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PART II

EVALUATION OF ELASTOMERS AS O-RING SEALS FOR LIQUID ROCKET FUEL AND OXIDIZER SYSTEMS

TECHNICAL DOCUMENTARY REPORT No. ASD-TDR-63-496, PART II

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RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

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(Prepared under Contract No. AF 33(616)-8483 by
Monsanto Research Corporation, Dayton, Ohio;
Carmen L. Bellanca, Ival O. Salyer, and Jay C. Harris, Authors)

FOREWORD

This report was prepared by Monsanto Research Corporation, Dayton Laboratory, under Air Force Contract No. AF 33(616)-8483. This contract was initiated under Project No. 7381, "Materials Evaluation," Task No. 738103, "Materials Information, Development, Collection, and Processing." The work was administered under the direction of the Materials Applications Division, Air Force Materials Laboratory, Research and Technology Division, Wright-Patterson Air Force Base, Ohio, with Mr. P. A. House acting as project engineer.

This report covers work performed during the period March 1963 to March 1964 at Monsanto Research Corporation's Dayton Laboratory.

The experimental work was performed by Carmen L. Bellanca and Ival O. Salyer. Jay C. Harris served as project manager.

ABSTRACT

O-Ring seals of selected elastomeric and compliant materials were evaluated for resistance to liquid rocket fuels in a simulated end-use test. The candidate elastomers were placed under compression in closed cells and exposed to the liquid and vapor of liquid rocket fuels and oxidizers for extended periods of time. Rate of fuel loss through the seal, and the change in physical properties of the seal materials were determined.

Nitrogen tetroxide, mixed hydrazines, chlorine trifluoride, 90% hydrogen peroxide, Hybaline A-5, and pentaborane were tested in direct contact with the O-ring seals at 73°F.

Metal clad and polyethylene encapsulated elastomeric O-rings were also tested for resistance to nitrogen tetroxide at 73°F.

The effect of temperature on elastomer endurance was determined by exposing the O-rings to nitrogen tetroxide, mixed hydrazines, and hydrogen peroxide at 160°F.

The effect of direct immersion in liquid rocket fuels on the physical properties of the seal materials was investigated by immersing promising O-ring candidates in nitrogen tetroxide, hydrogen peroxide, and mixed hydrazines.

The test methods used and the results obtained in this research are presented.

This technical documentary report has been reviewed and is approved.



W. P. Conrardy, Chief
Materials Engineering Branch
Materials Applications Division
AF Materials Laboratory

TABLE OF CONTENTS

	<u>Page No.</u>
I. INTRODUCTION	1
II. EXPERIMENTAL DETAILS	2
A. Test Cell Design and Fabrication	2
B. Test Procedure	2
III. TEST CELL EVALUATION OF ELASTOMERIC O-RINGS	4
A. Elastomers Tested as Seals for Nitrogen Tetroxide	4
B. Elastomers Tested as Seals for the Hydrazines	7
C. Elastomers Tested as Seals for Chlorine Trifluoride	8
D. Elastomers Tested as Seals for Hydrogen Peroxide	10
E. Elastomers Tested as Seals for Hybaline A-5	12
F. Elastomers Tested as Seals for Pentaborane	13
IV. IMMERSION TESTS	13
A. Test Method	13
B. Test Results	14
V. EVALUATION OF O-RING SEALS AT ELEVATED TEMPERATURE	17
A. Test Method	17
B. Test Results	18
VI. METAL CLAD ELASTOMERIC O-RING SEALS	20
A. Test Method	20
B. Test Results	20
C. Conductive Elastomers	23
VII. POLYETHYLENE ENCAPSULATED ELASTOMERIC O-RING SEALS	24

Table of Contents - Cont'd

	<u>Page No.</u>
VIII. SUMMARY AND CONCLUSIONS	25
REFERENCES	27
APPENDIX	28
Appendix 1. Formulations of Compounds Tested	29
Appendix 2. Glossary	32
Appendix 3. Tables 1-3	34
Appendix 4. Figures 1-66	37

LIST OF ILLUSTRATIONS

<u>Figure</u>		<u>Page No.</u>
1	Stainless Steel Test Cell	37
2	O-Ring Test Cell Cover	38
3	Test Cell with Relief Port	39
4	Polyethylene No. 39B: Exposure to Nitrogen Tetroxide	40
5	Polyethylene No. 39B: Nitrogen Tetroxide System	41
6	Polyethylene No. 39C: Exposure to Nitrogen Tetroxide	42
7	Uncrosslinked Low Pressure Polyethylene: Exposure to Nitrogen Tetroxide	42
8	Teflon: Exposure to Nitrogen Tetroxide	42
9	Filled Teflon: Exposure to Nitrogen Tetroxide	43
10	Viton B Formulation No. 76: Exposure to Nitrogen Tetroxide	43
11	Viton B Formulation No. 76: Nitrogen Tetroxide System	44
12	Butyl Formulation No. 34: Exposure to Nitrogen Tetroxide	45
13	Butyl Formulation No. 34: Nitrogen Tetroxide System	46
14	Teflon: Exposure to Mixed Hydrazines	47
15	Butyl Formulation No. 34: Exposure to Mixed Hydrazines (Liquid)	47
16	Butyl Formulation No. 34: Exposure to Mixed Hydrazines (Vapor)	47
17	EPR No. 2: Exposure to Mixed Hydrazines	47

List of Illustrations - Cont'd

<u>Figure</u>		<u>Page No.</u>
18	Polyethylene Formulation No. 39: Exposure to Mixed Hydrazines	48
19	Polyethylene Formulation No. 39B: Exposure to Mixed Hydrazines	48
20	Polyethylene Formulation No. 39C: Exposure to Mixed Hydrazines	48
21	Uncrosslinked Low Pressure Polyethylene: Exposure to Mixed Hydrazines	49
22	DX-954-GP: Exposure to Mixed Hydrazines	49
23	DX-954-GP: Mixed Hydrazines System	50
24	Teflon: Exposure to Chlorine Trifluoride	51
25	Filled Teflon: Exposure to Chlorine Trifluoride (Liquid)	51
26	Filled Teflon: Exposure to Chlorine Trifluoride (Vapor)	51
27	Polyethylene Formulation No. 39A: Exposure to Chlorine Trifluoride (Liquid)	52
28	Polyethylene Formulation No. 39A: Exposure to Chlorine Trifluoride (Vapor)	52
29	Polyethylene Formulation No. 39A: Chlorine Trifluoride System	53
30	Silicone No. PR-11555: Exposure to Hydrogen Peroxide	54
31	Silicone No. PR-11555: Hydrogen Peroxide System	55
32	Viton B Formulation No. 5187: Exposure to Hydrogen Peroxide	56
33	Viton B Formulation No. 5187: Hydrogen Peroxide System	57

List of Illustrations - Cont'd

<u>Figure</u>		<u>Page No.</u>
34	Teflon: Exposure to Hydrogen Peroxide	58
35	Polyethylene Formulation No. 39: Exposure to Hydrogen Peroxide	58
36	Butyl Formulation No. 34: Exposure to Hydrogen Peroxide	58
37	Butyl Formulation No. 34: Hydrogen Peroxide System	59
38	EPR No. 2: Exposure to Hydrogen Peroxide	60
39	Teflon: Exposure to Hybaline A-5	61
40	Polyethylene Formulation No. 39A: Exposure to Hybaline A-5	61
41	EPR No. 2: Exposure to Hybaline A-5	61
42	Viton A Formulation No. 3: Exposure to Pentaborane	62
43	Teflon: Exposure to Pentaborane	62
44	Test Cell Storage Container	63
45	Teflon: Exposure to Nitrogen Tetroxide at 160°F.	64
46	Polyethylene 39A: Exposure to Nitrogen Tetroxide at 160°F.	65
47	Butyl Formulation No. 34: Exposure to Nitrogen Tetroxide at 160°F.	66
48	Polyethylene Formulation No. 39A: Exposure to Mixed Hydrazines at 160°F.	67
49	Butyl Formulation No. 34: Exposure to Mixed Hydrazines at 160°F.	67

List of Illustrations - Cont'd

<u>Figure</u>		<u>Page No.</u>
50	Polyethylene Formulation No. 39 A: Hydrogen Peroxide at 160°F.	68
51	Polyethylene Formulation No. 39A: Exposure to Hydrogen Peroxide at 160°F.	69
52	Silicone No. PR-11555: Exposure to Hydrogen Peroxide at 160°F.	70
53	Viton B Formulation No. 5187: Exposure to Hydrogen Peroxide at 160°F.	71
54	Test Cell for Metal Clad O-Ring Seal Testing	72
55	Lead Coated O-Rings After Nitrogen Tetroxide Exposure	73
56	Copper/Lead Coating O-Rings After Nitrogen Tetroxide Exposure - 30% Compression	74
57	Copper/Lead Coated O-Rings: Exposure to Nitrogen Tetroxide (Liquid)	75
58	Copper/Lead Coated O-Rings After Nitrogen Tetroxide Liquid Exposure - 15% Compression	76
59	Copper/Lead Coated O-Rings: Exposure to Nitrogen Tetroxide (Vapor)	77
60	Copper/Lead Coated O-Rings After Nitrogen Tetroxide Vapor Exposure - 15% Compression	78
61	Silver/Lead Coated O-Rings: Exposure to Nitrogen Tetroxide	79
62	Crimped Lead Coated O-Rings: Exposure to Nitrogen Tetroxide	79
63	Crimped Lead Coated O-Rings After Nitrogen Tetroxide Exposure	80
64	Lead Coated O-Rings After Nitrogen Tetroxide Exposure	81

List of Illustrations - Cont'd

<u>Figure</u>		<u>Page No.</u>
65	Crimped Aluminum - Slit Copper/lead: Nitrogen Tetroxide Exposure	82
66	Conductive Elastomer Copper Plated	83

I. INTRODUCTION

Advanced liquid propellant systems for spacecraft and missiles are generally characterized by high reactivity and high solvent power, especially toward polymeric materials of construction. Because of their resilience, flexibility, and compressibility, organic elastomers are well suited to serve as functional components in missile systems. However, the susceptibility of these organic materials to propellant attack leads to degradation and subsequent failure.

Since the properties of elastomers are requisites in applications such as seals, hoses, and expulsion bladders, the need exists for elastomeric materials resistant to highly reactive fuels and oxidizers.

This report describes the further investigation of the effects of various fuels and oxidizers on selected elastomeric O-ring seals. Fuels included in the program are hydrazine/unsymmetrical dimethyl hydrazine (1:1 mixture), Hybaline A-5, and Pentaborane. Oxidizers are nitrogen tetroxide, chlorine trifluoride, and 90% hydrogen peroxide.

The major effort of the past year was directed toward continuing the long term evaluation of seal materials under simulated service conditions at ambient temperature (Ref. 1). Service conditions in this case consist of the seal under static compression in a closed cell, in direct contact with the test media.

Other work initiated and completed the past year is as follows:

1. Evaluation of elastomeric O-rings under simulated service conditions at 160°F in nitrogen tetroxide, mixed hydrazines, and 90% hydrogen peroxide.
2. Evaluation of the effect of nitrogen tetroxide on metal clad and polyethylene encapsulated elastomeric O-rings under simulated in-use conditions.
3. Immersion tests of the most promising materials in nitrogen tetroxide, mixed hydrazines, and hydrogen peroxide.

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Many of the items compared in this report were commercial items that were not developed or manufactured to meet any Government specification, to withstand the tests to which they were subjected, or to operate as applied during this study. Any failure to meet the objectives of this study is no reflection on any of the commercial items discussed herein or on any manufacturer.

II. EXPERIMENTAL DETAILS

A. TEST CELL DESIGN AND FABRICATION

The evaluation of elastomeric O-ring seals under simulated service conditions was accomplished through the use of two component test cells. The design consists of a flanged cup with a grooved flange cover for O-ring seating (Figure 1). When secured together the desired O-ring compression (ca. 20%) is obtained. Four pressure relief slots are spaced at 90° intervals on the flange cover to allow fluid and/or vapor leaking by the O-ring to be vented (Figure 2). In testing O-rings in contact with the vapor phase the cup is placed upright. For liquid phase evaluation, the cell is inverted.

Relief port modifications in the test cell were required in hydrogen peroxide, Hybaline A-5, and Pentaborane systems due to possible high pressure gas buildup within the cell. A chamfered relief port is located in the test chamber and a Teflon ball seated in the port (Figure 3). A rise in internal pressure to 25-30 psi causes the ball to rise and thereby allow pressure relief.

Aluminum alloy No. 1060 was utilized as test cell material for hydrogen peroxide systems; stainless steel No. 304 used as test cell material for the other fluids.

B. TEST PROCEDURE

The evaluation of O-ring seals in test cells was accomplished as follows: the test cells were assembled with their respective O-rings and tare weight established. The cells were filled approximately half-full of test fluid and the net weight determined. All of the current exposure tests were conducted with the O-ring exposed to the liquid phase. Generally three O-rings in each elastomer/fluid system were tested.

Weighings were made daily for the first week, then weekly for two months. If little loss of fuel or oxidizer was observed at this time, weighings were subsequently conducted monthly. Room temperature evaluations were conducted in vented laboratory hoods; test temperature was maintained at $73 \pm 3^{\circ}\text{F}$.

The extreme sensitivity of Pentaborane toward oxygen, resulting in spontaneous ignition, necessitated revisions in the standard procedure of test initiation.

To eliminate possible oxygen exposure, Pentaborane was transferred into an evacuated single stopcock glass bulb from the original storage container and subsequently vacuum distilled into an evacuated graduated double stopcock glass bulb seated in dry ice. To minimize the need of opening the second stopcock during cell filling, an argon blanket was placed over the distilled fuel to enforce complete drainage.

The filled bulb was placed in a stainless steel dry box under dry ice. An outer wooden box vented to the atmosphere surrounds the dry box. Nitrogen was fed continuously through the dry box until gas chromatograph analyses of effluent samples indicated an extremely low oxygen content. The subsequent filling and assembling of test cells and test O-rings was conducted under these inert nitrogen conditions. The filled test cells were stored and weighed in the dry box.

The cleaning and pickling of test cells prior to test was as follows:

Cells Used for Nitrogen Tetroxide and Chlorine Trifluoride Systems (Ref. 2)

1. Solvent degreasing (Triclene - 1 hour at 120°F).
2. Detergent cleaning (4% soln. Santomerse® - 1 hour at 120°F). Rinse.
3. Nitric acid immersion (50% - 1 hour). Rinse with distilled water and dry.

Cells Used for Mixed Hydrazine Systems (Ref. 2)

1. Solvent degreasing (Triclene - 1 hour at 120°F).
2. Detergent cleaning (4% soln. Santomerse® - 1 hour at 120°F). Rinse.
3. Nitric acid immersion (50% - 1 hour). Rinse.
4. Ammonium hydroxide immersion (20% - 1 hour). Rinse with distilled water and dry.

Cells Used for 90% Hydrogen Peroxide (Ref. 3)

1. Solvent degreasing (Triclene - 1 hour at 120°F).
2. Detergent cleaning (4% soln. Santomerse® - 1 hour at 120°F). Rinse.
3. Nitric acid immersion (35% - 24 hours). Rinse
4. Hydrogen peroxide immersion (35% - 48 hours).
5. Hydrogen peroxide immersion (90% - 72 hours).

Cells Used for Hybaline A-5

1. Solvent degreasing (Triclene - 1 hour at 120°F).
2. Detergent cleaning (4% soln. Santomerse® - 1 hour at 120°F). Rinse.
3. Nitric acid immersion (50% - 1 hour). Rinse with distilled water.
4. Rinse with toluene.
5. Blow dry with nitrogen. Store in polyethylene bag.

Cells Used for Pentaborane (Ref. 2)

1. Solvent degreasing (Triclene - 1 hour at 120°F).
2. Detergent cleaning (4% soln. Santomerse® - 1 hour at 120°F). Rinse.
3. Nitric acid immersion (50% - 1 hour). Rinse with distilled water.
4. Alcohol immersion for 15 minutes.
5. Blow dry with nitrogen. Store in vacuum desiccator.

III. TEST CELL EVALUATION OF ELASTOMERIC O-RINGS

A. ELASTOMERS TESTED AS SEALS FOR NITROGEN TETROXIDE

Hydrocarbon and fluorocarbon type polymers, containing little or no unsaturation, have shown the most resistance to oxidative attack by nitrogen tetroxide contact (Ref. 1,4,5). Elastomeric and compliant materials selected for further evaluation as seals for nitrogen tetroxide systems during the past year were: high and low pressure polymerized polyethylene, Teflon, calcium fluoride filled Teflon, Viton B, and butyl rubber.

Teflon and filled Teflon were selected for evaluation based on the inherent resistance of the polymers to oxidative degradation. In addition the crystallinity arising from the closely packed order of the chains should inhibit permeation of oxidizer through the O-ring.

Test results of the above material selections are as follows:

1. High Pressure Polyethylene Formulation No. 39B (Ref. 6)

O-Ring seals of carbon black filled and vulcanized high pressure polyethylene formulation No. 39B were molded and tested in contact with nitrogen tetroxide. Very low loss of fluid was observed through the first month's exposure; however, as can be observed in Figure 4, a steady increase in rate of loss was noted. After eight months' exposure, an average fluid loss of 67% was observed. The test cells were opened at this time and the action of the oxidizer caused the O-ring composition to become very brittle with complete loss of physical properties (Figure 5).

2. Low Pressure Polyethylene Formulation No. 39C (Ref. 6)

In order to further define the merits of polyethylene as a seal material for nitrogen tetroxide systems, O-rings of carbon black filled, vulcanized low pressure polymerized (high density and crystallinity) polyethylene were molded.

O-Ring seals of low pressure polyethylene No. 39C have been under test eight months and have allowed only 3.5% loss (Figure 6).

This is a significant improvement in sealing effectiveness compared to high pressure polyethylene. The highly crystalline nature of the low pressure polymerized polyethylene (85% in LPPE vs 60% in HPPE) is believed to be principally responsible for the improved resistance to degradation by nitrogen tetroxide. This increased resistance arises from the fact that either liquids or gases diffuse with much difficulty through closely packed crystal domains of the polymer, and considerable energy is required to destroy the crystal lattice. Thus, in a highly crystalline polymer, permeation of oxidizer through the seal, and subsequent loss, would depend upon progressive degradation of the crystallites and subsequent attack of the amorphous sections of the polymer.

Additionally, the lower level of unsaturation and fewer reactive branch sites found in low pressure polyethylene may also contribute significantly to the improved stability of this highly crystalline polyethylene.

3. Low Pressure Polyethylene (Uncrosslinked) (Ref. 6)

Uncrosslinked unfilled low pressure polyethylene O-rings, (high density and high crystallinity Marlex 6015), have been exposed to nitrogen tetroxide for eight months and have allowed only 0.2% loss (Figure 7). It is evident an excellent seal has been effected. The polymer has shown surprising resistance to attack, considering the lack of filler loading and crosslinking which is generally required in high pressure polyethylene for optimum resistance (Ref. 1). As previously noted, it is felt the high degree of crystallinity present and closely packed chains have imparted an effect similar to crosslinking.

4. Teflon

Teflon O-rings have been under exposure test 16 months and have allowed 1.0% loss in fluid (Figure 8).

An excellent seal has been effected and Teflon is a prime candidate for long term seal applications in nitrogen tetroxide systems.

5. Filled Teflon

Calcium fluoride filled-Teflon O-rings have been under test 16 months and have allowed 4% fluid loss (Figure 9). No loss in fluid was observed through the first three months' exposure; however, a very slow rate of loss thereafter is indicated.

6. Viton B Formulation No. 76 (Ref. 5)

O-ring seals of Viton B formulation No. 76 were exposed to nitrogen tetroxide for 9.5 months and sustained an average loss of 21.8% (Figure 10). Two cells were opened at that time while a third (5% loss) was allowed to continue. The O-rings had degraded to a gummy mass (Figure 11). The third cell allowed 19% loss after one year's exposure, indicative of degradation. Since the high reactivity of nitrogen tetroxide toward organic elastomers usually leads to much earlier degradation of elastomeric seals (Ref. 1), these results indicate Viton B may be suitable for short term application.

7. Butyl Formulation No. 34 (Ref. 4)

O-ring seals molded of butyl formulation No. 34 allowed complete loss of oxidizer after 8.5 months (Figure 12). An effective seal was noted for a limited period (6.5% loss/2 months) but thereafter a rapid rate of oxidizer loss was observed. Degradation of the O-rings was visually observed as a gummy

mass containing small brittle particles (Figure 13). It is felt the gummy state is contributed by the butyl polymer, which degrades through a chain scission mechanism. The brittle particles are probably due to polyethylene present in the formulation which degrades by oxidative crosslinking.

B. ELASTOMERS TESTED AS SEALS FOR THE HYDRAZINES

Previous work completed during the first year of this contract and the last work period indicated excellent compatibility of saturated nonpolar hydrocarbon polymers toward the highly polar mixed hydrazines (Ref. 1).

The following polymers have been under long term exposure: Teflon, butyl, ethylene/propylene copolymer, high and low pressure polyethylene, and Monsanto vinyl copolymer DX-954-GP.

O-ring seal test results are as follows:

1. Teflon

Teflon O-rings have been under exposure 19 months and have allowed 0.8% fluid loss (Figure 14). It is evident an excellent seal has been effected.

2. Butyl Formulation No. 34

Butyl O-rings have been under test 17 months versus mixed hydrazines liquid and vapor phases. O-rings in contact with liquid have allowed 1.1% loss (Figure 15); seals versus the vapor phase have allowed 1.3% loss (Figure 16).

It appears butyl rubber is an excellent seal material for hydrazines and is a prime candidate for long term applications in this system.

3. Ethylene-Propylene Formulation No. 2 (Ref. 1)

O-rings molded of EPR No. 2 during 17 months of test have allowed 3.8% loss of fuel (Figure 17). An excellent seal has been effected, presenting further evidence of the apparent compatibility of saturated nonpolar hydrocarbon polymers with highly polar hydrazines.

4. High Pressure Polyethylene Formulation No. 39

O-ring seals molded of polyethylene formulation No. 39 appear to be excellent seals for mixed hydrazine systems. Seals have been under test 19 months and have allowed 0.6% fuel loss (Figure 18).

5. High Pressure Polyethylene Formulation No. 39B

O-ring seals of polyethylene formulation No. 39B have been under test 9 months and have allowed 0.8% fuel loss indicating a satisfactory seal has been effected (Figure 19).

6. Low Pressure Polyethylene Formulation No. 39C

O-ring seals of polyethylene formulation No. 39C have allowed 0.7% loss through 9 months' exposure (Figure 20).

7. Low Pressure Polyethylene (Uncrosslinked)

O-rings molded of uncrosslinked low pressure polyethylene, Marlex 6015, have been under hydrazine exposure 9 months and have allowed 0.5% fluid loss (Figure 21).

8. DX-954-GP

O-ring seals molded of DX-954-GP polymer under test 8 months allowed an average loss of 22.7% (Figure 22). Two cells were opened at this time and compression set had occurred allowing fluid loss through the seal (Figure 23).

Degradation of the polymer was expected to some degree in this case due to its polar nature. Though its polarity is low compared to commonly known polar elastomers, such as the butadiene-acrylonitrile copolymers and acrylates, it appears sufficient to render the polymer incompatible with the polar hydrazines.

Compression set was determined to be 28% (Table 1); ultimate tensile and elongation decrease was 14.5% and 53% respectively. Shore "A" hardness increased 24 points. Volume and weight changes were +2.6% and +2.9% respectively.

C. ELASTOMERS TESTED AS SEALS FOR CHLORINE TRIFLUORIDE

Chlorine trifluoride, to date, has presented a problem in finding satisfactory elastomeric seal materials due to its high reactivity toward organic rubber materials. As reported in

progress reports to the project engineer and in Reference 1, fluorinated hydrocarbon elastomers, such as Viton A and B, show limited utility as seals for chlorine trifluoride systems. Compliant materials currently under evaluation are: Teflon, calcium fluoride filled Teflon, and crosslinked, carbon black loaded high pressure polyethylene. Low pressure polyethylene has not been tested in chlorine trifluoride.

Test results are as follows:

1. Teflon

Teflon O-rings have been under test 20 months and no weight loss has been observed (Figure 24). However, deposits have been noted at the seal flange area. Qualitative emission analysis of the deposit indicated iron and chromium as the major constituents; infrared analyses indicated the presence of water, metallic hydrates, and possibly metal fluorides. A possible explanation of the cause of deposit formation is an absorption of oxidizer by the O-ring, subsequent attack of the stainless steel test cell resulting in metal corrosion and, upon contact with moisture, formation of metal hydrates. A slight weight gain has been observed and is believed due to water present as a result of moisture absorption from the atmosphere.

2. Filled Teflon

Filled Teflon O-rings have been under test 20 months versus chlorine trifluoride liquid and vapor and no weight loss observed (Figures 25 and 26). Here again, a slight weight gain and deposits were noted.

3. Polyethylene Formulation No. 39A

Three O-ring seals of polyethylene formulation No. 39A were exposed to liquid chlorine trifluoride; two exposed to vapor. One cell from each phase was opened after 12 months due to high oxidizer loss. Complete degradation of the O-rings was observed (Figure 29). Of the two remaining O-rings exposed to liquid, one cell was opened after 16 months because of high loss; the third has been under test 20 months and has allowed 20% loss (Figure 27). The remaining O-ring was exposed to vapor for 19 months and allowed 33% loss (Figure 28). The cell was opened at this time and complete degradation, such as that of Figure 29, was observed.

As was noted for nitrogen tetroxide, the resistance of high density polyethylene seals to ClF_3 would be expected to be significantly better than obtained with the O-rings of HPPE.

D. ELASTOMERS TESTED AS SEALS FOR HYDROGEN PEROXIDE

Concentrated (90%) hydrogen peroxide presented a problem in material selection due to its high oxidative properties, susceptibility to catalytic decomposition due to contamination and resulting pressure buildup and decrease in peroxide concentrations. Materials selected for evaluation as seals were silicone rubber, Viton B, Teflon, polyethylene, butyl rubber, and ethylene/propylene copolymer.

Test results of the above elastomer selections are as follows:

1. Silicone Rubber Formulation PR-11555

O-ring seals of silicone rubber PR-11555 were exposed to 90% hydrogen peroxide for 10 months at which time 31.4% loss in weight was noted (Figure 30). Upon opening the cells, a white deposit was noted (Figure 31) which was identified as hydrated alumina. Analysis of the remaining solution showed the peroxide concentration had decreased to 27.9%.

Ultimate O-ring tensile strength decreased 31%; a decrease in elongation (45%) was observed. Shore "A" hardness increased 15 points (43 to 58). As observed in Table 1, a volume change of -2.36% was found and final compression set was 22%.

2. Viton B Formulation No. 5187 (Ref. 7)

Viton B No. 5187 O-rings allowed 21% weight loss after 10 months (Figure 32). Upon opening the cells, the O-rings appeared to be in excellent condition and no deposits were found (Figure 33). As may be observed in Table 1, excellent retention of physical properties was found. Ultimate tensile and elongation increased (0.8% and 2% respectively) accompanied by a five-point increase in Shore "A" hardness. Volume and weight changes were slight (+0.22% and +0.19% respectively) and compression set of 12% had taken place. The resultant peroxide concentration was found to be 58.3%.

3. Teflon

O-ring seals of Teflon have been under hydrogen peroxide exposure 17 months. As seen in Figure 34 weight loss at 10 months was 6.6%. At this time, the peroxide was replaced by fresh 90% solution on the assumption that the loss in weight was due solely to oxygen release as opposed to leakage through

the seal. Assay of the resultant peroxide concentration found it to be in close agreement with the theoretical concentration based on the known loss in weight (actual 81.5% calculated 83%). Weight loss for the remaining 7 months was 14.03%.

4. Polyethylene Formulation No. 39

O-ring seals of polyethylene formulation No. 39 have been under test 17 months. Weight loss after 10 months was 2.6% (Figure 35). At this time, the cells were emptied and fresh 90% peroxide added. Weight loss for the remaining seven months was 4.17%.

The peroxide concentration of the solution removed from the cells was found to be 83% as opposed to the calculated value of 85%. A five day time lapse between emptying the cells and determination of the concentration possibly accounts for the difference in values due to continual release of oxygen accompanied by decrease in concentration.

5. Butyl Formulation No. 34

O-ring seals of butyl formulation No. 34 allowed 45.4% fluid weight loss through 10 months exposure (Figure 36). A white deposit was noted upon cell opening (Figure 37) and determined to be hydrated alumina. The residual peroxide concentration was 0.028%, indicative of vigorous peroxide decomposition. Determination of test physical properties (Table 1) showed excellent retention of physical properties. Ultimate tensile loss was 5%, elongation increased 5%, and Shore "A" hardness decreased from 82 to 79. Volume and weight changes were -0.34% and +0.36% respectively. Very low compression set (4.5%) was noted.

The relatively small change in physical properties of the O-ring was unexpected based on the vigorous peroxide decomposition and subsequent large decrease in concentration. It is believed decomposition of the peroxide was due to interaction with Hypalon 20, a chlorosulfonated polyethylene present in the formulation, rather than the butyl elastomer. It has been shown the presence of chlorides in aluminum/concentrated hydrogen peroxide systems leads to peroxide decomposition and localized pitting of the aluminum (Ref. 10).

6. Ethylene/Propylene Formulation No. 2

O-ring seals of EPR formulation No. 2 have been under peroxide exposure 5 months and have allowed 43% loss (Figure 38).

It appears the compliant polymers, Teflon and polyethylene, are excellent sealant materials for hydrogen peroxide systems. Viton B formulation No. 5187 could be considered for limited utility as O-ring seals, however, the decrease in peroxide concentration could prohibit usage of this material for long term application. Some of the rubber formulations (e.g., butyl, EPR) may contain ingredients which catalyze peroxide decomposition.

E. ELASTOMERS TESTED AS SEALS FOR HYBALINE A-5

The following materials were selected as O-ring seal candidates: Teflon, polyethylene, and ethylene/propylene.

Test results of the above material selections are as follows:

1. Teflon

O-ring seals of Teflon have been under Hybaline exposure seven weeks and have allowed 3.05% weight loss (Figure 39).

2. Polyethylene Formulation No. 39A

Polyethylene O-ring seals during seven weeks under test have allowed 3.9% weight loss (Figure 40).

3. Ethylene/Propylene (EPR) Formulation No. 2

O-ring seals of EPR No. 2 have allowed 3.4% loss during seven weeks of test (Figure 41).

In all cases, there was no evidence of fluid leakage past the flange seal area. However, traces of white deposits were discernible at the relief port of most cells indicative of an apparent reaction occurring within the cells. It is felt the loss in weight is due to relief of gas buildup within the cell (venting) rather than failure of the seal.

F. ELASTOMERS TESTED AS SEALS FOR PENTABORANE

Pentaborane, a high energy fuel, has been found to have a degrading effect on most elastomers (Ref. 8). A literature search showed only the fluorocarbon polymers, Viton A and Teflon, suitable for long term utility in pentaborane systems. As a result, O-ring seals of Viton A formulation No. 3 and Teflon were selected for long term seal evaluation.

Test results are as follows:

1. Viton A Formulation No. 3

Viton A O-rings have been under test for four weeks and allowed 4% loss (Figure 42). No leakage was apparent at the flange seal area, however, it appeared internal gas relief had occurred. A white stain was noted at the relief port. Gas chromatograph analysis has indicated minute traces of hydrogen present in the dry box, possibly as a result of the interaction between pentaborane and the test cell or O-ring.

2. Teflon

O-ring seals of Teflon during four weeks of test have allowed 3.2% weight loss (Figure 43). No evidence of leakage past the seal area was apparent, however, a white stain at the relief port as noted above indicated gas release had occurred.

IV. IMMERSION TESTS

Immersion tests of promising seal materials were conducted to assist RTD formulate bulletins defining seal material recommendations for nitrogen tetroxide, hydrazines, and 90% hydrogen peroxide systems. Selection of materials was based on results of work completed during the first year of this contract and described in progress reports to the project engineer.

In addition, immersion tests were conducted to determine candidate O-ring seal materials for Hybaline A-5 systems (Ref. 6).

A. TEST METHOD

The high volatility of the test media prohibited the use of the standard method of elastomeric immersion in liquids (ASTM D-471), "Method of Test for Change in Properties of Elastomeric

Vulcanizates Resulting from Immersion in Liquids." All swelling data from D-471 is obtained from the specimen removed from the test media. Due to the high volatility of the liquids, this operation would be difficult.

This necessitated the use of ASTM D-1460, "Change in Length of an Elastomeric Vulcanizate Resulting from Immersion in a Liquid." This method gives limited information of the effect of elastomeric immersion, by observing through the transparent wall of the container the change in specimen length by use of graph paper background. Based on the assumption the specimen swells or shrinks proportionally in all directions, a table is included in D-1460 denoting the relationship between the percentage difference in length and the corresponding change in volume expressed as a percentage of the original volume of the specimen.

Test conditions were 14 days at $73 \pm 3^{\circ}\text{F}$. Tensile, elongation, and Shore "A" hardness were determined after evaporation of test fluid from the specimen.

Evaluation of specimens in nitrogen tetroxide was conducted as follows: Test specimens were placed in Carius tubes and sufficient nitrogen tetroxide introduced to reach a level double the specimen length. The oxidizer was then frozen and the loaded tubes heat sealed. After completion of the test period, the tubes were broken and the specimens allowed to degas for 72 hours in a vented laboratory hood.

Immersion tests in mixed hydrazine, hydrogen peroxide, and Hybaline A-5 were carried out as follows: Test specimen and sufficient fluid to cover twice the length of the specimen were placed in test tubes and capped with a polyethylene stopper. Periodically throughout the two-week test period the stoppers were removed to relieve any pressure buildup which may have occurred. Upon completion of the test period, test specimens were removed from the respective fluids and allowed to degas overnight in a vented hood. Hybaline samples were rinsed in toluene after removal and allowed to degas.

B. TEST RESULTS

1. Nitrogen Tetroxide

Materials selected for immersion testing in nitrogen tetroxide were Teflon, polyethylene formulation No. 39A, and butyl rubber formulation No. 34. Test results are as follows:

a. Teflon

Teflon exhibited no volume change upon 14 days' immersion (Table 2). An average tensile loss of 18.8% was observed, accompanied by 10.8% decrease in elongation. Shore "A" hardness change was difficult to measure due to the fact that all readings were near the upper maximum limit of the durometer.

As noted in Table 2, Shore "A" durometer readings in excess of 100 were observed as opposed to an original hardness of 98.

b. High Pressure Polyethylene Formulation No. 39A

Polyethylene No. 39A exhibited no volume change upon immersion testing. However, complete loss of physical properties was observed as the test specimens broke upon assembling in the tensile test jaws. No change in hardness was noted (Table 2). Low pressure, high density polyethylene should be more resistant, and will be tested.

c. Butyl Formulation No. 34

Butyl formulation No. 34 showed a volume change of +33.1% after 14 days' immersion. After degassing, a decrease in tensile of 70% was observed; elongation increased 25%; Shore "A" hardness decreased 3 points (Table 2).

2. Mixed Hydrazines

Promising seal materials selected for immersion studies in hydrazines were polyethylene formulation No. 39A, ethylene/propylene No. 2, and butyl formulation No. 34.

Test results are as follows:

a. High Pressure Polyethylene Formulation No. 39A

Polyethylene formulation No. 39A exhibited no volume change upon 14 days' immersion. An increase in ultimate tensile and elongation was observed (1.5% and 1.7% - Table 2). Shore "A" hardness was determined immediately upon removal from the test fluid and no change was observed. After degassing overnight, the hardness increased one point (98 to 99).

b. Ethylene/Propylene Formulation No. 2

EPR No. 2 exhibited no volume change upon 14 days' immersion (Table 2). Ultimate tensile strength increased 1.2% and elongation decreased 41%. Shore "A" hardness dropped from 70 to 67 as

determined immediately after removal of specimens from the fluid. After drying overnight hardness increased to 70.

c. Butyl Formulation No. 34

Butyl formulation No. 34 swelled 11.4% after 14 days' immersion (Table 2). Ultimate tensile strength decreased 2.6% accompanied by a 6.6% loss in elongation. Shore "A" hardness decreased 5 points (87 to 82) as determined immediately after removal; however, after drying overnight, hardness increased to 84, a 3-point decrease from the original.

3. Hydrogen Peroxide (90%)

Materials selected for immersion tests in 90% hydrogen peroxide were polyethylene formulation No. 39A, silicone rubber PR-11555, and EPR No. 2.

Test results are as follows:

a. Polyethylene Formulation No. 39A

Polyethylene formulation No. 39A exhibited 1.7% volume swell after 14 days' immersion in hydrogen peroxide (Table 2). Ultimate tensile and elongation increases were noted (4.7% and 5% respectively). Shore "A" durometer readings taken immediately after removal were in excess of 100 compared to an original hardness of 98. After allowing to dry overnight, the hardness decreased to 98.

b. Silicone Rubber PR-11555

Silicone rubber PR-11555 swelled 7.3% after 14 days' immersion (Table 2). A decrease in ultimate tensile strength of 8.4% was noted, accompanied by a decrease of 6.5% in elongation. No change in Shore "A" hardness was noted.

c. Ethylene/Propylene Formulation No. 2

EPR No. 2 exhibited no volume change after hydrogen peroxide immersion (Table 2). Ultimate tensile strength decreased 11%; elongation decreased 22%; Shore "A" hardness showed no change prior to drying and increased two points (70 to 72) after allowing to dry overnight.

4. Hybaline A-5

Sealant materials selected for immersion studies were Teflon, polyethylene No. 39A, butyl No. 34, and EPR No. 2.

Test results are as follows:

a. Teflon

Teflon exhibited no volume change upon 14 days' immersion (Table 2). Tensile loss was 16.5% accompanied by 8% decrease in elongation. Shore "A" hardness decreased two points (98 to 96).

b. High Pressure Polyethylene No. 39A

Polyethylene No. 39A exhibited no volume change upon immersion testing (Table 2). Ultimate tensile strength decreased 4%, elongation decreased 16% and no change in Shore "A" hardness was observed.

c. Butyl No. 34

No volume change was observed after 14 days' immersion of butyl No. 34 specimens (Table 2). Tensile strength loss was 54%; elongation decreased 73%; Shore "A" hardness increased two points.

d. EPR No. 2

EPR formulation No. 2 exhibited no volume change after 14 days' immersion (Table 2). Ultimate tensile strength decreased 12.8%; elongation decreased 59.5%; Shore "A" hardness increased nine points (70 to 79).

V. EVALUATION OF O-RING SEALS AT ELEVATED TEMPERATURE

Test cell evaluation of elastomeric O-ring seals was conducted at 160°F in contact with nitrogen tetroxide, mixed hydrazines, and 90% hydrogen peroxide.

A. TEST METHOD

High temperature testing of elastomeric O-ring seals under exposure to fuels and oxidizers was effected through the use of the test cells previously described.

The potential toxicity and/or explosive hazard, associated with vapor generated through failure of test O-rings, necessitated the isolation of filled test cells of individual propellant systems. The possibility of ineffective dissipation of vapor

from the ovens thus led to the design of test cell storage containers (Figure 44). The filled cells were placed in the respective storage container, the container lid secured in place, and the container placed in an oven. Air, from an external source and regulated by a flowmeter, at the rate of 2.5 cubic feet per hour, was introduced into coiled 3/16" aluminum tubing leading into the container. The heated air was directed into the container, consequently, test temperature was maintained inside the container and any vapor present was swept out to a vent stack. A thermocouple well was located at the air outlet point and the outlet air temperature checked periodically. A Teflon gasket was placed at the lid area to insure against vapor escaping into the oven area.

The cells were filled and weighed as previously described.

B. TEST RESULTS

1. Nitrogen Tetroxide

Materials selected for elevated temperature evaluation as O-ring seals for nitrogen tetroxide systems were Teflon, polyethylene formulation No. 39A, and butyl formulation No. 34.

Test results are as follows:

a. Teflon

O-ring seals of Teflon have been under test six months and have allowed 5.4% loss (Figure 45).

b. Polyethylene Formulation No. 39A

O-ring seals of polyethylene formulation No. 39A failed completely after four days' exposure. Essentially 100% loss of oxidizer was observed. The cells were opened and degradation of the O-rings was evident (Figure 46). The appearance of the degraded O-rings was indicative of excessive crosslinking as evidenced by embrittlement.

c. Butyl Formulation No. 34

Seals molded of butyl formulation No. 34 allowed complete loss of oxidizer after four days' exposure. O-ring degradation was noted (Figure 47); the gummy consistency was indicative of chain scission type degradation.

Thus, it appears Teflon is the only seal material tested suitable for long term nitrogen tetroxide exposure at 160°F. Low pressure, high density polyethylene should, however, also be evaluated.

2. Mixed Hydrazines

Polyethylene formulation No. 39A and butyl formulation No. 34 were selected for elevated temperature evaluation as O-ring seals for mixed hydrazines. Test results are as follows:

a. High Pressure Polyethylene Formulation No. 39A

O-ring seals of polyethylene formulation No. 39A have been under hydrazines exposure six months and have allowed 17.9% fuel loss (Figure 48).

b. Butyl Formulation No. 34

O-ring seals of butyl formulation No. 34 have been under test six months and have allowed 7.6% loss (Figure 49).

It appears that butyl is satisfactory for seal application in mixed hydrazine systems at 160°F under long term exposure; polyethylene is suitable, but only for limited service.

3. Hydrogen Peroxide (90%)

Materials selected for O-ring seal evaluation in hydrogen peroxide at elevated temperature were polyethylene formulation No. 39A, silicone rubber PR-11555, and Viton B formulation No. 5187. Test results of the above evaluations are as follows:

a. Polyethylene Formulation No. 39A

Three O-ring seals molded of polyethylene formulation No. 39A were exposed to 90% hydrogen peroxide. One O-ring allowed 100% loss within 12 days; a second, after one month's exposure (Figure 50); the third was under exposure six months and allowed 64% weight loss (Figure 51). No evidence of peroxide leakage past the seal area was noted. To investigate the possibility of peroxide decomposition due to reaction with the cell wall rather than reaction with the O-ring, residual peroxide was removed from the third cell and fresh 90% peroxide added. Little data are available at this time. Low weight loss should be encountered if decomposition is the result of reaction with the cell wall. Passivation of the cells should be in an advanced state resulting in decreased reactivity.

b. Silicone Rubber PR-11555

O-ring seals of silicone rubber formulation PR-11555 have been under test six months and have allowed 76.5% loss (Figure 52). No evidence of peroxide leakage past the seal area was noted therefore fresh 90% peroxide was added to the emptied cells as above. Little additional data are available at this time.

c. Viton B Formulation No. 5187

Viton B O-ring seals of formulation No. 5187 have been exposed to hydrogen peroxide six months and sustained 69.4% loss (Figure 53). Here again, as described above, new 90% solution was added to the emptied cells to determine the cause of peroxide decomposition.

VI. METAL CLAD ELASTOMERIC O-RING SEALS

This section describes the investigation into the effects of nitrogen tetroxide exposure on metal coated elastomeric O-ring seals in closed, static systems under compression.

A. TEST PROCEDURE

Testing of metal clad O-rings under compression in closed static systems in contact with nitrogen tetroxide was effected through the use of stainless steel test cells (Figure 54). The groove dimension was based on 15% compression of the O-ring seal allowing for a nominal 10-mil coating.

The cleaning and passivation of the test cells and the method of determining rate of loss followed procedures described previously. Coated O-rings had been prescreened in MEK by the fabricator as a means for quality control.

B. TEST RESULTS

Metal systems evaluated as coatings were 10-mil lead, copper-lead, silver-lead, crimped aluminum, crimped lead, 10-mil lead on a wire-reinforced Viton B base, 10-mil lead on high modulus Viton B base (XF-30), 10-mil lead on still higher modulus Viton B base (XF-31), and lead-copper coated O-rings with 1/32" wide slit removed from the outside circumference. Test results were as follows:

1. Ten-mil Lead

Upon assembling test cells with 10-mil lead coated O-rings, immediate vapor leakage through the seals was noted. Visual inspection showed the lead had flattened (Figure 55) and essentially a metal-to-metal contact existed. A flange cover was remachined to allow 30% compression, but immediate leakage was again observed. The coating had flattened and showed evidence of cracking at the inner circumference (Figure 55).

Microscopic observation of the coatings showed very rough surfaces. The coating of one O-ring was slit around the inner and outer circumferences and immersed in MEK overnight. Swelling of the elastomer allowed easy removal of the coating from the O-ring but microscope observation of the metal did not reveal any voids or holes.

It was possible a difference in pressure at the sealing surface existed during test, allowing vapor to escape. The top and bottom surfaces of two O-rings were sanded smooth by lightly rubbing the first on Masonite[®], and the second on ordinary tablet paper. Testing showed immediate leakage in the first case but no leakage in the second. The O-rings were under 30% compression. Pursuing this approach further, three O-rings were paper sanded and assembled under 30% compression. However, all three leaked immediately. Visual observation showed all three had flattened.

The coating appeared to be too heavy; since lead is soft enough to flatten under load, resilience imparted by the elastomeric base was not utilized.

2. Copper-Lead

Three O-rings coated with 8-mil copper/2-mil lead were assembled in test cells under 30% compression in contact with nitrogen tetroxide. The cells were placed under a beaker, thus, any vapor leakage could be readily observed. No apparent leakage was observed over a 2-day period. Visual inspection of the O-rings showed cracking of the coating at the inner circumference; one indicated degradation of the elastomer had begun (Figure 56). However, cracking of the coating was believed due to excessive compression, causing the O-ring to be pinched, rather than through oxidizer attack.

One O-ring was evaluated under 15% compression and appeared suitable for effective sealing, therefore, five additional O-ring seals were set up in test cells. Two of the five leaked immediately. However, O-ring distortion led to the belief the O-rings were not seated properly

An additional seven copper-lead coated seals were set up for exposure and four leaked immediately. Consequently, four O-rings were evaluated versus the liquid phase and three versus vapor.

One O-ring had been exposed to the liquid phase 9.5 months and allowed 2.5% oxidizer loss. The remaining three were under liquid exposure 9 months and allowed 11% average loss (Figure 57). However, one of the three had sustained 43.5% loss at this time.

All cells were opened; as observed in Figure 58 all were cracked at the inner circumference and showed evidence of elastomer degradation.

The three O-rings exposed to vapor sustained 2.9% oxidizer loss through seven months (Figure 59). The cells were opened at this time. Two O-rings were cracked at the inner circumference and evidence of copper nitrate deposits was noted (Figure 60).

3. Silver-Lead

Silver-lead coated O-ring seals (8-mil silver/2-mil lead) have allowed 7.5% oxidizer loss through eight months' nitrogen tetroxide liquid exposure (Figure 61). Test O-rings are under 15% compression.

4. Crimped Lead

Crimped lead coated O-rings sustained 10% oxidizer loss through six months' exposure under 15% compression (Figure 62). O-rings were evaluated versus nitrogen tetroxide liquid. Test cells were opened at this time and the O-rings appeared in good condition (Figure 63).

5. Others

Exposure tests on crimped aluminum; 10-mil lead coatings on wire-reinforced Viton B, high modulus Viton B (XF-30) and still higher modulus Viton B (XF-31); and lead-copper coated O-rings with 1/32" wide slit removed from the outer circumference, were initiated. All samples leaked immediately under 15 and 30% compression, therefore, testing was discontinued. All O-rings had flattened and subsequent formation of metal-to-metal seals prohibited effective sealing action (Figures 64 and 65).

C. CONDUCTIVE ELASTOMERS

The heavy coatings (ca. 10 mil) required to ensure reproducible void-free coatings appear to decrease the sealing effectiveness of coated O-rings. Flattening and subsequent formation of metal-to-metal surfaces, in addition to cracking under compression, have prohibited the utilization of most evaluated coatings as seals.

Therefore, if the elastomeric core were of sufficient conductivity to allow uniform metal deposition through plating, a consistent film could be applied and should be essentially void-free. In addition, this void-free film could be of a minimum thickness (2-3 mils), and could be controlled by immersion time and current density.

Accordingly, development of a highly conductive O-ring formulation was undertaken. The addition of conductive carbon black to elastomers is a means for increasing the conductivity of elastomeric formulations. Though an increase in carbon black loading increases the conductivity of the elastomer, the amount of added carbon black is limited by the filler receptivity of the elastomer and the resilience of highly loaded formulations.

1. Conductive Elastomer Formulations

Since high percentages of carbon black would be required to attain sufficient conductivity, ethylene/propylene copolymer was chosen as the base elastomer. Ethylene/propylene copolymer is highly receptive to high black loadings (100-200 phr).

Ethylene/propylene formulations 8, 9, and 10 were compounded containing 60, 100 and 150 phr Vulcan XC-72, a highly conductive carbon black. Shore "A" hardness of 67, 82, and 94 respectively were obtained (Table 3).

Volume resistivity was determined according to ASTM D 991-48T. As observed in Table 3, a volume resistivity of 1.6 ohm-cm was obtained from EPR No. 10. Resistivity of this order was considered suitable for plating studies.

2. Plating Procedure

O-rings of EPR No. 10 were molded and an electroplating solution formulated as follows (Ref. 9):

Copper sulfate	150 grams
Sulfuric acid	25 grams
Distilled water	1000 ml

An O-ring (cathode) was connected to the negative pole of a 12-volt battery charger used as the power supply; copper sheet (anode) was connected to the positive terminal. A rheostat was utilized to control the amperage to maintain a current density of 25-30 amps/square foot or 0.625 amps.

Studies indicated the use of conductive elastomers to be feasible for effective plating as evidenced by Figure 66. However, molding difficulties were encountered and no defect-free O-rings were obtained. The difficulties were attributed to the high viscosity of the unvulcanized, highly loaded elastomer.

3. Conductive Encapsulated Elastomeric O-rings

The molding difficulties encountered led to the development of conductive encapsulated elastomeric O-rings for two reasons: less difficulty would be encountered in molding thin, conductive shells from the highly loaded formulation; and high conductivity was only necessary at the O-ring surface rather than through its cross-section. It was felt that encapsulating a nonconductive elastomer with a thin shell (0.010") of the conductive formulation could impart sufficient conductivity for plating.

Encapsulation was accomplished by molding half shells of EPR No. 10 at temperatures suitable for forming but lower than the crosslinking temperature. Two shells were then assembled with an elastomeric O-ring between them. The shell assembly was placed in an O-ring mold having the same dimensions as the shells. The "welding" of the seams and subsequent crosslinking of the shell was carried out at elevated temperature.

Plating studies carried out as previously described indicated the encapsulation approach feasible. Copper coatings in thicknesses of 1-2 mils were successfully applied and plated O-rings will be evaluated as seals.

VII. POLYETHYLENE ENCAPSULATED ELASTOMERIC O-RING SEALS

Carbon black filled, crosslinked polyethylene has shown apparent resistance to attack from nitrogen tetroxide (Ref. 1). Although polyethylene is compliant rather than elastomeric and its resistance is of a limited nature, it lends itself to the possibility of encapsulating an elastomeric O-ring. This combination imparts elastomeric resilience to a resistant, otherwise nonresilient material.

Encapsulating EPR O-rings with polyethylene shells was accomplished in the same manner described in Section VI-3, "Conductive Encapsulated Elastomeric O-rings." As reported in Reference 1, encapsulated O-rings exposed to nitrogen tetroxide under 10, 20, 25, and 30% compression in test cells allowed rapid loss of oxidizer. In all cases, however, inspection of the O-rings revealed no degradation of the polyethylene, but attack of the elastomer. The inner circumference of the O-ring appeared to be pinched upon securing the test cells, thus cracking the shell.

O-rings of the encapsulated type are larger than the standard rings used throughout this program. Flange cover grooves were remachined to maintain the same O-ring/groove relationship used in the evaluation of elastomeric O-rings. Utilizing the remachined flange covers, encapsulated O-rings under nitrogen tetroxide exposure allowed 20% loss after one week and 100% loss after five weeks' exposure. Visual inspection showed degradation of the shell had occurred and subsequent elastomer failure.

In view of the superior resistance of high crystallinity (low pressure process) polyethylene solid O-rings, the encapsulation approach will be re-evaluated using this type material. If, as indicated by the recent exposure results, uncrosslinked high crystallinity polyethylene is inherently resistant, the molding of encapsulating shells would be a very simple and economical operation.

VIII. SUMMARY AND CONCLUSIONS

Teflon appears to be a prime candidate as O-ring seal material suitable for use in all the test media. Very low loss of propellant has been observed through Teflon seals in long-term testing at $73 \pm 3^\circ\text{F}$.

Crosslinked polyethylene (high pressure polymerized) has shown compatibility with all test media, though of limited effectiveness in nitrogen tetroxide.

Polyethylene of increased crystallinity (low pressure) appears to be superior in oxidation resistance to high pressure polyethylene and to approach Teflon in overall utility.

Butyl and ethylene/propylene rubber continue to show excellent seal effectiveness for the mixed hydrazines system and are recommended for service.

Immersion tests and short-term test cell exposure at $73 \pm 3^{\circ}\text{F}$ indicate Teflon, polyethylene, and ethylene/propylene rubber suitable as seal materials for Hybaline A-5.

Short-term test cell exposure to pentaborane suggests Viton A formulation No. 3 may be recommended for utility as a seal material due to low weight loss observed.

Simulated O-ring tests at 160°F indicate Teflon the only seal material resistant to attack from nitrogen tetroxide with low weight loss observed. Low pressure, high density polyethylene has not been tested but is expected to approach Teflon in resistivity. Polyethylene and butyl have proven effective seal materials for mixed hydrazines at the elevated temperature. Viton B and silicone rubber appear the most effective seal materials at 160°F for hydrogen peroxide systems.

Test cell exposure evaluation indicates silver-lead coatings to hold the most promise as seal materials for nitrogen tetroxide allowing very low loss of oxidizer. Inconsistencies in sealing effectiveness have hindered the application of copper-lead as elastomeric seal coatings.

The concept of encapsulation of elastomeric O-rings with polymeric shells has proven successful. Resistance to nitrogen tetroxide attack was increased through the use of resistant polyethylene shells. Successful copper plating of conductive elastomeric shells was accomplished. Copper plated O-rings will be evaluated as seals.

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A P P E N D I X

- Appendix 1. Formulations of Compounds Tested
- Appendix 2. Glossary
- Appendix 3. Tables 1 - 3
- Appendix 4. Figures 1 - 65

APPENDIX 1. Formulations of Compounds Tested

Formulation No. 2

EPR 404	100 parts	Cure 40 min. at 330°F
Statex R	50	
Zinc Oxide	1	
Sulfur	0.45	
Varox	8	

Formulation No. 3

Viton A	100 parts	Cure 30 min. at 300°F
Lead Monoxide	15	Step bake 24 hr. at 400°F
Sterling MT	2.5	
Diak No. 1	1	

Formulation No. 8

EPR 404	100 parts	Cure 40 min. at 330°F
Vulcan XC-72	60	
Zinc Oxide	1	
Sulfur	0.90	
Varox	8	

Formulation No. 9

EPR 404	100 parts	Cure 40 min. at 330°F
Vulcan XC-72	100	
Zinc Oxide	1	
Sulfur	0.90	
Varox	8	

Formulation No. 10

EPR 404	100 parts	Cure 40 min. at 330°F
Vulcan XC-72	150	
Zinc Oxide	1	
Sulfur	0.90	
Varox	8	

Formulation No. 34

Butyl 218	100 parts	Cure 45 min. at 350°F
Polyethylene	25	
Statex R	75	
Zinc Oxide	5	
Amberol ST-137	12	
Diocetyl Sebacate	10	
Hypalon 20	5	

Formulation No. 39

Polyethylene		
Grade 600-2415	100 parts	Cure 40 min. at 320°F
Varox	10	

Formulation No. 39A

Polyethylene		
Grade 600-2415	100 parts	Cure 40 min. at 320°F
Statex R	50	
Varox	10	

Formulation No. 39B

Polyethylene		
Grade 600-2415	100 parts	Cure 40 min. at 320°F
Statex R	65	
Varox	10	

Formulation No. 39C

Polyethylene		
Marlex 6015	100 parts	Cure 40 min. at 320°F
Statex R	50	
Varox	10	

Uncrosslinked Polyethylene

Marlex 6015	100 parts
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Formulation No. 76

Viton B	100 parts	Cure 30 min. at 300°F
Thermax MT	25	Step bake 24 hr. at 400°F
Lead Oxide	15	
Diak No. 1	1	

Formulation No. 5187

Viton B	100 parts	Cure 30 min. at 300°F
Kel-F 827	25	Step bake 24 hr. at 400°F
Dyphos	10	
Diak No. 1	2	
Maglite D	10	

Formulation PR-11555 (Precision Rubber Products
Proprietary Silicone Rubber Compound)

G. E. Silicone SE-555

DX-954-GP

DX-954-GP	100 parts	Cure 30 min. at 320°F
Sterling S	65	
BLE	0.10	
DiCup 40C	6	

Teflon

Teflon	100 parts
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Filled Teflon

Teflon	100 parts
Calcium Fluoride	25

APPENDIX 2. Glossary

<u>Name</u>	<u>Chemical Name</u>	<u>Supplier</u>
Amberol ST-137	Phenol-Formaldehyde Resin	Rohm and Haas Co.
BLE	Diphenylamine-Acetone reaction product	Naugatuck Chemical Division
Butyl 218	Isobutylene-Isoprene Co-polymer containing 1.5-2.0 mole percent unsaturation	Enjay Chemical Co.
Diak No. 1	Hexamethylenediamine Carbamate	DuPont
DiCup 40C	Dicumyl Peroxide 40% Calcium Carbonate 60%	Hercules Powder Co.
DX-954-GP	Saturated, low polarity vinyl copolymer	Monsanto
Dyphos	Dibasic Lead Phosphite	National Lead Co.
EPR 404	Ethylene-Propylene Rubber	Enjay Chemical Co.
Hypalon 20	Chlorosulfonated Polyethylene	DuPont
Kel-F 827	Copolymer of Chlorotri-fluoroethylene and Vinylidene Fluoride	Minnesota Mining and Manufacturing Co.
Maglite D	Magnesium Oxide	C. P. Hall Co.
Marlex 6015	Low Pressure Polyethylene	Phillips Chemical Co.
Polyethylene Grade 600-2415	High Pressure Polyethylene Grade 600-2415 Low Density (0.92)	Monsanto
Silicone SE-555	Methylphenyl vinyl-type Silicone Rubber	General Electric Co.
Statex R	High Abrasion Furnace Carbon Black	Columbian Carbon Co.

<u>Name</u>	<u>Chemical Name</u>	<u>Supplier</u>
Sterling S	Semi-Reinforcing Furnace Carbon Black	Godfrey L. Cabot, Inc.
Teflon	Polytetrafluoroethylene	DuPont
Thermax MT	Medium Thermal Carbon Black	R. T. Vanderbilt and Co.
Varox	2,5-Bis(tert-butylperoxy)- 2,5-dimethyl hexane (50%)- Inert Carrier (50%)	R. T. Vanderbilt and Co.
Viton A	Vinylidene fluoride and Hexafluoropropylene Copolymer	DuPont
Viton E	Fluorinated Elastomer	DuPont
Vulcan XC-72	CF Carbon Black	Godfrey L. Cabot, Inc.

APPENDIX 3. Tables 1 - 3

Table 1

O-RING TEST DATA

Elastomer	Tensile, psi		Elongation, %		Durometer, Original		Durometer, Test		Volume Change, %		Weight Change, %		Compression Set, %	
	Original	Test	Original	Test	Original	Test	Original	Test	Original	Test	Original	Test	Original	Test
<u>Hydrazines</u>														
DX-954-GP	2115	1809	430	202	96	75			+2.6		+2.9		28	
<u>Hydrogen Peroxide</u>														
Silicone PR-11555	682	466	484	207	58	43			-2.36		-2.1		22	
Viton 5187	2243	2261	407	415	77	72			+0.22		+0.19		12	
Butyl 34	1913	1806	319	335	79	82			-0.34		-0.36		4.5	

Table 2

IMMERSION TEST DATA

<u>Elastomer</u>	<u>Tensile, psi Original</u>	<u>Tensile, psi Test</u>	<u>Elongation, % Original</u>	<u>Elongation, % Test</u>	<u>Durometer, Original</u>	<u>Durometer, Test</u>	<u>Volume Change, %</u>
<u>Nitrogen Tetroxide</u>							
Teflon	3993	3243	435	388	98	100+	0
P.E. 39A	2477	- *	118	- *	98	98	0
Butyl No. 34	1882	550	546	693	87	84	+33.1
<u>Mixed Hydrazines</u>							
P. E. 39A	2477	2514	118	120	98	98/99**	0
EPR No. 2	2710	2369	965	774	70	67/71**	0
Butyl No. 34	1882	1829	546	510	87	82/84**	+11.4
<u>Hydrogen Peroxide</u>							
P. E. 39A	2477	2593	118	124	98	100+/98**	+1.7
Silicone PR-11555	1295	1187	1225	1147	50	50/50**	+7.3
EPR No. 2	2609	2324	633	495	70	70/72**	0
<u>Hybaline A-5</u>							
Teflon	3993	3335	435	400	98	96**	0
P.E. 39A	2477	2380	118	99	98	98**	0
Butyl No. 34	1882	809	546	146	87	89**	0
EPR No. 2	2710	2363	965	396	70	79**	0

* Samples broke prior to test

** Determined after drying

Table 3

PHYSICAL AND RESISTIVITY PROPERTIES OF EPR FORMULATIONS

<u>EPR No.</u>	<u>Tensile, psi</u>	<u>Elongation, %</u>	<u>Shore "A"</u>	<u>Surface Resistivity, ohms</u>	<u>Volume Resistivity, Ohm-cm</u>
8	2025	590	67	6500	159
9	2100	430	82	100	10
10	1600	300	94	17	1.6

APPENDIX 4. Figures 1 - 66

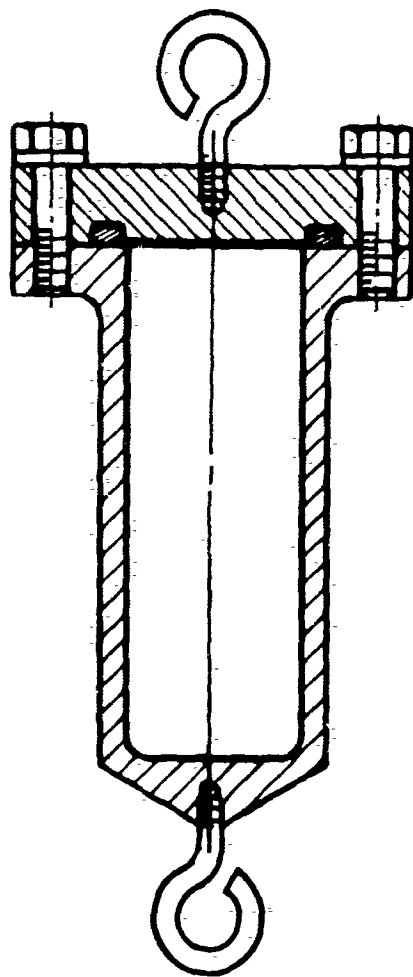


Figure 1. Stainless Steel Test Cell

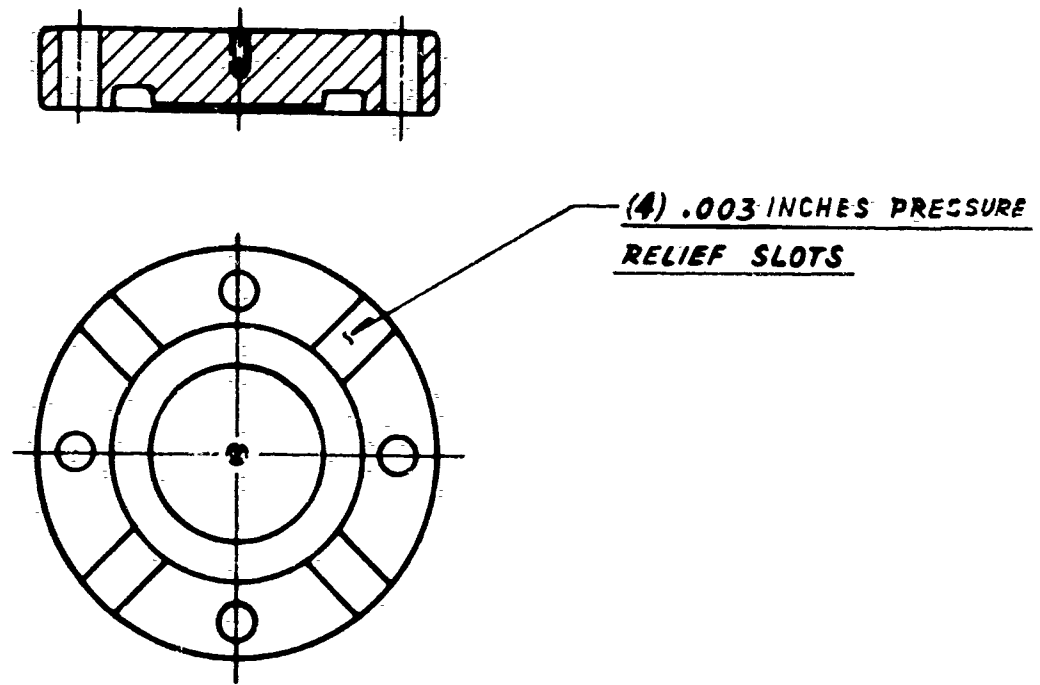


Figure 2. O-Ring Test Cell Cover

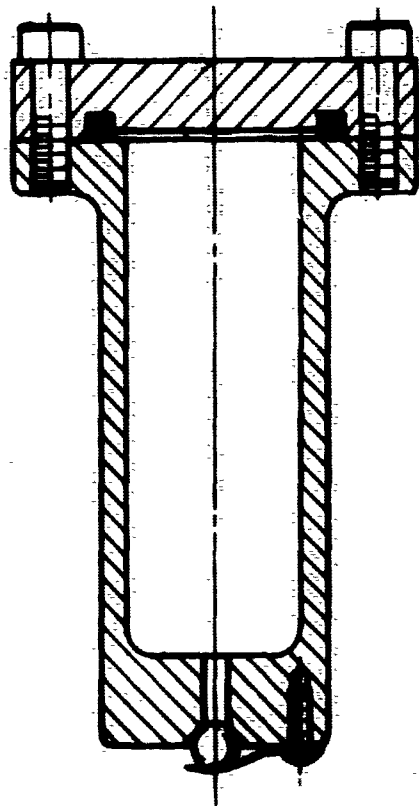


Figure 3. Test Cell with Relief Port

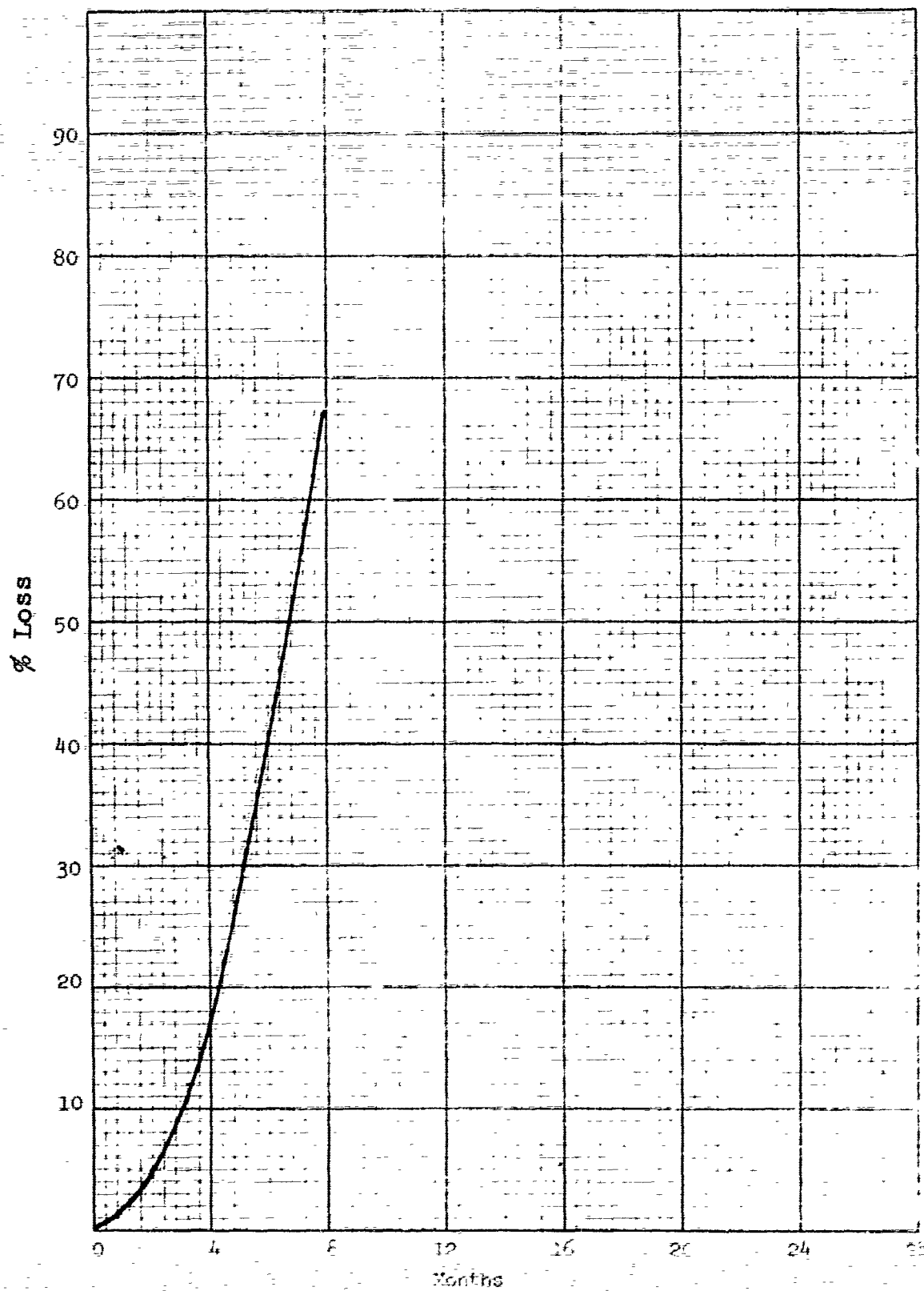


Figure 4. Polyethylene No. 39B: Exposure to Nitrogen Tetroxide

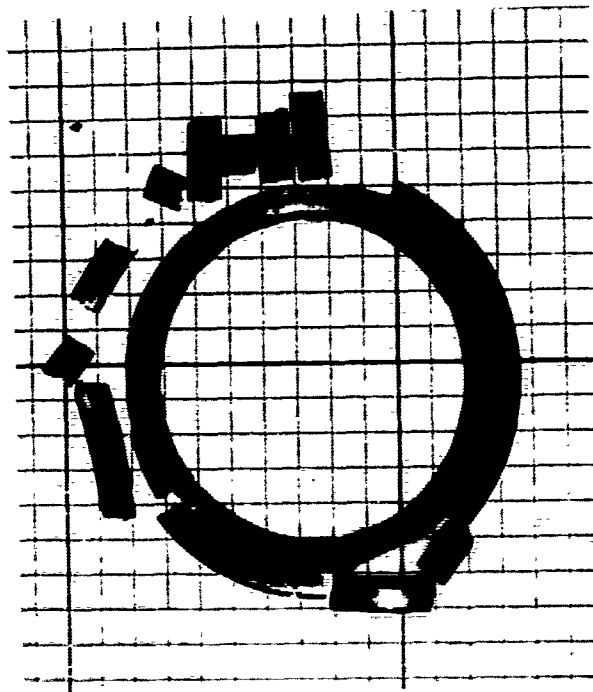
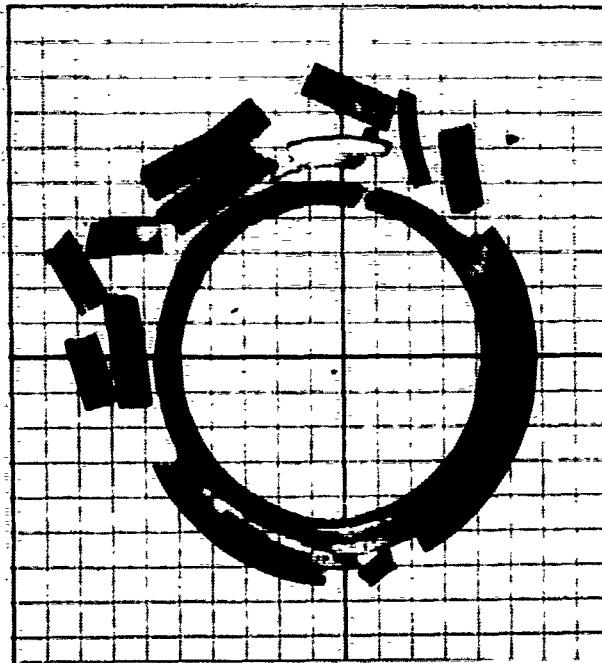


Figure 5. Polyethylene No. 39B: Nitrogen Tetroxide System

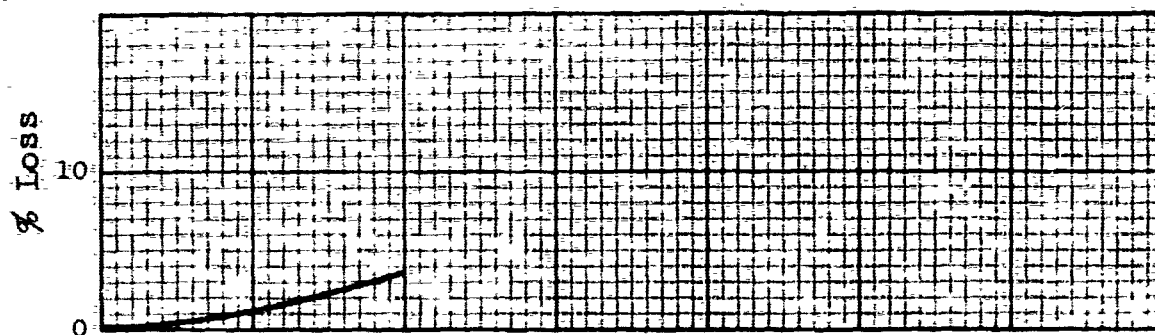


Figure 6. Polyethylene No. 39C:
Exposure to Nitrogen Tetroxide

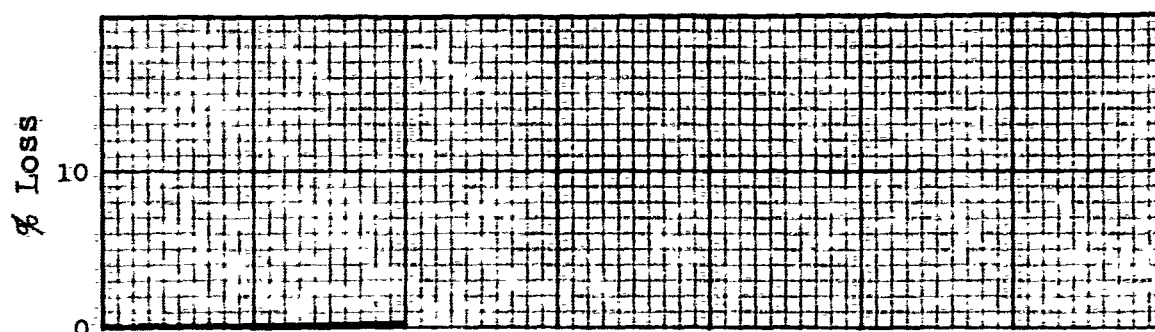


Figure 7. Uncrosslinked Low Pressure Poly-
ethylene: Exposure to Nitrogen Tetroxide

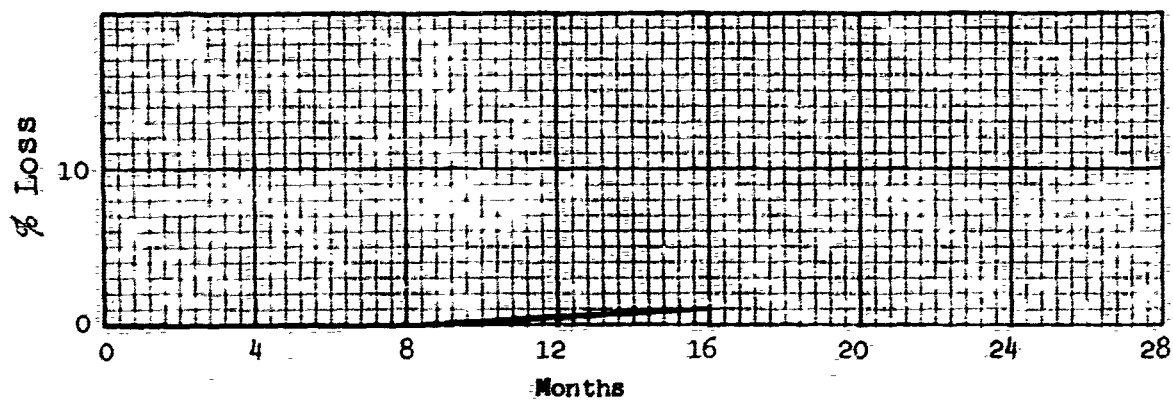


Figure 8. Teflon: Exposure to
Nitrogen Tetroxide

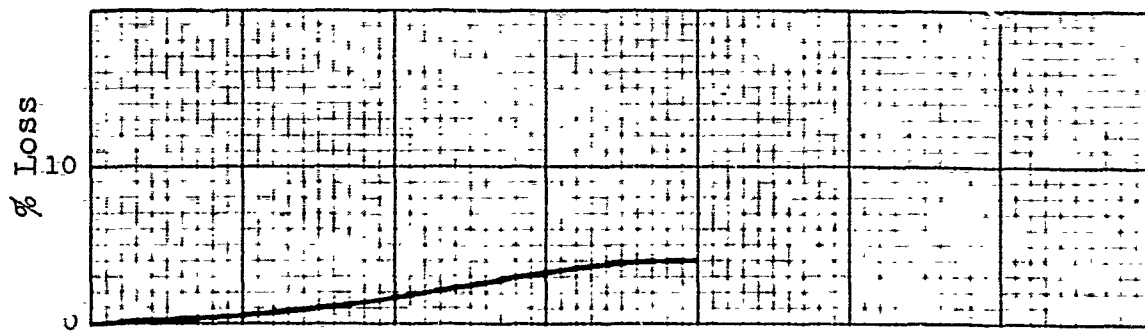


Figure 9. Filled Teflon: Exposure to Nitrogen Tetroxide

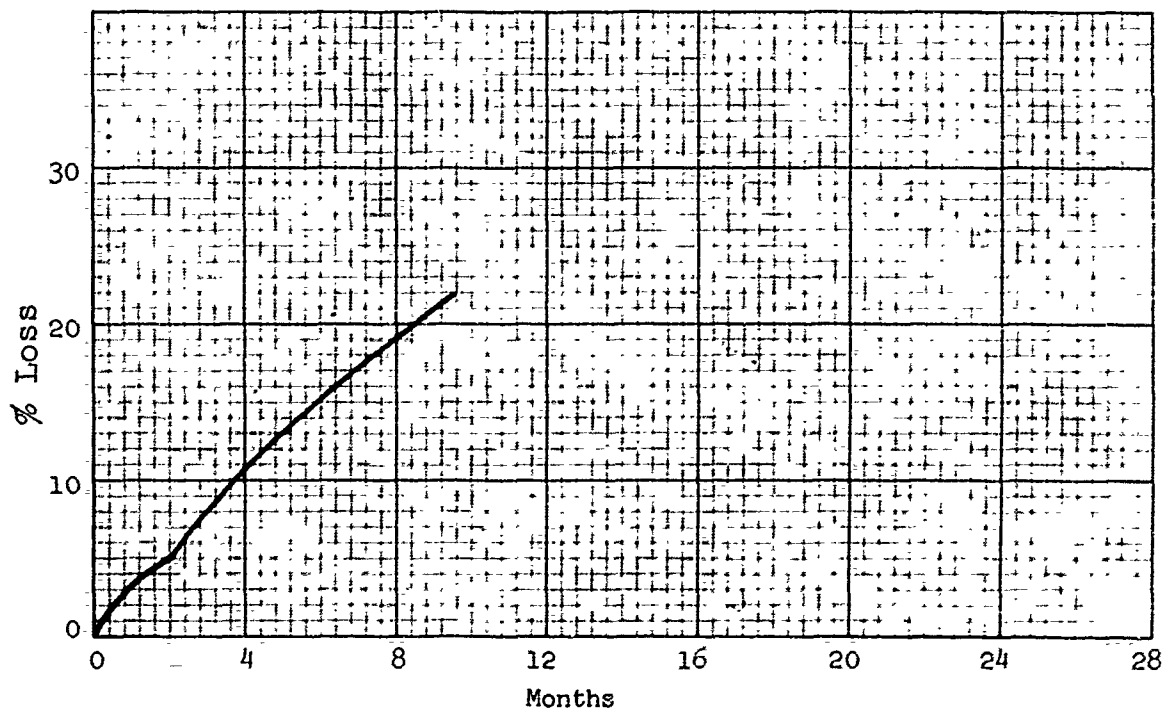
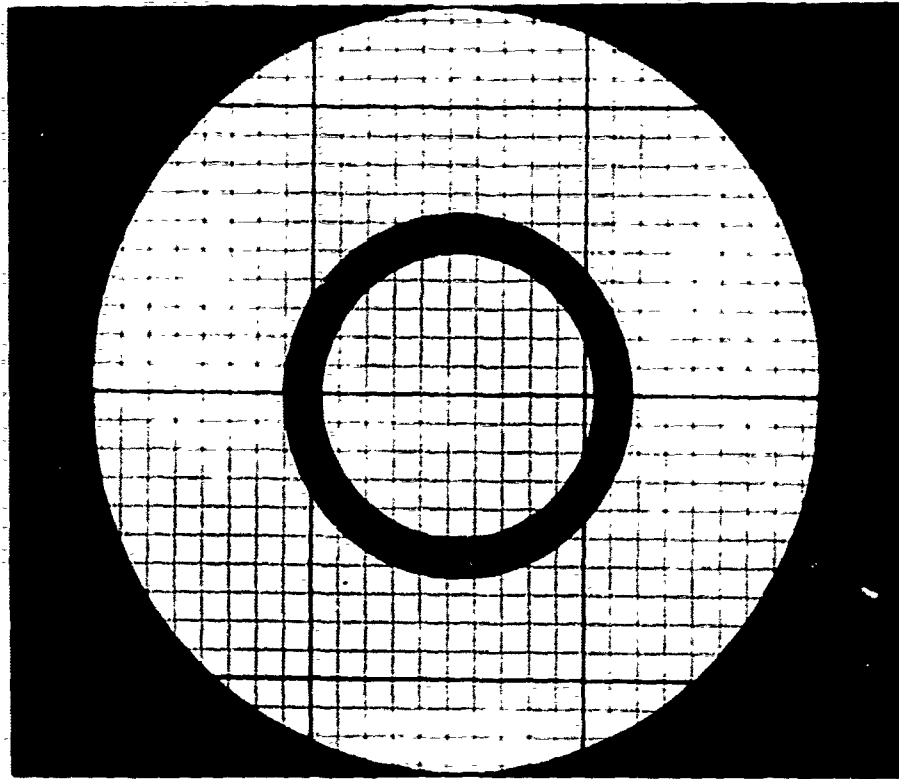
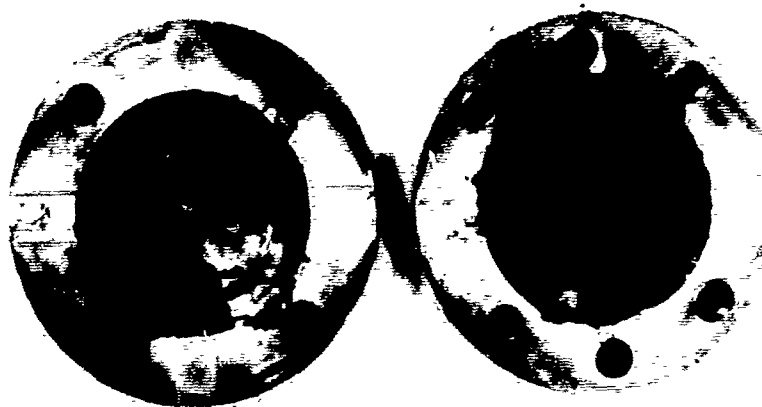


Figure 10. Viton B Formulation No. 76: Exposure to Nitrogen Tetroxide



Before



After

Figure 11. Viton B Formulation No. 76:
Nitrogen Tetroxide System

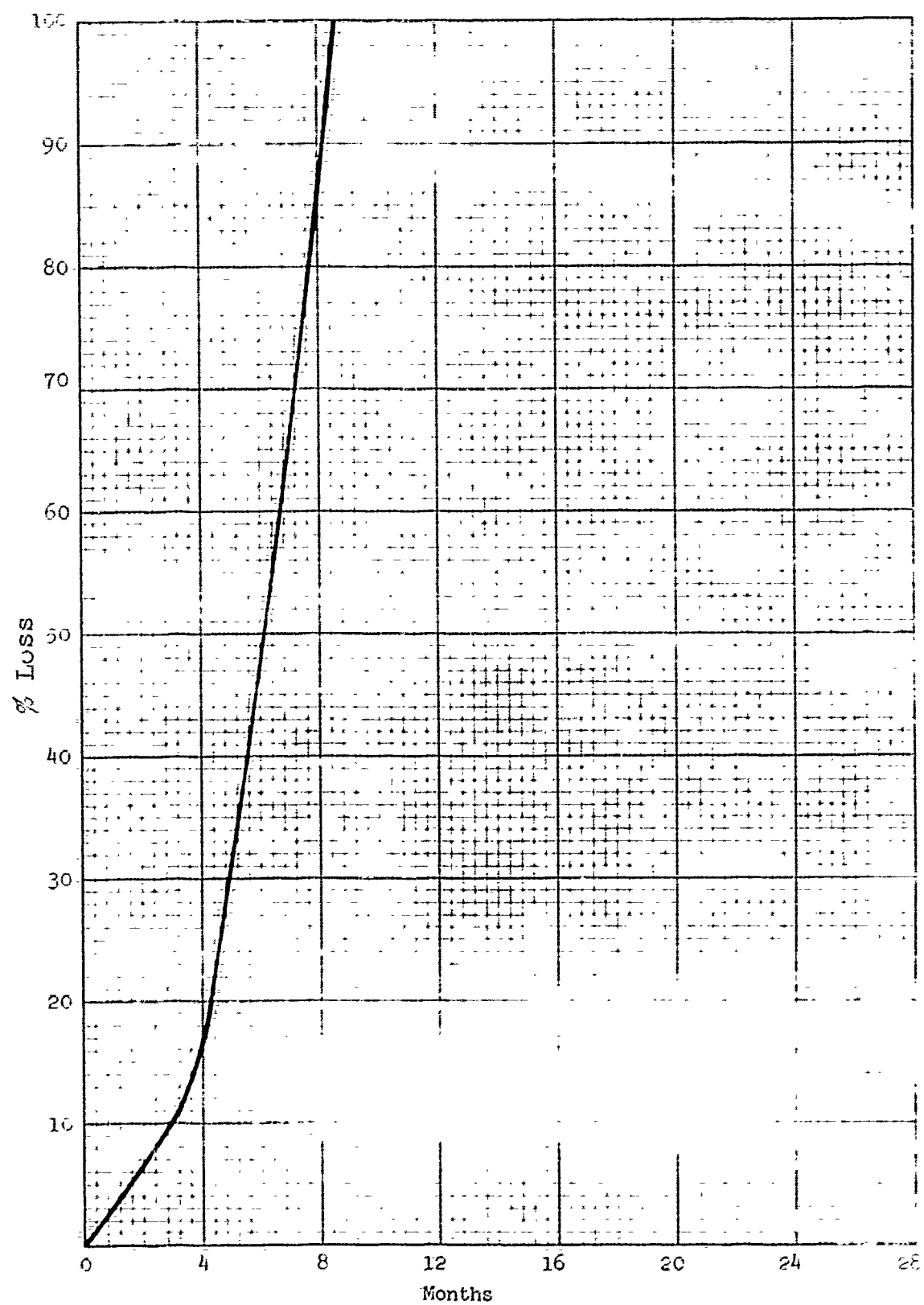
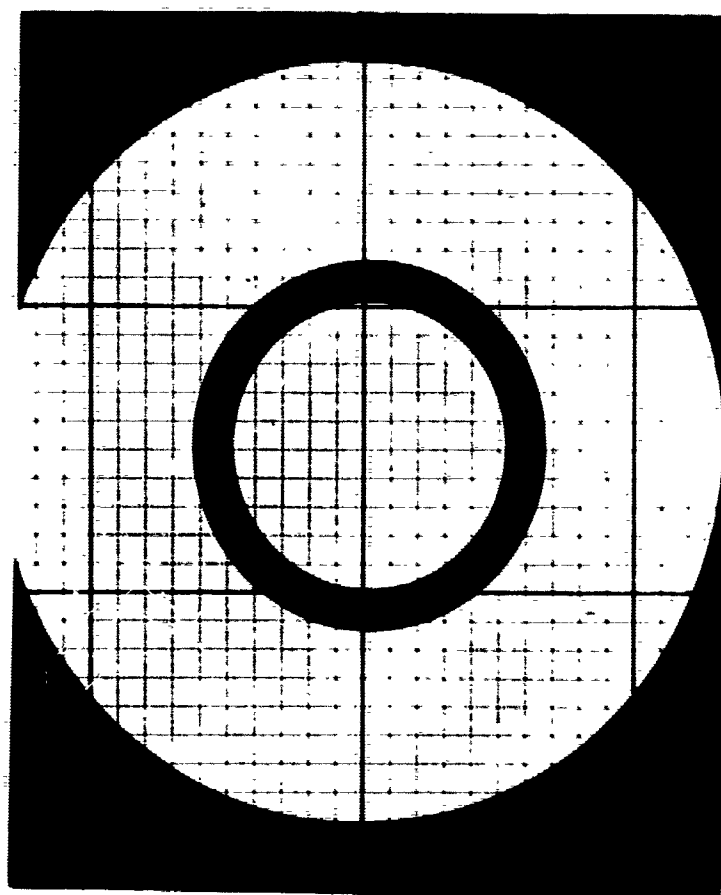


Figure 12. Butyl Formulation No. 34:
Exposure to Nitrogen Tetroxide



Before



After

Figure 13. Butyl Formulation No. 34:
Nitrogen Tetroxide System

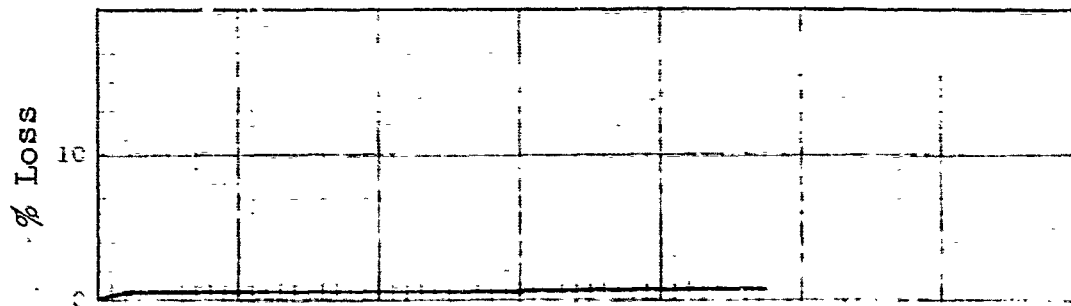


Figure 14. Teflon: Exposure to Mixed Hydrazines

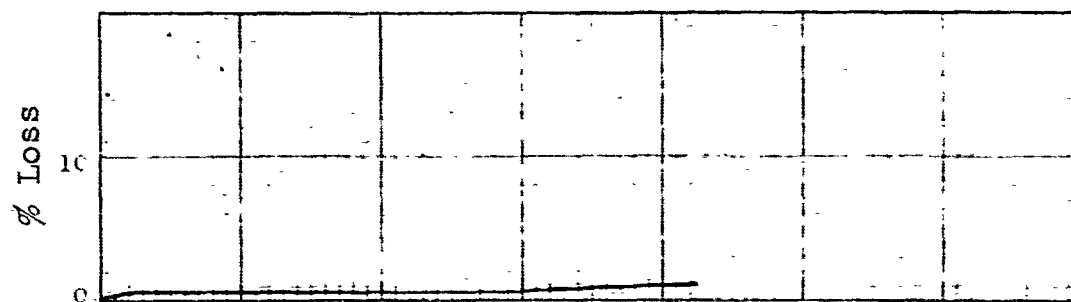


Figure 15. Butyl Formulation No. 34: Exposure to Mixed Hydrazines (Liquid)

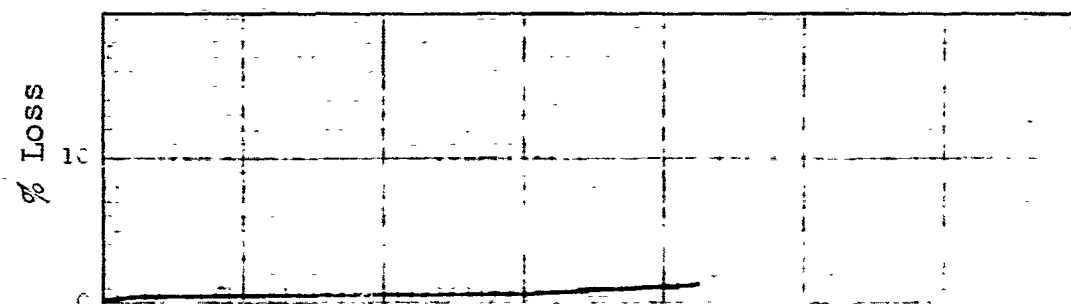


Figure 16. Butyl Formulation No. 34: Exposure to Mixed Hydrazines (Vapor)

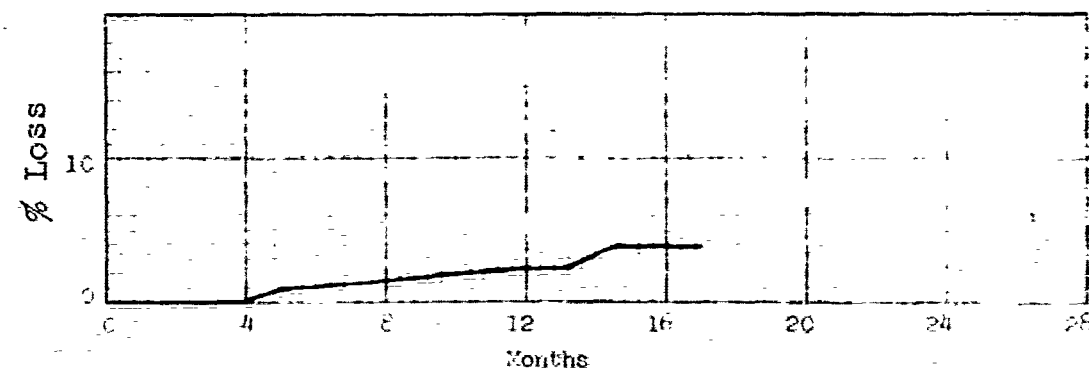


Figure 17. EPR No. 2: Exposure to Mixed Hydrazines

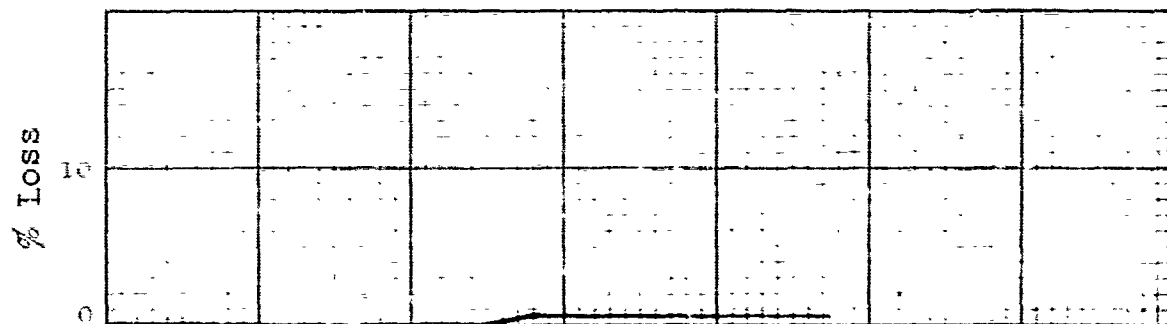


Figure 18. Polyethylene Formulation No. 39:
Exposure to Mixed Hydrazines

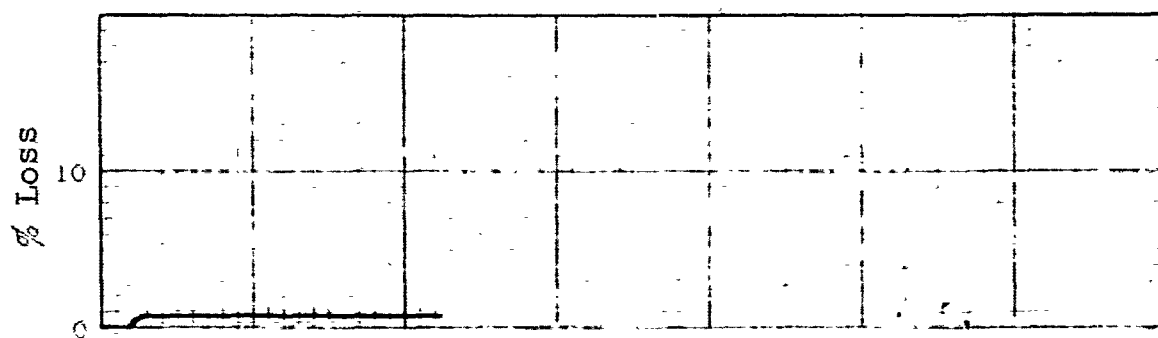


Figure 19. Polyethylene Formulation No. 39B:
Exposure to Mixed Hydrazines

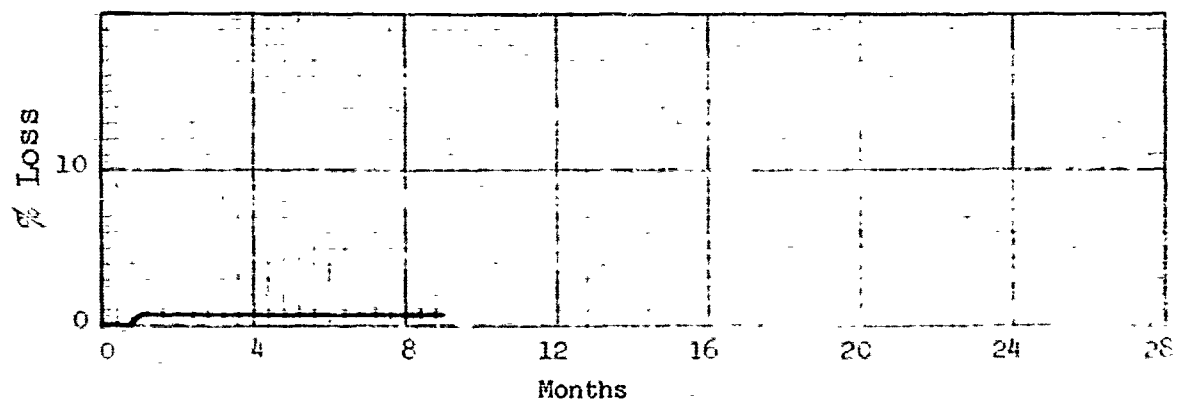


Figure 20. Polyethylene Formulation No. 39C:
Exposure to Mixed Hydrazines

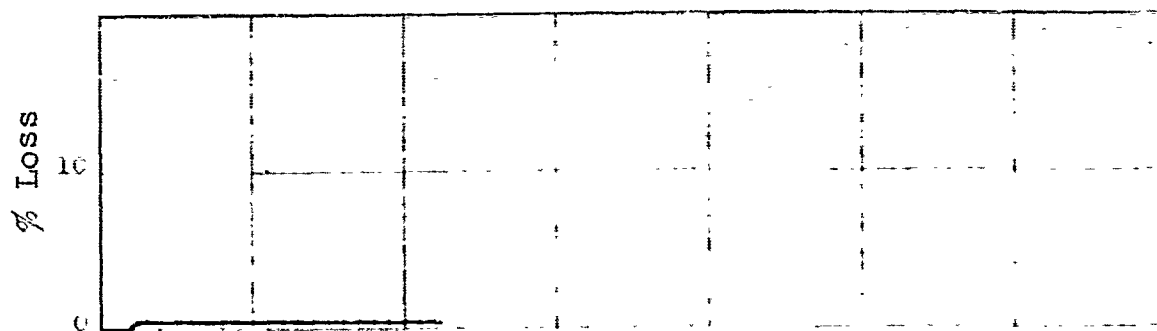


Figure 21. Uncrosslinked Low Pressure Polyethylene.
Exposure to Mixed Hydrazines

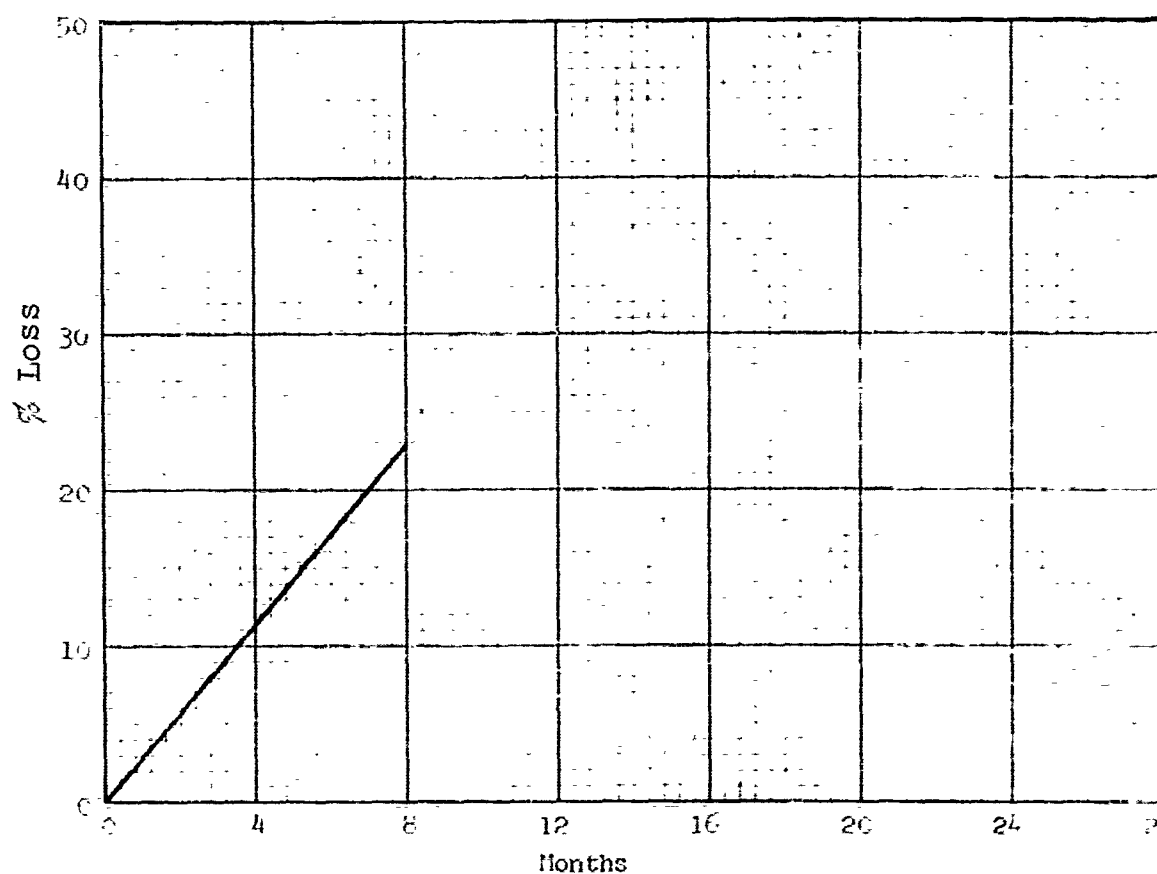
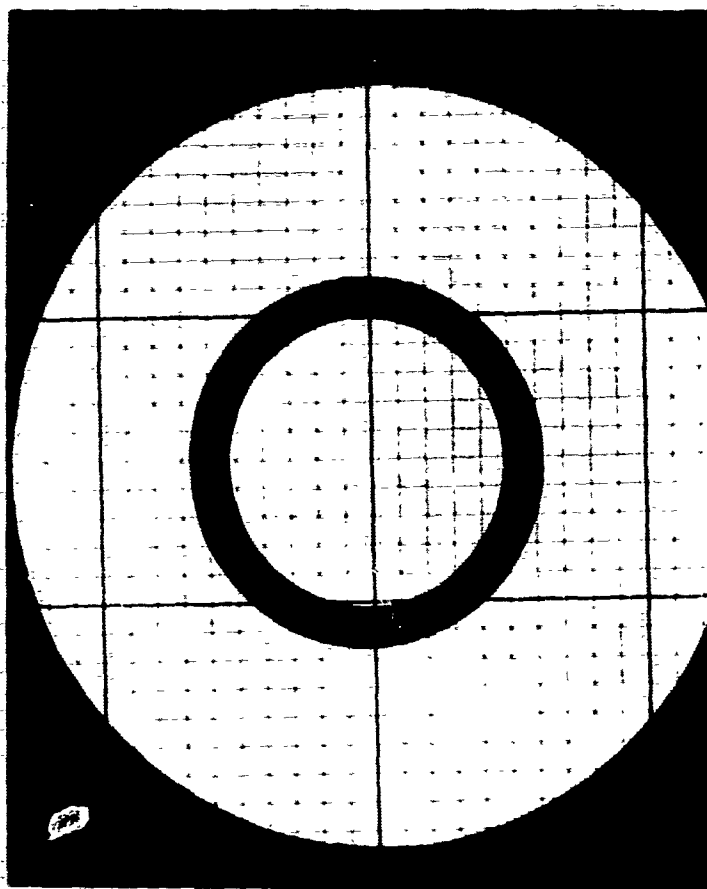
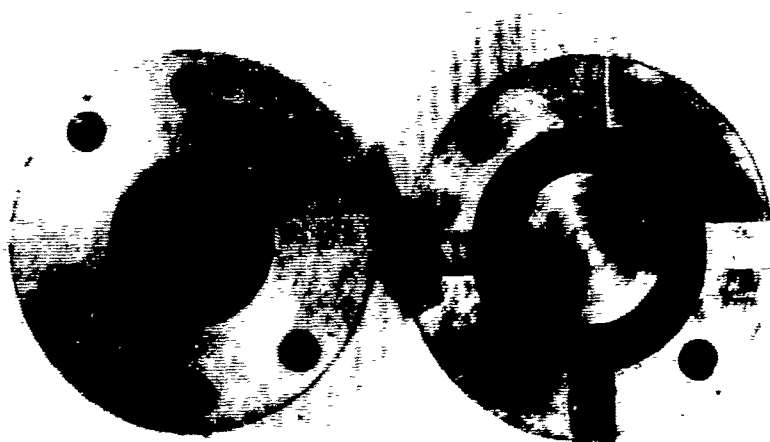


Figure 22. DX-954-GP: Exposure
to Mixed Hydrazines



Before



After

Figure 23. DX-954-GP: Mixed Hydrazines System

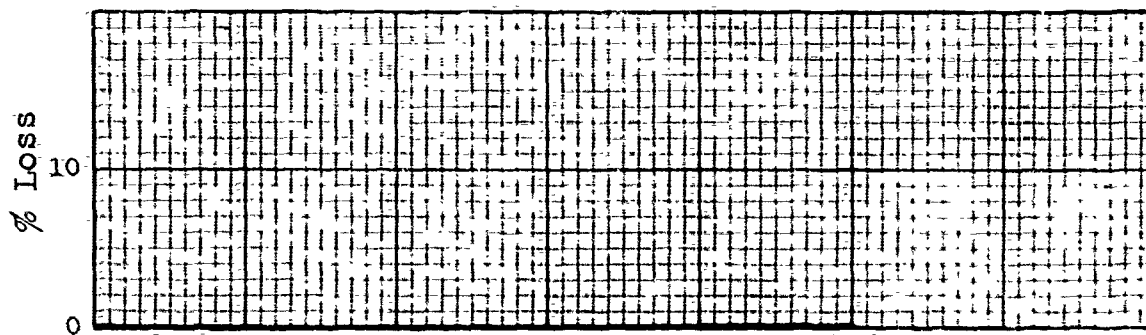


Figure 24: Teflon: Exposure to Chlorine Trifluoride

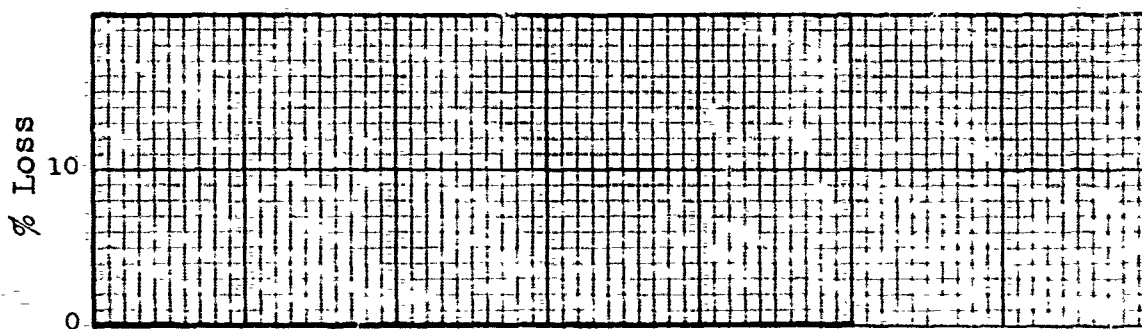


Figure 25. Filled Teflon: Exposure to Chlorine Trifluoride (Liquid)

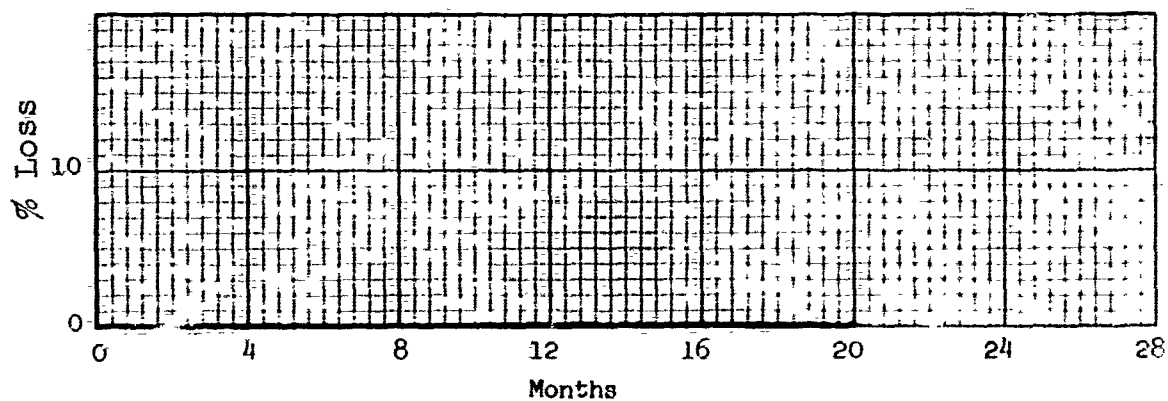


Figure 26. Filled Teflon: Exposure to Chlorine Trifluoride (Vapor)

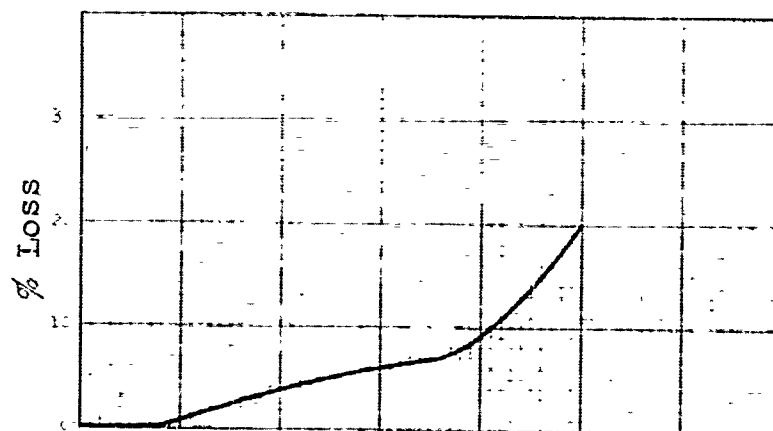


Figure 27. Polyethylene Formulation No. 39A: Exposure to Chlorine Trifluoride (Liquid)

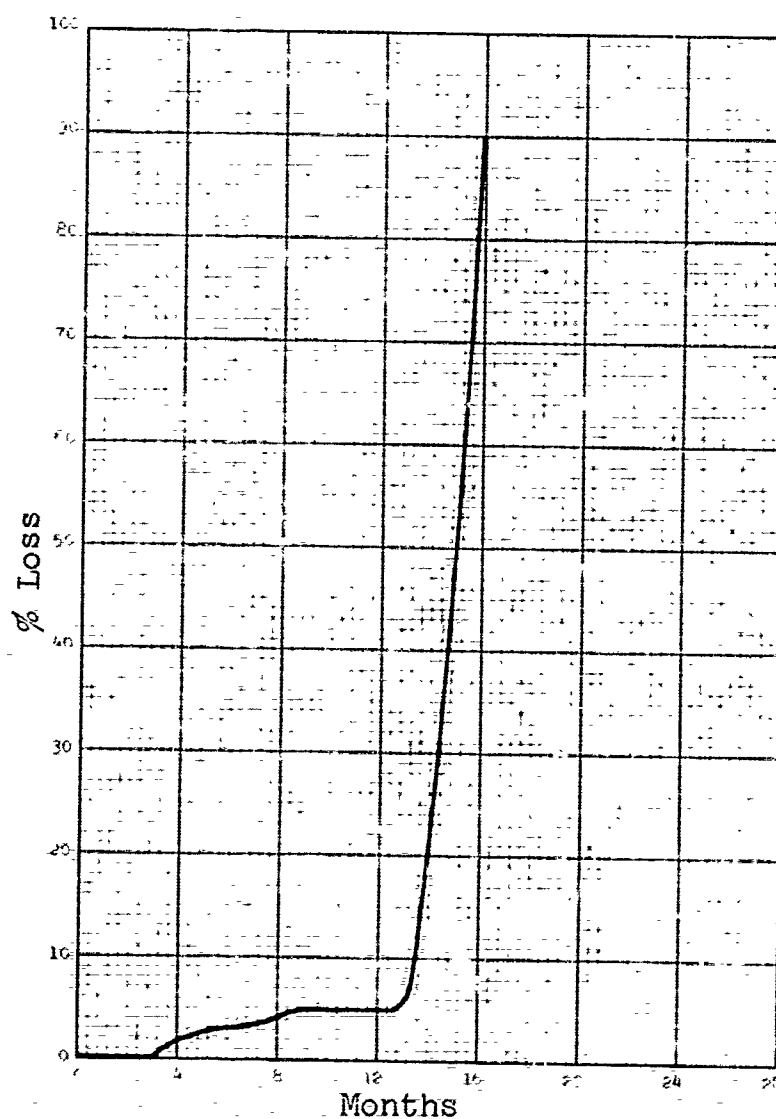
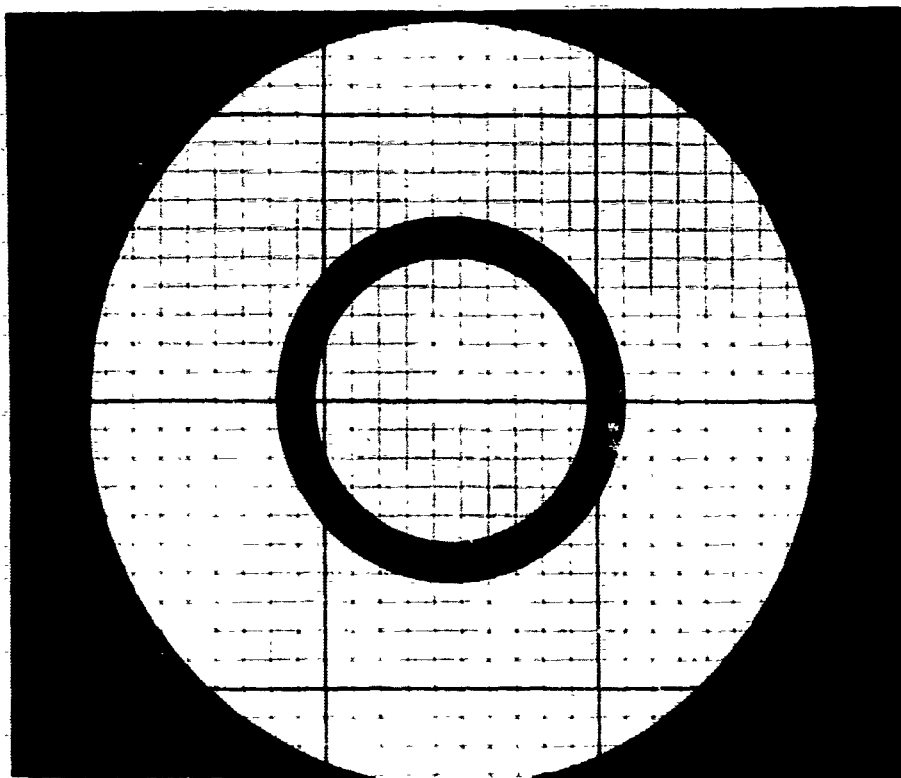


Figure 28. Polyethylene Formulation No. 39A: Exposure to Chlorine Trifluoride (Vapor)



Before



After Liquid Phase



After Vapor Phase

Figure 29. Polyethylene Formulation No. 39A:
Chlorine Trifluoride System

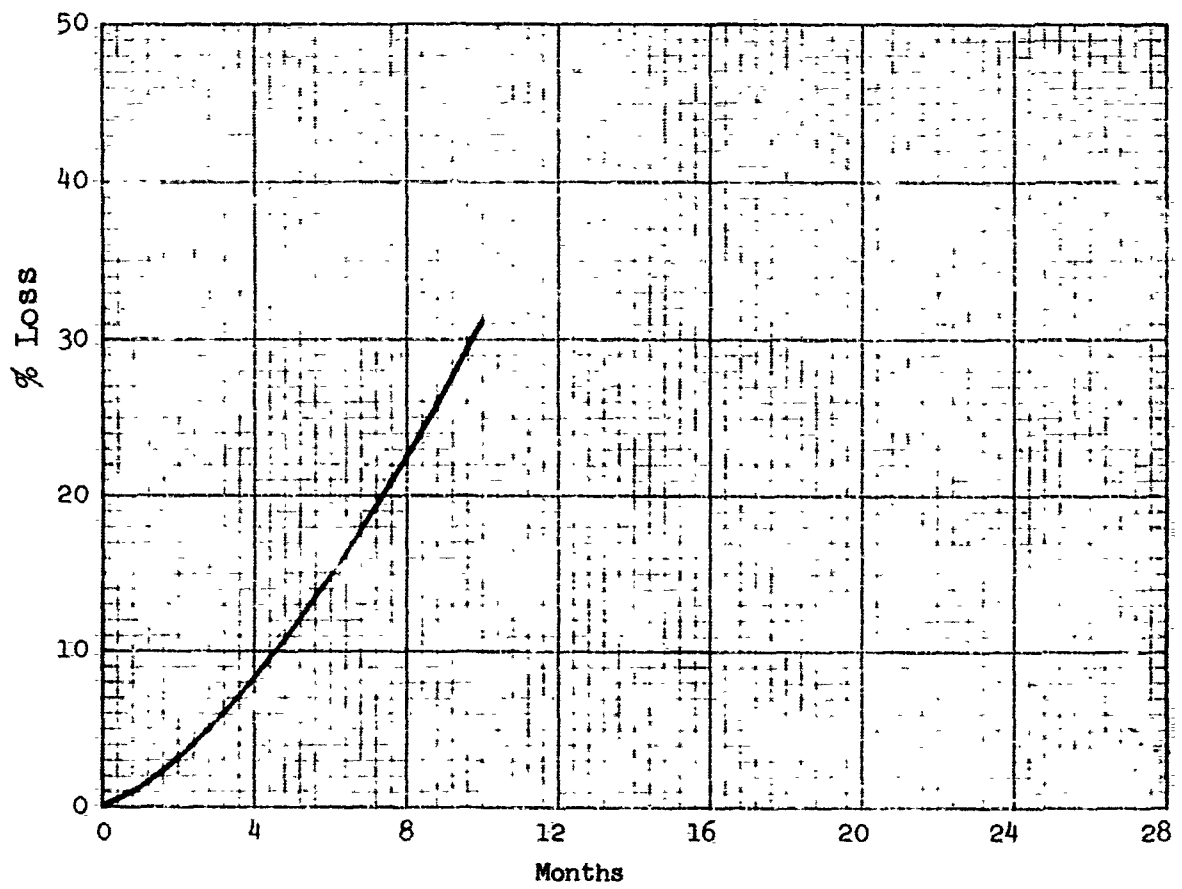
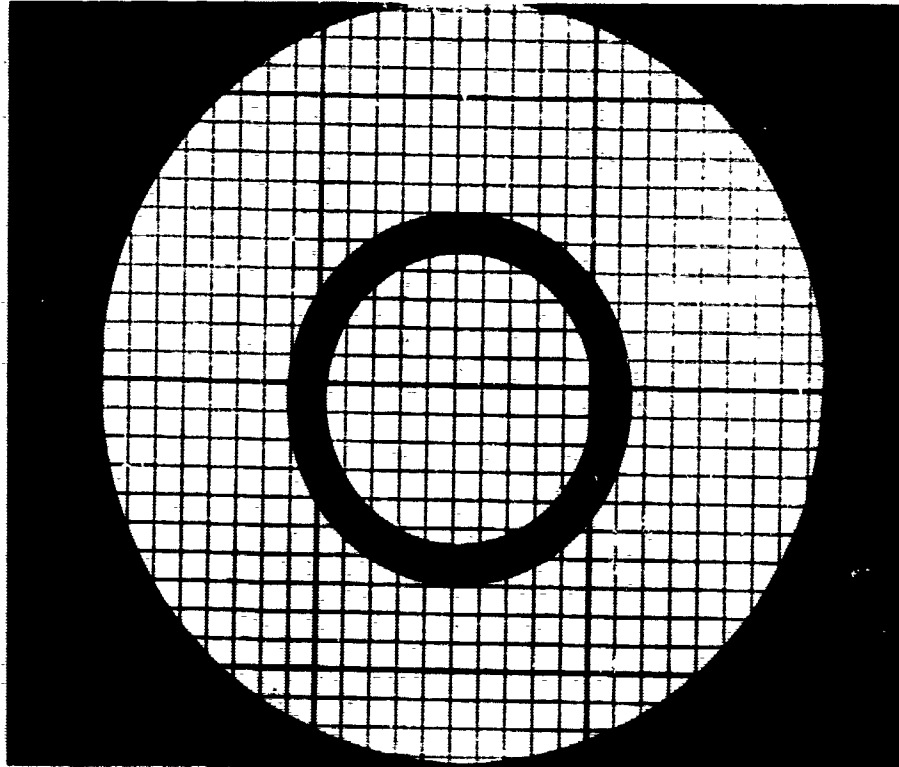
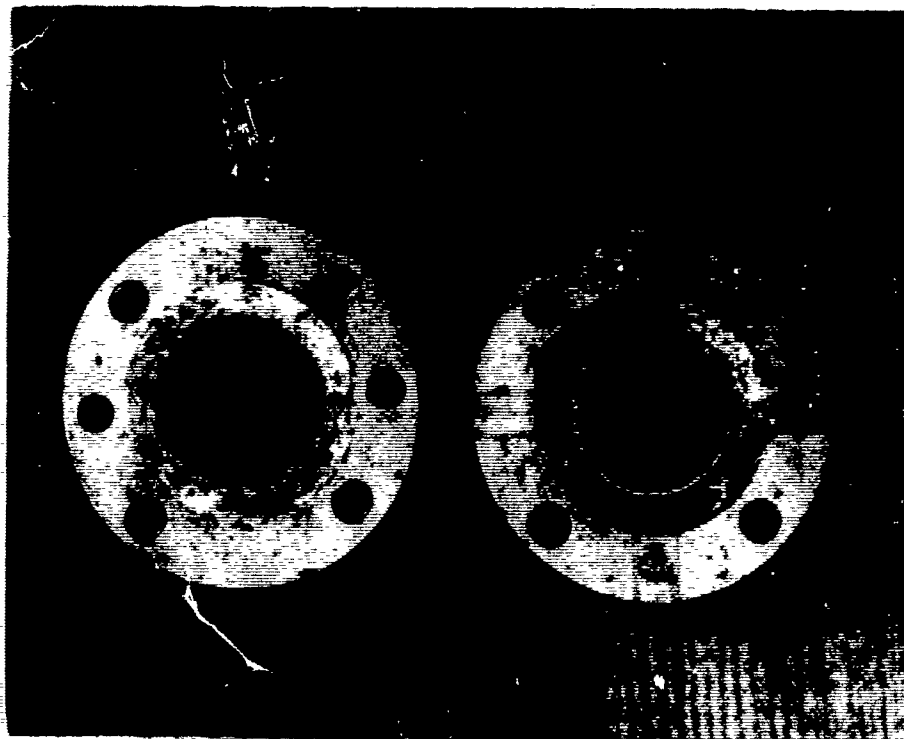


Figure 30. Silicone No. PR-11555:
Exposure to Hydrogen Peroxide



Before



After

Figure 31. Silicone No. PR-11555:
Hydrogen Peroxide System

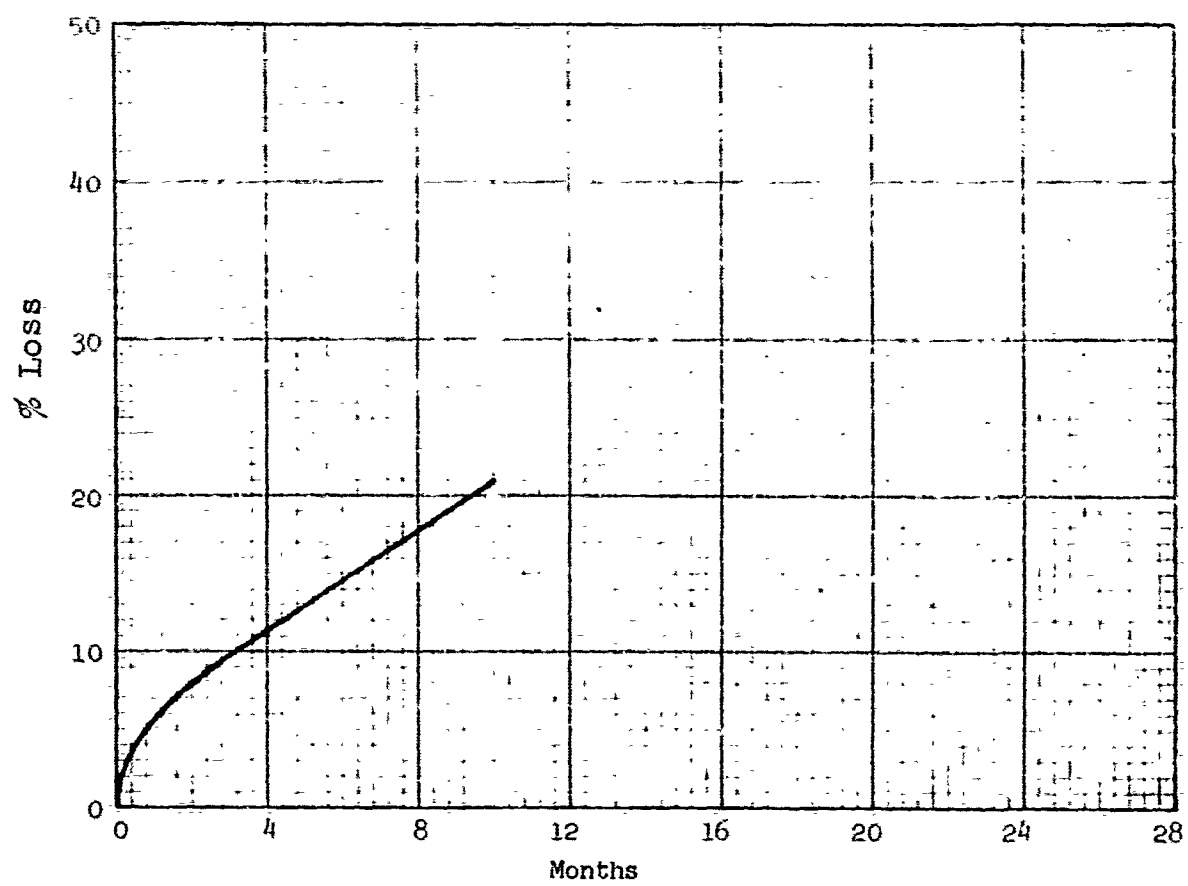
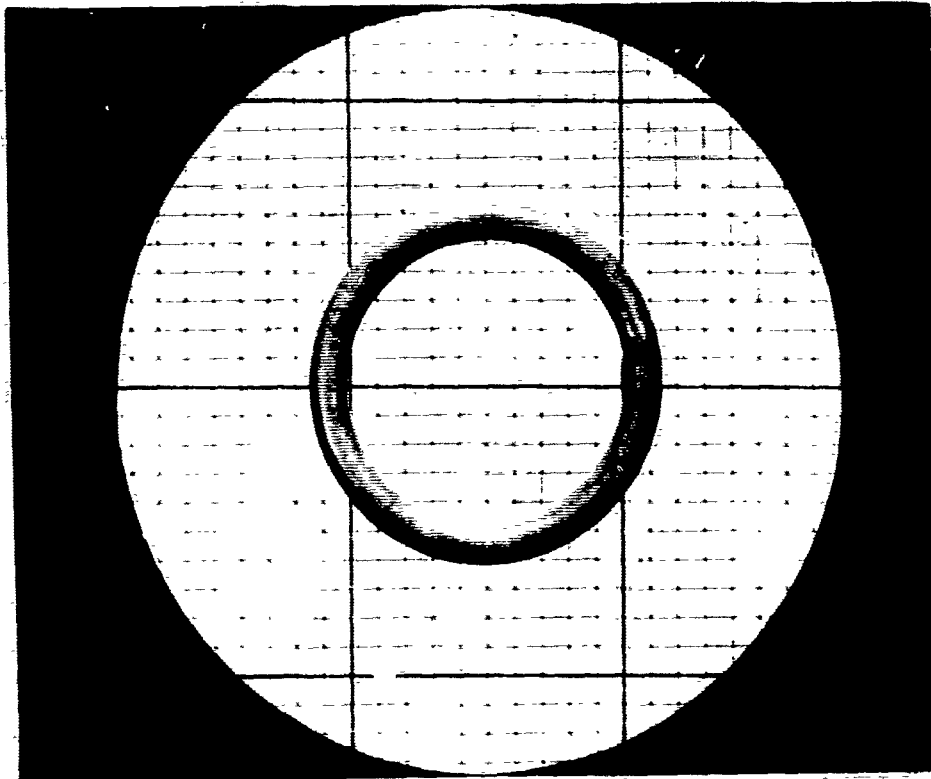
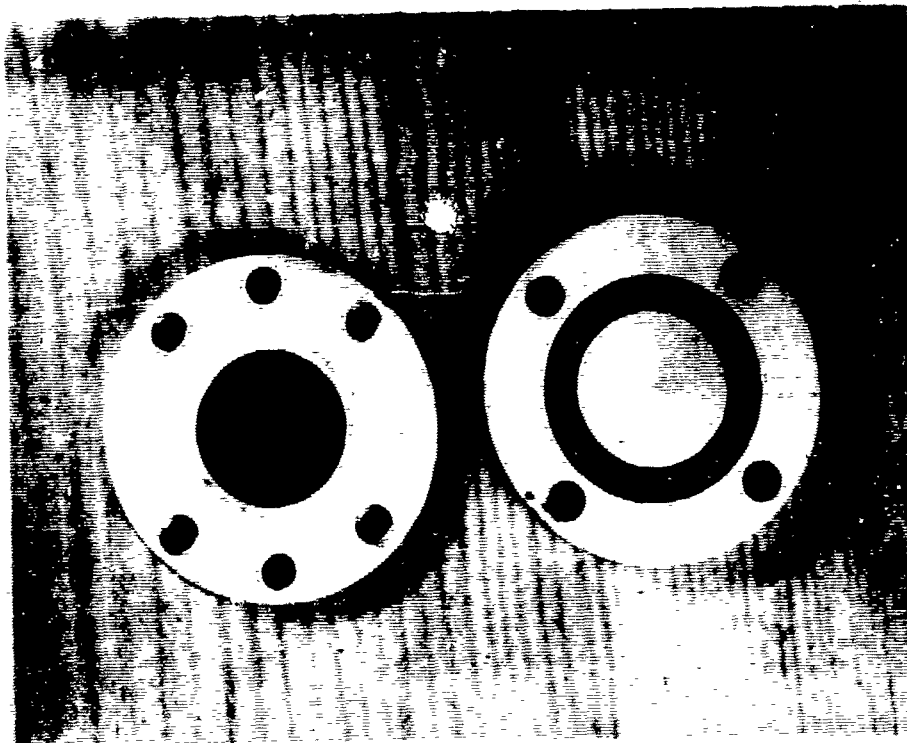


Figure 32. Viton B Formulation No. 5187:
Exposure to Hydrogen Peroxide



Before



After

Figure 33. Viton B Formulation No. 5187:
Hydrogen Peroxide System

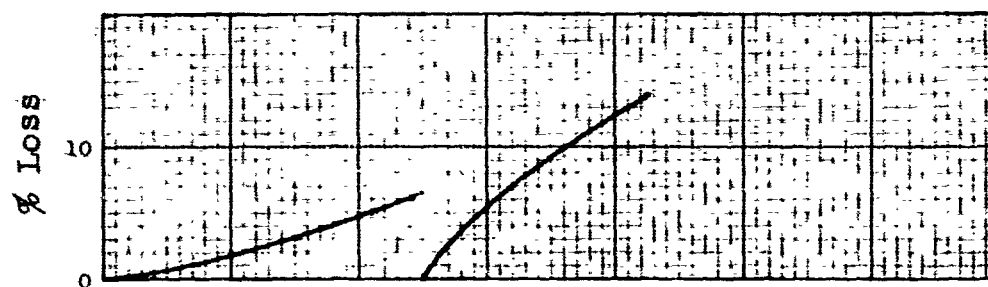


Figure 34. Teflon: Exposure to Hydrogen Peroxide

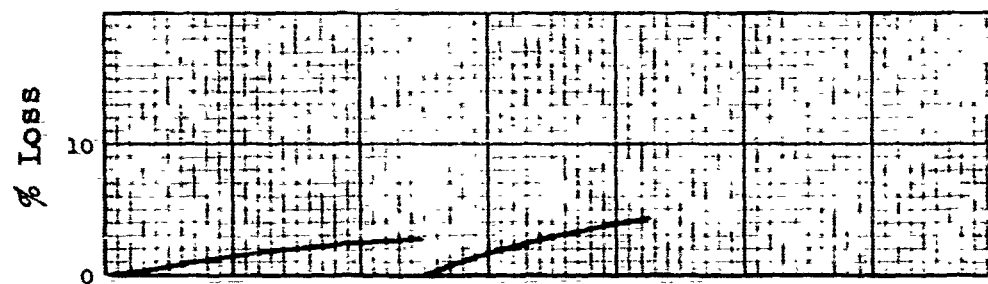


Figure 35. Polyethylene Formulation No. 39: Exposure to Hydrogen Peroxide

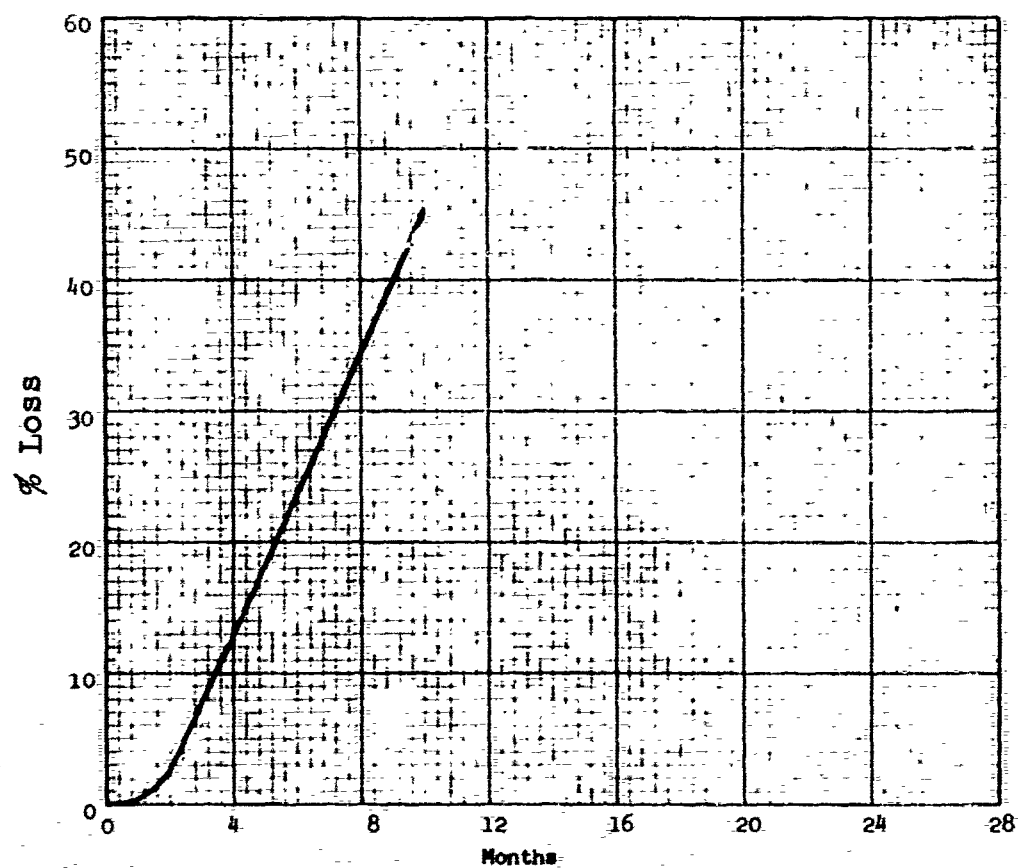
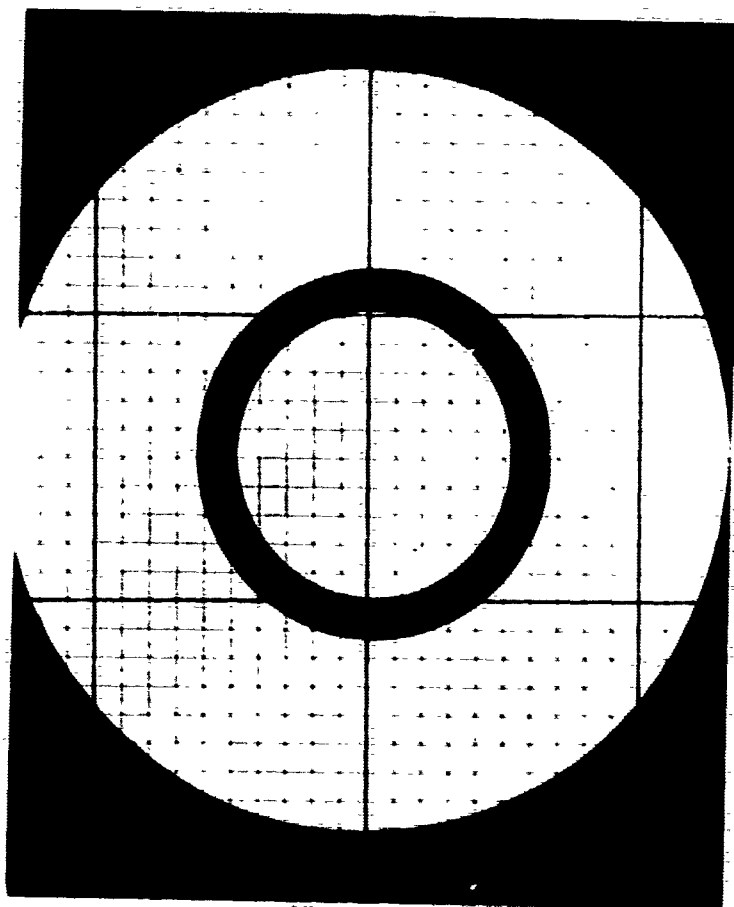
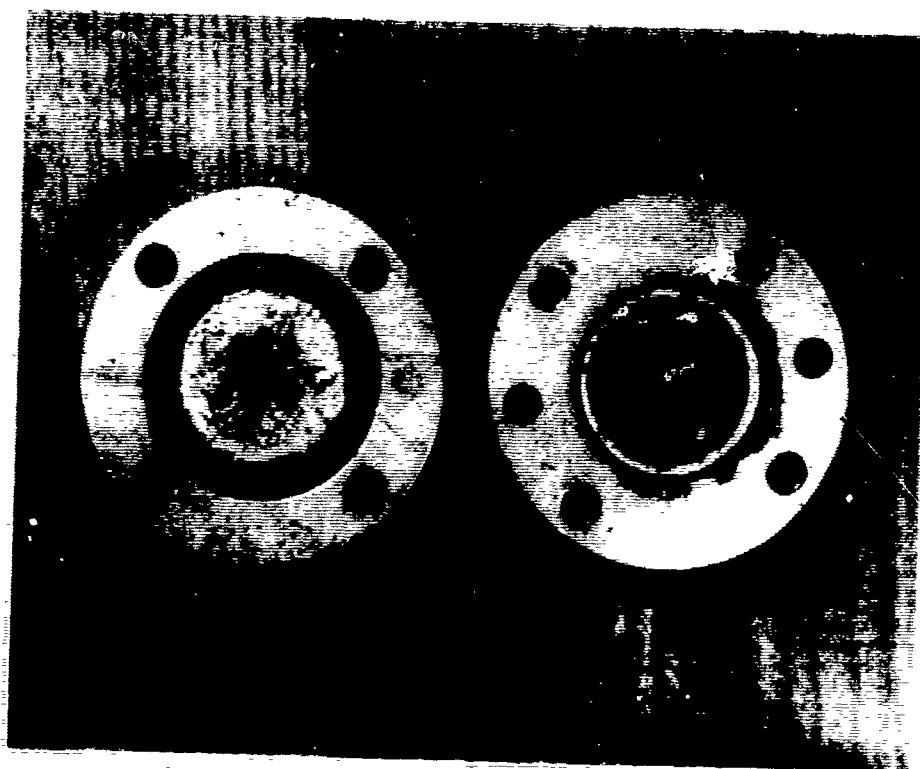


Figure 36. Butyl Formulation No. 34: Exposure to Hydrogen Peroxide



Before



After

Figure 37. Butyl Formulation No. 34:
Hydrogen Peroxide System

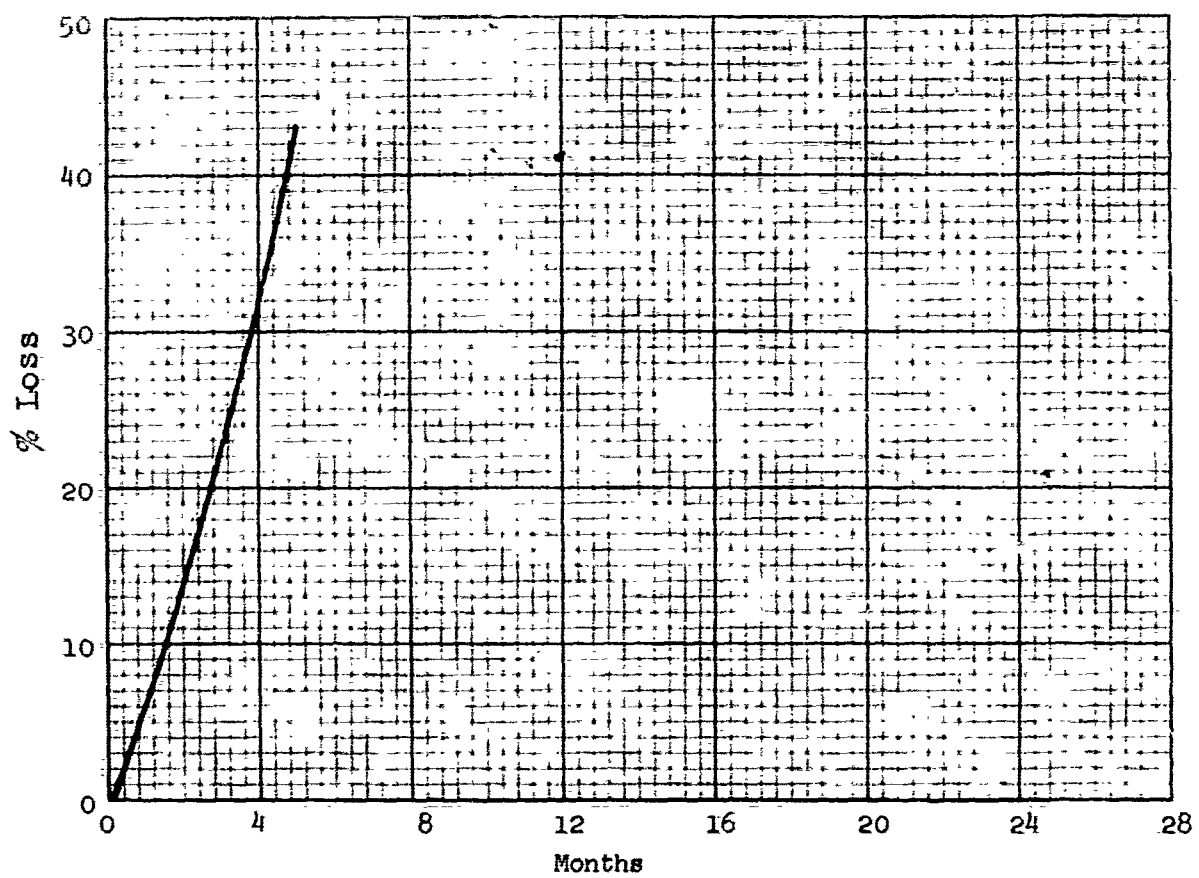


Figure 38. EPR No. 2: Exposure to Hydrogen Peroxide

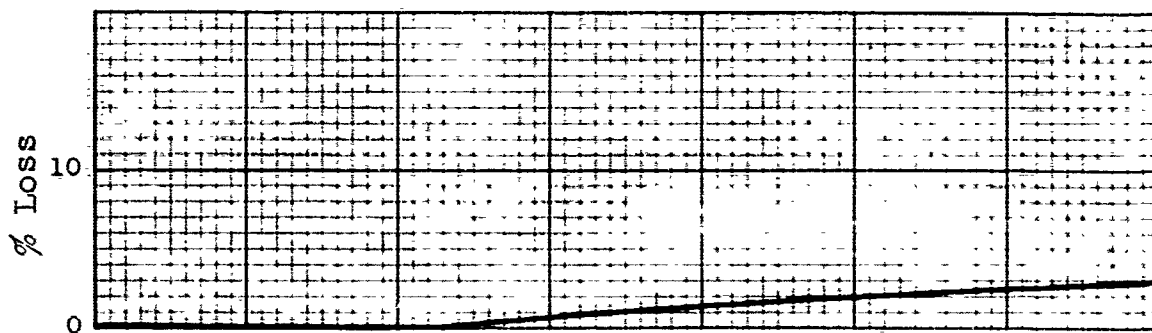


Figure 39. Teflon: Exposure to Hybaline A-5

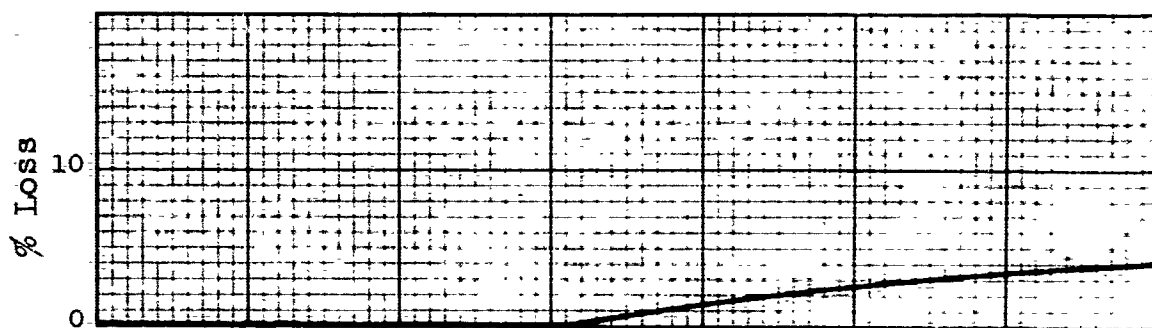


Figure 40. Polyethylene Formulation No. 39A: Exposure to Hybaline A-5

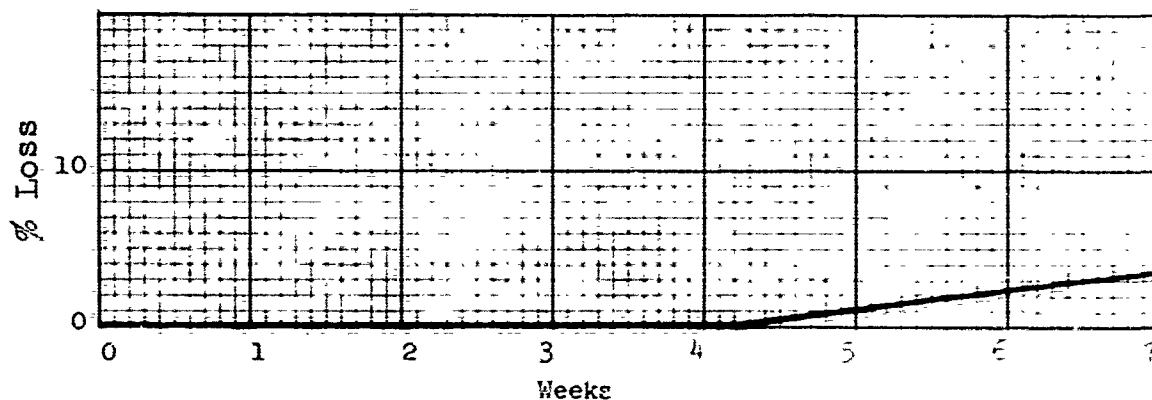


Figure 41. EPR No. 2: Exposure to Hybaline A-5

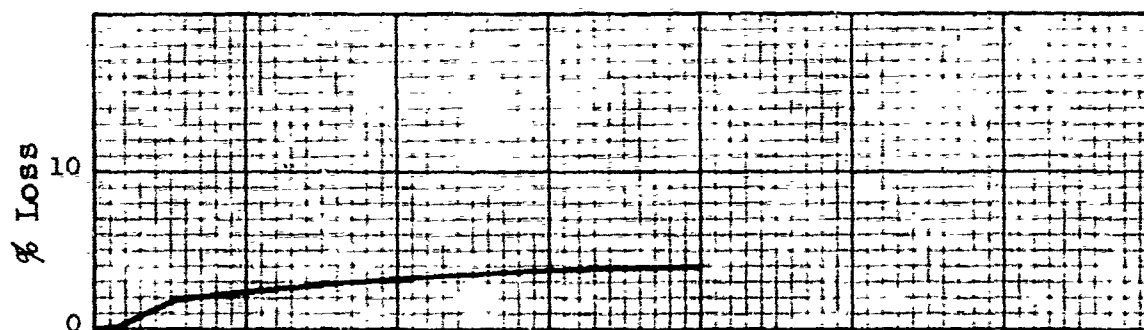


Figure 42. Viton A Formulation No. 3:
Exposure to Pentaborane

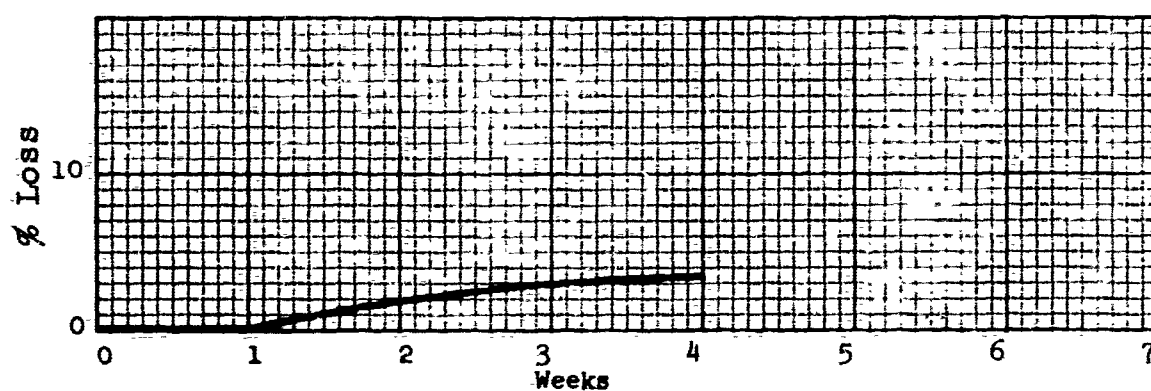


Figure 43. Teflon: Exposure
to Pentaborane

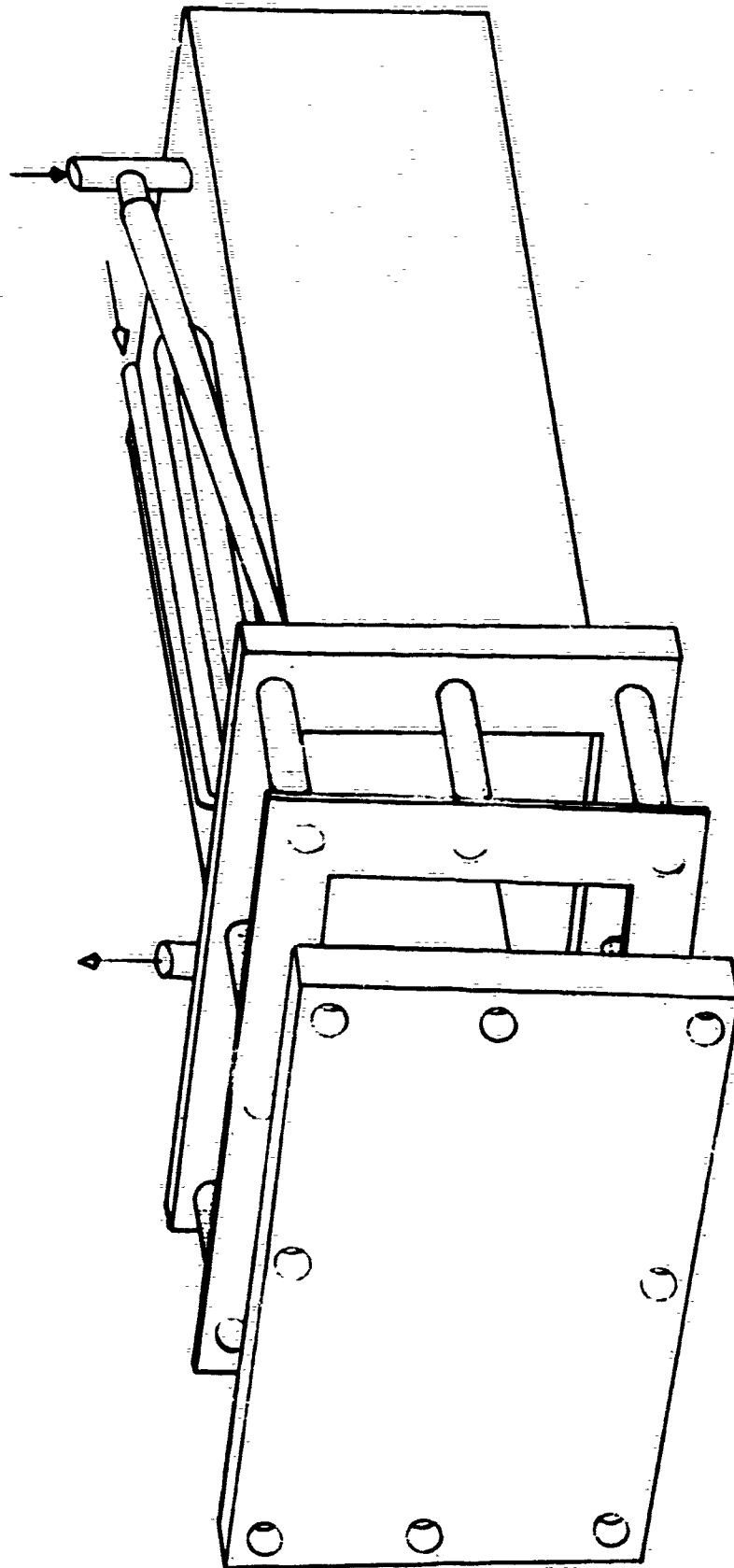


Figure 44. Test Cell Storage Container

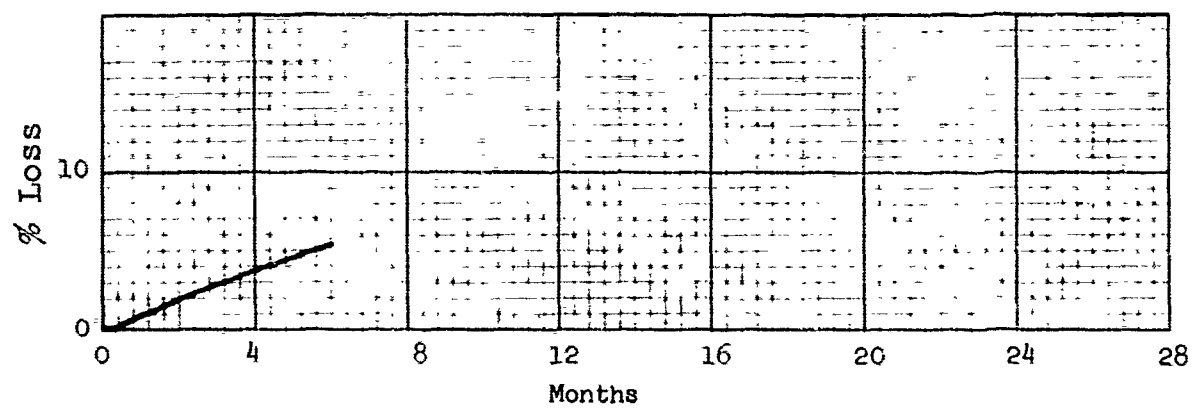


Figure 45. Teflon: Exposure to Nitrogen Tetroxide at 160°F



Figure 46. Polyethylene 39A: Exposure to Nitrogen Tetroxide at 160°F

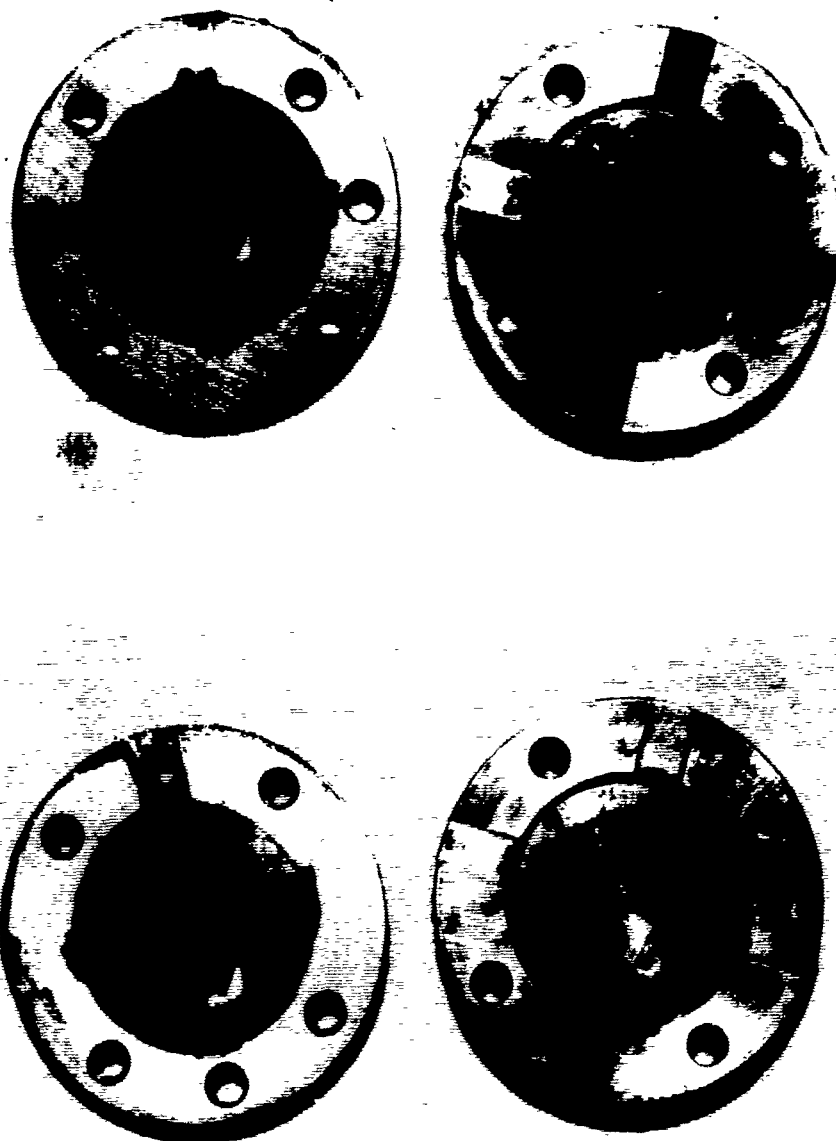


Figure 47. Butyl Formulation No. 34: Exposure to Nitrogen Tetroxide at 160°F

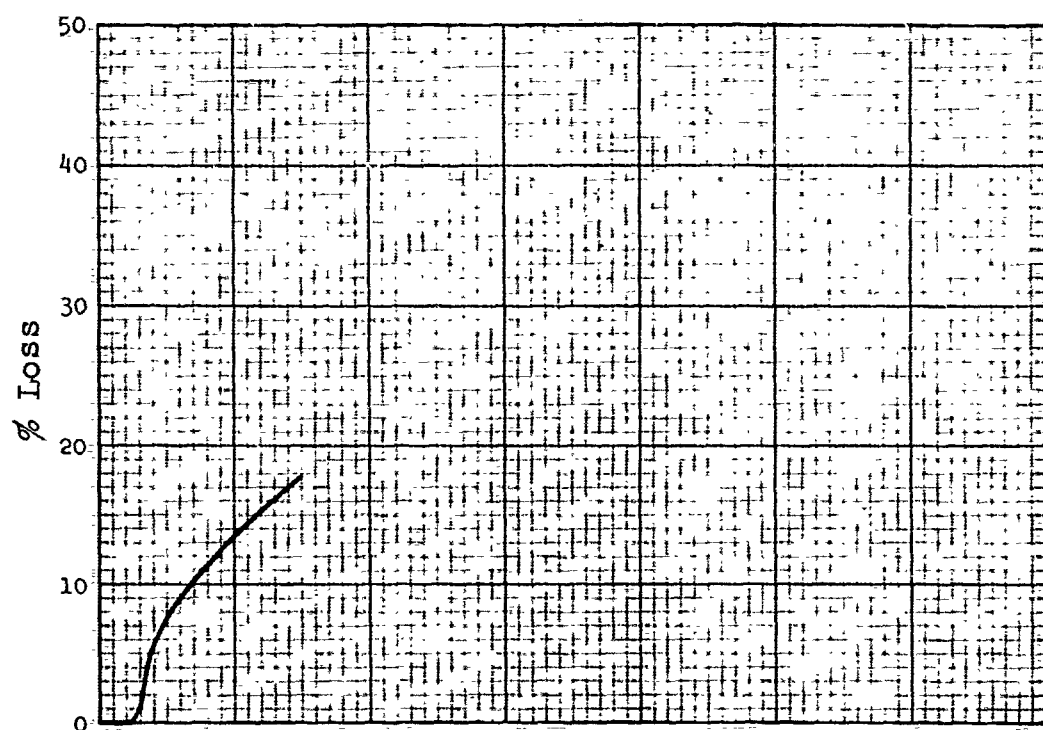


Figure 48. Polyethylene Formulation No. 39A:
Exposure to Mixed Hydrazines at 160°F

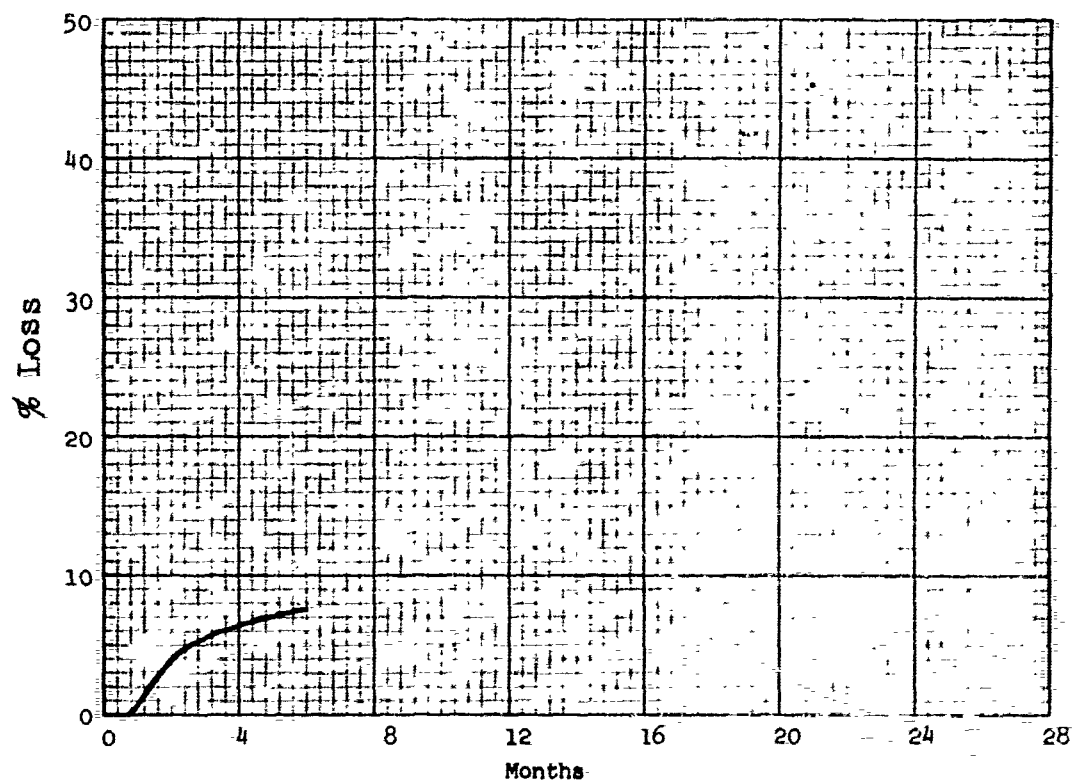


Figure 49. Butyl Formulation No. 34: Exposure
to Mixed Hydrazines at 160°F

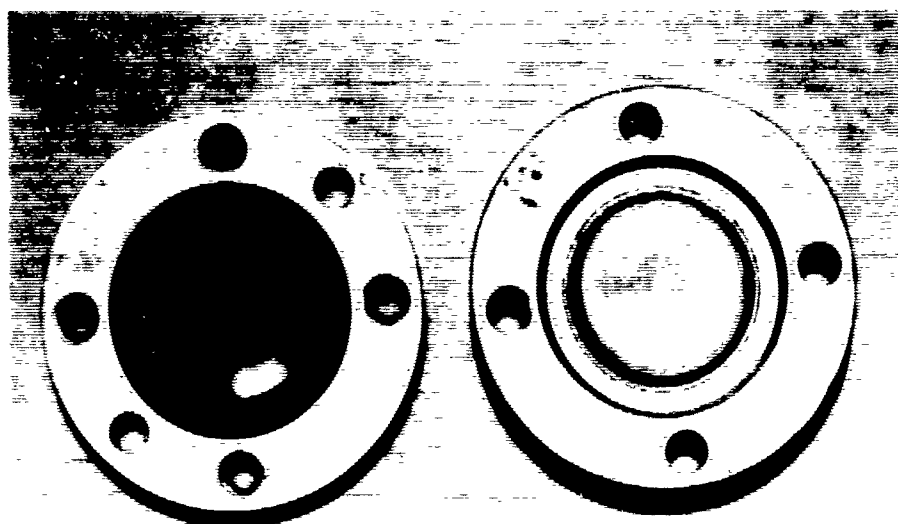
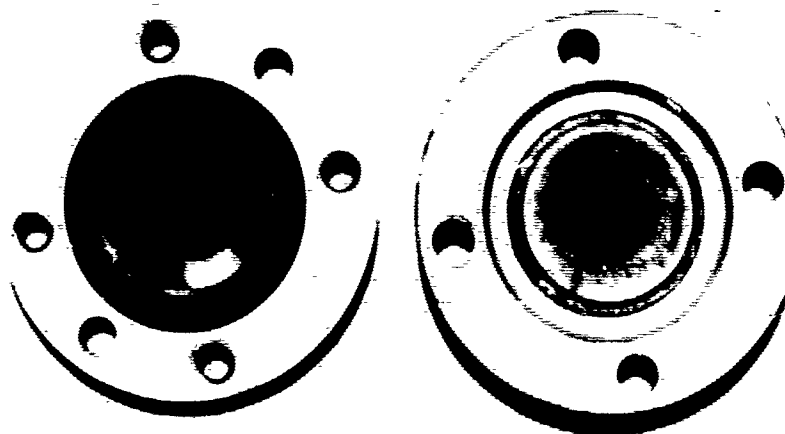


Figure 50. Polyethylene Formulation No. 39A:
Hydrogen Peroxide at 160°F

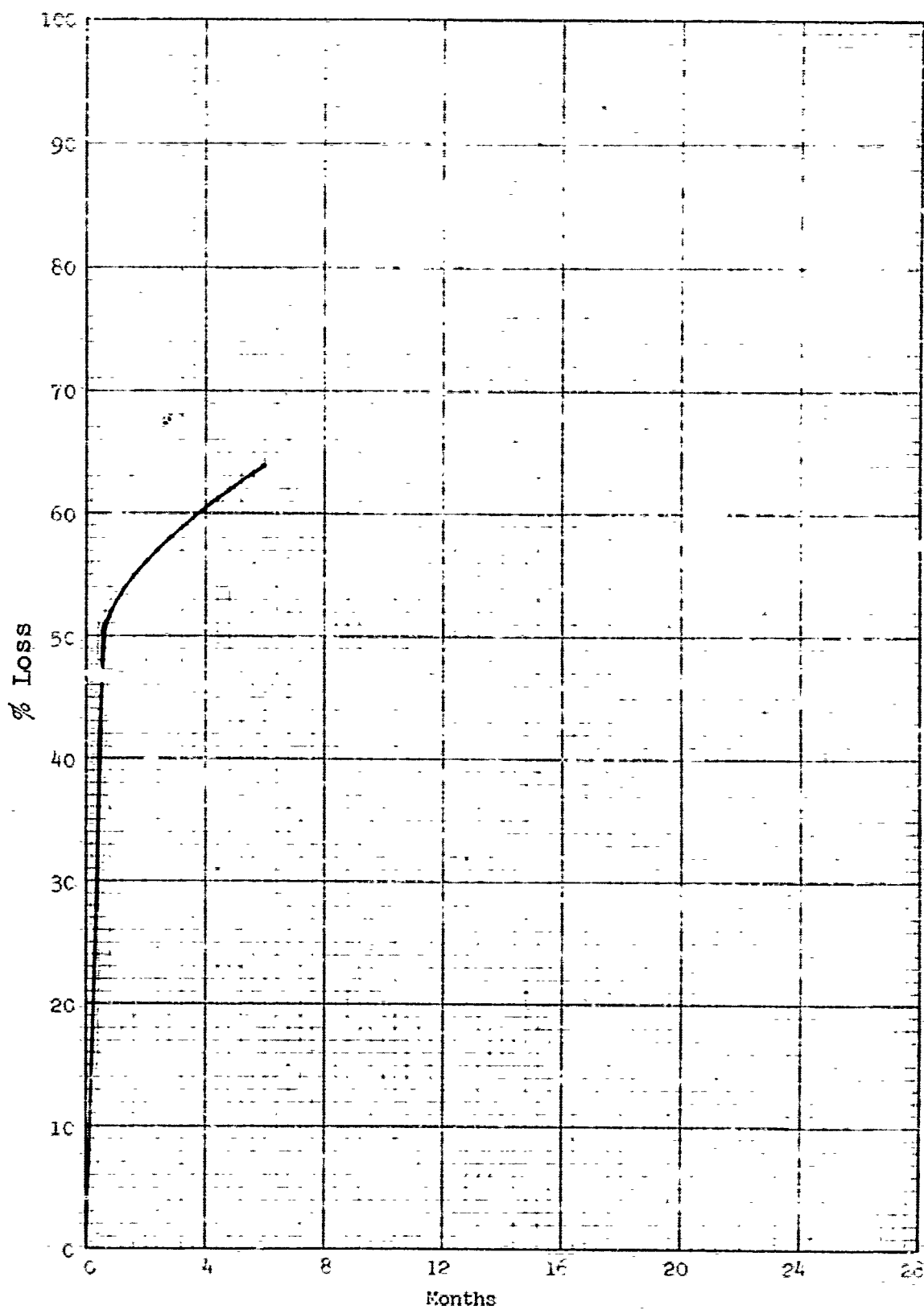


Figure 51. Polyethylene Formulation No. 39A:
Exposure to Hydrogen Peroxide at 160°F

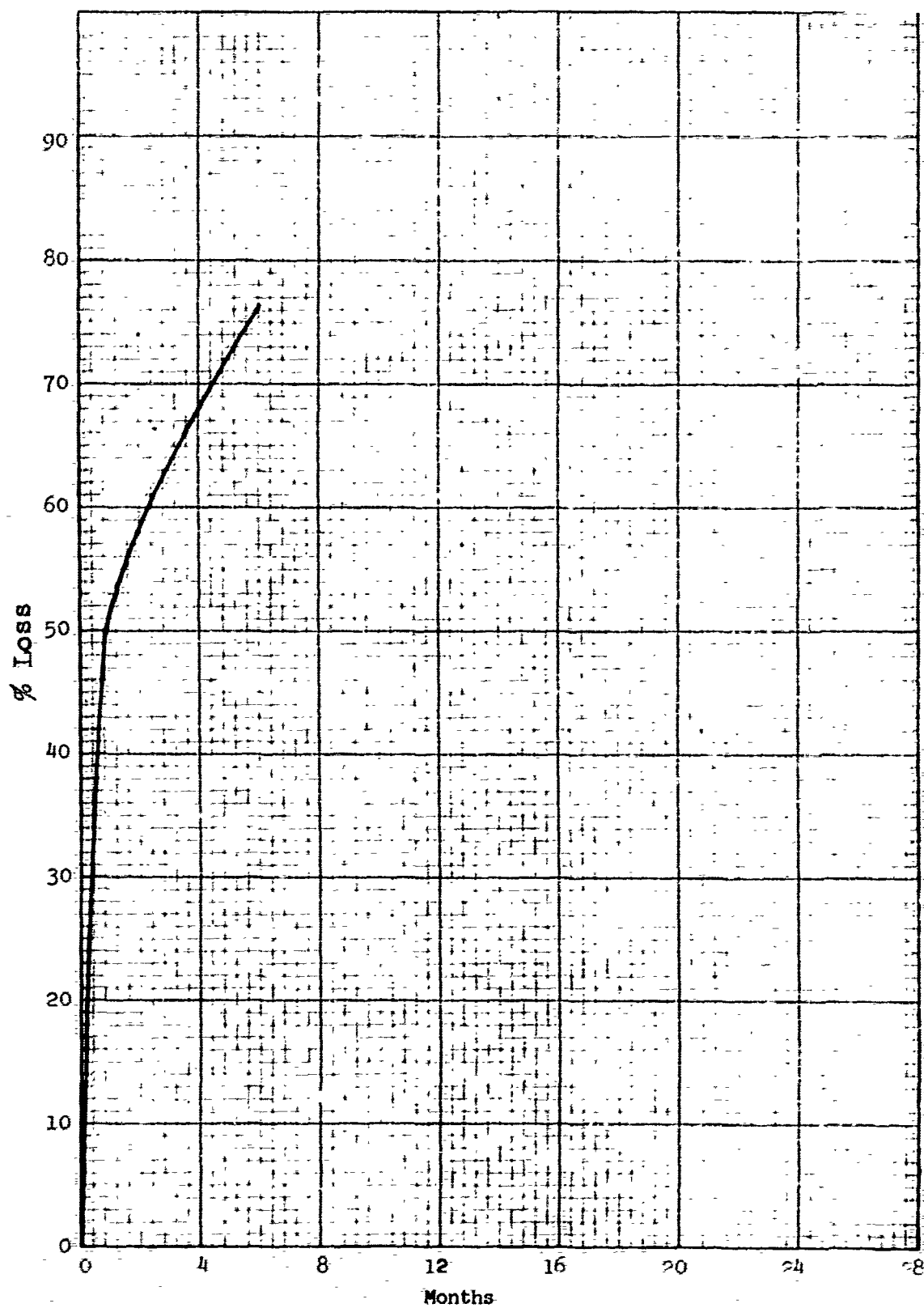


Figure 52. Silicone No. PR-11555: Exposure to Hydrogen Peroxide at 160°F

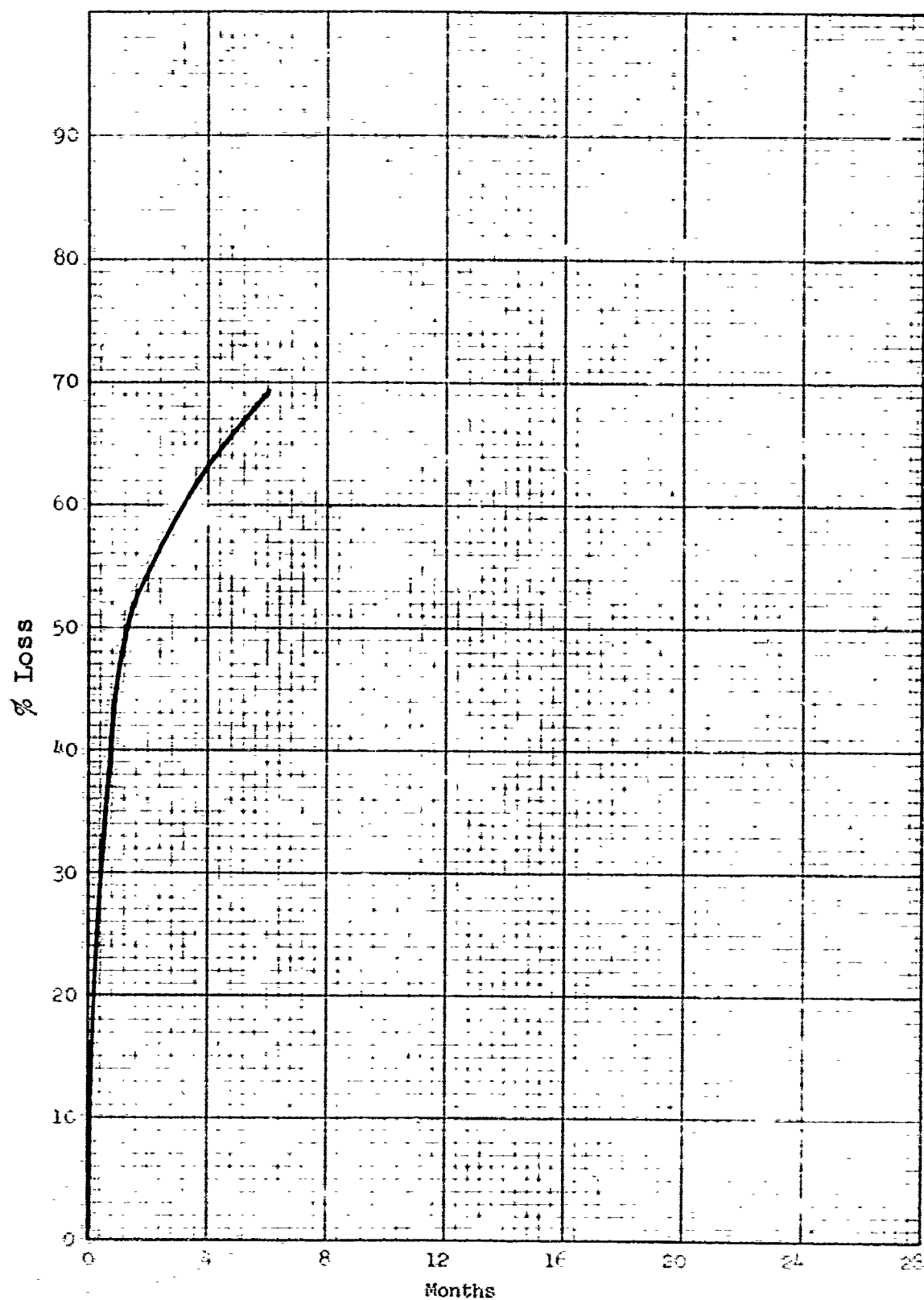


Figure 53. Viton B Formulation No. 5187:
Exposure to Hydrogen Peroxide at
160°F

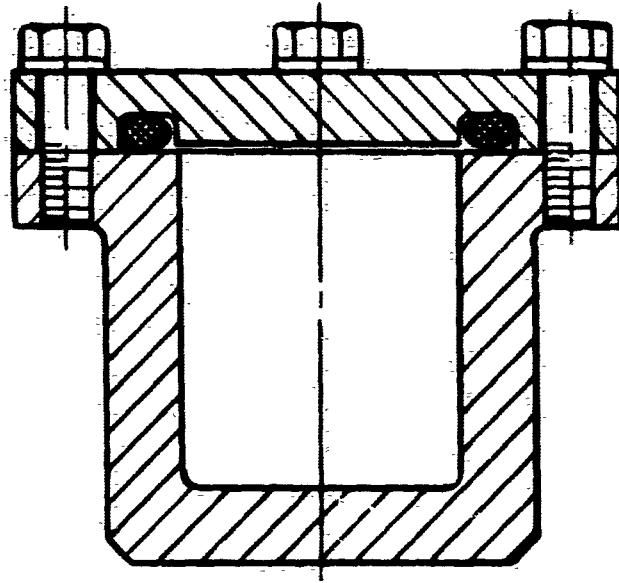
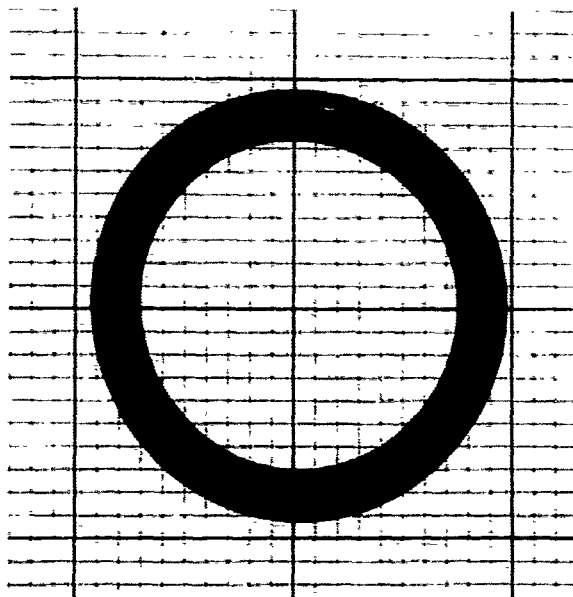
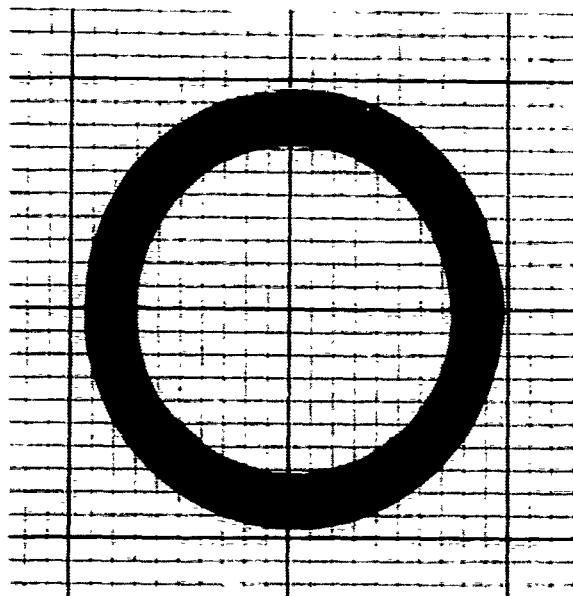


Figure 54. Test Cell for Metal Clad
O-Ring Seal Testing



15% Compression



30% Compression

Figure 55. Lead Coated O-Rings After Nitrogen Tetroxide Exposure

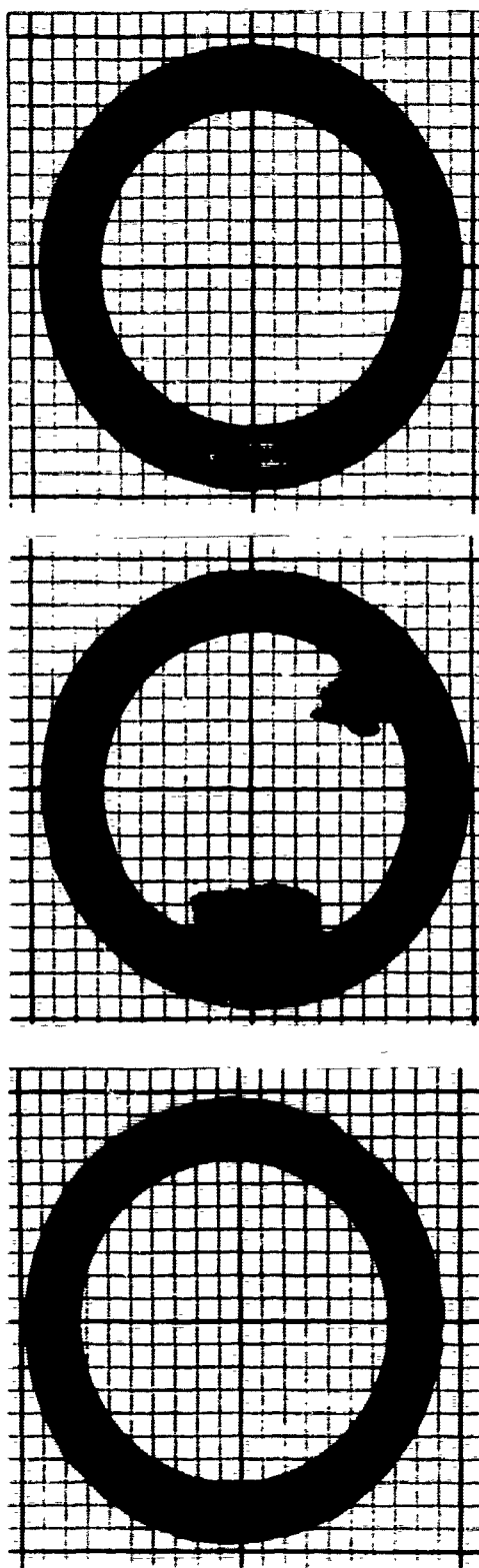


Figure 56. Copper/Lead Coating O-Rings After Nitrogen Tetroxide Exposure - 30% Compression

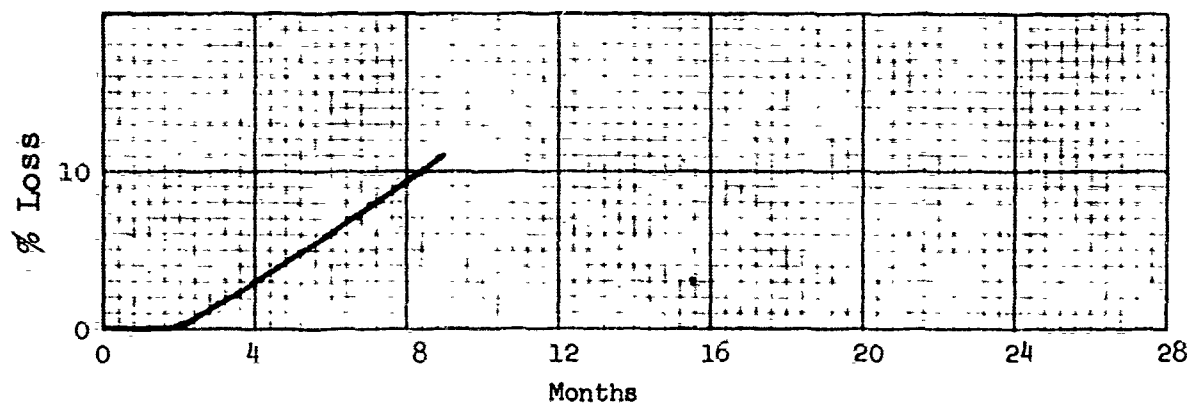


Figure 57. Copper/Lead Coated O-Rings:
Exposure to Nitrogen Tetroxide
(Liquid)



Figure 58. Copper/Lead Coated O-Rings After Nitrogen
Tetroxide Liquid Exposure - 15% Compression

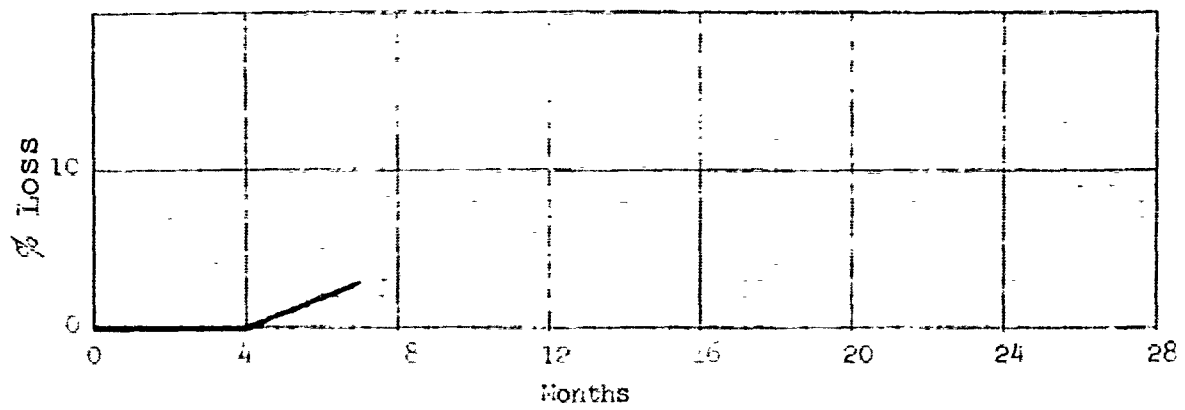


Figure 59. Copper/Lead Coated O-Rings:
Exposure to Nitrogen Tetroxide (Vapor)

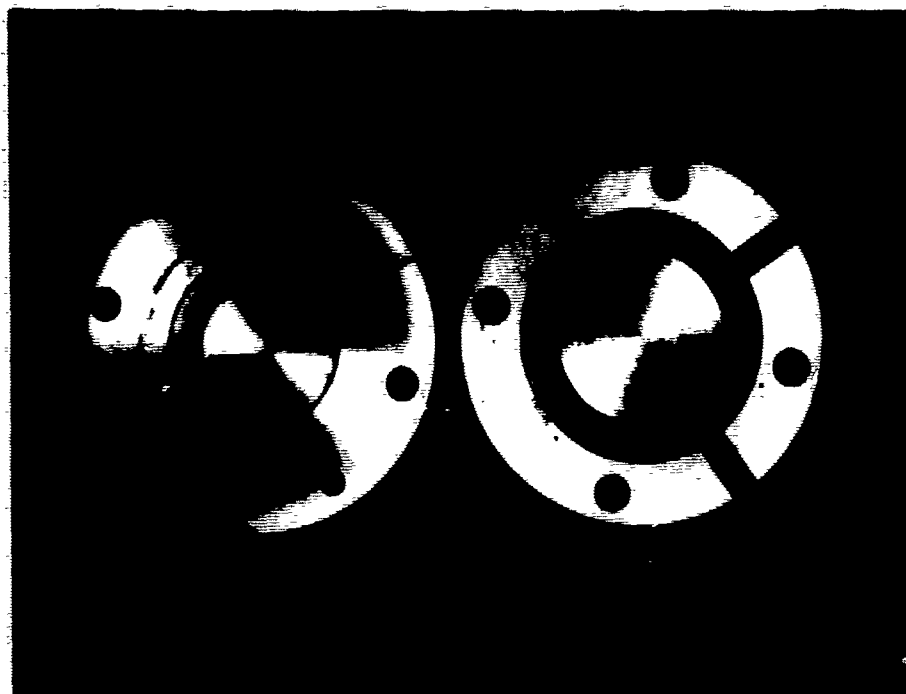


Figure 60. Copper/Lead Coated O-Rings After Nitrogen Tetroxide Vapor Exposure - 15% Compression

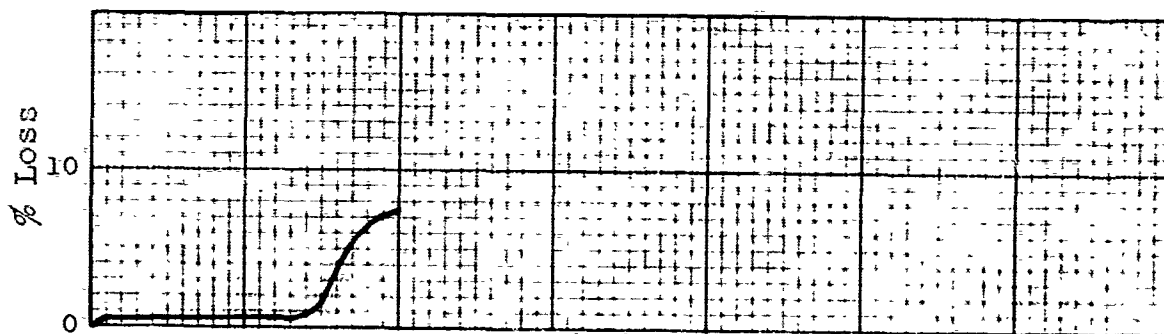


Figure 61. Silver/Lead Coated O-Rings:
Exposure to Nitrogen Tetroxide

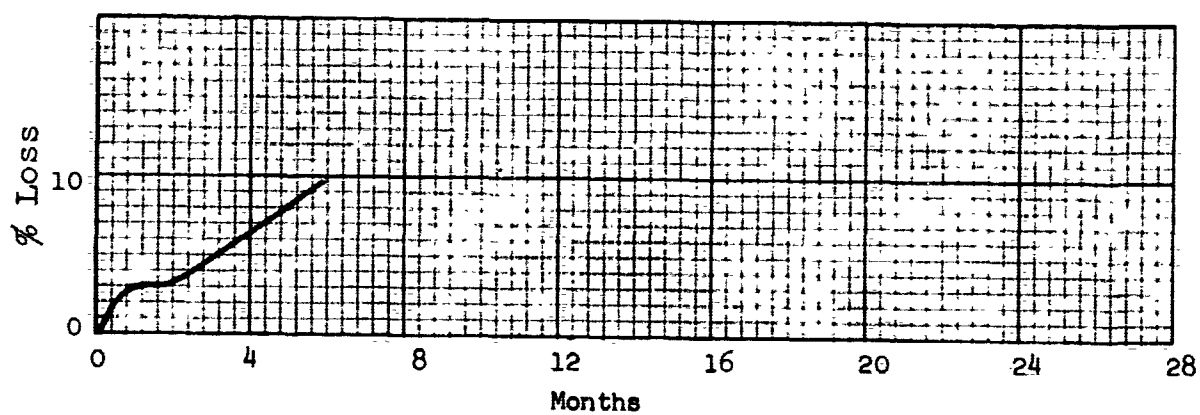


Figure 62. Crimped Lead Coated O-Rings:
Exposure to Nitrogen Tetroxide

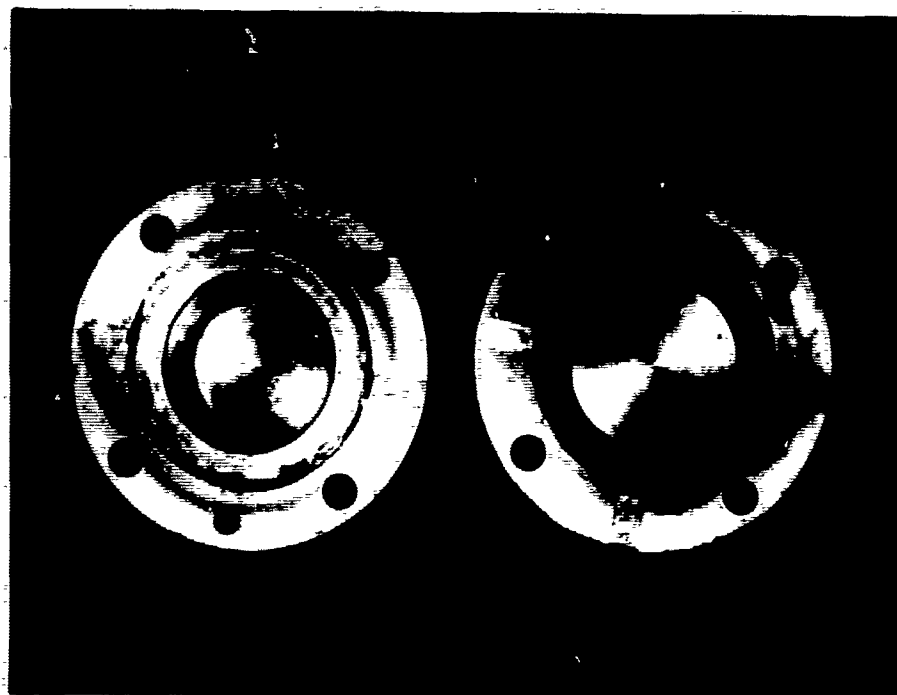


Figure 63. Crimped Lead Coated O-Rings After Nitrogen Tetroxide Exposure

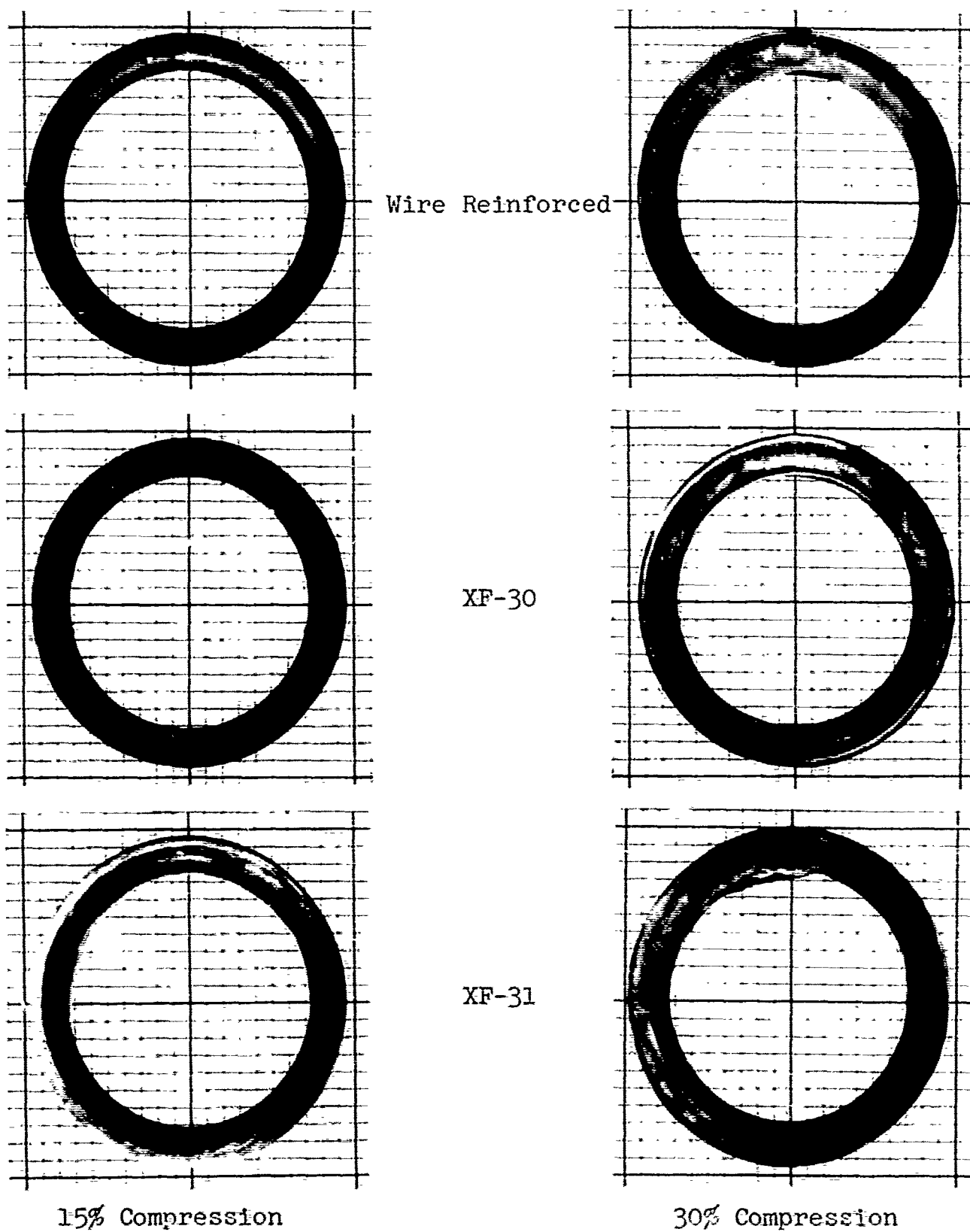
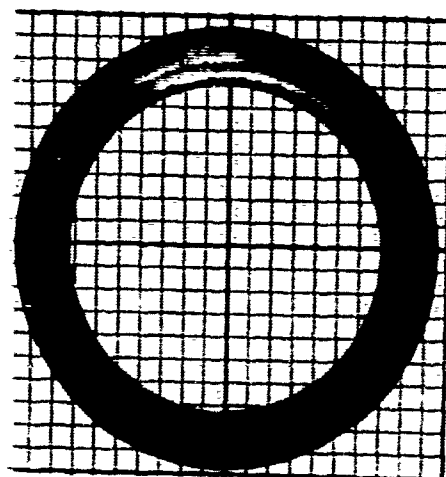
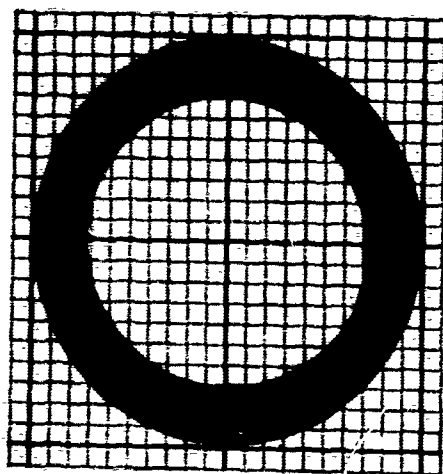


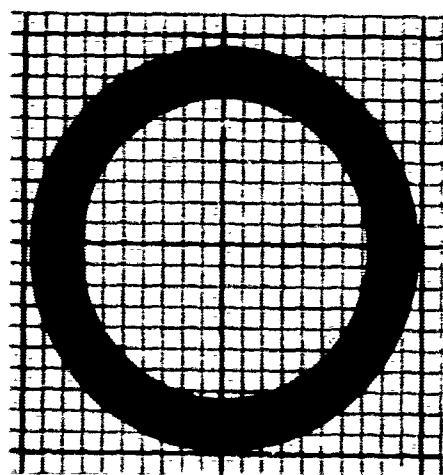
Figure 64. Lead Coated O-Rings After Nitrogen Tetroxide Exposure



Crimped Aluminum - 30% Compression

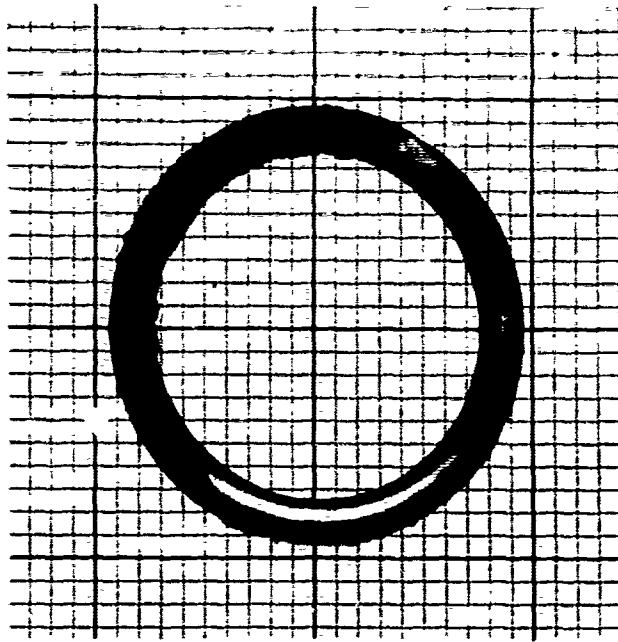


Slit Lead - 30% Compression

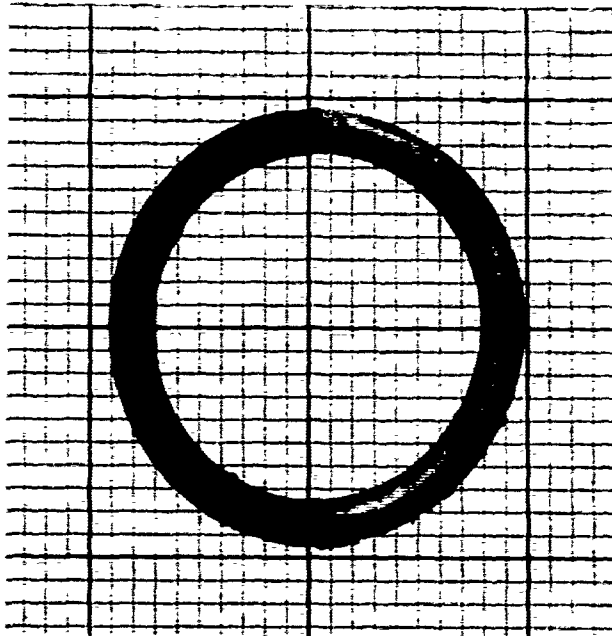


Slit Lead - 15% Compression

Figure 65. Crimped Aluminum - Slit Copper/Lead:
Nitrogen Tetroxide Exposure



15 Minute Immersion



30 Minute Immersion

Figure 66. Conductive Elastomer Copper Plated