the solution became very alkaline after 24 hours. The results of these pH measurements are presented in Figure 20. One observation of these tests was that the samples began to precipitate some scale which can affect an accurate determination of the weight loss.

4. Test No. 4. In an attempt to provide a better means of discriminating between coatings, it was decided to increase the temperature of the pressurized Baroid cells to 130° F. The three-



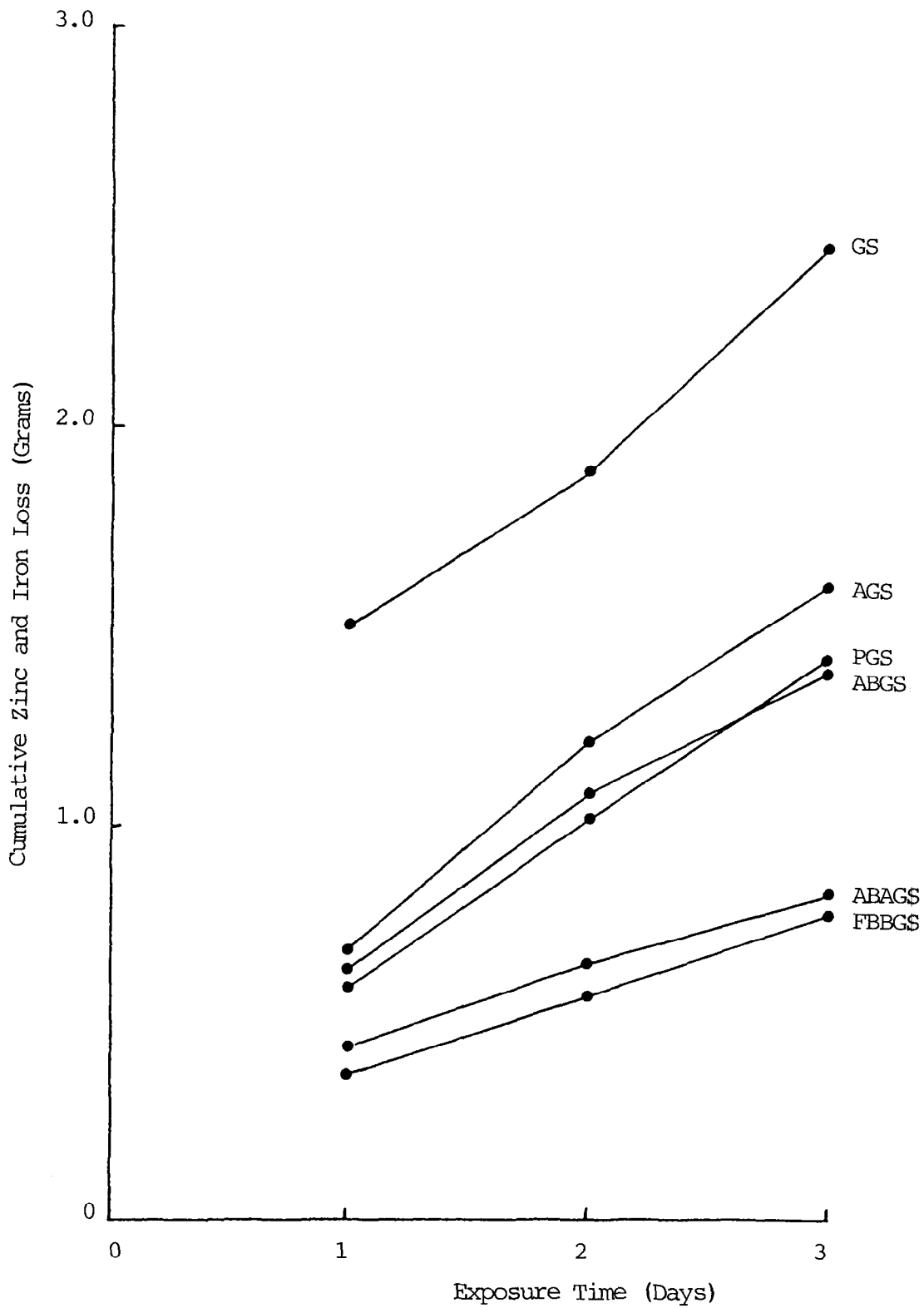
Cumulative Zinc Weight Loss in High-Pressure Oxygen Test No. 2

FIGURE 13



Cumulative Iron Weight Loss in High-Pressure Oxygen Test No. 2

FIGURE 14



Cumulative Zinc and Iron Loss in High-Pressure Oxygen Test No. 2

FIGURE 15

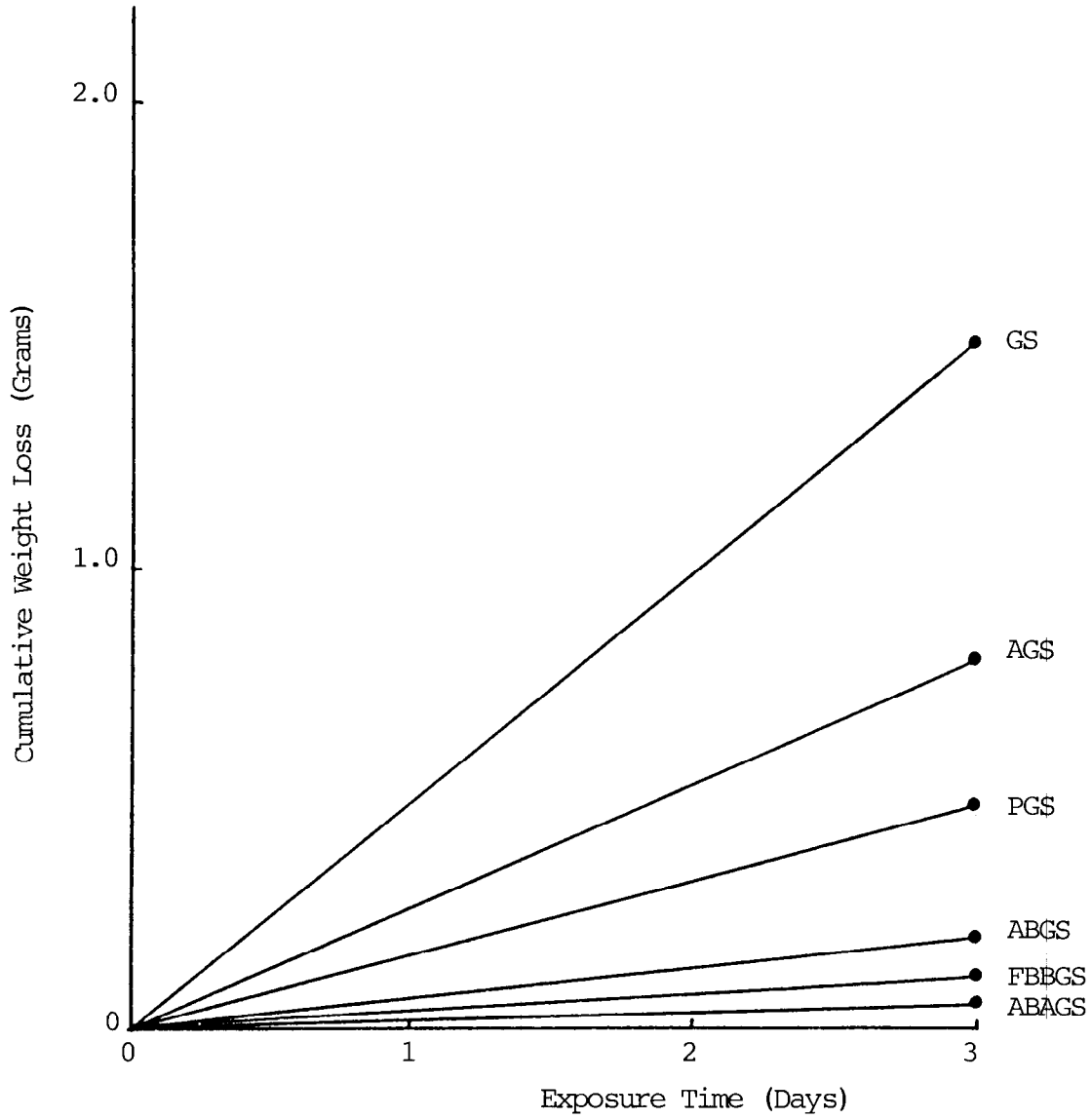
Table 6

HIGH PRESSURE OXYGEN TEST NO. 3

Weight Loss from Weighing and Atomic Absorption (AA)

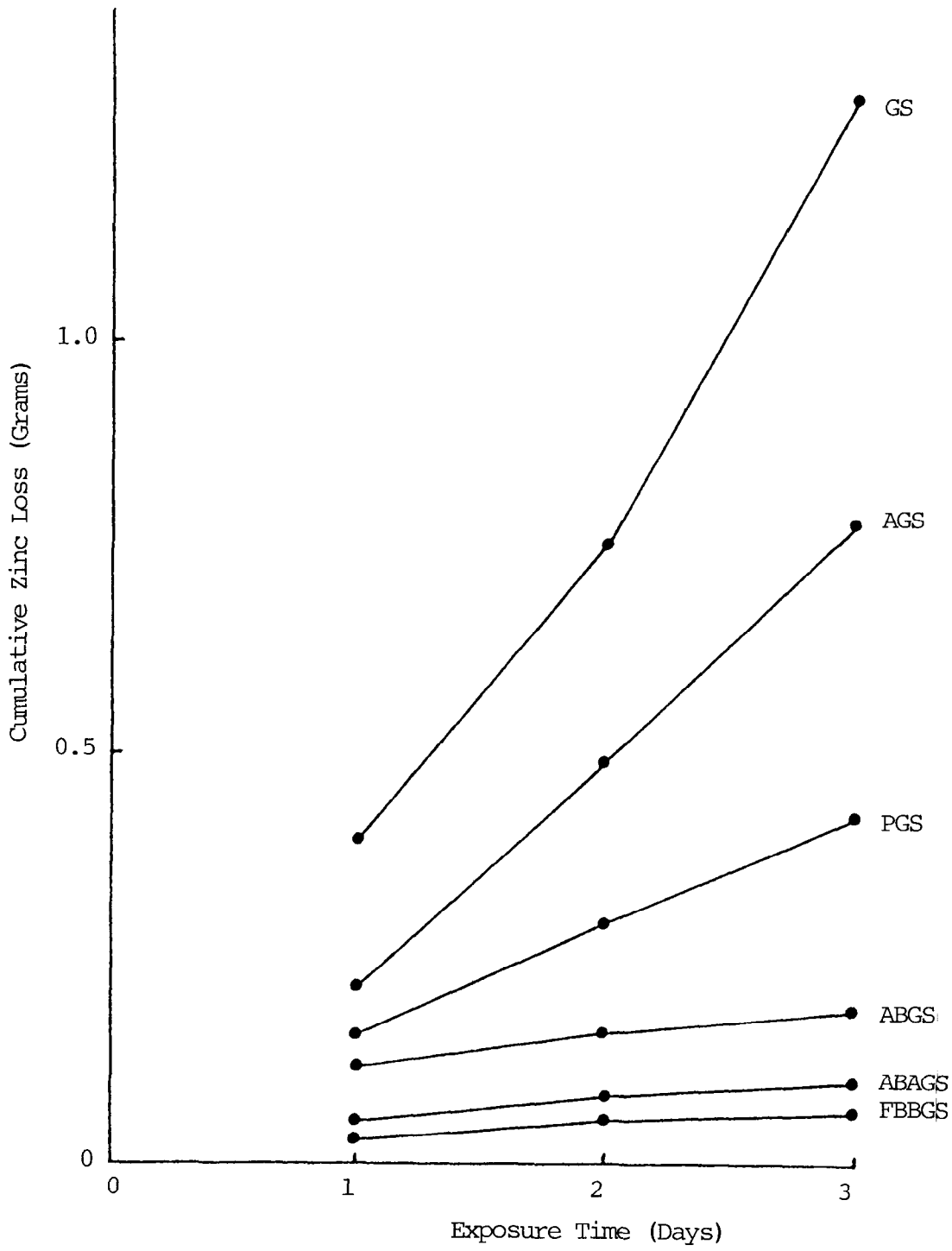
<u>Coupon</u>	<u>Weighing, g</u>	<u>AA, g</u>	<u>% difference*</u>
Galvanized	1.48	1.31	11.0
Asphalt	0.810	0.784	3.2
Blackclad	0.486	0.430	11.5
Asbestos	0.196	0.239	18.0
Fiber-Bituminous	0.105	0.104	1.0
Asbestos-Asphalt	0.078	0.112	30

* % difference is defined as the difference between the two methods divided by the larger number.



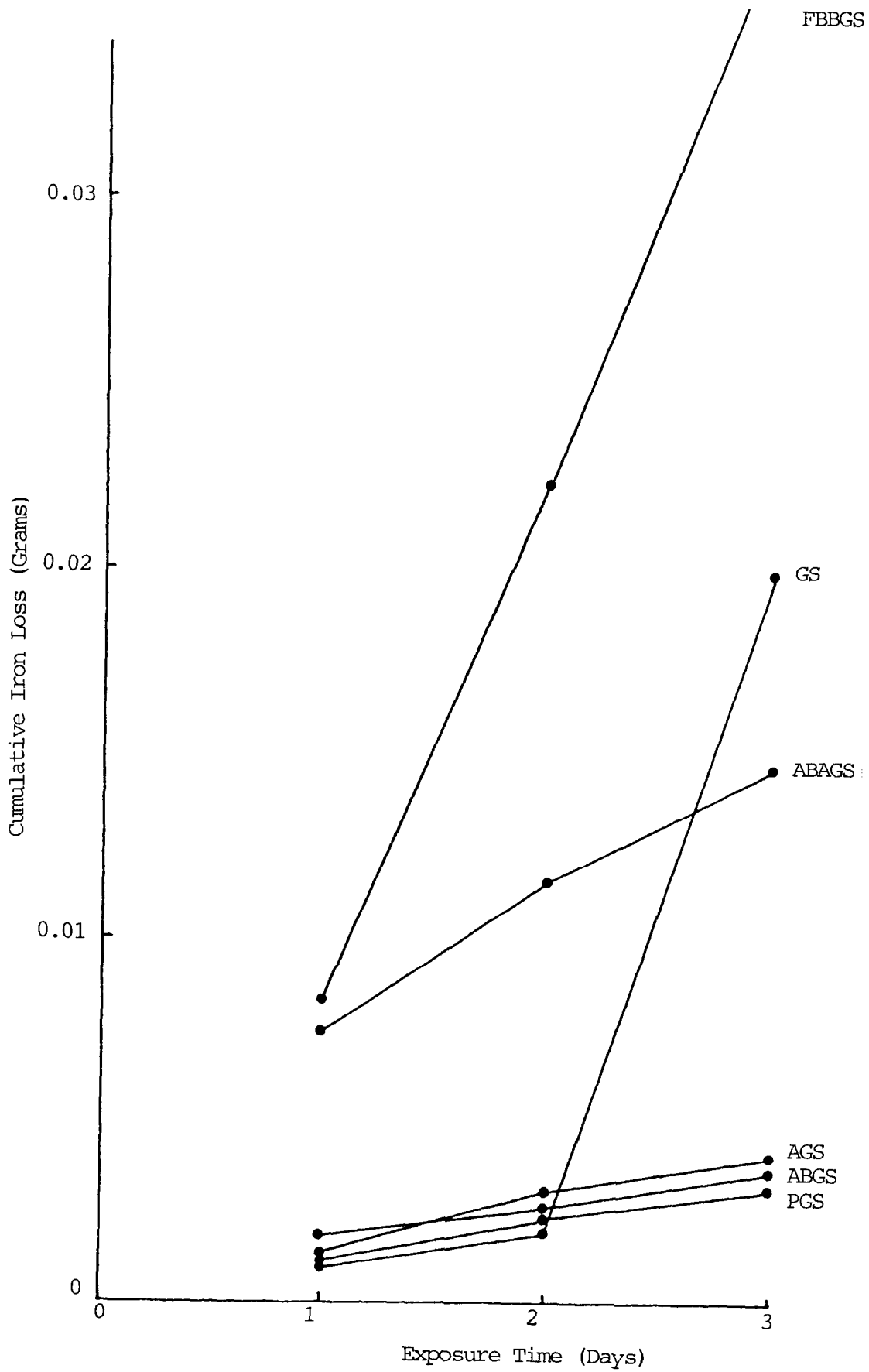
Cumulative Weight Loss in High-Pressure Oxygen Test No. 3

FIGURE 16



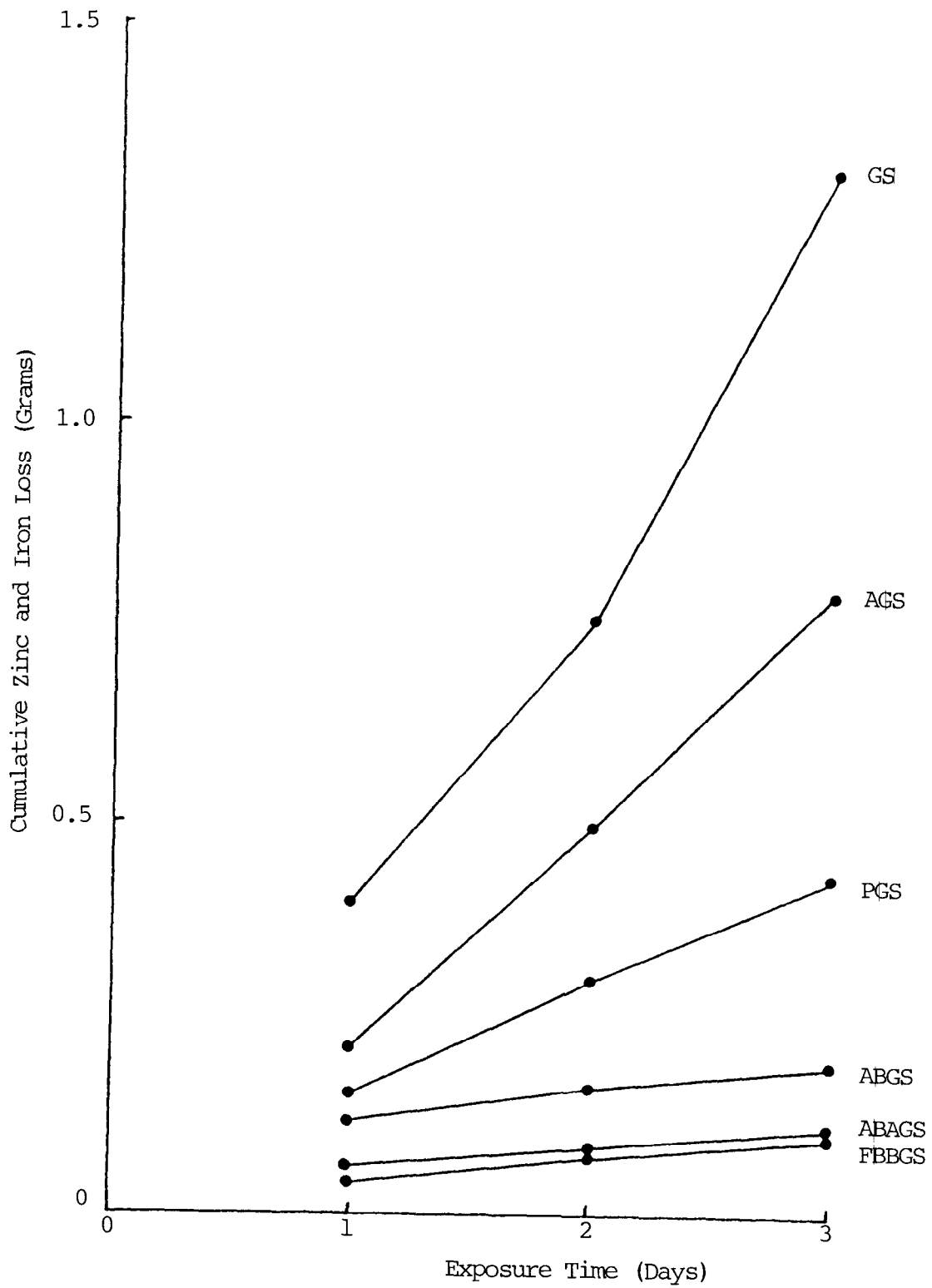
Cumulative Zinc Weight Loss in High-Pressure Oxygen Test No. 3

FIGURE 17



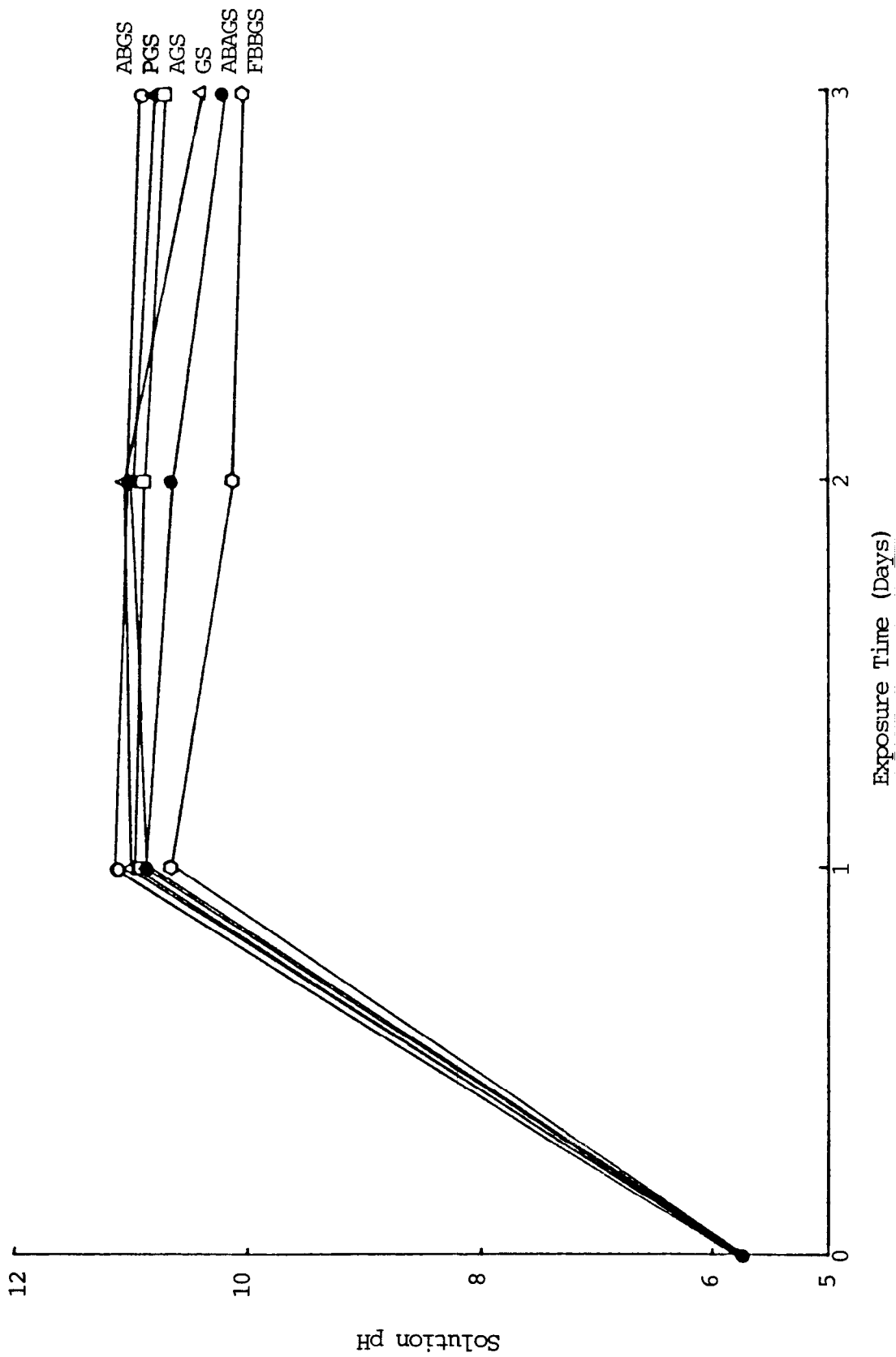
Cumulative Iron Weight Loss in High-Pressure Oxygen Test No. 3

FIGURE 18



Cumulative Zinc and Iron Loss in High-Pressure Oxygen Test No. 3

FIGURE 19



Solution pH of High Pressure Oxygen Test No. 3

FIGURE 20

day experiments used the same procedure as Test No. 1, with the Baroid cells being pressurized with oxygen to 500 psi before being placed inside of an oven. The cumulative weight loss of the six culverts under examination is given in Table 7, and a plot of these data is shown in Figure 21.

These results are somewhat different than those obtained at room temperature. The asphalt-asbestos material was still the best but the blackclad performed much better, and the asbestos was much worse. Figure 22 shows the pH results for this test. As seen previously, there does appear to be a difference between pH values after the first day of exposure. On the second and third days this difference does not exist.

5. Test No. 5. Since the test coupons are touching the 316SS Baroid cells, there is a galvanic effect that should be examined. In this experiment a 2" X 2" coupon was weighed and placed inside a plastic cup which fit tightly inside the Baroid cell. The same procedure as Test No. 1 was used. The standard amount of test solution, 200 ml, was placed inside the cell. The cell was then pressurized to 500 psi with oxygen. In this fashion, it was clear that there was no possible electrical contact between the coupon and the cell. Figure 23 shows the results of the no-contact test in which the weight loss of asbestos, asphalt-asbestos, and fiber-bituminous coupons were determined each day for a three-day period. The weight losses were very low and the curves tend to overlap with no significant difference between them.

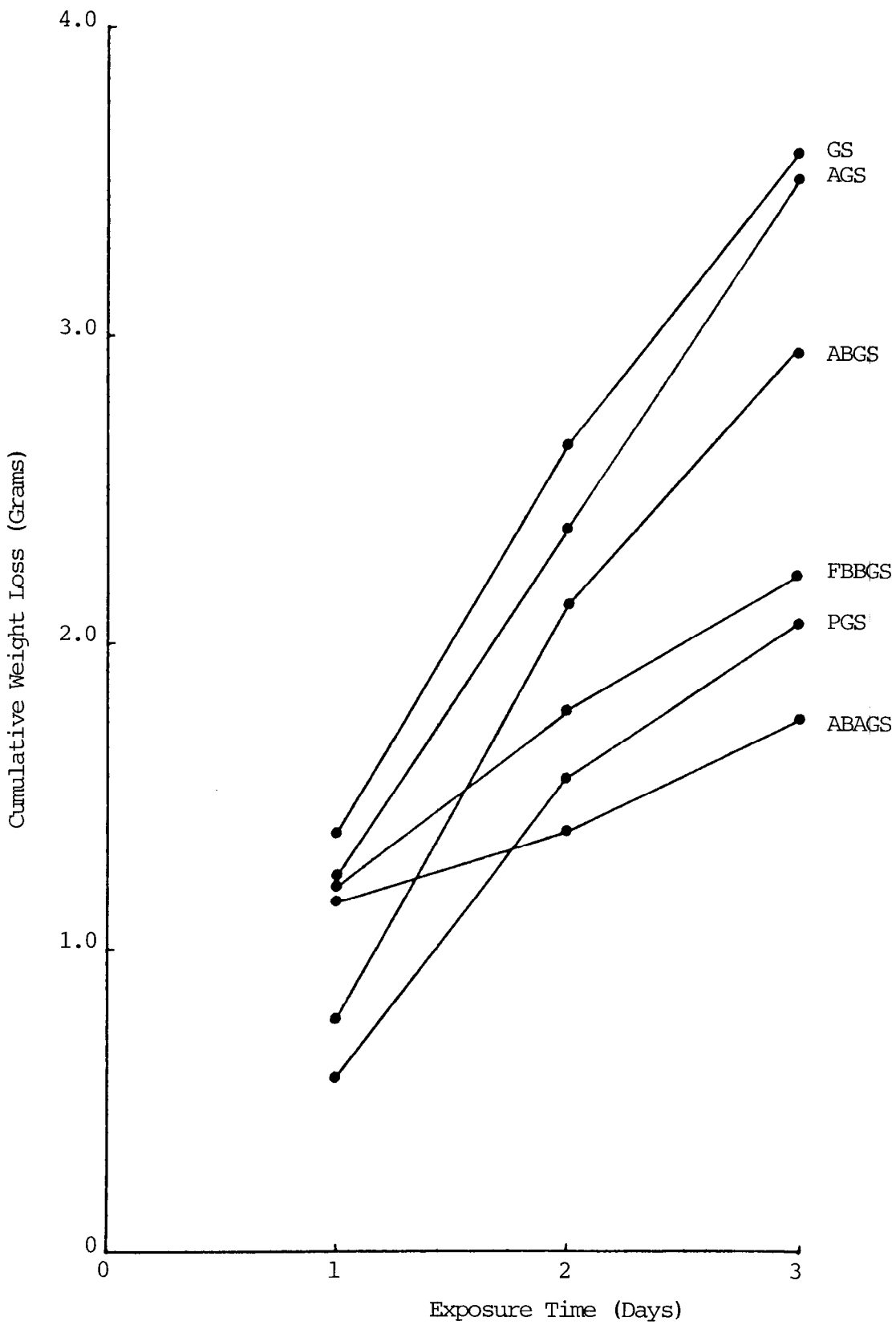
The same three types of coupons were then tested for three days, making sure that electrical contact was made with the Baroid cells. This is the condition under which all previously run experiments were conducted. The 200 ml of solution was again used and the oxygen pressure was 500 psi. These results shown again in Figure 23 are higher than the no-contact results by a factor of four. These tests were conducted during December break when there was no heat in the Engineering building, so these results are not directly comparable to previously obtained data.

Table 7

HIGH-PRESSURE OXYGEN TEST AT ELEVATED TEMPERATURE (130° F)

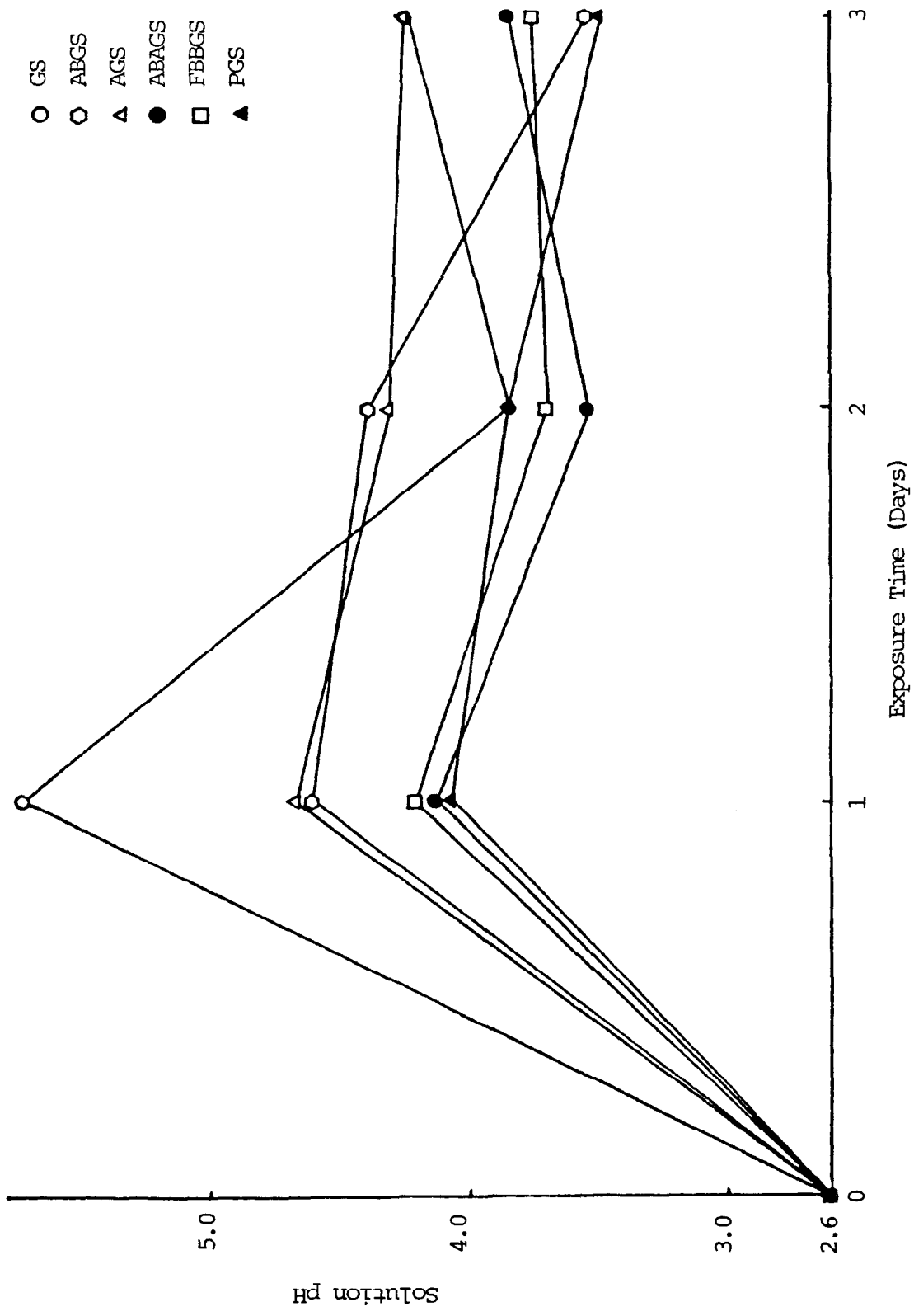
Cumulative Weight Loss, g

	<u>Day 1</u>	<u>Day 2</u>	<u>Day 3</u>
Galvanized Steel	1.278	2.663	3.580
Asbestos-bonded Asphalt-coated	1.173	1.366	1.786
Fiber-bonded Bituminous-coated	1.225	1.783	2.208
Asphalt coated	1.235	2.410	3.503
Asbestos coated	0.756	2.131	2.940
Blackclad	0.575	1.573	2.076



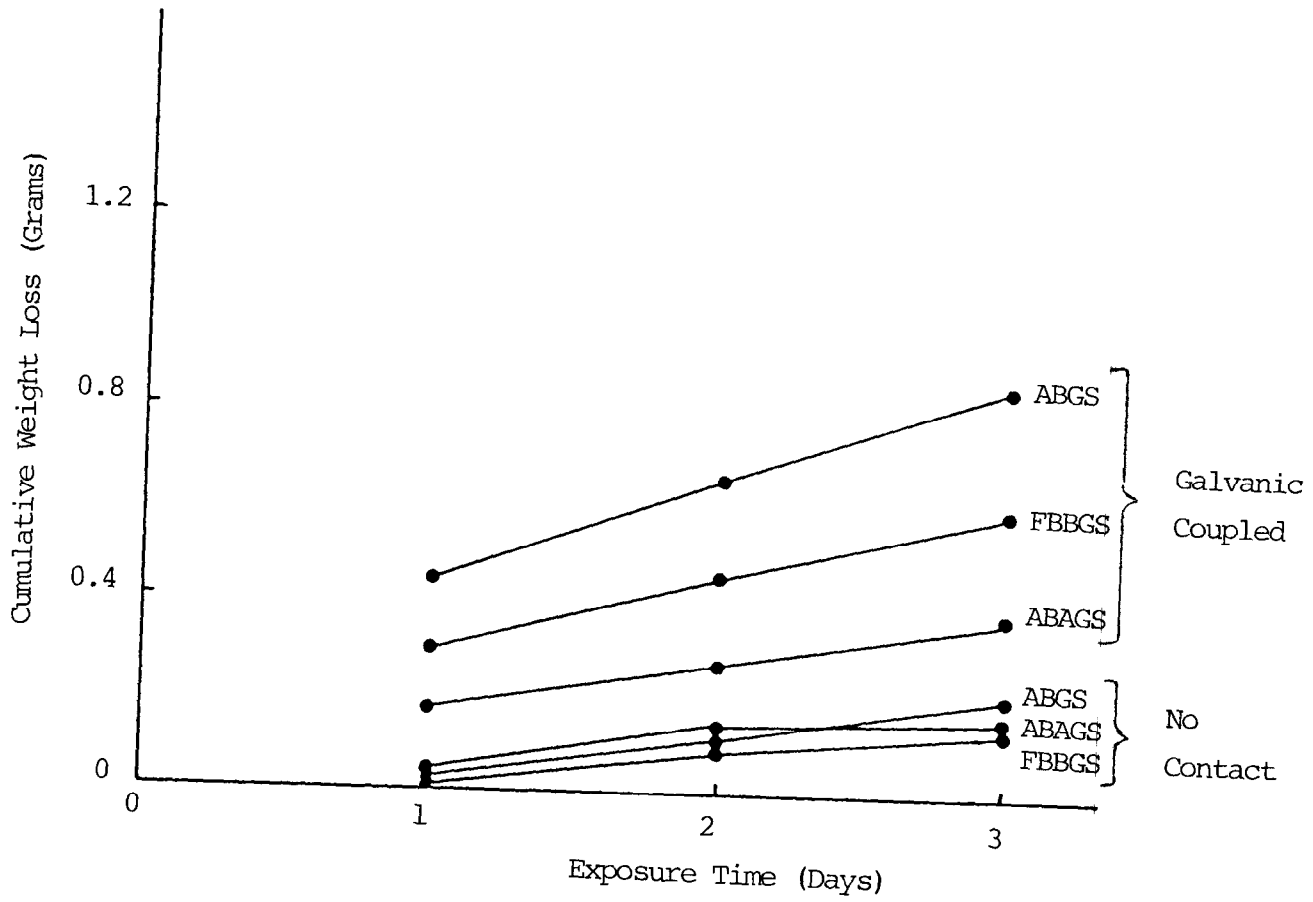
Cumulative Weight Loss in High-Pressure Oxygen Test No. 4

FIGURE 21



Solution pH of High Pressure Oxygen Test No. 4

FIGURE 22



Galvanic Effects of Coupons in 316SS Baroid Cells

FIGURE 23

These results are very significant since they show the importance of how coupons are prepared when placed in the Baroid cells. Since the objective of this project is to accelerate the corrosion rate of coupons, it is suggested that the galvanic corrosion be allowed to occur. However, this requires that the cells be carefully cleaned between each run, and that all four corners of the steel in the coupon must be in electrical contact with the cell at all times. All previous tests were run with the coupon in full electrical contact with the steel.

Anodic Undercutting of Coupons

In both the potentiostat and high-pressure oxygen tests, the metal inside the coating has had its natural potential increased. In the case of low carbon steel or iron, this procedure greatly accelerates the corrosion rate of the metal under the coating. It is our contention that the amount of undercutting which occurs on these coupons should be a consideration in evaluating the effectiveness of coatings. In both test methods, the coupons were 2" X 2" and the edges of the coupons were left exposed to the environment.

The approach taken was to remove the remaining coating from the exposed coupon after the drying and weighing was completed, and measure the exposed area. In most cases, the remaining area of the zinc was highly visible. The coupons were photographed at a power of 1X and the area measured using a planimeter. The result reported was the % zinc left on the coupon after exposure. The asbestos and asbestos-asphalt were the most difficult to measure by this procedure since the attack was more random on these coupons.

The photographs of the coupons exposed to the 1-volt Potentiostat Test after the coating was removed are shown in Appendix A (page 75). The percentage of zinc left on the coupons was measured on both sides for each coupon for the 1-volt potential difference tests. The results of this test are presented in Table 8. The photographs of the 2-volt test are not shown, but the % zinc remaining on each side is given in Table 8. The % zinc reported on both the 1 and 2 volt tests was based on the area of zinc remaining divided by the original surface area of 8 in.².

Table 8

% ZINC REMAINING ON COUPONS*
AFTER POTENTIOSTATIC TEST

1-Volt Potential Difference

<u>Coupon</u>	<u>Figure #**</u>	<u>Side 1</u>	<u>Side 2</u>	<u>Average</u>
Galvanized	A-1	0	--	0
Asphalt	A-2	0	--	0
Blackclad	A-3	16	16.6	16.3
Asbestos	A-4	51.8	28.1	39.9
Fib/Bit.	A-5	40.1	50.8	46.4
Asb/Asphalt	A-6	71.5	--	71.5

2-Volt Potential Difference

<u>Coupon</u>	<u>Side 1</u>	<u>Side 2</u>	<u>Average</u>
Asphalt	2.7	15.4	4.5
	0	--	
Asbestos	28	26.3	28.2
	28.1	30.4	
Blackclad	38	30	34.4
	38	30.5	
Fib/Bit.	29.3	26	37
	49.4	43	
Asb/Asphalt	42.5	45.6	44.1

* Expressed as a percentage of surface area

** See Figures in Appendix A

The photographs of coupons exposed to the High-Pressure Oxygen Test No. 2 are shown in Appendix B (page 82). The coatings were removed after exposure, and the zinc remaining was clearly visible. In this test the initial solution pH was equal to 2.7 and the test was run at ambient temperature. Since the attack was less severe than the Potentiostat Test, the coupon dimensions did not change. The results from examining both sides of the coupon are presented in Table 9. The results show the same general order as seen in the weight loss data (page 34). The weight loss data and photographs of these repeated tests show that there is a high degree of consistency in the High-Pressure Oxygen Test No. 2.

Coupons from the High-Pressure Oxygen Test No. 3 were examined in the same fashion as described in Test No. 2. The % zinc remaining on the coupons has been determined and is given in Table 10. The initial pH of 5.7 used in this test reduced the general level of zinc removal. The samples experienced some scaling due to the high pH levels produced during the test.

Test coupons from the High-Pressure Oxygen Test No. 4 conducted at an elevated temperature of 130° F and initial pH of 2.7 were examined for the % zinc remaining. Table 11 shows that in spite of the higher temperature, the % zinc remaining is higher than it was at lower temperatures. This can be clearly seen by comparing the results in Table 11 with those of Test No. 2 shown in Table 9 (page 54).

Examination of Culvert Metal and Coating

The six culvert materials that were used in this project were all low carbon steel, essentially iron, with zinc on the outside of the steel. Since zinc is a very important component in the corrosion process, it was decided to determine the zinc content on the various culverts. Zinc was removed from the 2" X 2" coupons by placing them in concentrated hydrochloric acid for 24 hours. The entire liquid sample was digested and run on the Atomic Absorption Unit (AA). Duplicate runs were made and in some cases the test was performed three times until representative results were obtained. Table 12 shows the final results and the average values. Most of the samples have very similar zinc content except for the fiber bituminous, which contained less than half of the zinc of the other samples. Blackclad also showed a slightly

Table 9

% ZINC REMAINING ON COUPONS* AFTER
HIGH PRESSURE OXYGEN TEST NO. 2

<u>Coupon</u>	<u>Figure #**</u>	<u>Side 1</u>	<u>Side 2</u>	<u>Average</u>
Galvanized	B-1	0	--	0
	B-2	0	--	0
Asphalt	B-3	31.3	31.1	
	B-4	22.1	21.8	22.6
	B-5	0	30.2	
Blackclad	B-6	20.1	33.1	
	B-7	31.9	26.1	
	B-8	32.6	32	29.2
	B-9	25.9	31.9	
Asbestos	B-10	65.5	42.2	
	B-11	54.6	39.5	50.5
Fib/Bit.	B-12	63.2	63.5	
	B-13	64.4	58.8	60.2
	B-14	55.4	56.1	
Asb/Asphalt	B-15	89.5	88.6	
	B-16	83.5	88.5	85.9
	B-17	80.0	85.5	

* Expressed as a percentage of surface area

** See Figures in Appendix B

Table 10

% ZINC REMAINING ON COUPONS* AFTER
HIGH PRESSURE OXYGEN TEST NO. 3

<u>Coupon</u>	<u>Side 1</u>	<u>Side 2</u>	<u>Average</u>
Galvanized	0	--	0
Asphalt	28.3	71.5	49.9
Blackclad	67.1	70.9	69.0
Asbestos	72.0	73.8	72.9
Fib/Bit	82.0	84.1	83.0
Asb/Asphalt	84.0	92.4	88.2

* Expressed as a percentage of surface area

Table 11

% ZINC REMAINING ON COUPONS* AFTER
HIGH PRESSURE OXYGEN TEST NO. 4

<u>Coupon</u>	<u>Side 1</u>	<u>Side 2</u>	<u>Average</u>
Galvanized	0	--	0
Asphalt	0	--	0
Blackclad	68.8	70.5	69.7
Fib/Bit.	78.2	81.0	79.6
Asbestos	71.3	91.2	81.2
Asb/Asphalt	84.8	93.1	89.0

* Expressed as a percentage of surface area

Table 12

ZINC CONTENT OF A 2" X 2"
COUPON FROM EACH CULVERT MATERIAL

<u>Coupon</u>	<u>Zinc Content, g</u>	<u>Average</u>	<u>Grams Zinc/in²</u>
Galvanized	1.52, 1.52, 1.75	1.60	0.20
Asphalt	1.70, 1.54, 1.72	1.65	0.21
Blackclad	1.48, 1.58, 1.56	1.54	0.19
Asbestos	1.64, 1.66	1.65	0.21
Asb/Asphalt	1.70, 1.58	1.64	0.21
Fib/Bit.	0.71, 0.78, 0.58	0.69	0.086

lower value than the average. Table 12 also contains a factor of grams zinc/in² which allows a quick comparison between the various galvanized culverts. The majority of the steels contain 0.21 g. zinc/in².

Another important consideration in this testing was the effect that various solutions have on the coatings. Literature on asphalt revealed that it is insoluble in water, alcohol, acid, and alkali solutions; however, no information could be found specifically on bituminous coatings. A series of experiments was conducted to check the solubility of this coating in the various test solutions. The experiment was conducted in the Baroid cells for three days at room temperature and 500 psi pressure. The results of bituminous coating weight loss in the two solutions used in the High-Pressure Oxygen Test Are:

<u>Solution</u>	<u>Coating Weight Loss, g</u>
Standard Solution, pH = 2.7	0.012
Standard Solution, pH = 5.7	0.009

These numbers reveal that the weight loss of the bituminous coating at the conditions of the Baroid cell tests is low. However, the weight loss of the fiber bituminous culvert in these solutions was 0.802 g and 0.104 g, respectively. Therefore, the coating loss at pH of 2.7 is 1.5% while at the pH of 5.7 it is 9% of the total weight loss. Therefore, these numbers are significant in the High-Pressure Oxygen Test No. 3.

DISCUSSION OF RESULTS

The objective of this project was to develop an accelerated test to help the Louisiana Highway Department select the best culvert coating material from among the large group that is available. The primary criteria was that the test results must correlate with results found in the field. This study considered the following six culverts provided by the Louisiana Department of Transportation: galvanized, asphalt galvanized, blackclad galvanized, asbestos galvanized, fiber-bituminous galvanized, and asbestos-asphalt galvanized. Each of these culvert metals was checked microscopically and found to be a very fine-grained, low carbon steel (essentially iron). Elemental analysis using the scanning electron microscope revealed the existence of only zinc and iron on the metal. Their zinc content was determined by atomic absorption (AA), and the results, presented in Table 12 (page 57) show that they averaged 0.21g zinc/in² except for fiber-bituminous and blackclad, which averaged 0.086 and 0.19g zinc/in², respectively.

Since the time for testing had to be relatively short (a matter of days), it was decided to use test methods which raised the potential of the culvert metal above its natural potential and expose it to a corrosive environment. A discussion of two test methods and an interpretation of their results follows.

Potentiostat Test

The Model 363 Potentiostat has the capability of raising and maintaining the potential of a metal several volts above its natural potential if necessary. The first test performed using this equipment involved placing two 2" X 2" test coupons in two liters of the test solution at ambient temperature and maintaining a 1-volt potential difference between them. The coupons were weighed and dried every 24 hours for the duration of each run. Figure 5 (page 21) shows that based on weight loss, this test was able to reveal the poorest coating, which is asphalt. However, it had a more difficult time distinguishing between the better coatings, such as blackclad, asbestos, fiber-bituminous, and asbestos-asphalt. Measurement of the amount of zinc remaining on the coupons after testing was performed. The results, presented in

Table 8 (page 52), look promising. The galvanized and asphalt coupons had all of their zinc completely removed during testing and there was a distinct difference between the other four coatings, with blackclad losing the most zinc, and asbestos-asphalt losing the least.

In an attempt to better discriminate between coatings, three additional potentiostat tests were developed, which produced the following results.

1. Elevating the temperature to 125° F and using a 1-volt potential difference produced a significant spread in the coatings (Figure 7, page 23). This method showed the asbestos-asphalt to be the best material and the asbestos to be the worst. The blackclad material looked much better at this higher temperature. There is concern that at the higher temperature there are signs of deterioration of the organic coating. Also, the zinc is known to become cathodic to steel as the temperature increases. For these reasons, the high temperature results are somewhat suspect.
2. Since a more aggressive environment will cause rapid deterioration of the galvanized steel, it was decided to use a 5% hydrochloric acid solution at ambient temperature for testing. The potential of the coupon was elevated 0.5 volts above its natural potential by means of a reference electrode. The two-day test (Figure 8, page 25) shows results which are very different than those seen previously. The blackclad material looks the best while the fiber-bituminous looks worst. Again there was considerable damage to the coating with a heavy sediment forming at the bottom of the test cell. This deterioration of the organic coatings most likely led to these materials performing so poorly in this corrosive environment.
3. Returning to the original conditions of ambient temperature and the standard test solution, it was decided to run the potentiostat at a 2-volt potential difference between coupons. Table 2 (page 26) indicates the weight loss numbers were very large, with the asbestos-asphalt and blackclad showing the lowest weight loss and the asbestos and asphalt showing the greatest loss. After testing, the coupons were approximately half their

original size. Therefore, the test is extremely hard on all the coupons, and for this reason it may not be possible to discriminate between good and poor coatings. Based on the original coupon area of 4 in², the % zinc remaining ranged from 4.5% for asphalt to 44.1% for asbestos-asphalt (Table 8, page 52). It should be noted that this test method caused damage to the asphalt and bituminous coatings. Some coating fell off since the coupon size was reduced so dramatically.

High-Pressure Oxygen Test

Based on the literature survey (21), it is clear that the dissolved oxygen concentration in the solution has a very significant impact on increasing the corrosion rate. In addition to this, it was demonstrated that by having the culvert metal in electrical contact with the 316SS Baroid cells, there is a galvanic couple which occurs that accelerates the corrosion rate of the culvert metal by a factor of four above the non-contact condition. Therefore, when using this testing method, it was necessary to make sure that all four edges of the coupon were in electrical contact with the cell.

A total of four different high-pressure oxygen tests were conducted on the culvert materials. All of the tests described below were run at a total oxygen pressure of 500 psi.

1. Test No. 1. In this test, the coupons were removed from the cell every 24 hours, dried for two hours at 130° F, weighed, and placed back into the cell with a fresh solution. The results shown in Figure 10 (page 31) are similar to those seen in the 1-volt potentiostat test (Figure 5, page 21). However, these results show a better spread of the culvert materials and allow for a better evaluation to take place. The asbestos-asphalt and fiber-bituminous coatings appear to perform the best while the asphalt is the worst. In the ten-year field study, the asphalt material was much worse than the asbestos-asphalt. Therefore, from known field results, this procedure appears to give good agreement.

The pH of the resultant test solution for each day is shown in Figure 11 (page 32). Examination of this data, tabulated in

Table 4 (page 33), reveals a good spread after the first day. However, there does not appear to be any consistency between these numbers and the weight loss values. For example, the pH of the blackclad solution had changed the least, but it showed the third largest weight loss in Figure 10 (page 31). Also, there is no spread between the pH values of blackclad, fiber-bituminous, and asbestos-asphalt. For these reasons, the pH method of evaluating coatings does not appear to be a viable approach in this test.

2. Test No. 2. This test procedure was developed for three reasons:
- (1) this method is less labor-intensive than Test No. 1,
 - (2) there was some concern that heating the coupon for two hours at 130° F between 24-hour runs could affect the results, and
 - (3) repeated handling of the coupons could cause some coating to be lost, which would produce a higher weight loss value.
- This method also introduced the use of atomic absorption (AA) to measure the zinc and iron in the test solution. Since zinc and iron are the only elements in the culvert material, their measurement should allow a material balance to be performed.

Table 5 (page 35) lists the results of several sets of runs which were performed using the same conditions of temperature, pressure and test solution as Test No. 1. The only difference is that the coupons were left in the cell until after the third day of testing. The results show that the method gives consistent results. The results of the test are similar to Test No. 1, except that the fiber-bituminous lost significantly less weight in Test No. 2 (Figure 12, page 34). The asbestos-asphalt also lost less weight with this procedure. These results were somewhat expected since it is believed that handling coupons with heavy coatings can cause pieces to fall off, which contributes to larger weight loss values.

The solution from each 24-hour test period was completely digested and analyzed for zinc and iron using atomic absorption. In this manner it is possible to monitor the corrosion rate of the metal on a daily basis. Figures 13 (page 37) and 14 (page 38) show the daily amount of zinc and iron, respectively, which went

into solution. The sum of these two figures is plotted in Figure 15 (page 39) as the total weight loss by atomic absorption. Although Figures 13, 14, and 15 are for a specific run, the values of average weight loss by AA in Table 5 (page 35) are seen to be within $\pm 8\%$ of the weight loss values which were obtained by weighing the coupon. The order of the coupons from lowest to highest weight loss exhibited by Figures 13 and 15 are the same as for the actual measured weight loss in Figure 12.

Most of the test coupons listed in Table 5 were photographed at 1X and examined visually to determine the location of zinc on the coupon. The photographs, shown in Appendix B (page 82), were measured for the amount of zinc remaining and these results are tabulated in Table 9 (page 54). These results generally agree with the weight loss data except that the asbestos-asphalt is significantly better than the fiber-bituminous material and asbestos is significantly better than the blackclad.

The difference between the % zinc removed numbers and the weight loss numbers, either by weighing or atomic absorption, can be explained on the basis that the fiber-bituminous and blackclad materials have less zinc on the coupons than the other culvert materials. Since zinc is the most significant element lost in the corrosion process, these coupons lose proportionally less zinc, as is shown in Figure 13 (page 37). This means that fiber-bituminous and blackclad look better when comparing total weight loss data as seen in Figure 12 (page 34).

3. Test No. 3. The effect that pH has on culverts in the High-Pressure Oxygen Test has been examined in this test. The same procedure as Test No. 2 was followed with the exception that the acetic acid was left out of the standard test solution. This means that the initial solution pH was 5.7 instead of 2.7. Figure 16 (page 41) shows the weight loss values for the various culvert materials. These values are substantially less than the values in Test No. 1, but the order of coupon weight loss is essentially the same, except that asbestos-asphalt is slightly better than fiber-bituminous. The daily atomic absorption

values for zinc and iron are shown in Figures 17 (page 42) and 18 (page 43), respectively. Figure 19 (page 44) shows the cumulative zinc and iron weight loss in this test. It can be seen from Figure 18 that very little iron was lost from most of the coupons. Zinc was again the primary corrosive element. A comparison of weight loss values from weighing and atomic absorption are given in Table 6 (page 40) and the agreement is reasonably good considering the magnitude of the numbers involved.

The test coupons were photographed, visual examination was performed, and the amount of zinc remaining was measured with a planimeter. Table 10 (page 55) shows that the average values of % zinc remaining are significantly higher than those obtained in Test No. 2. This method shows the asbestos-asphalt and fiber-bituminous to be very close. The asbestos value is somewhat lower than expected, which probably arises from the difficulty of measuring the area of zinc remaining on this coupon.

In this test, the pH values at the end of each run were measured. The resulting values of pH were very alkaline as can be seen from Figure 20 (page 45). The values were all very close, and it was not possible to see any correlation in the data. It was noted that at these high pH values there is considerable precipitation that occurs. This produces a problem in trying to make a material balance on the coupons. This one problem makes the results of this test method suspect.

4. Test No. 4. As in the case of the potentiostat test, it was decided to elevate the temperature to see how this parameter affected the High-Pressure Oxygen Test. The procedure was the same as Test No. 1, except that the Baroid cells were placed in an oven. The weight loss from each day was determined and recorded in Table 7 (page 47). Figure 21 (page 48) shows a graph of this three-day test, which again showed the asbestos-asphalt to be the best coating and asphalt the worst. The blackclad showed great improvement over the ambient temperature result in Figure 10 (page 31).

The % zinc remaining on the coupons was measured. These results, listed in Table 11 (page 56), are much higher than expected. The blackclad, fiber-bituminous, asbestos, and asbestos-asphalt all show % zinc values of 70% or higher. These results verify the literature statement that at temperatures above 120° F the zinc will begin to become cathodic to steel. In these systems, the steel will cathodically protect the zinc. It is for this reason that it is believed that high-temperature tests are a poor way to evaluate coatings on galvanized steel culverts.

The pH of the test solutions was evaluated and the results are shown in Figure 22 (page 49). There is a large spread in the pH after the first day of testing, and in general the coatings with the lowest weight loss showed the smallest change in pH. The only discrepancy is that the pH of the blackclad was lower than the asbestos-asphalt.

Comparison of Weighing, AA, and Area Analysis

It is clear that there are several methods available to evaluate the results of the Potentiostat and High-Pressure Oxygen Tests. The most common and easiest method is the weight loss, which is excellent when using a single metal having no coating. However, in a coated sample, if the coating can crumble during handling or deteriorate during the test, this will lead to a higher than actual weight loss. If galvanized steel is being used it is important that each of the metals have approximately the same amount of zinc/in², or it will not be possible to directly compare the various culverts. In this study, it was stated that most of the metals contained 0.21g zinc/in², blackclad- 0.19g and Fib/Bit- 0.086g zinc/in². These problems mean that using only weight loss as an indicator could lead to some difficulties in evaluation.

Since a chemical analysis has been performed on the galvanized steel, and it is known to contain only iron and zinc, it is possible to use an analytical method such as atomic absorption to measure these elements and establish the amount of metal loss. In the High-Pressure Test Nos. 2 and 3, the amount of zinc and iron was determined for each

of the three days. Table 5 (page 35) shows that for Test No. 2, the weight loss from atomic absorption was within $\pm 8\%$ of the actual weight loss. Most importantly, the amount of total zinc loss by each sample can be found by this method. If the original amount of zinc on the sample is known, it is then possible to calculate the % zinc left on the coupon.

The third method of evaluating the exposed coupon is to physically measure the amount of zinc remaining on the coupon. The photographs in Appendices A and B allow one to use a planimeter to measure the area of zinc remaining on the coupon. Tables 8 (page 52), 9 (page 54), 10 (page 55), and 11 (page 56) show the results of this area analysis which is a laborious task. On some of the coupons, such as galvanized steel, asphalt, blackclad, and fiber-bituminous, it is very easy to see the zinc remaining. The asbestos samples are very difficult to analyze by this method since the attack is more at random on the metal surface. The asbestos-asphalt is not as much a problem, but it can be difficult to remove all of the coating. The asphalt is often times attacked inside a remaining area of zinc, making it difficult to measure the area accurately.

A comparison was made of the % zinc remaining on a coupon by the methods of atomic absorption and area analysis. The coupons from the High-Pressure Oxygen Test Nos. 2 and 3 were used for comparison. The calculation using AA results was made as follows:

$$\% \text{ zinc remaining} = 100 - \frac{\text{g zinc in solution}}{\text{g zinc on 8 in.}^2 \text{ sample}} \times (100)$$

The area numbers were taken from Tables 9 and 10. The results shown in Table 13 (page 67) reveal the two methods agree very well. The results were very good in Test No. 2, in which a lot more zinc was lost. The weight loss results in Figure 12 (page 34) suggest that the fiber-bituminous was better than the asbestos-asphalt; however, the area method showed 60% zinc left compared to 86% zinc left for asbestos-asphalt. The AA results on zinc predicted 68% of the zinc left for fiber-bituminous and 79% zinc left for asbestos-asphalt. Thus, the AA method was able to correct for the fact that the fiber-bituminous contains less zinc/in² than other samples.

Table 13

COMPARISON OF % ZINC REMAINING ON A COUPON
USING ATOMIC ABSORPTION (AA) AND AREA

<u>Coupon</u>	<u>Test No. 2</u>		<u>Test No. 3</u>	
	<u>AA</u>	<u>Area</u>	<u>AA</u>	<u>Area</u>
Asphalt	29	22.6	53	49.9
Blackclad	33	29.2	73	69
Asbestos	40	50	88	72
Fib/Bit.	68	60	91	83
Asb/Asphalt	79	86	94	88

The results for Test No. 3 are equally impressive since the two methods agree with each other and the order of prediction is the same. Realizing how difficult it is to measure the area of zinc removed with a planimeter, it is clear that the calculated % zinc from AA is a more appropriate method. It also significantly reduces the amount of labor involved.

CONCLUSIONS

Based on the experimental results obtained in this project, a number of conclusions can be made. Some conclusions are of a general nature while others are for a specific test method.

General

1. It appears that the best way to accelerate the corrosion of culvert coupons is to elevate the potential of the metal above its natural potential and place it in a corrosive environment.
2. It was found that the modified NACE solution, which has an initial pH of 2.7 and contains chloride and sulfate ions, gives a corrosive environment which is ideal for evaluating galvanized steel.
3. The high-temperature runs produced results which were significantly different from those at ambient temperature. There was evidence that the elevated temperature caused some damage to the asphalt and bituminous coatings.
4. It was also verified in this work that at a temperature of 130° F zinc starts to become more cathodic to the steel. This tends to change the mechanism of attack, and thus makes the results of high-temperature testing suspect.
5. The method of atomic absorption provides a good technique to determine the amount of zinc and iron in each test solution. It is best to run AA on the entire solution rather than to run it on a portion of the sample.
6. The amount of zinc on each of the culverts was measured to obtain a baseline value. It was found that all of the culverts contained about 0.21 g/in² except blackclad, which contained 0.19 g/in², and fiber-bituminous, which contained 0.089 g/in².
7. Post-test examination of coupons and measurement of the area of zinc remaining with a planimeter appears to be a good way to discriminate between good and poor coatings. However, this method is laborious

and some coupons such as asbestos are attacked at random instead of only at the edges, which makes the method somewhat subjective.

8. Once a good baseline number for the amount of zinc on the coupon is known, it is possible to measure the amount of zinc in the test solution and calculate the % zinc remaining on the coupon. This method is able to adjust for the fact that not all of the coupons contain the same amount of zinc initially.
9. The procedure of drying the coupon only after the three-day test is completed appears to reduce the possibility of coating loss due to handling. Since the coupons with the thickest coatings usually lose the least amount of weight, this can be important.
10. In each test, the pH of the various test solutions shows a differentiation after the first day. Unfortunately, in general the results did not correlate with weight loss or % zinc loss data.
11. The results of all the test methods that were tried correlated well with the results of the ten-year field study conducted by the State of Louisiana Department of Transportation.
12. From an overall view of the project results, it appears that the best test method to use for evaluating coating on galvanized steel is the High-Pressure Oxygen Test No. 2.

Potentiostat Test

1. The 5% hydrochloric acid solution was too aggressive since it appeared to attack the coating as well as the coupon. The acid is also more selective for zinc than for the coupon in general.
2. The 1-volt potential difference test provided some discrimination between coatings, but had difficulty distinguishing between the better coatings. It appears that an area analysis on the % zinc remaining can provide the degree of discrimination needed.
3. The 2-volt potential difference test appears to be too extreme a test condition. The coupon sizes were reduced to half their original size during this test. There was considerable sediment at the bottom

of the test cell, which indicated that the test damaged the organic coating. The area analysis method on the % zinc remaining appears to correlate with the 1-volt test results, although the weight loss numbers did not correlate.

High Pressure-Oxygen Test

1. There is a galvanic couple which occurs when a culvert coupon is placed inside one of the 316 SS Baroid cells. All four edges of the coupon must be in electrical contact with the cell in order to obtain reproducible results.
2. The values of weight loss obtained from zinc and iron measurements on atomic absorption compared within $\pm 8\%$ of the actual weight loss values in Test No. 2.
3. The % zinc remaining on the coupons which was calculated from the atomic absorption values of zinc compare very well with the numbers obtained from area analysis.
4. The standard test solution without acetic acid developed a high pH at the end of the run which allowed scale to form on the coupons. Scale formation means that a material balance cannot be performed.
5. The % weight loss of the bituminous coating alone was found to be about 1.5% at a pH of 2.7 and 9% at a pH of 5.7. This is another reason to use atomic absorption instead of total weight loss as a measure of corrosion rate.

RECOMMENDATIONS

The purpose of this study was to develop an accelerated test to evaluate corrosion of culvert/coating combinations in a relatively short period of time. The study has been completed and the following two test methods are recommended for use to the Louisiana Department of Transportation.

1. It is believed that the High-Pressure Oxygen Test No. 2 is the best accelerated test method developed in this study. The test reduces the coupon handling problem that occurred in Test No. 1. After each day the solution is retained and run for total zinc using atomic absorption. It is recommended that the entire solution be digested so that any problem in sampling will be eliminated. Knowing the zinc baseline value (g/in^2) for the culvert, the % zinc remaining on the sample can be calculated. Appendix C (page 100) gives the recommended AA procedure.

It is necessary that the samples be in electrical contact with the cell, and it is suggested that the tests be run in triplicate. The baseline zinc determination should also be run in triplicate, but need only be done once.

This method does not require that coupon weight loss or % zinc area analysis be performed. These analyses can be added to the test procedure if desired. However, it is believed that comparing the % zinc remaining on the coupon is sufficient.

2. An alternative method which appears to give good results is the 1-volt Potentiostat Test. This method will easily distinguish between the good and poor coatings. If a % zinc remaining area analysis is performed on the coupon after testing is complete, the results will additionally allow discrimination between the good coatings. The large volume of solution used in this test will prevent an accurate zinc determination from being made, since only a portion of the sample can be digested.

BIBLIOGRAPHY

1. W. H. Temple and S. L. Cumbaa, "Evaluation of Metal Drainage Durability Analysis after 10 years," Louisiana Transportation Research Center, Jan. 1986.
2. M. G. Fontana and N. D. Greene, "Corrosion Engineering," 2nd ed., p. 12 McGraw-Hill, 1978.
3. Transportation Research Board, "Durability of Drainage Pipe," p. 7, 1978.
4. F. E. Kulman and S. N. Palica, "Fundamental Parameter in Underground Pipe Corrosion," Pipeline News. Dec., 1963.
5. H. Leidheiser, "Corrosion Control by Organic Coatings," NACE, 1981.
6. C. G. Munger, "Corrosion Prevention by Protective Coatings," NACE, 1984.
7. M. Pourbaix, "Atlas of Electrochemical Equilibria in Aqueous Solutions," NACE, 1974
8. B. Husock, "Appalachian Underground Corrosion Short Course," West Virginia Univ., p. 113, 1971.
9. Transportation Research Board, Report 50, "Durability of Drainage Pipe," p. 5, 1978.
10. Test Method No. California 643-C, Oct. 1972.
11. B. H. Welch, "Pipe Corrosion and Protective Coatings," Transportation Research Record No. 604, p. 20, 1976.
12. K. M. Jacobs, "Durability of Drainage Structures in Maine," Final Report, Materials and Research Division, June, 1982.
13. R. P. Brown and R. J. Kessler, "Performance Evaluation of Corrugated Metal Culverts in Florida," Florida Department of Transportation, April, 1976.
14. K. J. Boedecker and J. Bauner, "Performance Evaluation of Corrugated Metal Culverts in Georgia," Southeastern Corrugated Steel Pipe Association, April, 1977.
15. P. J. Bellair and J. P. Ewing, "Metal-Loss Rates of Uncoated Steel and Aluminum Culverts in New York," Research Report 115, New York State Department of Transportation, October, 1984.
16. American Iron and Steel Institute in the Handbook of Steel and Highways Construction Products, 2nd edition, Chapter 5.
17. F. E. Kulman and S. N. Palica, "Fundamental Parameters in Underground Pipe Corrosion," Pipe Line News, pp. 40-49, November, 1963.

18. J. R. Caris, "Performance of Aluminum Corrugated Culvert in Mississippi," Kaiser Aluminum and Chemical Corporation, Houston, Texas, March, 1984.
19. NACE Standard, TM-01-84.
20. T. Liu, "Is the Salt Fog Test an Effective Method to Evaluate Corrosion Resistant Coatings?" Corrosion Control by Organic Coating, p. 247, 1981.
21. R. A. Pesigan and J. E. Singley, "Evaluation of Water Corrosivity Using the Langelier Index and Relative Corrosion Rate Models," Materials Performance, p. 26, April, 1985.
22. D. A. Jones and T. A. Lowe, "Polarization Methods for Measuring the Corrosion of Metals Buried Underground," J of Materials, 4 (3), p. 600, Sept. 1969.
23. R. L. Martin, "Application of Electrochemical Polarization to Corrosion Problems," Petrolite Corp. 1977.
24. J. R. Scully and K. J. Bundy, "Electrochemical Methods for Measurement of Steel Pipe Corrosion Rates in Soil," Materials Performance, p. 18, April 1985.
25. J. R. Scully and K. J. Bundy, "Laboratory Electrochemical Testing of Pipe Surfaces in Soils," Materials Performance, p. 50, July 1984.
26. EG&G Princeton Applied Research, "Basics of AC Impedance Measurement," Application Note AC-1, 1985.
27. J. C. Padget and Moreland, "Use of AC Impedance In the Study of the Anticorrosive Properties of Chlorine-Containing Vinyl Acrylic Latex Copolymers," J. of Coating Technology, Vol. 55, No. 698, March, 1983.
28. J. J. Sudol, "Pipe Coating Study," Indiana Department of Highways, Final Report, September, 1982.

APPENDIX A

PHOTOGRAPHS OF COUPONS AFTER EXPOSURE TO 1-VOLT
POTENTIOSTAT TEST

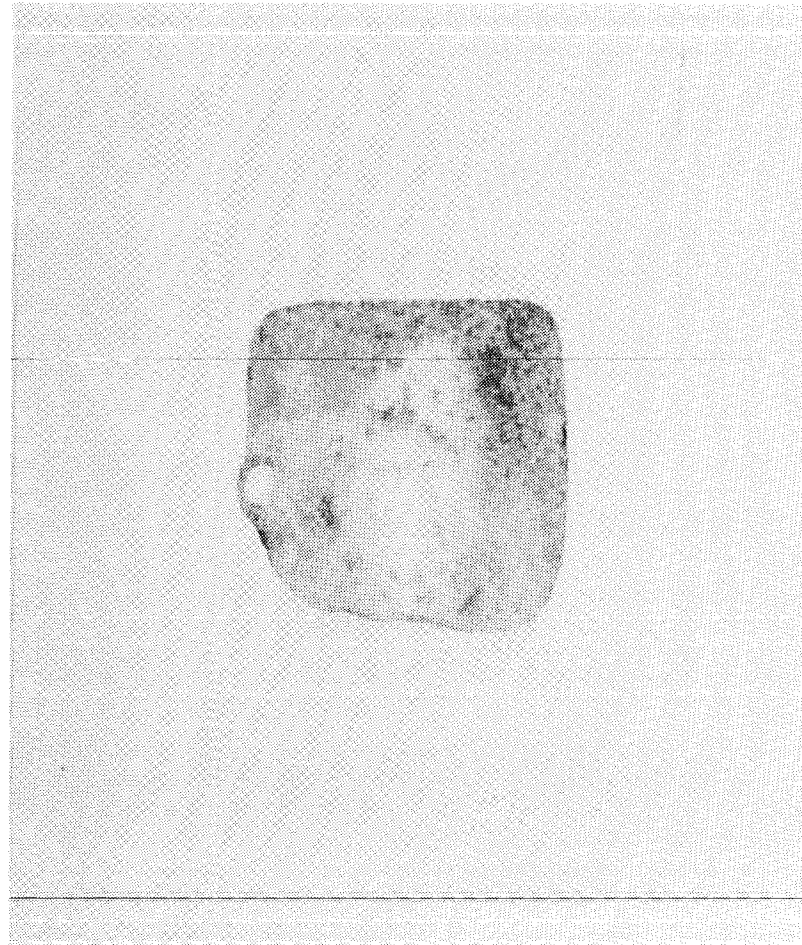


Figure A-1. Galvanized Steel Exposed to 1-Volt Potentiostat Test

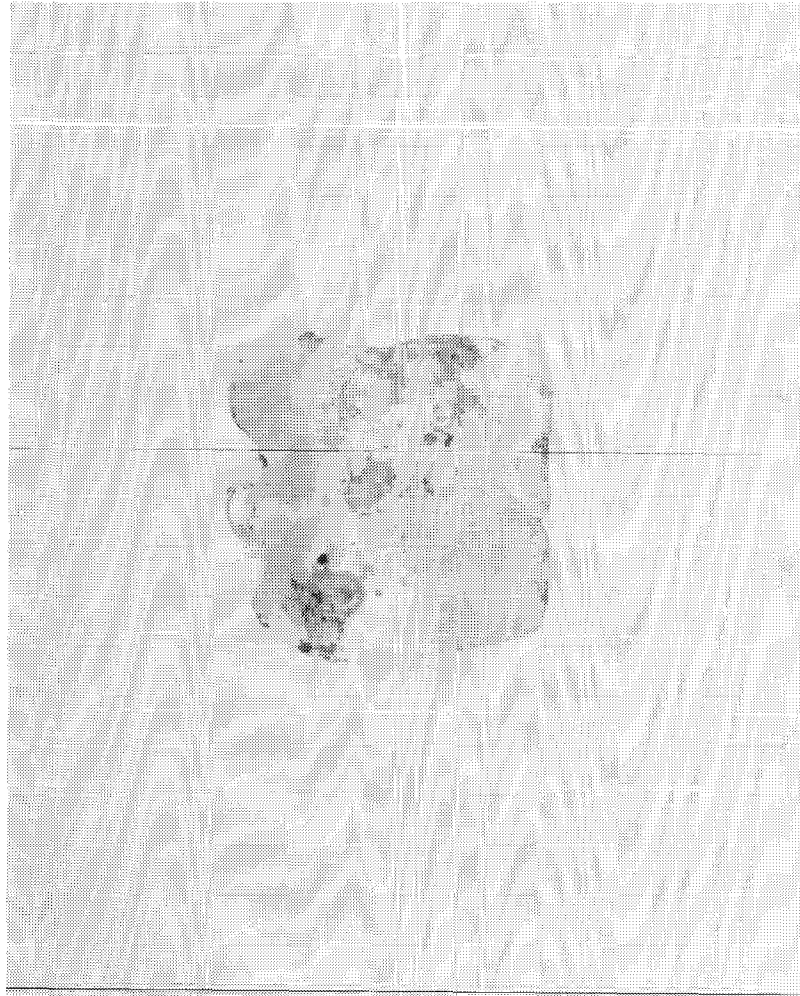
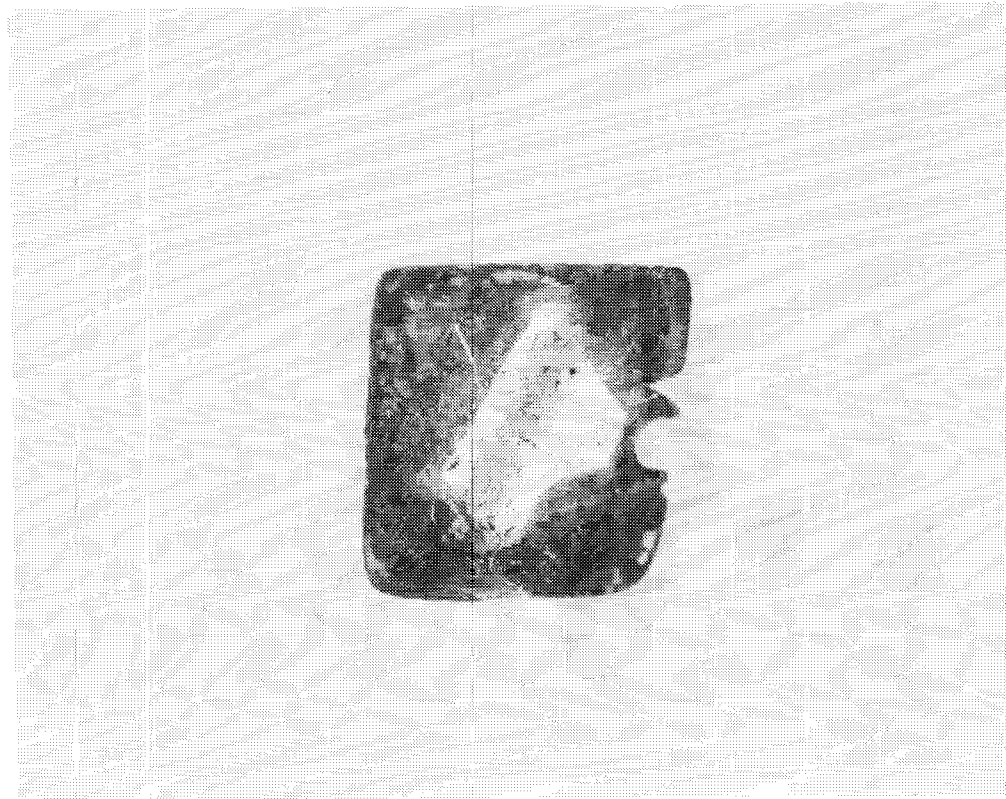
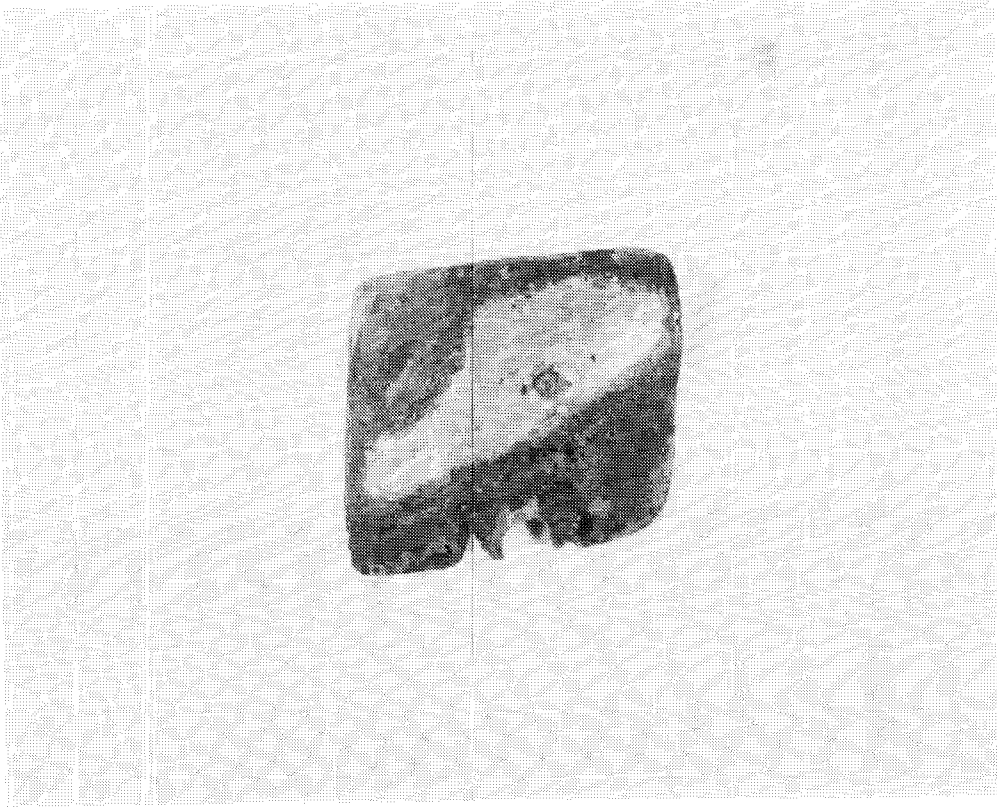


Figure A-2. Asphalt Galvanized Steel Exposed to 1-Volt Potentiostat Test

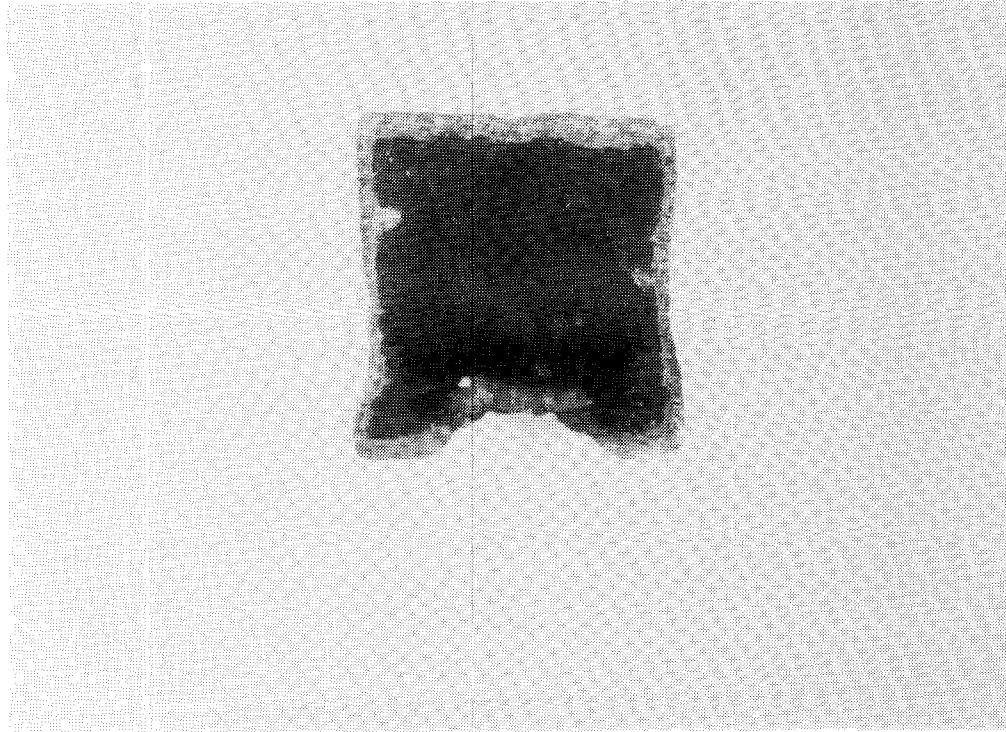


Side 1

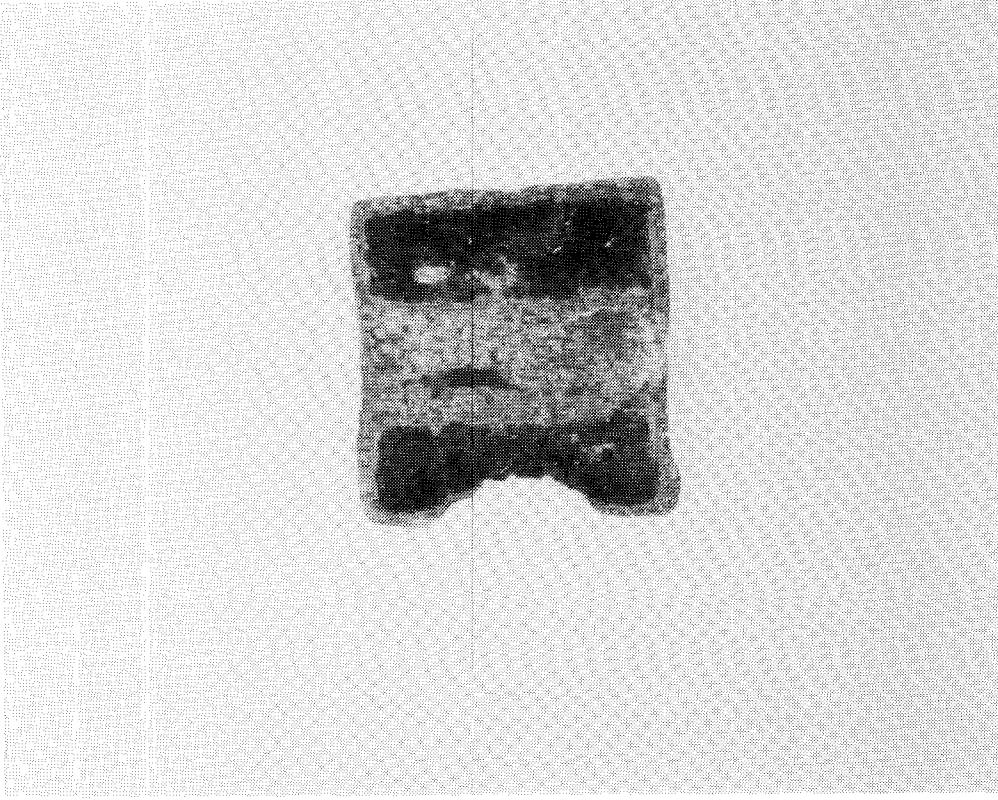


Side 2

Figure A-3. Blackclad Galvanized Steel Exposed to 1-Volt Potentiostat Test

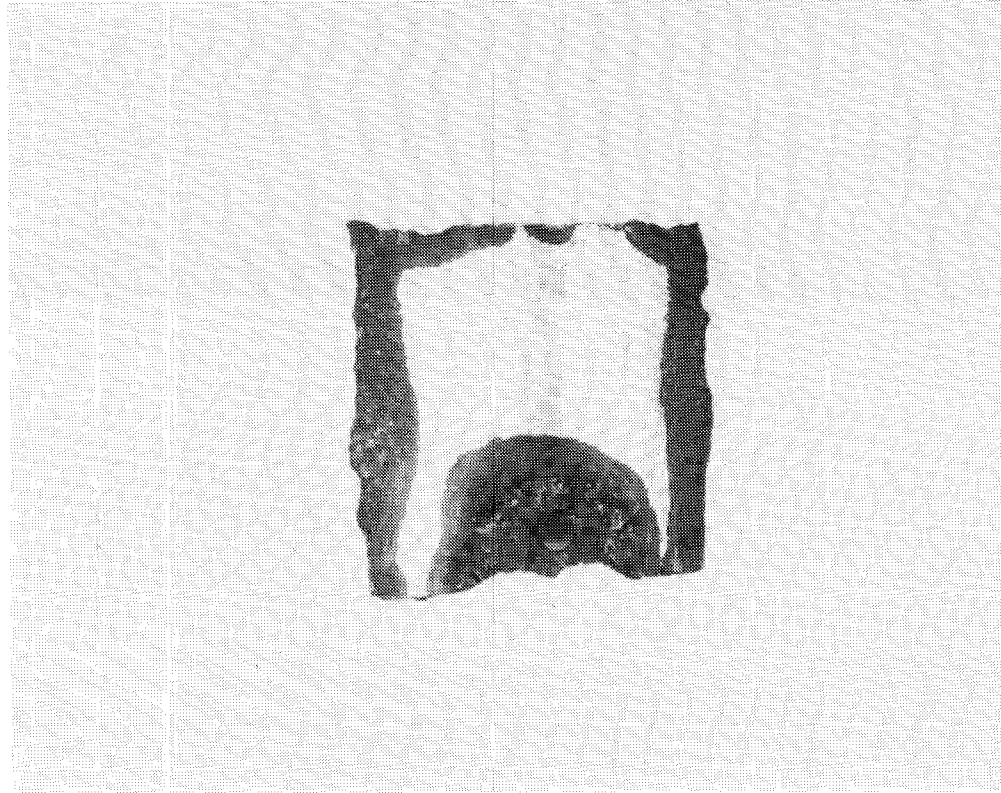


Side 1

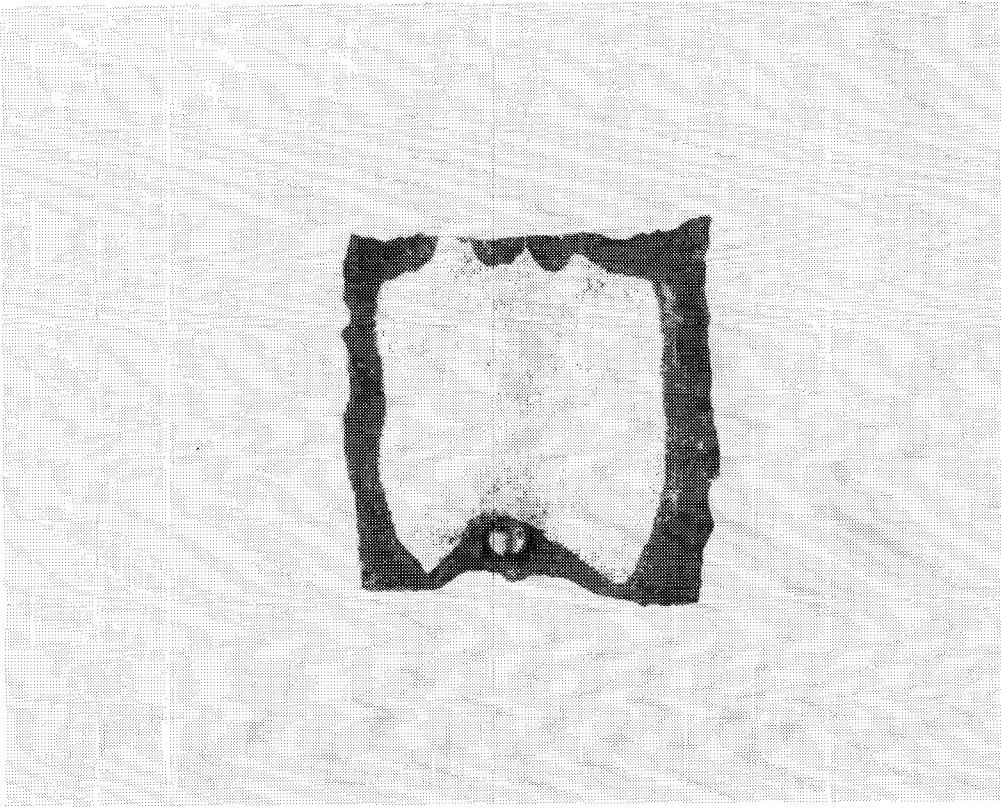


Side 2

Figure A-4. Asbestos Galvanized Steel Exposed to 1-Volt Potentiostat Test



Side 1



Side 2

Figure A-5. Fiber-Bituminous Galvanized Steel Exposed to 1-Volt Potentiostat Test

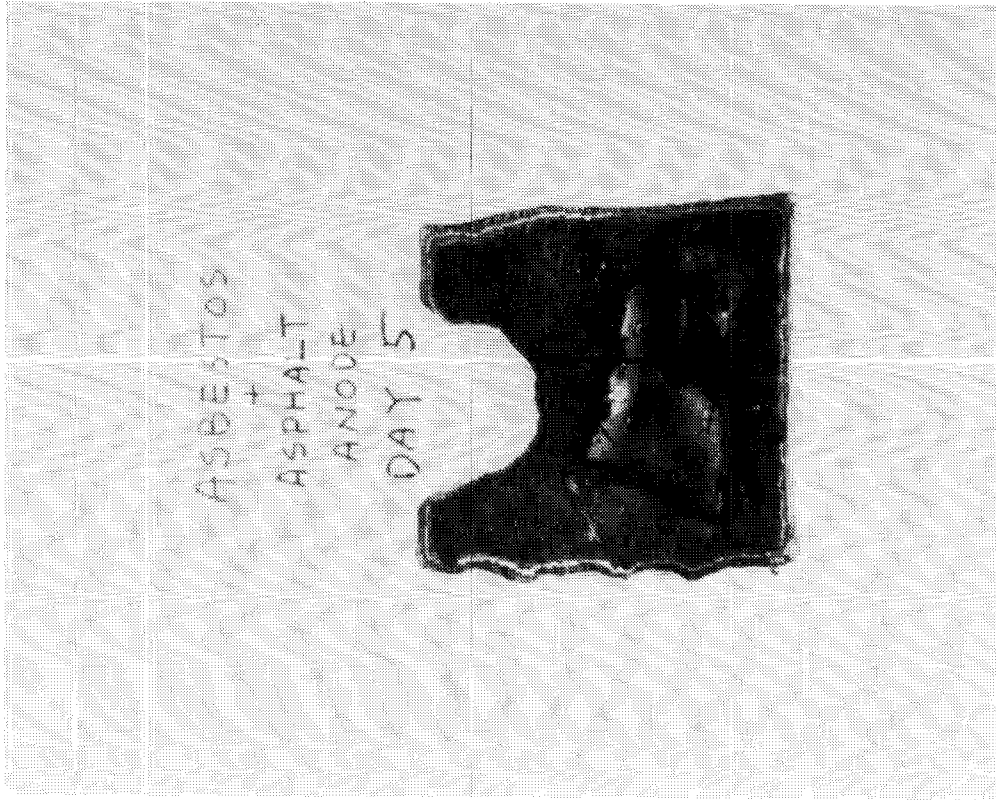


Figure A-6. Asbestos-Asphalt Galvanized Steel Exposed to 1-Volt Potentiostat Test

Appendix B

PHOTOGRAPHS OF COUPONS AFTER EXPOSURE
TO HIGH-PRESSURE OXYGEN TEST NO. 2

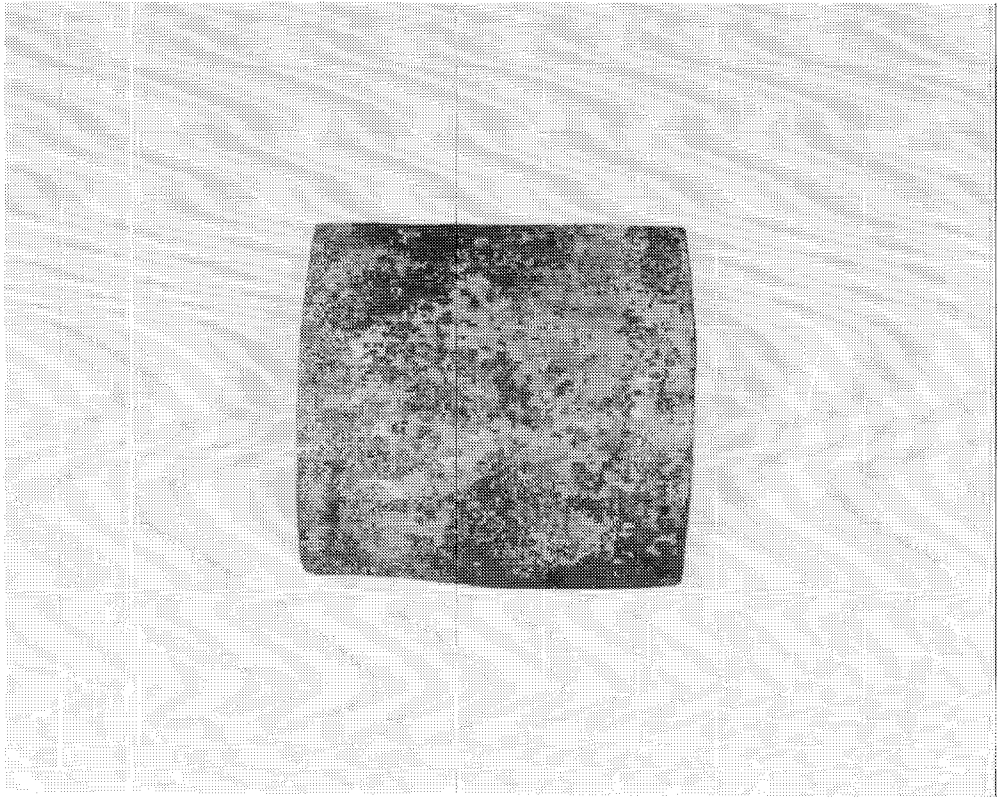


Figure B-1. Galvanized Steel Exposed to High-Pressure Oxygen Test No. 2

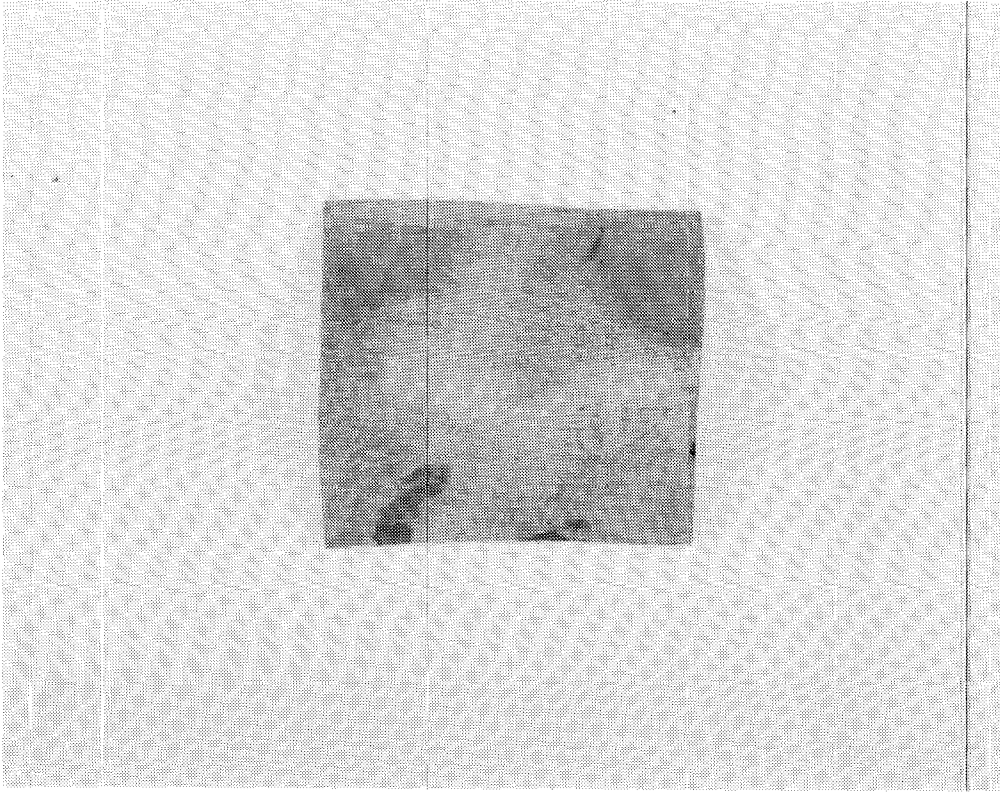
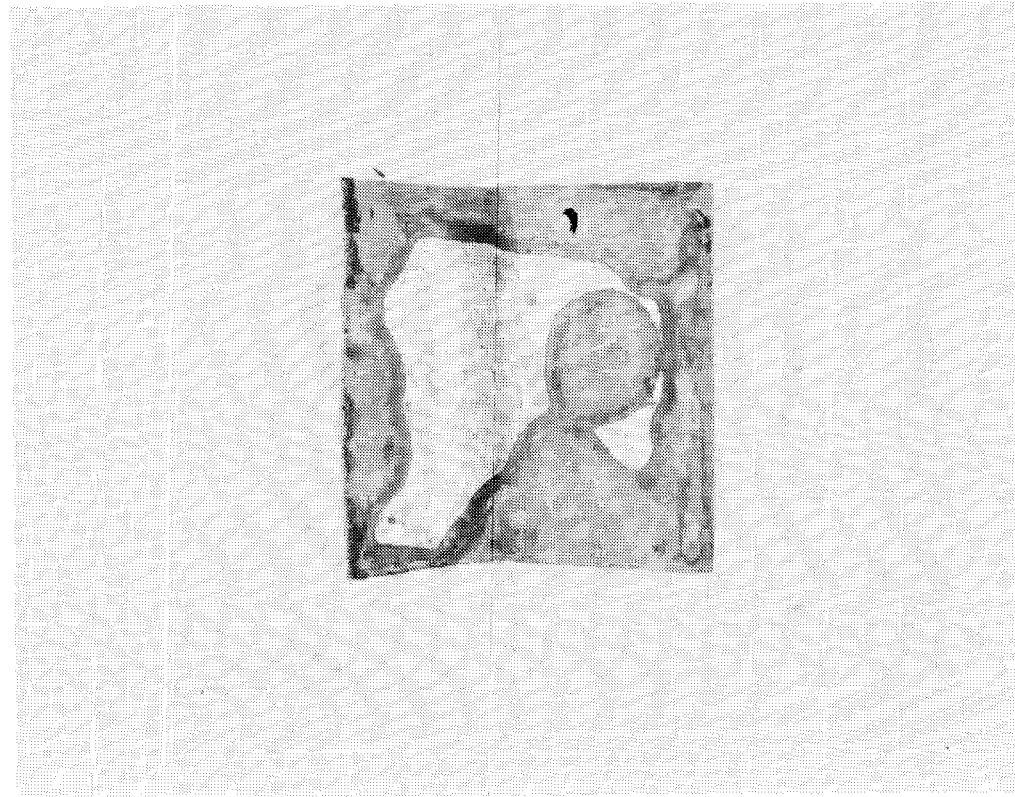
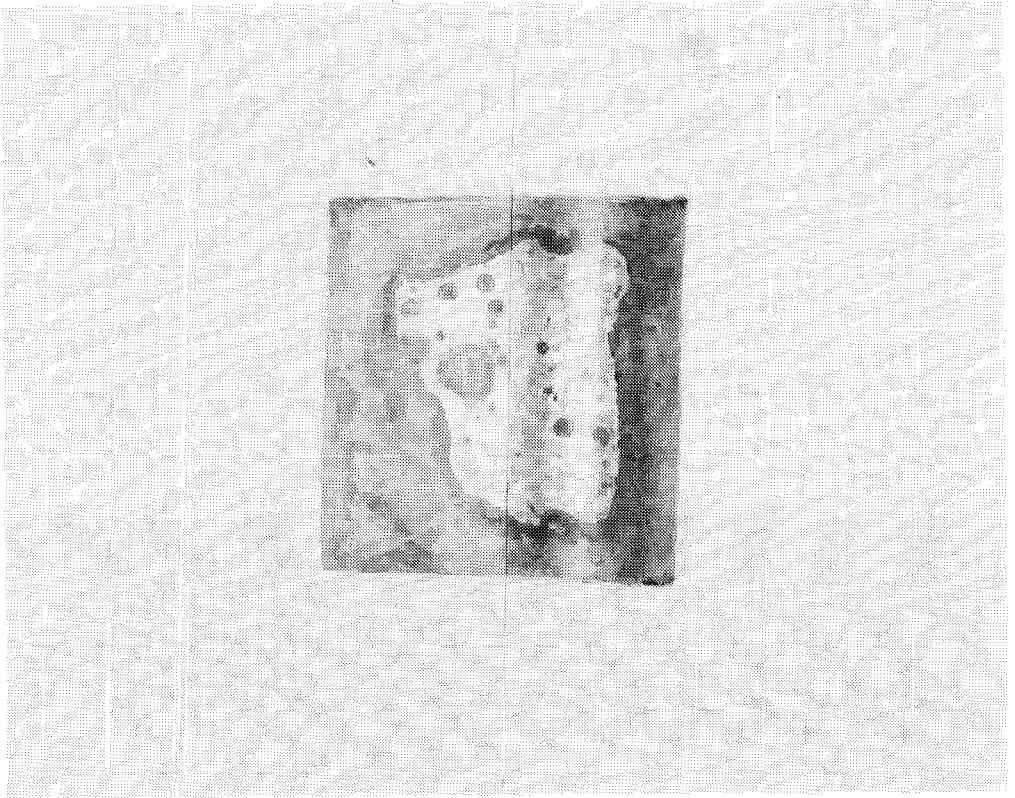


Figure B-2. Galvanized Steel Exposed to High-Pressure Oxygen Test No. 2

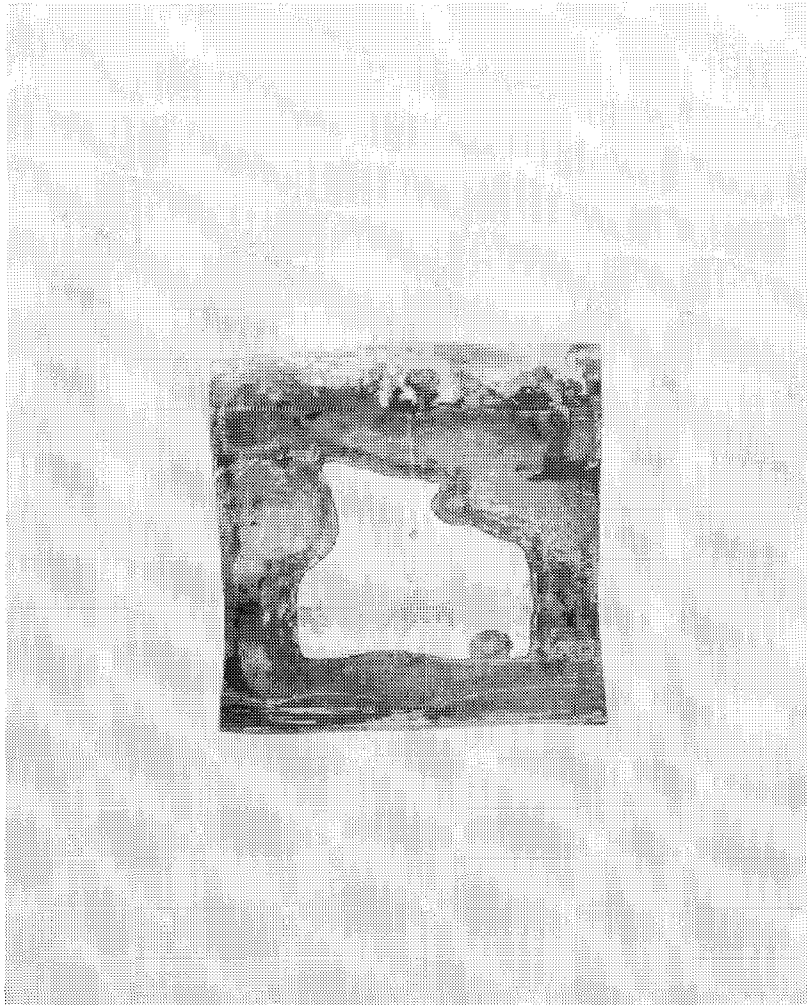


Side 1

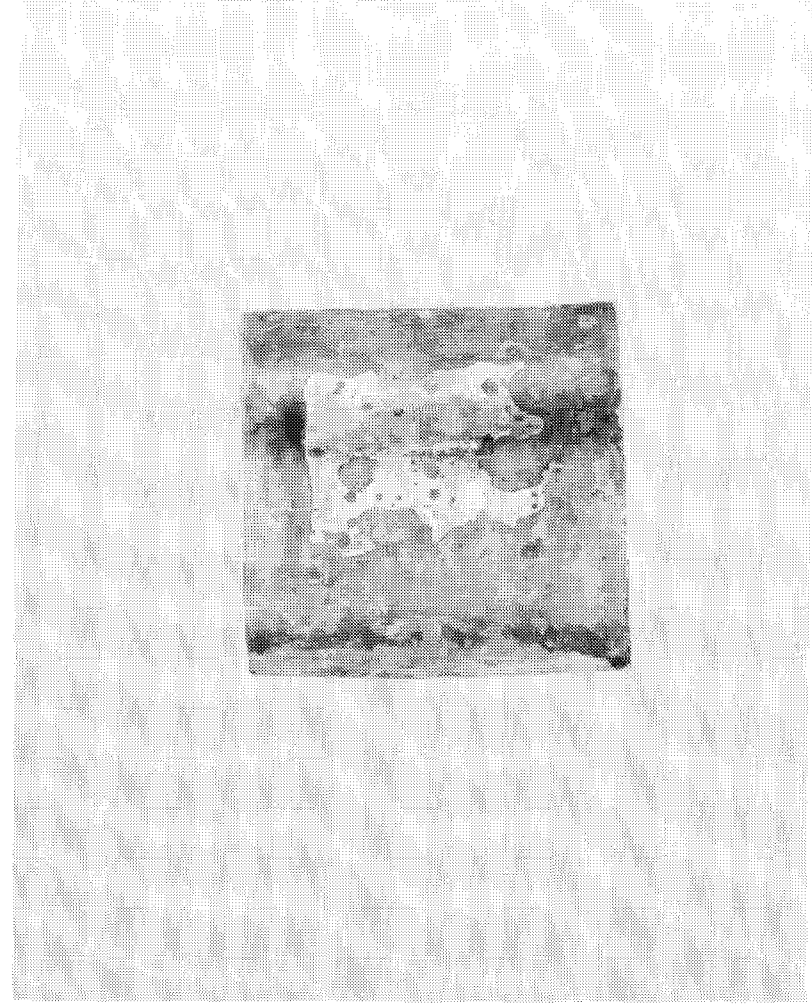


Side 2

Figure B-3. Asphalt Galvanized Steel Exposed to High-Pressure Oxygen Test No. 2

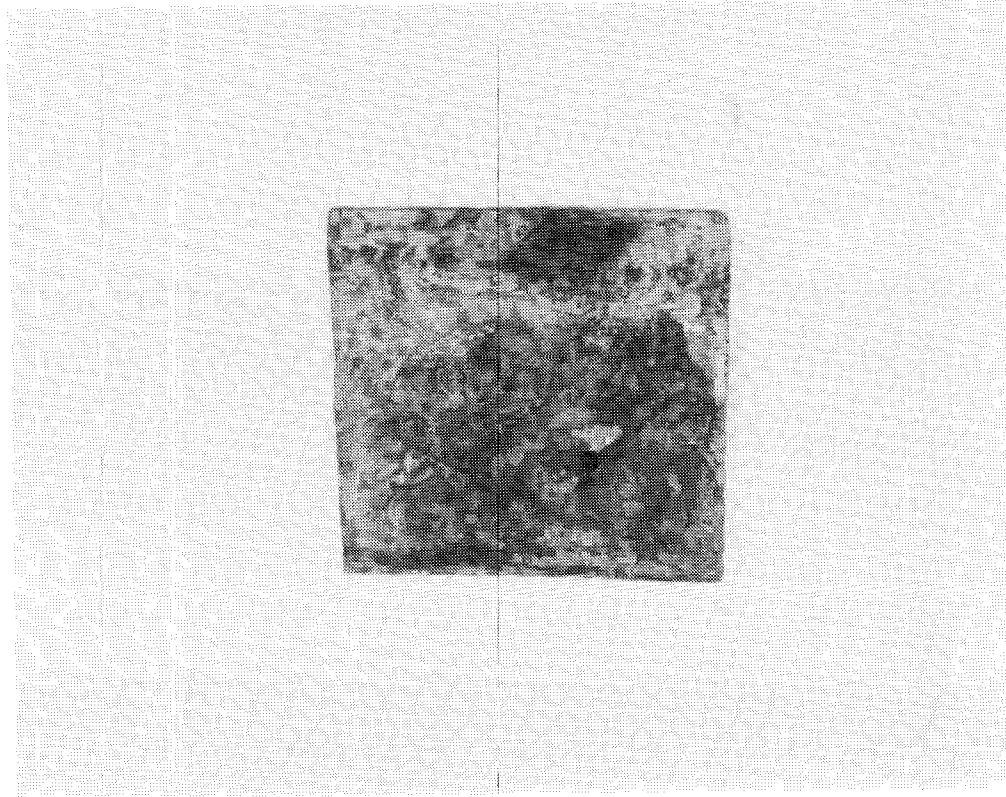


Side 1

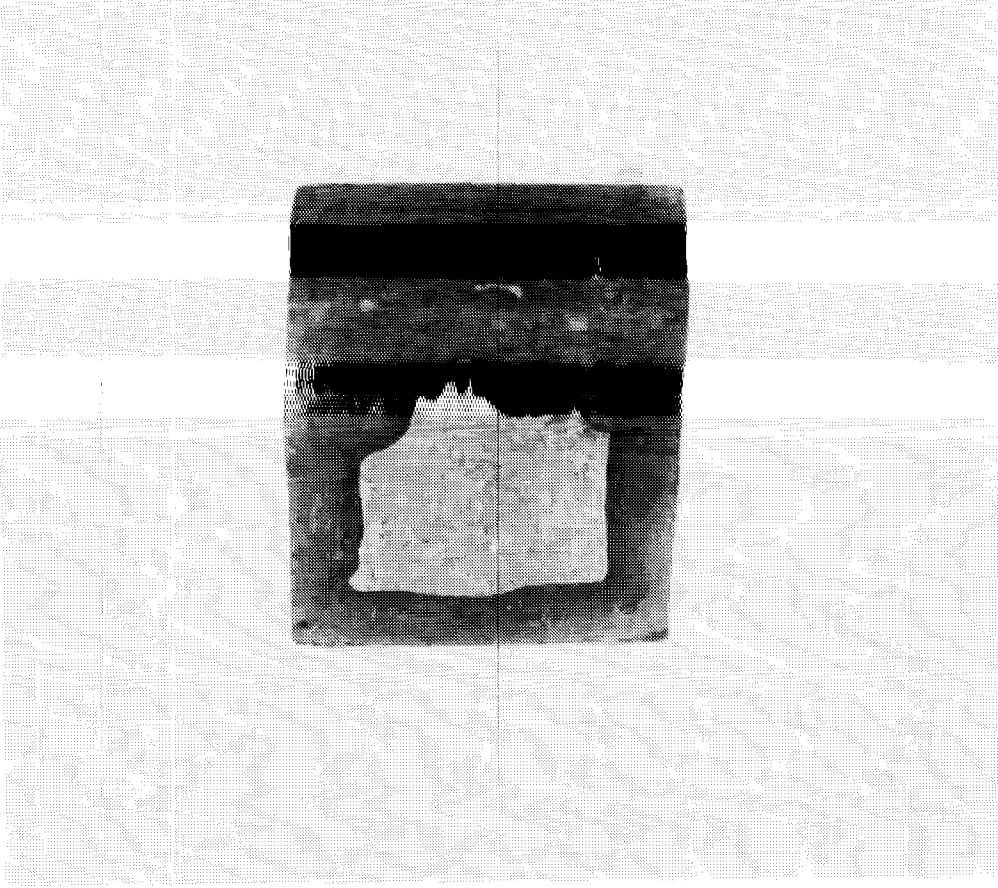


Side 2

Figure B-4. Asphalt Galvanized Steel Exposed to High-Pressure Oxygen Test No. 2

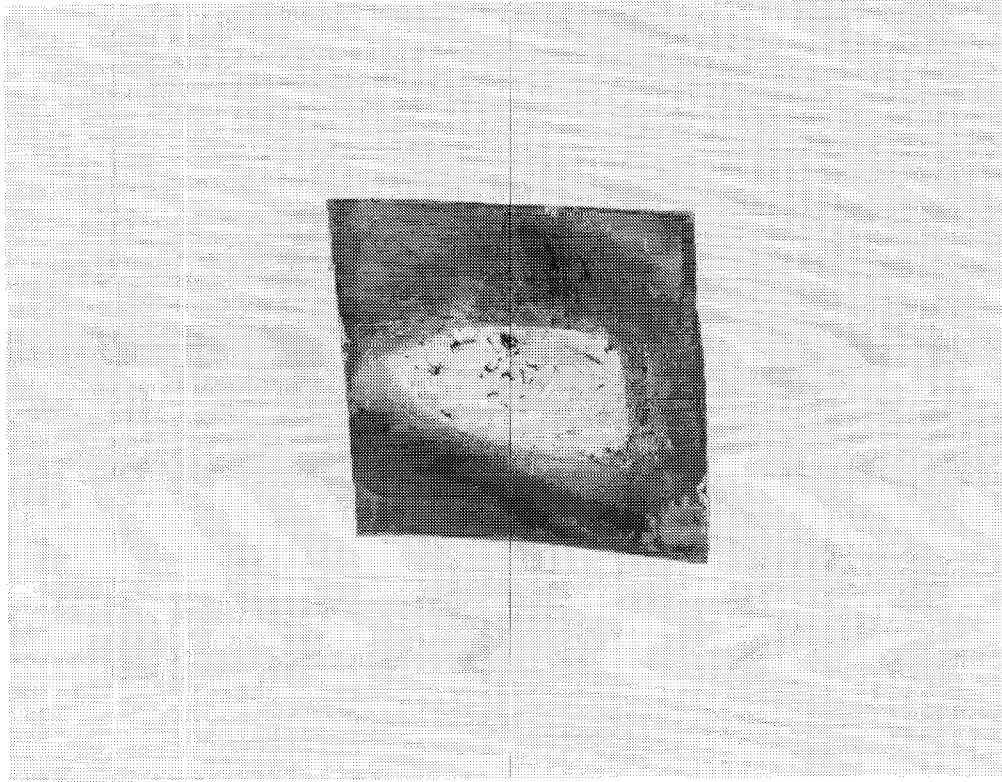


Side 1

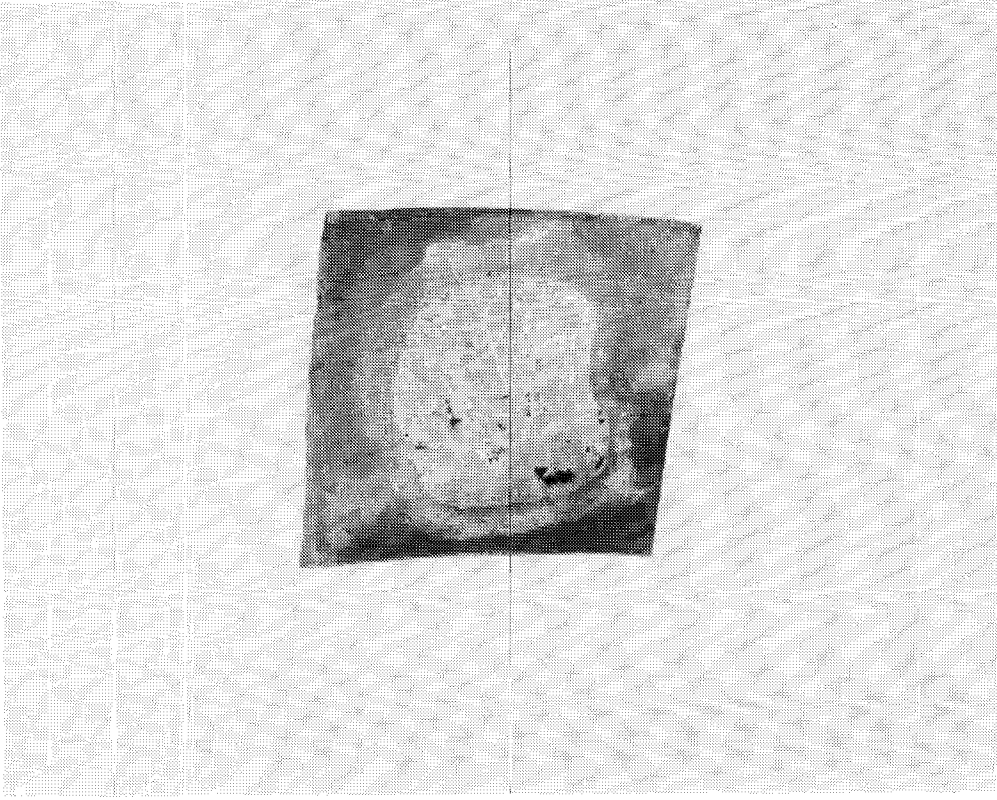


Side 2

Figure B-5. Asphalt Galvanized Steel Exposed to High-Pressure Oxygen Test No. 2

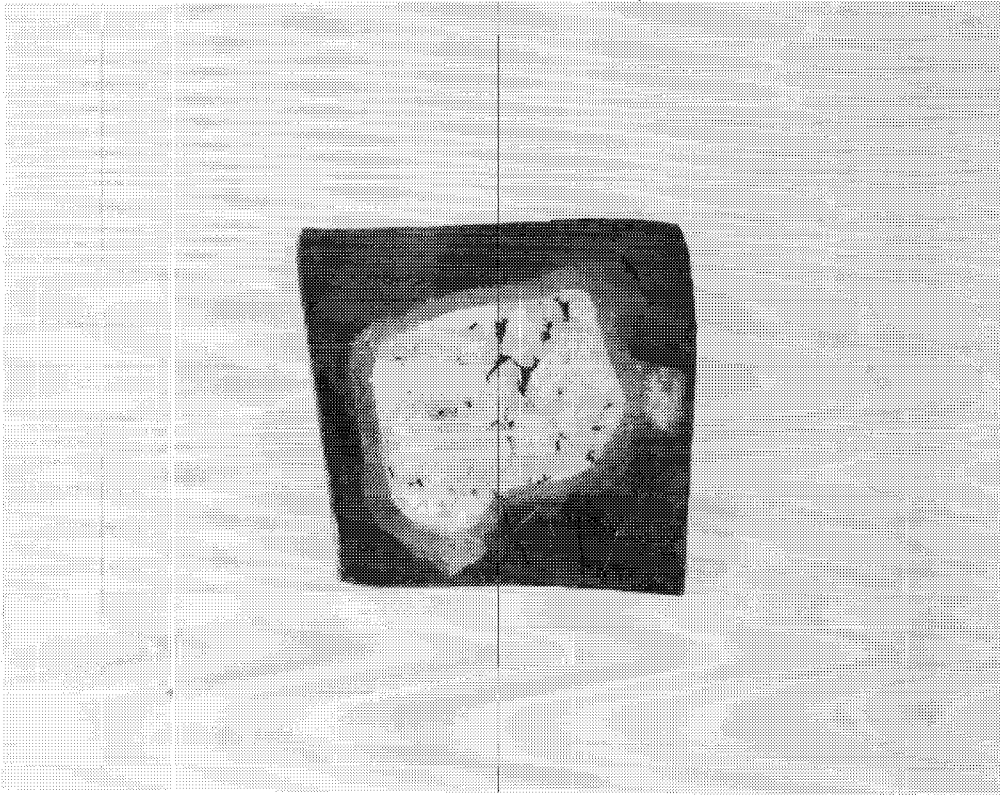


Side 1

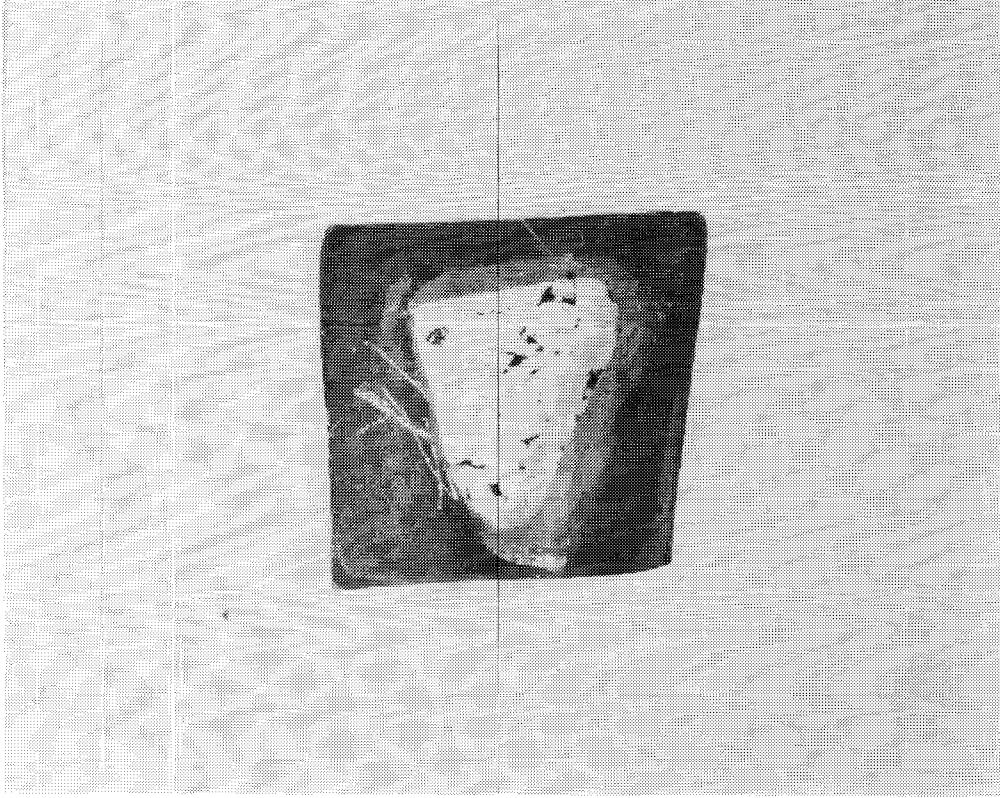


Side 2

Figure B-6. Blackclad Galvanized Steel Exposed to High-Pressure Oxygen Test No. 2

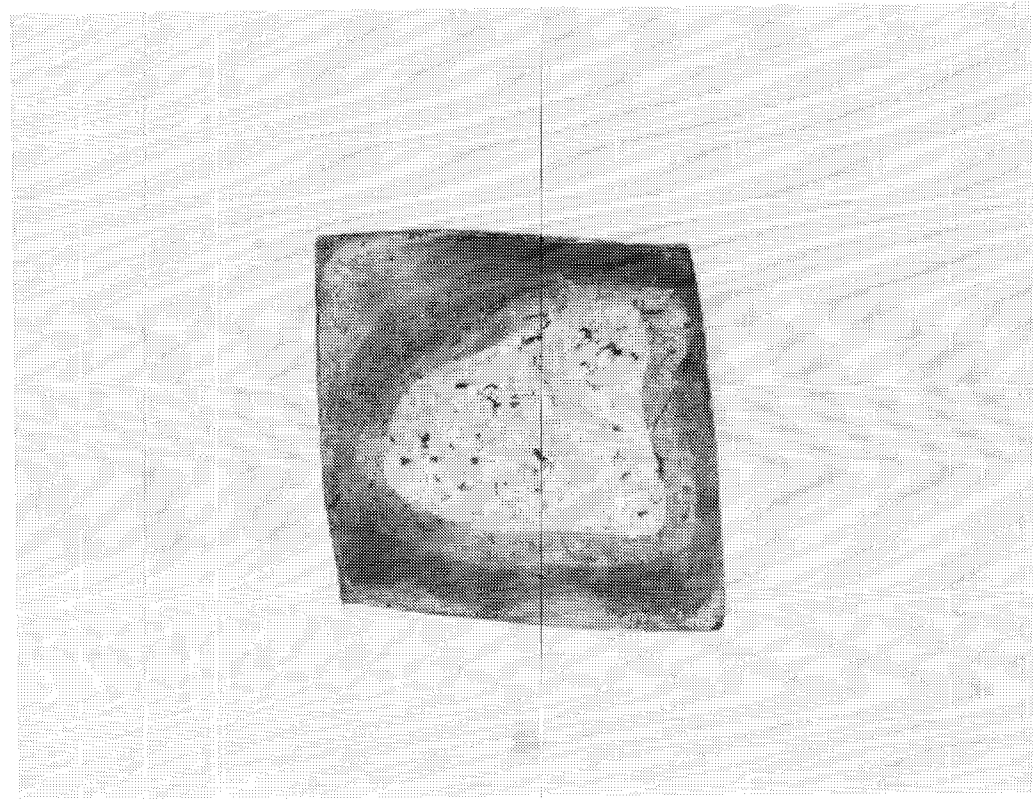


side 1

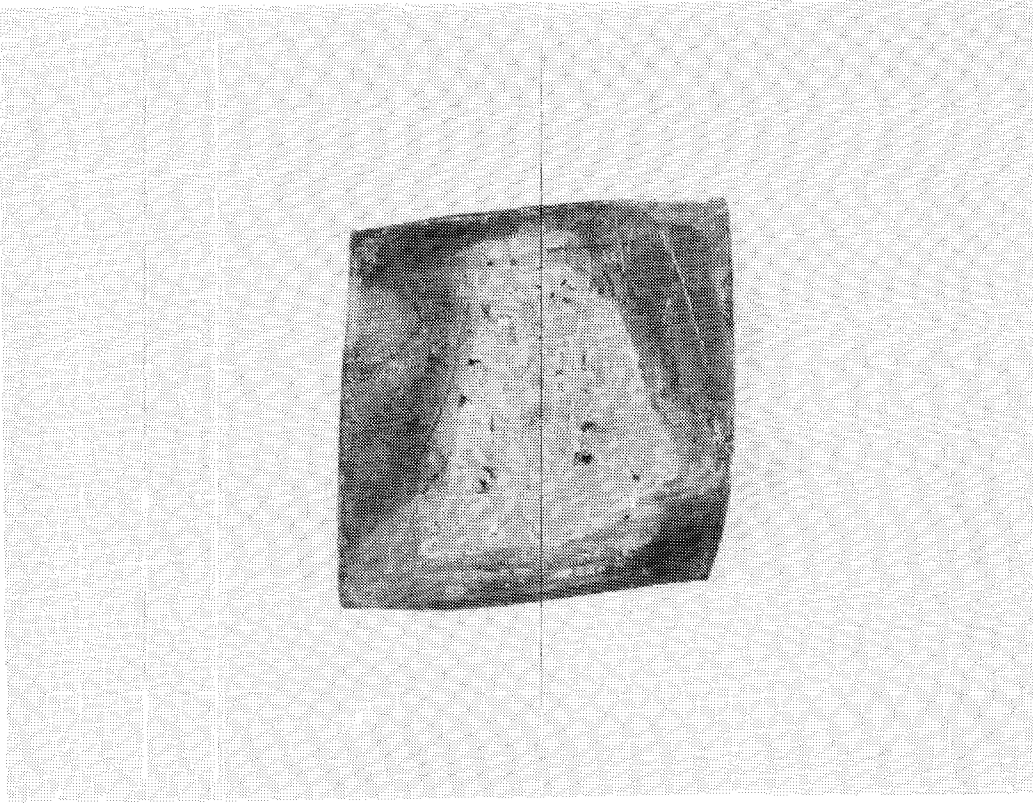


side 2

Figure B-7. Blackclad Galvanized Steel Exposed to High-Pressure Oxygen Test No. 2

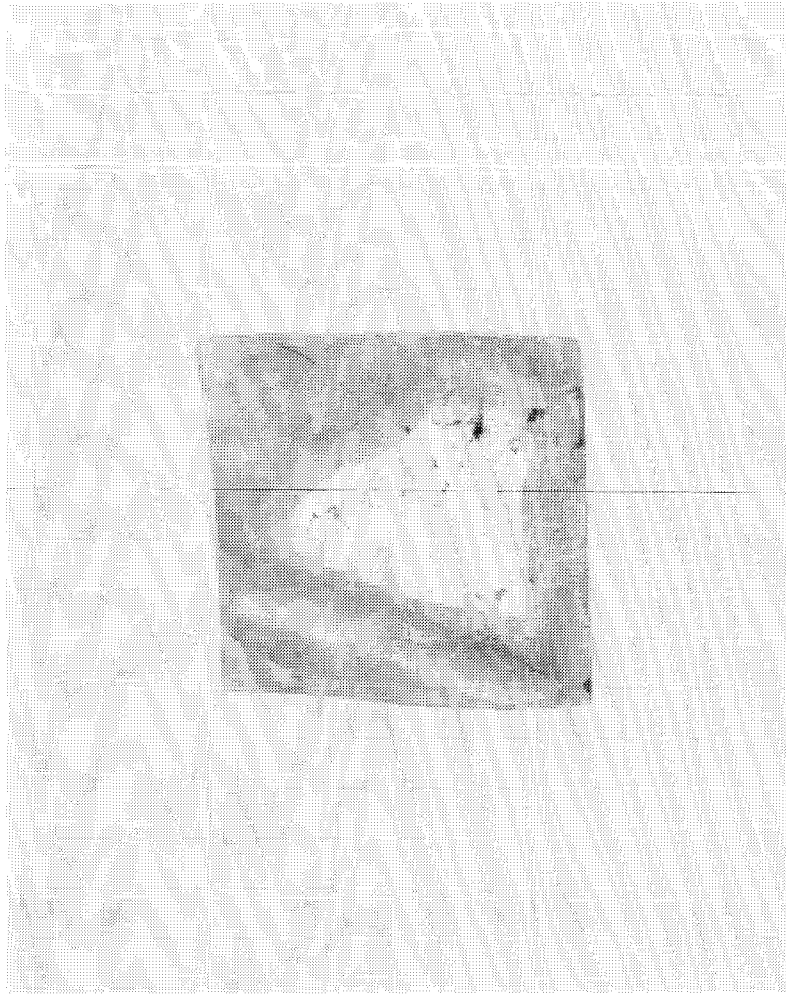


Side 1

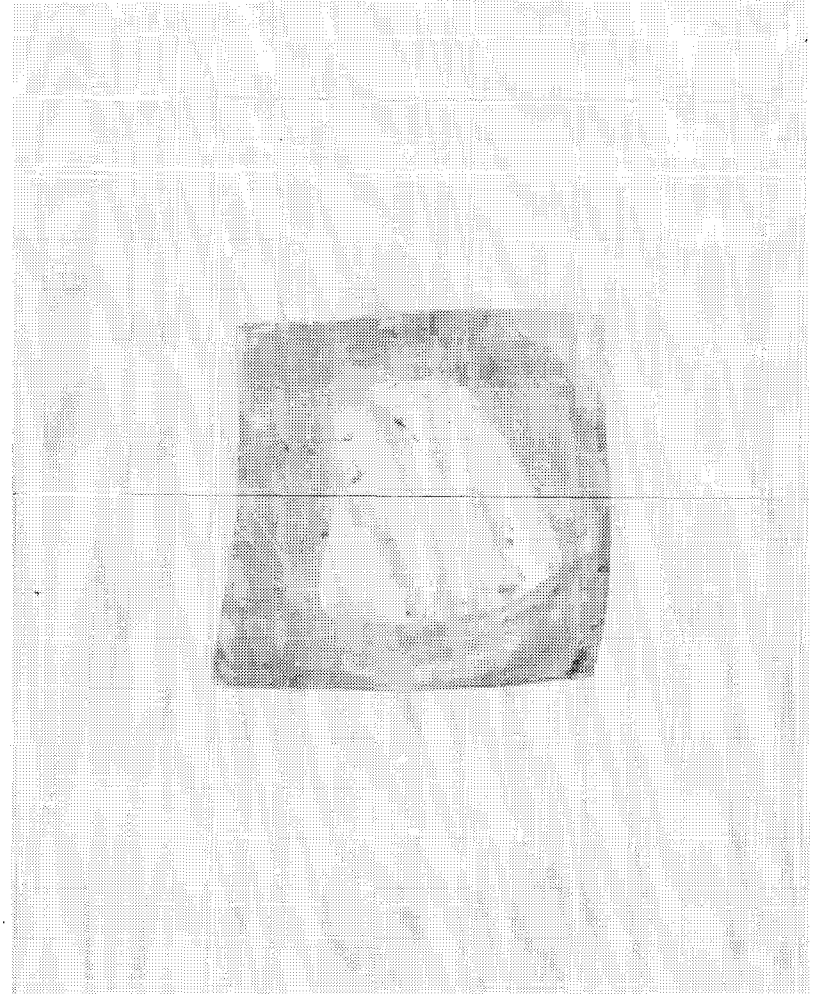


Side 2

Figure B-8. Blackclad Galvanized Steel Exposed to High-Pressure Oxygen Test No. 2

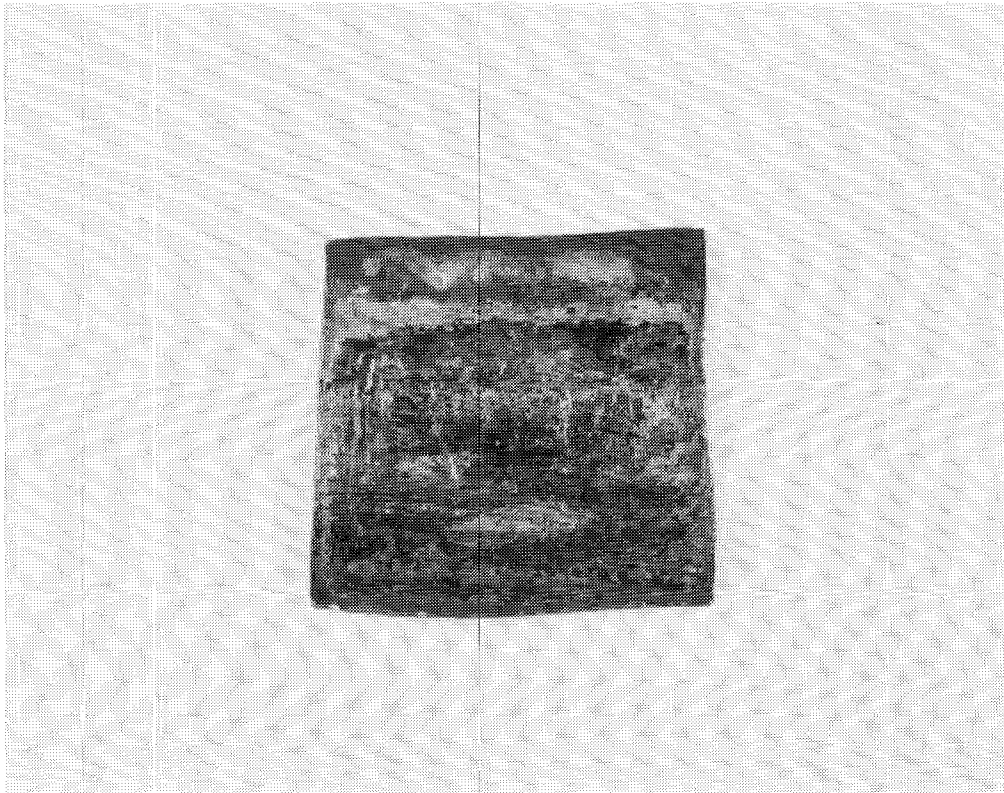


Side 1

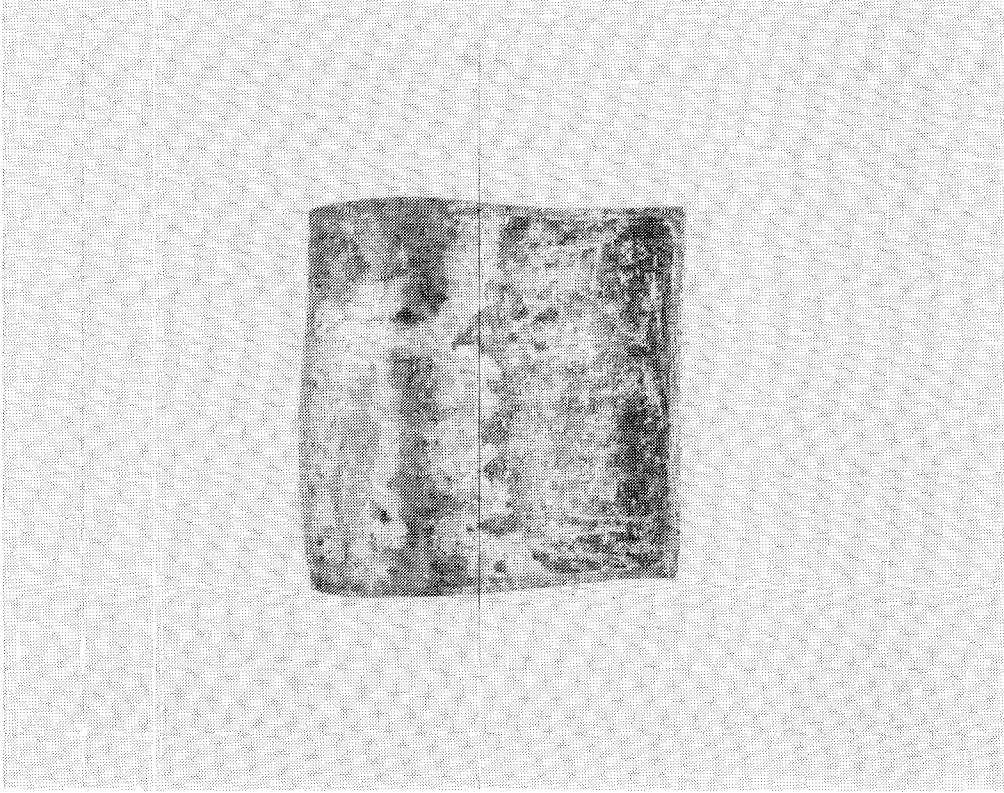


Side 2

Figure B-9. Blackclad Galvanized Steel Exposed to High-Pressure Oxygen Test No. 2

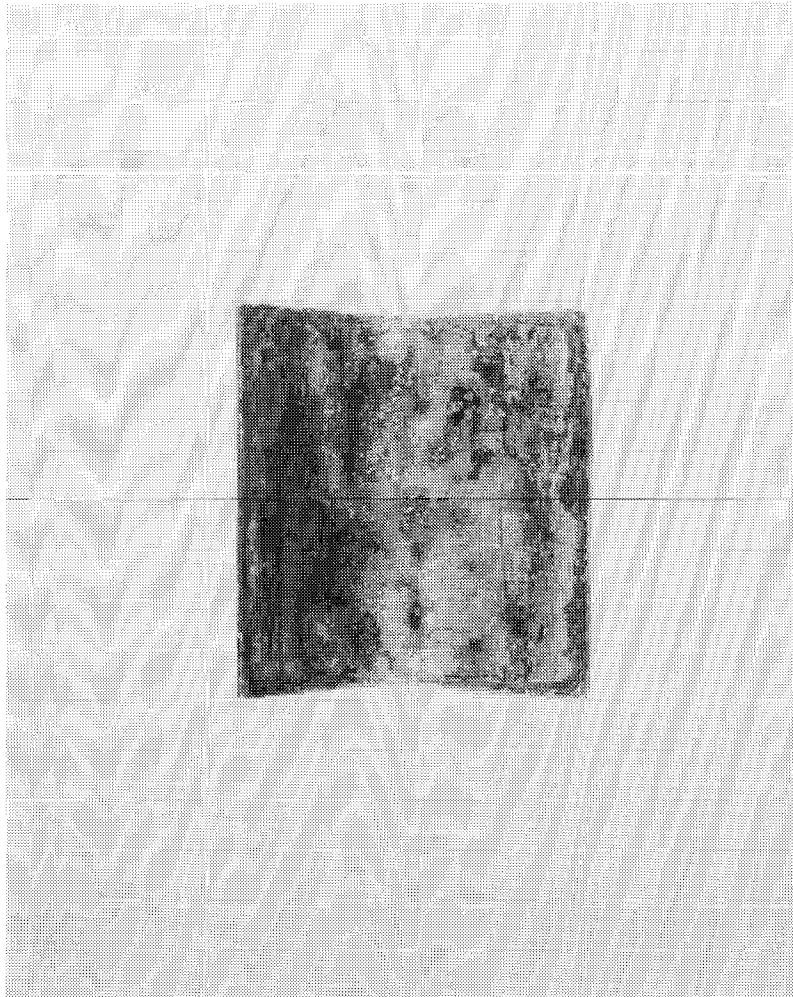


Side 1

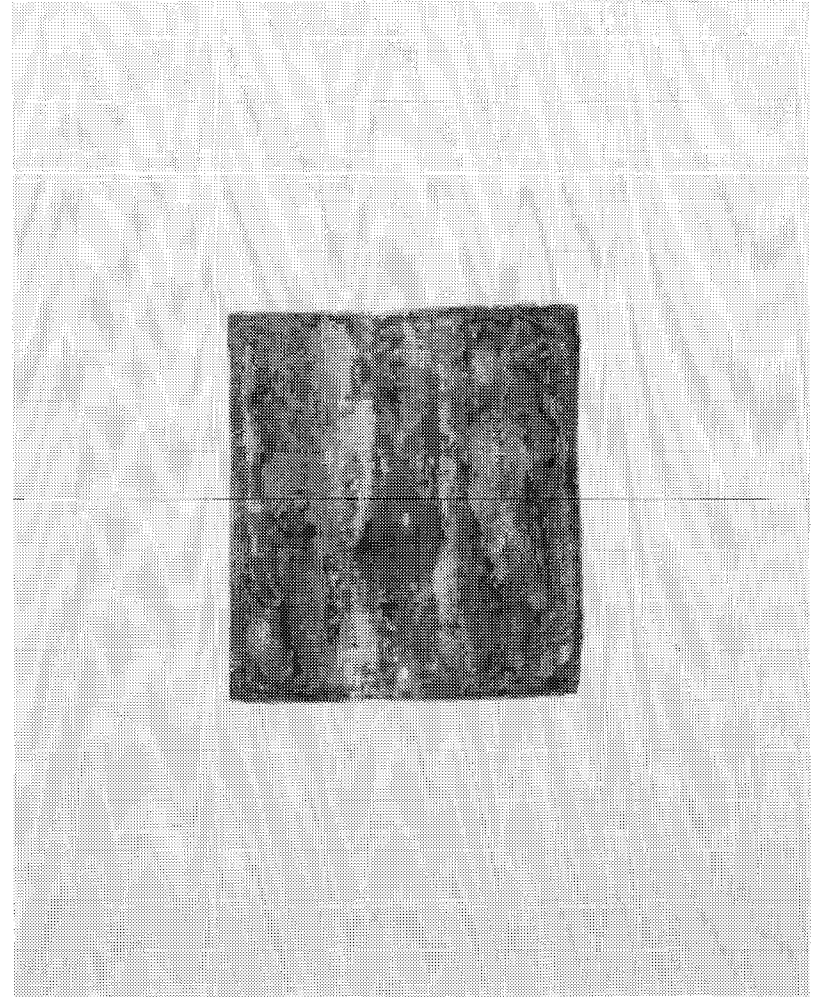


Side 2

Figure B-10. Asbestos Galvanized Steel Exposed to High-Pressure Oxygen Test No. 2

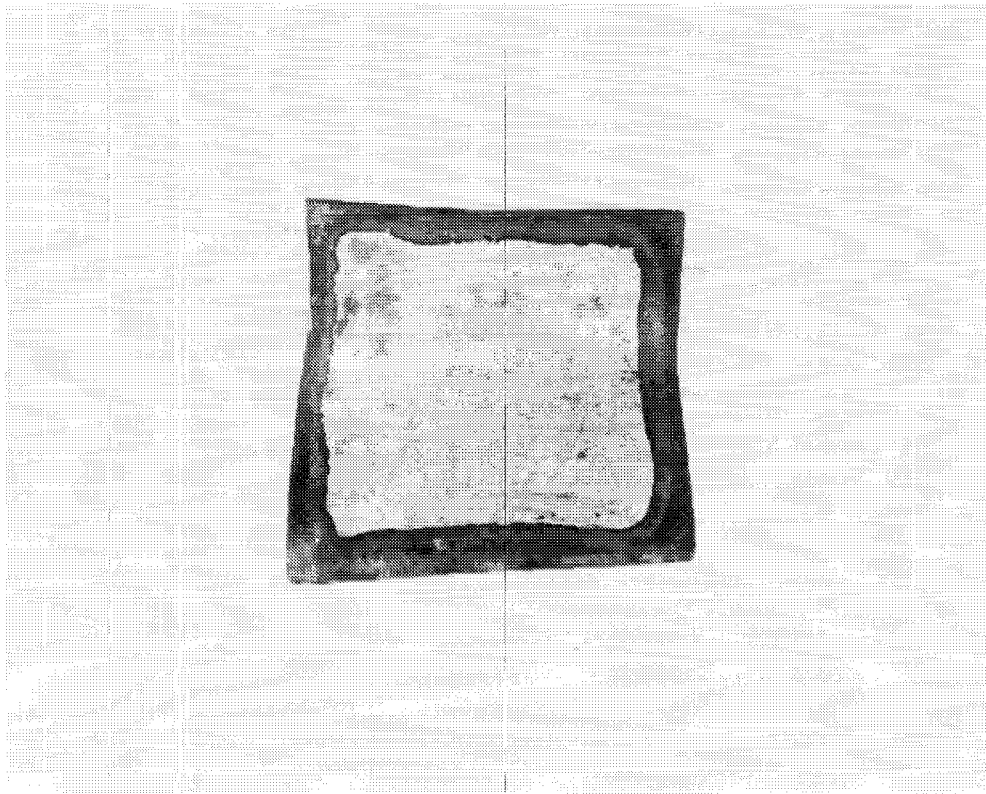


Side 1

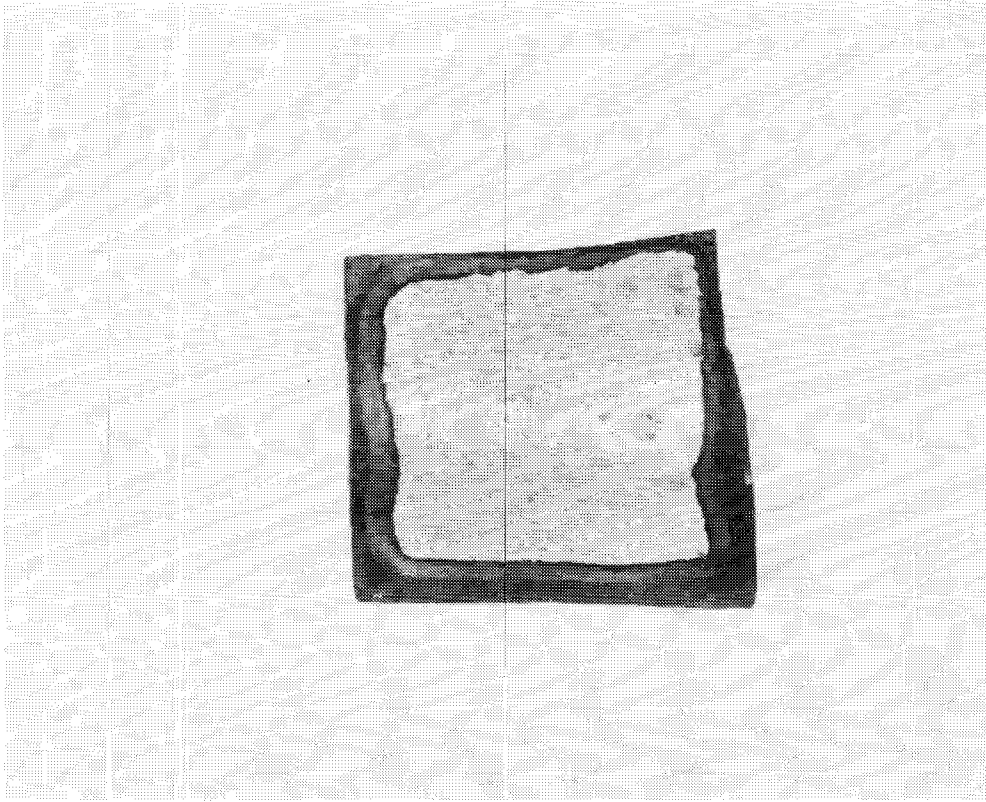


Side 2

Figure B-11. Asbestos Galvanized Steel Exposed to High-Pressure Oxygen Test No. 2

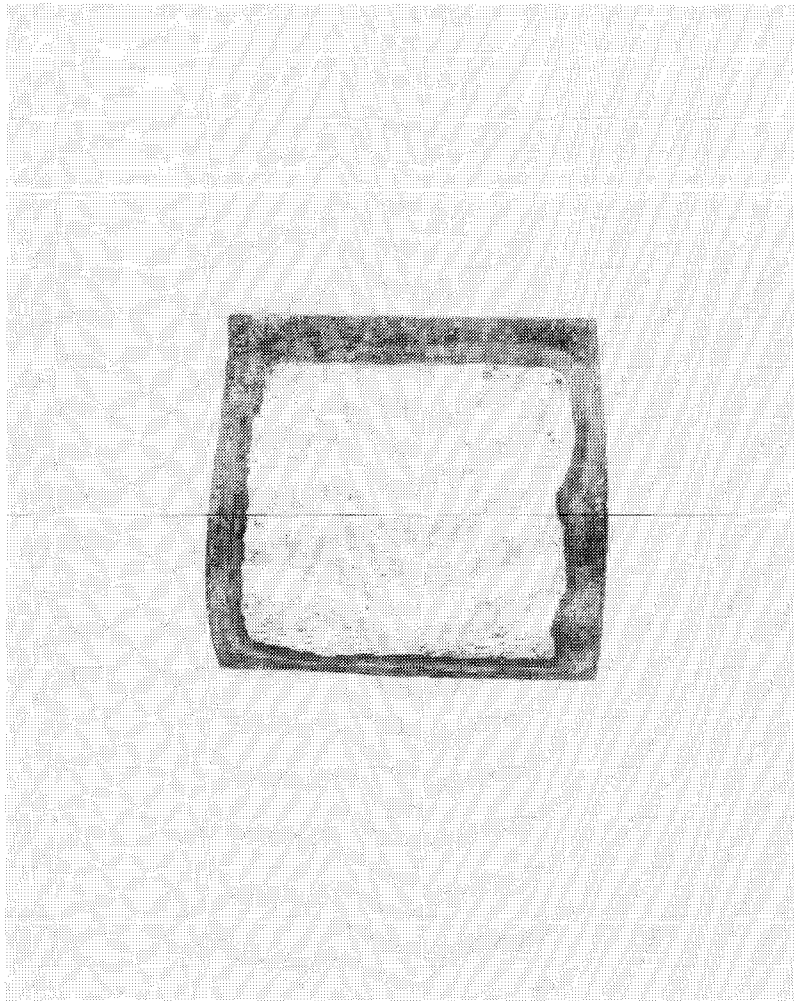


Side 1

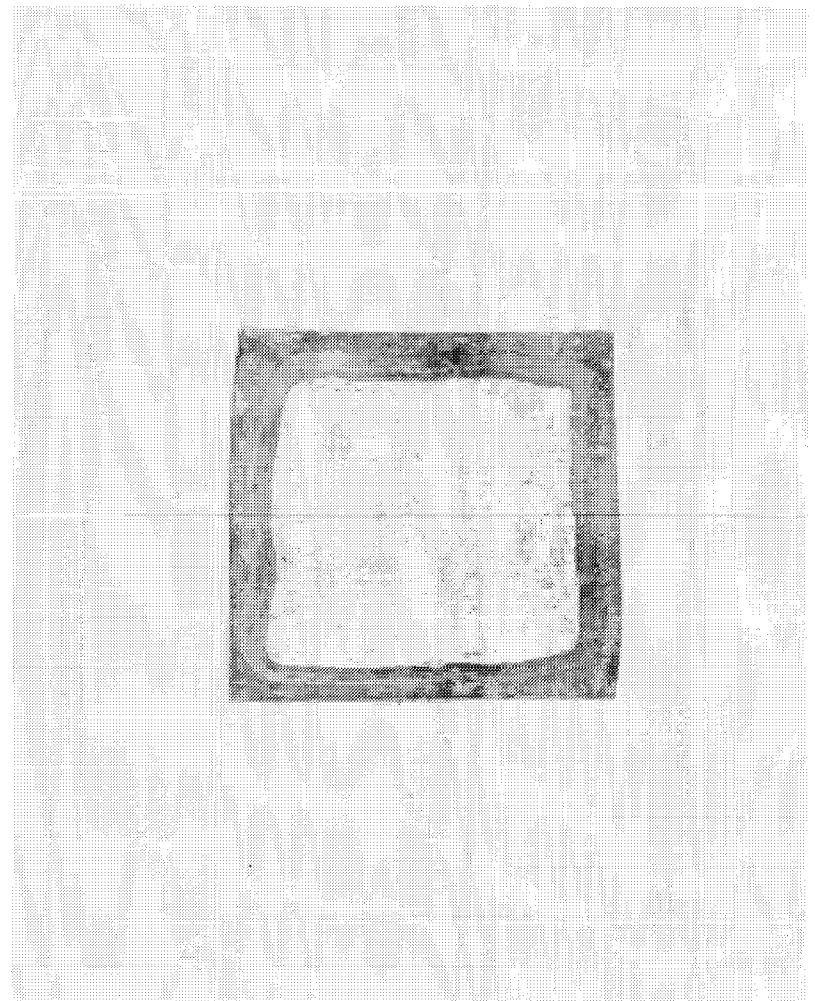


Side 2

Figure B-12. Fiber-Bituminous Galvanized Steel Exposed to High-Pressure Oxygen Test No. 2

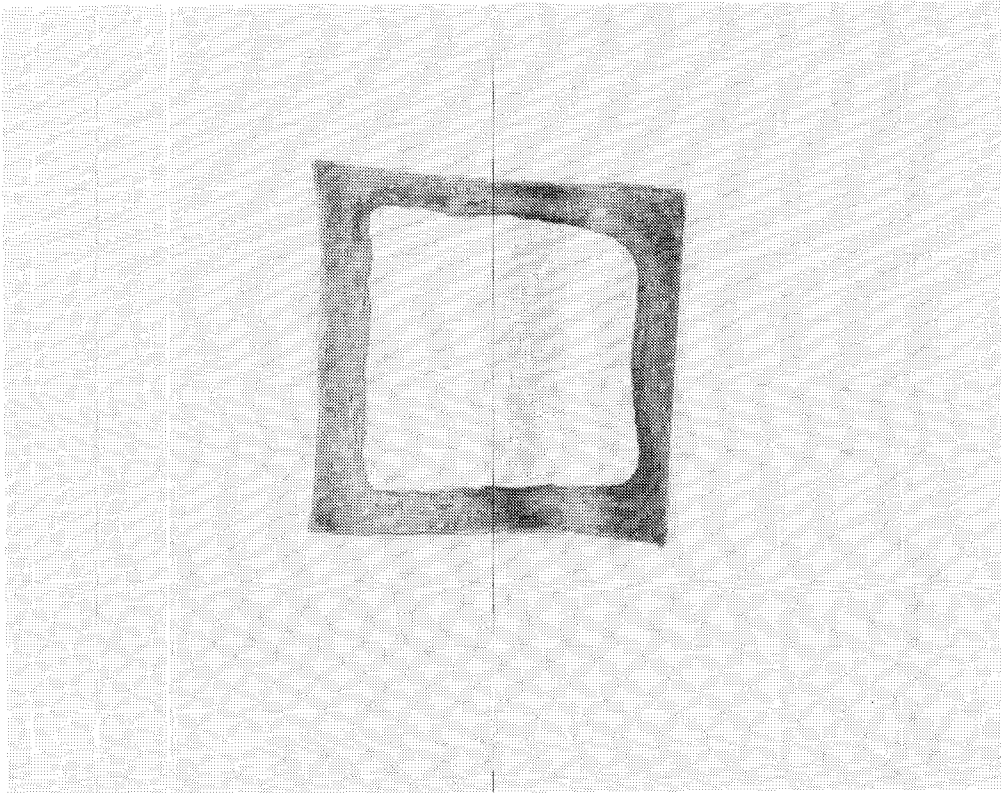


Side 1

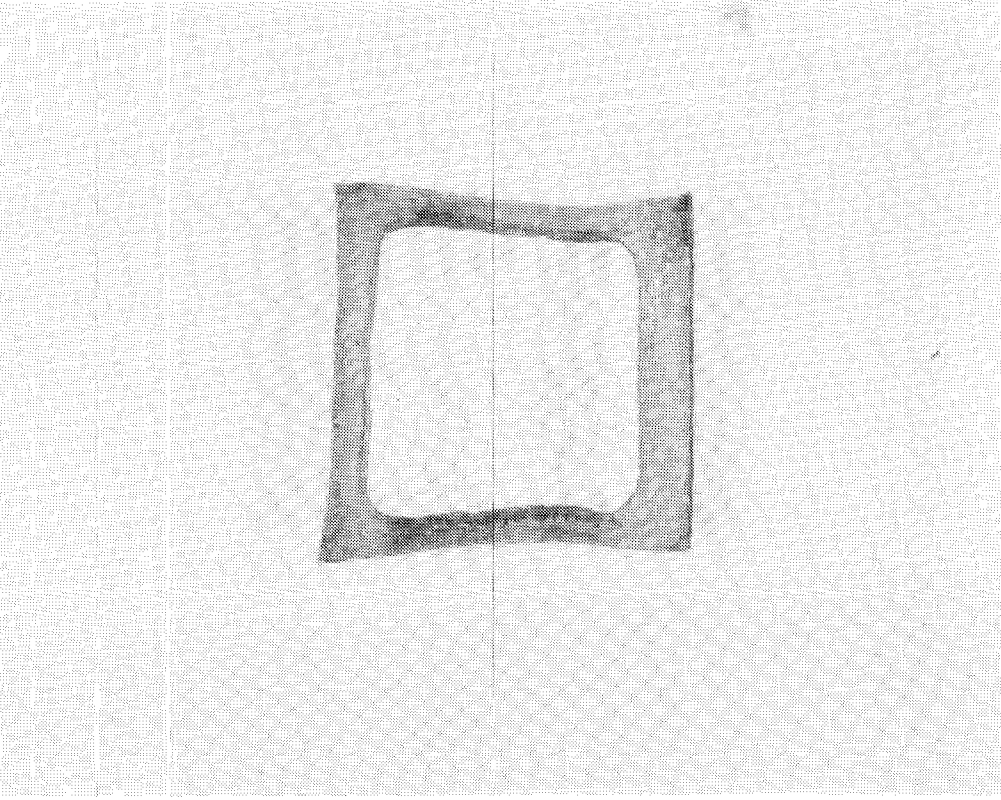


Side 2

Figure B-13. Fiber-Bituminous Galvanized Steel Exposed to High-Pressure Oxygen Test No. 2

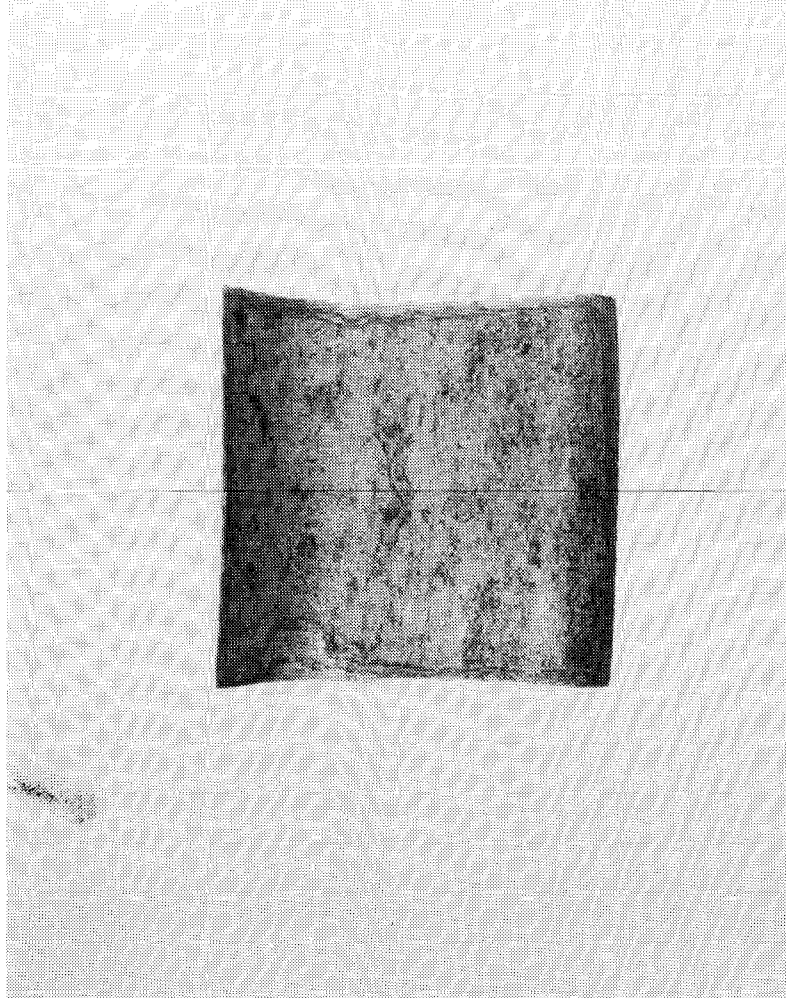


Side 1

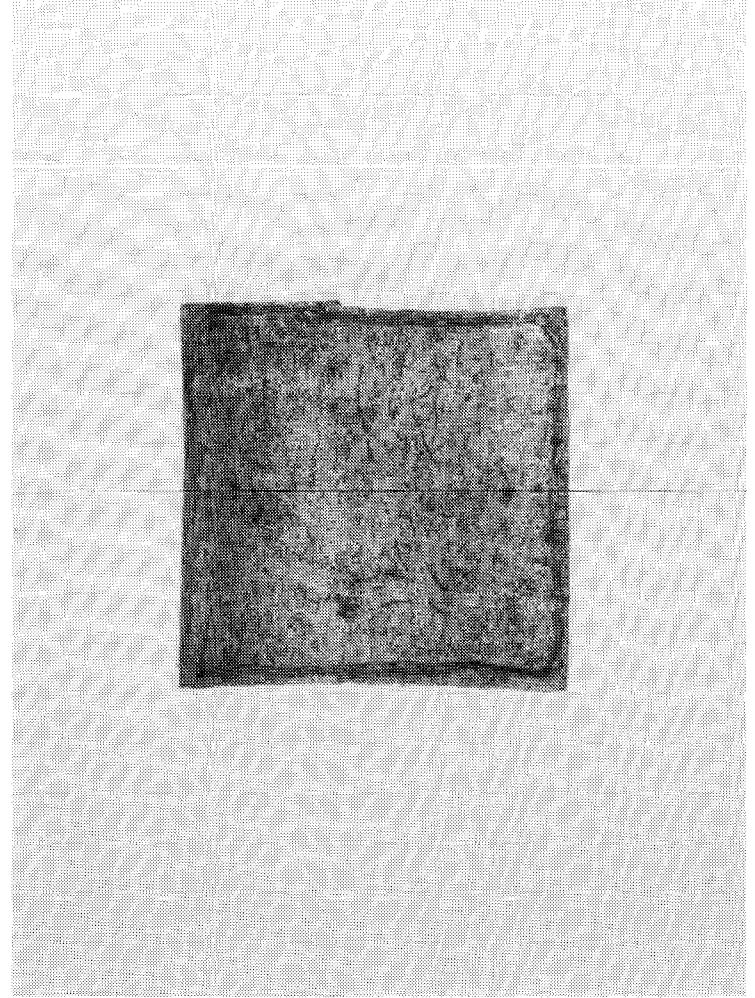


Side 2

Figure B-14. Fiber-Bituminous Galvanized Steel Exposed to High-Pressure Oxygen Test No. 2

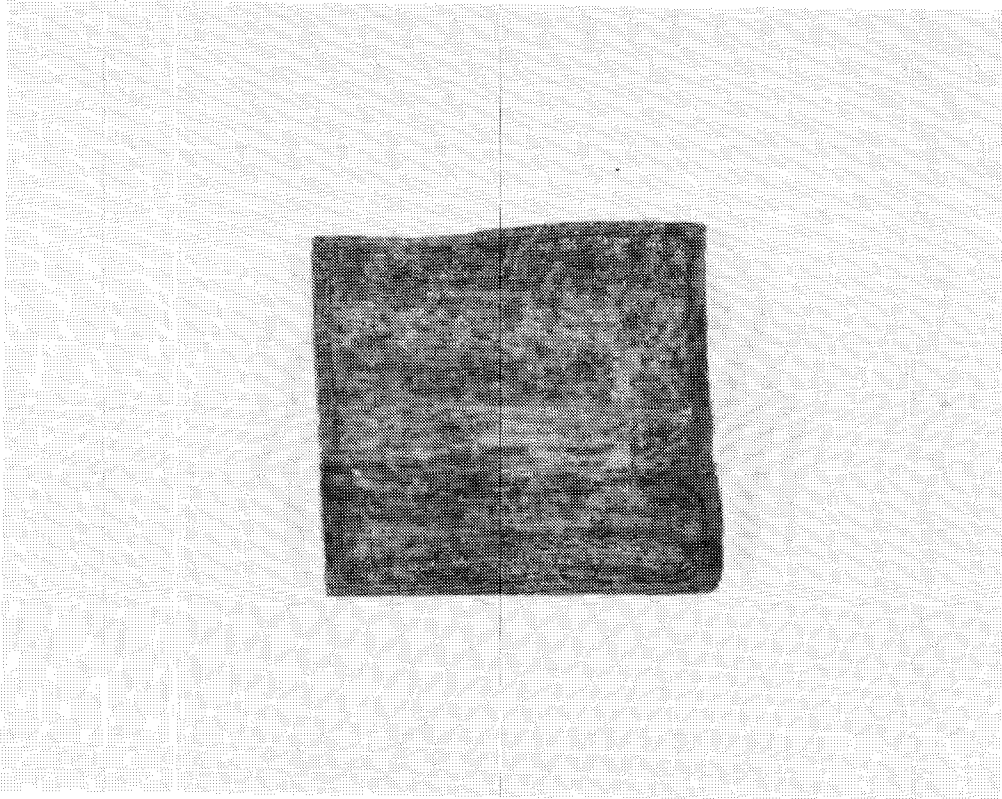


Side 1

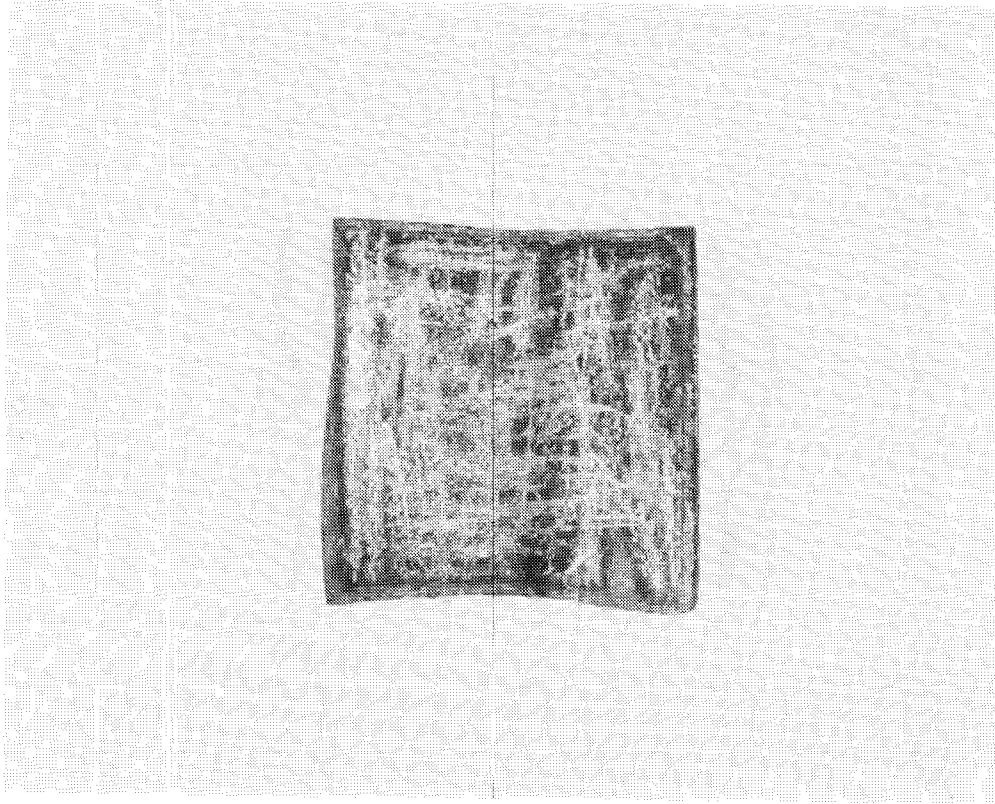


Side 2

Figure B-15. Asbestos-Asphalt Galvanized Steel Exposed to High-Pressure Oxygen Test No. 2

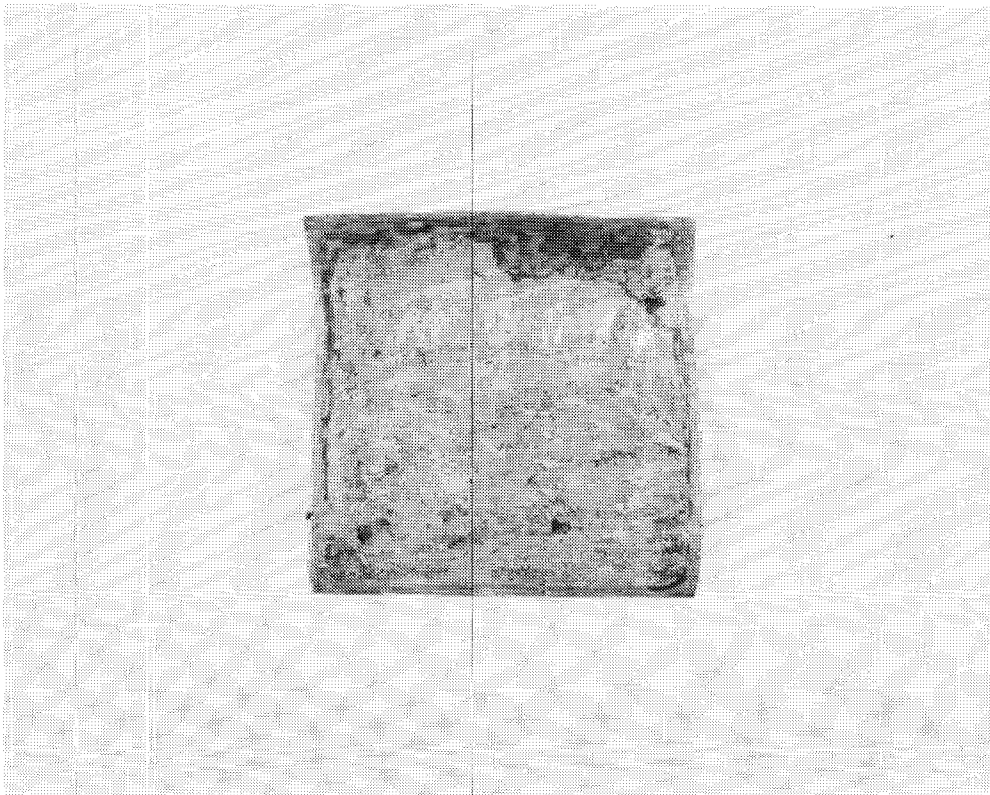


Side 1

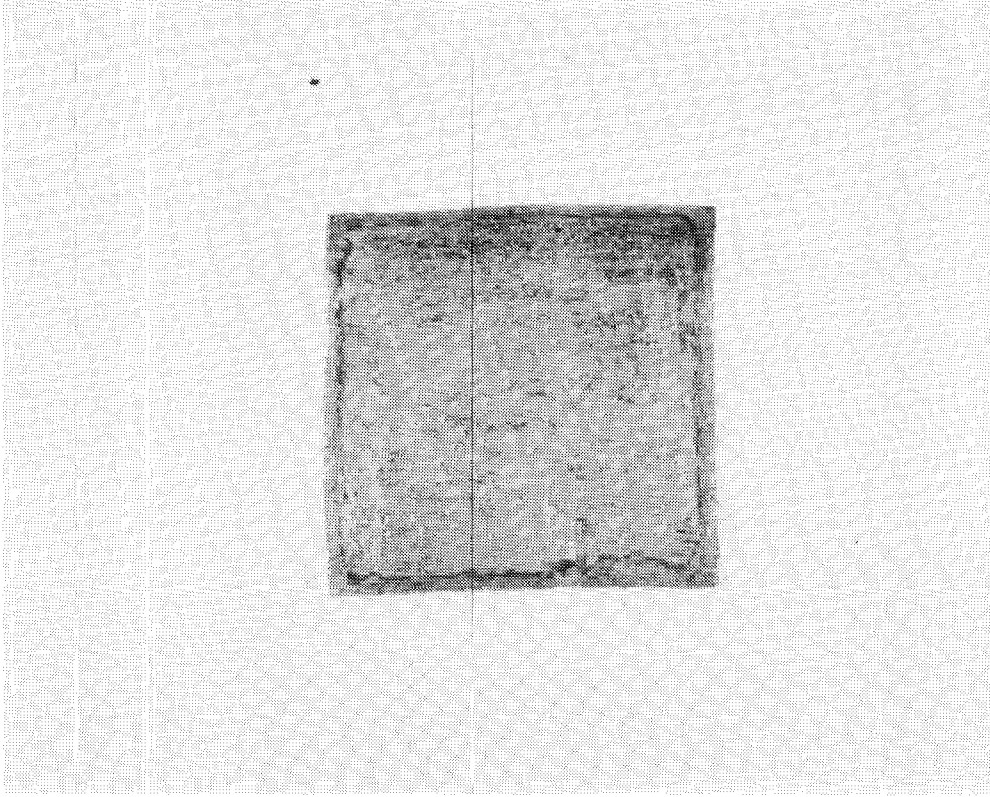


Side 2

Figure B-16. Asbestos-Asphalt Galvanized Steel Exposed to High-Pressure Oxygen Test No. 2



side 1



side 2

Figure B-17. Asbestos-Asphalt Galvanized Steel Exposed to High-Pressure Oxygen Test No. 2

APPENDIX C

ATOMIC ABSORPTION DETERMINATION OF
ZINC AND IRON IN SOLUTION

ATOMIC ABSORPTION (AA) METHOD

The following procedure was used when determining the amount of zinc and iron in the test solution after a test was completed. The procedure is described as it was performed on the contents of High-Pressure Oxygen Test No. 2, but it is general enough to be used for any test.

General Procedure

- (1) 250 ml of solution from the high pressure baroid cells was transferred into a 500 ml beaker.
- (2) Ten ml of concentrated nitric acid was added and the beaker was covered with a watch glass.
- (3) This solution was placed on a hot plate set for 140°C.
- (4) The solution was digested until all solids were removed and the solution became clear. Approximately 30 minutes was required.
- (5) The watch glass was then rinsed with 1% nitric acid and removed.
- (6) The solution was then evaporated on the hot plate to about 30 ml.
- (7) The remaining solution was transferred to a 50 ml volumetric flask and diluted to volume with 1% nitric acid.
- (8) The test solution was then ready for AA determination of zinc and iron.

Zinc Determination

- (1) 0.1 grams of pure zinc was dissolved in 1 ml of concentrated nitric acid and diluted to 100 ml with deionized water.
- (2) This produced a zinc standard (stock) solution of 1000 µg/ml zinc.
- (3) Two ml of this stock solution was taken and diluted to 200 ml using 1% nitric acid.
- (4) A calibration curve of 4 different concentrations of zinc was established using various dilutions of the above solution. The curve was obtained on the Atomic Absorption Spectrometer using a zinc lamp set at 213.9 nm.
- (5) The test solution was run on the spectrometer and the calibration curve was used to obtain the experimental value.
- (6) If the experimental value fell outside of the calibration range, it was diluted and rerun.

Iron Determination

- (1) 0.1 grams of pure iron was dissolved in 1 ml of concentrated nitric acid and diluted into 100 ml with deionized water.
- (2) This produced an iron standard (stock) solution of 1000 μ g/ml iron.
- (3) The dilution of the stock solution was the same as in the zinc standard solution except using deionized water instead of 1% nitric acid.
- (4) A calibration curve of 4 different concentrations of iron was established using the iron lamp on the Atomic Absorption Spectrometer set at 248.3 nm.
- (5) The test solution was run on the spectrometer and the calibration curve was used to obtain the experimental value.
- (6) If the experimental value fell outside of the calibration range, it was diluted and rerun.