WADC TECHNICAL REPORT 55-381

POLY FBA
A FLUORINATED ACRYLIC ELASTOMER FOR HIGH TEMPERATURE SERVICE IN THE PRESENCE OF AIRCRAFT FUELS AND LUBRICANTS

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FOREWORD

This report was prepared by the Organic Materials Branch and was initiated under Project No. 7340, "Rubber, Plastic and Composite Materials", Task No. 73405, "Compounding of Elastomers", formerly RDO No. 617-12, "Compounding of Elastomers", and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Major Horace C. Hamlin acting as project engineer.

This report covers the period of work from December 1951 to August 1955.

ABSTRACT

A large number of fluorine containing polymeric systems have been investigated by the Minnesota Mining & Manufacturing Company under Air Force Contract No. 33(038)-515. One of the most successful developments, poly 1,1 dihydroperfluorobutyl acrylate (poly FBA) exhibits good rubbery characteristics, excellent resistance to many fuels, lubricants, solvents, chemicals, and ozone, plus very good stability at elevated temperatures. Compounding and processing studies have been made, and tests conducted in various media at temperatures up to 550°F. Results of laboratory and simulated service tests show poly FBA to be vulcanizable through the action of certain metal oxides or organic amines, reinforcible with carbon blacks and some inorganic fillers, and readily handled on standard rubber processing equipment. Tensile strength ranges from 1000 to 1400 pounds and elongation from 200 to 400%. It is highly resistant to the effects of hydrocarbon fuels, even at elevated temperatures, and has been successfully tested in the presence of synthetic lubricants for up to 300 hours at 400°F, and for shorter periods at higher temperatures.

Poly FBA is also highly resistant to the chemical action of fuming nitric acid, although quite permeable to it. It is completely resistant to ozone. Low temperature properties are limited to about 0°F unplasticized, although it retains good elasticity almost down to the brittle point.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. R. WHITMORE
Technical Director
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Directorate of Research

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INTRODUCTION

Developments in military aircraft and aeronautical science occurring during the past decade have resulted in increasingly severe material problems. This is particularly true in the field of elastomeric materials. Rubbery materials capable of functioning over a very wide temperature range and highly resistant to a variety of fuels, lubricants, solvents, chemicals, and ozone are now urgently required.

The purpose of the work herein reported has been threefold namely; to maintain more intimate contact with Minnesota Mining & Manufacturing Company's (3M's) work; to conduct additional developmental compounding studies supplementing like studies by the contractor; and through compounding studies to seek additional desirable properties of interest to the Air Force. For fundamental characteristics WADC Technical Report 52-197, Parts 2, 3, 4, and 5 should be consulted. These reports were prepared by Minnesota Mining & Manufacturing Company (Central Research Department) under contract and are available to qualified organizations from the Armed Services Technical Information Agency, Document Service Center, Knott Building, Dayton, Ohio.

Several samples of poly 1,1 dihydroperfluorobutyl acrylate (poly FBA) made with slightly varying procedures have been received at Wright Air Development Center from 3M. All have been compounded and processed successfully without special difficulty, although samples which were alum coagulated contained a little more ash and were slightly more acidic requiring small adjustments in vulcanizing recipes.

Due to the relatively small quantities of poly FBA available for compounding, special small 2-1/8"x 3-1/8"x .040" sheet molds were made and compounding was done on either a 3"x 3"x 8" mill with adjustable guides or on a 1-1/4"x 1-1/4"x 4" mill also with adjustable guides. Tensile specimens were cut with a miniature die 2" in overall length with 1/2" wide tabs and a 1/2"x 1/8" restricted portion. Specimens were elongated at various rates with a rate of 12" per minute selected finally as a suitable speed at which modulus could also be read fairly accurately. Values obtained at this rate were found to be little, if any, different from those read at a rate of 20" per minute. It is likely that the miniature specimens produced values somewhat exceeding those which might be obtained with conventional samples, but such has not yet been demonstrated.

Poly FBA, uncompounded, is a translucent white snappy rubber which bands readily at processing temperatures around 140°-150°F and requires little breakdown. During compounding it has a strong tendency to adhere to both rolls. On completion of the process, however, cool rolls and proper handling permit relatively easy release and sheeting off. Fatty acids, such as stearic acid (1-2%), tend to reduce sticking and aid processing.

SECTION I

EARLY COMPOUNDING STUDIES

The earliest successful vulcanizing systems for poly FBA were devised at 3M and were based on a combination of sodium metasilicate nona hydrate and calcium hydroxide or on various metal oxides, the best of which were Litharge (PbO) and Magnesia (MgO). Magnesia in particular caked severely on the rolls unless great care was taken. Cool rolls and slow addition seemed to be the best preventative. At WADC other types of vulcanizing agents were tested, and it was found that Lead Peroxide was highly reactive and was accelerated by Polyac, although Polyac had no activity alone or with Litharge or Magnesia. Some recipes and typical properties are shown in Appendix I, Table 1. It was subsequently determined that compounds of equal quality could be produced with greatly reduced levels of both filler and vulcanizing ingredients (See Table 2). It is apparent from both tables, however, that aging stability, elasticity, and set (compression set and set at break) are far too high for a good elastomer.

A series of blends of other polymers was then tried. Three (3) examples are shown in Appendix I, Table 3. Only the Hypalon S-2 blend showed improved strength, and the volume swell of all samples in 70/30 Isooctane/Toluene test fluid was unsatisfactory. It is quite possible that actual blends were not obtained and the process resulted in mere mixtures.

Additional basic compounding studies conducted at 3M revealed that oxide vulcanizing agents for FBA were accelerated by some common rubber accelerators. Outstanding among these were zinc diethyldithio carbamate. Further studies at WADC showed that by the use of small quantities of this accelerator coupled with excellently dispersed Magnesia (added from a masterbatch) would produce a fairly well vulcanized rubber sample, provided the batch was partially set up in an oven (2 hrs at 300°F), then remilled and molded. The initial set-up provided sufficient cross linking to eliminate tendencies toward porosity during the slow curing reaction. A number of compounds of this type were made and partially evaluated. Of particular note is the reduction of swell on immersion in synthetic lubricant at 350°F, as well as the improvement in aged physical properties. It may be assumed that very small amounts of vulcanizing agents are adequate, provided they can be well enough dispersed and made to react adequately. The properties of such compounds are shown in Tables 5 and 6.

It was also found that similar procedures with Litharge were successful, the action of masterbatched fine Litharge being much faster than that of ELC Magnesia. Results are shown in Table 7.

One other vulcanizing agent, namely pulverized sodium hydroxide, was tried. It, somewhat surprisingly, proved very active and easy to employ, though not considered practical. Results of one recipe are shown in Table 8. No further work was done with it.

Although these compounds represent much improved properties over earlier ones, concurrent experimentation at both WADC and 3M resulted in the discovery that poly FBA could be vulcanized quite effectively by certain aliphatic poly amines to give compounds of equal fuel, oil, and solvent resistance, better heat resistance, much superior elasticity, more rapid cure, and lower compression set. Cyclic amines, such as hexamethylene tetramine, and aromatic amines, such as benzidine, showed much lower activity than the aliphatic amines and failed to vulcanize poly FBA.

SECTION II

COMPOUNDING STUDIES WITH AMINE VULCANIZING RECIPES

Triethylene tetramine and Trimene Base (a reaction product of ethyl chloride, formaldehyde, and ammonia) were first found to be very good cross linking agents for poly FBA. Of these, TETA (triethylene tetramine) produced better properties, its compounds being generally more thermally stable and more elastic than those using Trimene Base.

A large number of compounds have been made and tested and some of them are herein reported. These compounds were made from several experimental and pilot batches of poly FBA, which were produced by somewhat varied procedures. It is important to remember that such variations coupled with the very small samples employed may introduce variations in test results not accounted for by changes in recipe or test method. With the exception of variations already noted all tests were in conformity with ASTM Specifications, where applicable.

Compounding and processing techniques for carbon black reinforced, amine vulcanized poly FBA vary somewhat from previous methods. Roll temperatures remain about the same as with oxide loading (about 140°F). Carbon black, however, exhibits much less tendency to cake on the rolls and incorporates more readily. Generally, the polymer bands on both rolls almost immediately with some tendency to adhere to the hotter roll if there is a temperature differential. It also tends to adhere to the faster roll. Carbon black may be added slowly, almost immediately on banding, and should be continued until some band reinforcement has been achieved. Then add processing aids (stearic acid or paraffin) and sulfur, and the remainder of the filler, anti oxidant and other ingredients. After incorporation of all fillers, cool the rolls to a little above room temperature to facilitate blending and refining. Add amine slowly to prevent loss, since most amines are absorbed by poly FBA rather slowly. Final blending and sheeting is accomplished most easily on a cool (below RT) mill.

Among the variables tested in amine vulcanizing studies were processing aids. It was found that the replacement of one or two parts of stearic acid with paraffin wax resulted in a compound which hardened appreciably less on air oven aging. When employed in a later batch of poly FBA, which in itself had better aging characteristics, the paraffin compound actually became softer, a condition easily controlled with a little additional vulcanizing agent. Table 9 shows the effect of paraffin and demonstrates some typical poly FBA characteristics.

It was also noted that, as in other acrylics, sulfur, or a sulfur bearing agent is essential in developing optimum heat stability. Without sulfur, amine vulcanizates harden rapidly. It appears that about one part of sulfur represents an optimum amount since excess quantities are also detrimental. The data from Table 10 compared with Table 9 demonstrate this fact, with respect to original properties and even more noticeably in results of air and oil aging. Compound No. 456-91-1 in Table 11 shows the effect of two parts of Tetrone A replacing sulfur.

A number of amine containing agents have been tested as vulcanizing agents for poly FBA. None have been found which produced better properties than triethylene tetramine. Trimene Base is less active at low temperatures and is consequently less scorchy. Table 12 shows a brief evaluation of four other amines, which readily compound with, and vulcanize poly FBA. Of these, tetraethylene pentamine (TEPA) may be of value as a good vulcanizing agent with somewhat less low temperature reactivity than TETA. Further work with other amines is under way, but not completed.

Due, in part at least, to the alkaline nature of the vulcanizing system, the best reinforcing fillers for poly FBA seem to be furnace blacks ranging from neutral to highly alkaline pH. Fast Extruding Furnace (FEF) and High Abrasion Furnace (HAF) Blacks produce compounds with a good balance of mechanical properties exhibiting good stability under many conditions. Generally lower quantities of filler are required to produce optimum properties than with most other synthetic rubbers. Larger quantities of the coarser type fillers may be used. Little advantage is noted, however, except some reduction in volume cost of the compound and somewhat improved resilience. Table 13 shows poly FBA containing three loading levels of Medium Thermal (MT) Black. Ordinary acidic channel blacks interfere with the amine curing system in poly FBA and require additional amine to effect good cures. The high temperature aging qualities of such compounds tend to be poorer than with furnace blacks. A specially treated alkaline channel black, namely Texas 109 produced very good properties when compounded with poly FBA, and with the possible exception of shore hardness, exhibited high temperature aging properties almost equal to the furnace blacks. Table 14 shows the properties of one recipe containing alkaline channel black and one non black recipe containing Aerosil silica.

Conventional rubber antioxidants and accelerators produce a definite effect on poly FBA. The presence of small quantities of such materials noticeably enhances the retention of tensile strength after hot air and oil aging at the expense of reduced elongation and increased hardness. Table 15 shows the effect of Agerite Powder and Sulfasan R on original properties and after aging in air and synthetic lubricant at 350°F. Considerable additional work in this field has been carried out by the Minnesota Mining & Manufacturing Company and is detailed in their reports (previously referenced).

Although most of the work done on poly FBA has been pointed toward evaluation and improvement of properties in the presence of synthetic lubricants conforming to Specification MIL-L-7808 and other special fluids at very high temperatures, poly FBA has been shown to have excellent resistance to a variety of media over a wide temperature range. Table 16 shows its resistance to two types of hydrocarbon fuels and two lubricants at moderately elevated temperatures.

SECTION III

POLY FBA - COMPOUNDED SHELF LIFE

Manufacturers' literature has shown that other rubbery acrylic polymers undergo a change in properties if stored for an appreciable period subsequent to compounding and before curing. The change in the uncured compound consists mainly of some toughening due, no doubt, to cross linking. After curing the stored compound the changes are more noticeable and are, to some extent, dependent on time and conditions of storage. Increased precure storage results in cured compounds with increased elongation and decreased modulus and hardness. Tensile strength seems to be not greatly affected until after a considerable period, following which it too is reduced. Storage conditions of temperature and humidity appear to greatly affect the acceptable storage time for a given compound. Table 17 shows the effect on three (3) poly FBA compounds of precure storage up to three (3) days. This test was run in the summer under conditions of high temperature (90°-100°F) and high relative humidity. The effect of storage is self evident. It is also evident that variation in the amine content may partially overcome the effect of storage time and conditions.

A similar, but more complete, test was subsequently run in the winter under conditions of lower temperatures (65-70°F), and much lower relative humidity. This test was run over a considerably longer period of time with somewhat similar, but much less spectacular results on physical properties. The test was continued to determine the effect of storage on both hot air and hot synthetic lubricant aging. Original physical properties and recipes are shown in Table 18. Results of air oven aging at 350°F are shown in Table 19 and results of aging in synthetic lubricant at 350°F in Table 20. It is apparent that the conditions encountered in winter or in a properly controlled atmosphere are much less severe than those encountered during the previous test.

To obtain some indication of the effect of humidity alone, a brief additional test was run in which one sample was stored, before curing, in open atmosphere, and one was carefully wrapped in three (3) layers of polyethylene film. The results are shown in Table 21. This test was also run in the winter which probably reduced the difference in the results of open vs wrapped storage. Although the difference in results is slight, it appears that protection from moisture helps prevent changes in the compound during storage.

SECTION IV

POLY FBA COMPOUNDS FOR HIGH TEMPERATURE SERVICE

Since poly FBA has shown promise for high temperature service under various conditions, some work was devoted to determining the approximate temperature limit and how to extend service life at high temperatures. Tables 22 through 26 show the effect of temperatures ranging from 350°F to 550°F on poly FBA compounds. From the results it appears that 350°F is a very mild temperature for poly FBA for service in synthetic oil. As temperatures rise it appears that the slight tendency for hardness increase noted at 350°F is gradually reversed. This may be explained by slight additional cross linking which is overcome at higher temperatures, probably by chain scission and gradual decomposition. This effect is definitely retarded by the use of additional amine vulcanizing agent. Employment of such materials as Sulfasan R (previously discussed) should also help. Although properties degraded very rapidly at 500°F and above, some simulated service tests have indicated that poly FBA seals may be serviceable for considerably longer than indicated by the test data.

SECTION V

COMPRESSION SET OF POLY FBA

Compression set is an important characteristic of rubber materials intended for use as seals, diaphragms, etc. In this respect, also, poly FBA compounding is generally similar to that used for other acrylic rubbers. Factors affecting compression set are: type of filler used, period of rest prior to vulcanization, amount of filler used, amount of vulcanizing agent used, use of rubber antioxidant, and period of oven tempering before testing. Tables 27, 28, and 29 show the effect of some of the above variables. All samples were oven tempered prior to testing and all test discs, except where noted, were molded to one half the thickness and one half the diameter of Standard ASTM compression set test plugs. In these tests it appeared that the small size of the plug and/or the nature of the test jig (unplated) used produced more severe results than similar tests run elsewhere. It should be noted that compression recovery at or above test temperature resulted in quite favorable results. Since this condition may frequently occur in actual applications, it is quite significant. It should also be noted that the test temperature used was 250°F rather than 212°F commonly employed with most other synthetic rubbers. The test results show that reduced filler loading and coarser particle fillers (FEF Black) are favorable for low compression set. Some additional vulcanizing agent is also desirable. TETA vulcanized compounds produce lower compression sets than otherwise similar compounds vulcanized with Trimene Base. The presence of rubber antioxidants may be undesirable.

In order to provide more complete and useful information from the results of compression set tests, these compounds were aged in air and synthetic oil at 350°F. The results of aging are shown in Tables 30 through 32.

SECTION VI

RESISTANCE OF POLY FBA TO FUMING NITRIC ACID

The addition of fluorine to some other saturated organic compounds has resulted in improved resistance to red and white fuming nitric acid. Due to its saturated chemical composition and the presence of about 50% by weight fluorine, it is reasonable to suppose that poly FBA might withstand the action of the above acids quite well. Three (3) poly FBA compounds were immersed in Red Fuming Nitric Acid conforming to Specification MIL-N-7277 for 70 and 200 hours at room temperature. The results are shown in Table 33. All three compounds swelled at least 25% and suffered quite a reduction in physical properties, retaining, however, fairly reasonable tensile strength. It is of particular interest to note that on aging for 200 hours and then rinsing in water, virtually all of the swell was eliminated and physical properties returned practically 100%. This indicates that the loss of properties on immersion was due largely to the volume increase and there was little or no chemical degradation.

To determine the resistance of poly FBA to nitric acid or oxide penetration, a 15 mil film of compounded and cured poly FBA was tested in a standard H cell and the permeation measured by reduction in pH on the water side of the cell. The results are shown in Figure 1. Although chemically quite resistant to fuming nitric acid, poly FBA is quite transparent to it and forms only a poor barrier.

SECTION VII

LOW TEMPERATURE PROPERTIES OF POLY FBA

For military applications low temperature properties of materials are of extreme importance. The low temperature properties of two (2) poly FBA compounds (Peroxide cured and amine cured) were measured by the Temperature-Retraction Test and by the ASTM D746-52T Brittle Point method. The Temperature-Retraction Test was run essentially in confomity to the ASTM D599-40T procedure except that the entire retraction curve was recorded and methanol was substituted for acetone as the immersion medium. The results are shown in Figure 2. Although it is apparent that poly FBA is not a good low temperature material, two features can be noted. From the steepness of the curves it can be observed that poly FBA retains most of its original elasticity right down to the brittle point, and from the relation of the left hand portion of the curves to the brittle point it seems that, for some applications at least, poly FBA may have sufficient flexibility to be used a little below the measured brittle point. Poly FBA has been successfully plasticized with fluorine containing plasticizers to flexibility as low as -65°F. All plasticizers tested, however, have been readily extracted by common fuels and lubricants.

SECTION VIII

POLY FBA - MOLDING AND EXTRUSION

Generally, neither molding nor extruding poly FBA presents any particular problems. Early compounds vulcanized with oxides, peroxides, etc. were slow to exhibit incipient vulcanization. This, coupled with the extra polymer breakdown resulting from dispersing high oxide loadings, caused the heated polymer to become a very soft runny mass when placed in a hot mold. There was little or no back pressure exerted and consequently many moldings were badly blown. Oven preheating to incipient cross linkage before molding alleviated this difficulty and permitted relatively easy production of good molded sheets.

Amine vulcanized compounds do not exhibit the above difficulty and can be press cured or autoclave cured very readily. For all moldings, fairly generous application of a mold release such as Dow Corning DC 35B is essential, although it need not be repeated after each pressing. Unchromed molds will be discolored by the action of triethylene tetramine and some other vulcanizing agents. The discoloration has at no time been shown to damage the mold; although, the deposit may build up to appreciable thickness after some time. It can very readily be removed with wet steel wool and pumice. There is considerable experimental evidence that the amine stain acts as a corrosion preventative for the steel.

Molding temperatures may run from 280°F to 340°F or above; although, the higher temperatures may cause difficulty with large moldings. Relatively low molding pressures may be used without difficulty.

Experimental extrusion of poly FBA was initially done with a small plunger operated extruder which was preheated to about 165°F. A 40-50 gm slug of compounded stock was placed in the plunger well, the plunger partially inserted, and the assembly placed in a power driven heated molding press. As the press was closed at uniform speed, the extruder plunger was forced downward and approximately a two foot length of extrusion produced. This device was resorted to for reasons of economy of material. The extruding device assembled and disassembled is pictured in Figures 3 and 4.

Later extrusion tests with larger quantities of material were run on a No. 1/2 Royle rubber extruder. These extrusions were made very easily and many of the amine cured recipes herein reported have been successfully extruded. No extrusions were attempted with oxide cured stocks. All extrusions on the Royle extruder were run with a water cooled screw and a die heated to about 150-165°F. The extruder was otherwise uncontrolled. For long time continuous extrusions, the above figures will not necessarily hold true and extrusion conditions must be determined by experience. The extruder was operated from 1/2 speed to full speed (Linear extrusion rate not measured). Extrusion swell appeared to be minimized by faster extrusion rates.

Whereas in the plunger type extruder, only fresh stock could be smoothly extruded, in the Royle this problem was not particularly noticeable. The problem is believed to be due to the room temperature cross linking or scorching which occurs with TETA. It probably was reduced in the Royle extruder by the working which the stock received from the action of the screw.

Freshly compounded (or freshly extruded) poly FBA stocks are rather tacky and care should be exercised on handling fresh extrusions until soapstone can be applied. Extrusions can be readily cured in an autoclave at temperatures from 310°F to 340°F for 30' to 60'. Autoclave pans with a 1/2" or more layer of Talc or soapstone will prevent flattening one side of the extrusion.

A fifty (50) foot length of approximately .430" O.D. poly FBA tubing was run off at WADC and covered with two (2) layers of stainless steel wire braid and vulcanized through the courtesy of the B. F. Goodrich Company. Sections of the hose thus produced were coupled by the Aeroquip Corporation. A section of the tubing and the hose are shown in Figure 5.

Sections of this hose were subsequently tested on the Materials Laboratory, WADC, hose tester. The test was conducted by vibrating the hose mounting 1800 times per minute at 1/2" amplitude while circulating MIL-L-7808 synthetic lubricant through the hose at 350°F and 75 pounds pressure. Chamber temperature was maintained at 300°F. The test was carried on for over 300 hours and then discontinued without failure. Further hose development and testing is now being carried out by the Flexotube Division of the Meridan Corporation and the United States Rubber Company under Air Force Contract.

SECTION IX

POLY FBA LATEX

Although no work has been done at WADC with poly FBA Latex some studies carried out by 3M have shown poly FBA Latex capable of being compounded and processed without particular difficulty employing procedures and equipment generally used in handling latices.

SECTION X

SUMMARY

A new synthetic elastomer has been developed having a unique combination of fuel, oil, and solvent resistance and high temperature stability. The results of many tests suggest application in a variety of types of mechanical goods including seals, diaphragms, hose, coated fabrics, and other items.

For anyone interested in evaluating poly FBA or doing additional compound development, it is suggested that either Compound No. 257-86-1, described in Table 11 or Compound No. 257-18-3, described in Table 27 (if a soft stock is desired) exhibits a good balance of properties and makes a good starting point for further development.

Poly FBA is available in pilot quantities from the Minnesota Mining and Manufacturing Company, Fluorochemicals Department, St. Paul, Minnesota.

Poly FBA has been developed to the commercial stage and is finding increasing application in both military and non-military applications. Research and development work on improved materials is continuing with emphasis on promising polymers for equal chemical and high temperature stability and much improved low temperature flexibility.

APPENDIX I

TABLES

Table 1

Poly FBA - Properties of Peroxide-Polyac Cure

Compound No.	769-5-1	769-6-1	769-6-2	769-6-3	772-78-1
Poly FBA	100	100	100	100	100
Lead Peroxide	20	20	20	20	20
Polyac	3	3	3	3	3
EPC Black	10	-	-	-	SAF 20
FEF Black		10	30	30	
Cure	30'at 310°F	45'at 310°F	30'at 310°F plus 30'oven	35'at 310°F	60'at 310°F
100% Modulus Lbs/In^2	-	-	-	-	-
Tensile Strength Lbs/In^2	1320	1265	1310	1210	1256
Elongation, %	360	450	138	193	183
Set at Break %	55	32	12	14	32
Hardness Shore A					82
Vol. Increase 70 hrs in 70/30 Isooctane/Toluene at RT	18.8%				14.4
Compression Set 70 hrs at 212°F 30% Compression	85.5%				

Table 2

Poly FBA - Oxide Curing Recipes

Compound No.	000-18-1	000-19-8	000-19-9
Poly FBA	100	100	100
Magnesia	15	5(added from Masterbatch)	3
Lead Peroxide	3	3	2
Stearic Acid	1	1	-
Polyac	3	HAF Black 10	10
Cure	60' at 310°F	60' at 310°F	60' at 310°F

Original Properties

	000-18-1	000-19-8	000-19-9
100% Modulus, Lbs/In^2	432	-	183
Tensile Strength, Lbs/In^2	1420	1345	1062
Elongation, %	373	415	697
Set at Break, %	44	43	56
Hardness, Shore A	48	55	48
Compression Set 70 hrs at 212°F 30% Compression	52%	88.2	90.3

Aged 70 Hours at 350°F in Air

	000-18-1	000-19-8	000-19-9
Tensile Strength, Lbs/In^2	1701	1139	687
100% Modulus, Lbs/In^2	1442	-	456
Elongation, %	146	75	226
Hardness, Shore A	-	81	60
Set at Break, %	13	04	14
Volume Increase in MIL-L-7808 Oil 70 hrs at 350°F		60.0%	

Table 3

Poly FBA Blends with other Elastomers

Compound No. 772-46-1	772-53-1	772-55-3	772-55-2
Poly FBA - 60	Poly FBA - 60	Poly FBA - 100	Paracril 18-80 - 100
Neoprene GN - 40	Hypalon S-2 - 40	Magnesia - 25	Zinc Oxide - 5
Litharge - 25	Staybellite Resin - 10	Lead Oxide - 5	Stearic Acid - 1
Stearic Acid - 0.5	Magnesia - 25	Stearic Acid - 1	Sulfur - 2
Cure - 30' at 310°F	Lead Oxide - 5		Altax - 1.5
	HAF Black - 10		HAF Black - 40
	Cure - 45' at 310°F	Blend these two (2) compounds in ratios of 60% FBA and 40% Paracril 18-80 by polymer weight. Cure 60' at 310°F	

Properties

	772-46-1	772-53-1	772-55-3 / 772-55-2 blend
Tensile Strength, Lbs/In^2	1467	1804	1010
Elongation, %	435	148	455
Set at Break, %	15	5	15
Volume Increase, %, 70 hrs in 70/30 Isooctane/Toluene at RT	40	65	41

Table 4

Poly FBA - Oxide Cured - Volume Swell in MIL-L-7808 Lubricant at 350°F*

Compound No. 000-	42-1	42-5
Poly FBA	100	100
Stearic Acid	1	1
Magnesia (Masterbatched)	3	1.5
Ethyl Zimate	1	1
HAF Black	15	15

Cure - 90' at 310°F after 2 hr Oven Precure and Remill

Volume Increase in MIL-L-7808 Oil at 350°F

150 Hours	4.2%	-
300 Hours	3.2	10.8%
800 Hours	27.3	28.0

* See Table 5 for physical properties of similar compounds. This table included merely to show effect of advancing type cures on volume swell at elevated temperatures.

Table 5

Masterbatched Low Oxide Curing Recipes for Poly FBA

Compound No. 000-	46-1	46-3	46-5	58-2	62-1
Poly FBA	100	100	100	100	100
Stearic Acid	1	1	1	1	1
Magnesia (Masterbatched)	1.5	1.5	3	1.5	0.75
Ethyl Zimate	1	1	1	1	2
HAF Black	15	25	10	25	35
Sulfur	-	-	-	0.5	-

Cure - Oven precured to incipient cross linkage.
Press cured 90' at 310°F. Oven tempered 2 to 4 hours at 350°F

Original Properties

	46-1	46-3	46-5	58-2	62-1
100% Modulus, Lbs/In^2	299	569	352	584	858
Tensile Strength, Lbs/In^2	1255	1281	1323	1167	1314
Elongation, %	667	478	570	408	317
Set at Break, %	82	69	74	50	44
Hardness, Shore A	52	65	54	68	78

Air Oven Aged 70 Hrs at 350°F

	46-1	46-3	46-5	58-2	62-1
100% Modulus, Lbs/In^2	-	-	-	-	-
Tensile Strength, Lbs/In^2	674	680	693	424	-
Elongation, %	100	90	115	57	-
Set at Break, %	06	13	11	12	-
Hardness, Shore A	69	83	76	79	-

Table 6

Properties of Compounds in Table No. 5
After Aging in MIL-L-7808 Lubricant
at 350°F

Compound No. 000-	46-1	46-3	46-5	58-2	62-1
168 Hours					
100% Modulus, Lbs/In^2	-	-	-	-	-
Tensile Strength, Lbs/In^2	1077	1176	946	-	-
Elongation, %	152	147	85	-	-
Set at Break, %	07	06	-	-	-
Hardness, Shore A	50	58	60	-	-
340 Hours					
100% Modulus, Lbs/In^2	-	-	-	-	-
Tensile Strength, Lbs/In^2	1170	1238	-	1120	947
Elongation, %	160	165	-	203	146
Set at Break, %	09	09	-	11	05
Hardness, Shore A	-	70	-	-	64
570 Hours					
100% Modulus, Lbs/In^2	-	-	-	-	-
Tensile Strength, Lbs/In^2	865	870	-	-	-
Elongation, %	135	132	-	-	-
Set at Break, %	06	06	-	-	-
Hardness, Shore A	63	70	-	-	-

Table 7

Physical Properties of Accelerated Litharge Compounds - Masterbatched

Poly FBA	100	100	100
Stearic Acid	1	1	1
MB PbO (Fine)*	4	3	3
Ethyl Zimate	2	1	1
HAF Black	35	-	35
SAF Black	-	35	-
Cure - All precured 5' at 300°F in oven and remilled.			
Press Cure -	15' at 310°	60' at 310°	60' at 310°
Oven Bake -	60' at 300°	4 hrs at 300°	4 hrs at 300°

Physical Properties

100% Modulus, Lbs/In^2	860	736	739
Tensile Strength, Lbs/In^2	1416	1710	1429
Elongation, %	250	438	383
Set at Break, %	27	77	54
Hardness, Shore A	-	72	72

Due to poor Permanent Set - Not further tested

* Extra fine sublimed Litharge

Table 8

Sodium Hydroxide Vulcanized Poly FBA

Recipe

Compound No.	456-41-1
Poly FBA	100
HAF Black	40
Finely Pulverized Sodium Hydroxide	1
Cure - 60' at 310°F	

Physical Properties

100% Modulus, Lbs/In^2	757
Tensile Strength, Lbs/In^2	1108
Elongation, %	254
Set at Break, %	25
Hardness, Shore A	72

This compound was made solely to gain additional information on possible cross linking methods for poly FBA. No further work carried out.

Table 9

Effect of Processing Aid on Poly FBA

Recipes

Compound No. 456-	87-1	87-2	257-87-1A*
Poly FBA	100	100	100
Stearic Acid	1	-	-
Paraffin	-	1	1
HAF Black	35	35	35
TETA	1	1	1
Cure	60' at 310°F		30' at 320°F

Original Properties

	87-1	87-2	257-87-1A*
100% Modulus, Lbs/In^2	347	335	304
Tensile Strength, Lbs/In^2	1240	1315	1407
Elongation, %	366	349	395
Set at Break, %	25	17	17
Hardness, Shore A	63	60	57

Air Oven Aged 70 Hrs at 350°F in Open Test Tubes

	87-1	87-2	257-87-1A*
100% Modulus, Lbs/In^2	-	364	312
Tensile Strength, Lbs/In^2	541	758	765
Elongation, %	327	328	405
Set at Break, %	51	34	35
Hardness, Shore A	83	74	52

* Compound 257-87-1 was made from later batch of poly FBA than the others. Slight differences in method of producing polymer produced noticeable changes in aging characteristics.

Table 10

Effect of Variation of Amine and Sulfur Content in Poly FBA

Compound No. 456-	74-1	74-2	74-3	74-4
Poly FBA	100	100	100	100
Stearic Acid	1	1	1	1
HAF Black	35	35	35	35
Sulfur	.75	1.5	1.5	2
TETA	.75	1.5	1	1.5

Cured - 60' at 310°F after 24 hour Rest

Physical Properties

Original

	74-1	74-2	74-3	74-4
100% Modulus, Lbs/In^2	335	909	407	820
Tensile Strength, Lbs/In^2	1151	1325	1222	1307
Elongation, %	366	179	312	181
Set at Break, %	25	11	19	14
Hardness, Shore A	56	74	62	75

Aged in Air Oven 70 Hours at 350°F

	74-1	74-2	74-3	74-4
100% Modulus, Lbs/In^2	Yield Point	-	417	-
Tensile Strength, Lbs/In^2	369	747	516	667
Elongation, %	276	105	214	105
Set at Break, %	45	17	33	17
Hardness, Shore A	80(Plastic)	85	82	88

Table 10 (Cont'd)

Effect of Variation of Amine and Sulfur Content on Poly FBA

Compound No. 456-	74-1	74-2	74-3	74-4
Aged at 350°F in MIL-L-7808 Synthetic Oil in Open Test Tubes				
100 Hours				
100% Modulus, Lbs/In^2	473	-	591	-
Tensile Strength, Lbs/In^2	1025	1232	967	1188
Elongation, %	240	97	205	90
Set at Break, %	15	08	10	08
Hardness, Shore A	52	75	66	77
250 Hours				
100% Modulus, Lbs/In^2	427	-	551	-
Tensile Strength, Lbs/In^2	867	971	973	1106
Elongation, %	263	62	203	100
Set at Break, %	18	05	16	10
Hardness, Shore A	53	87	68	79
500 Hours				
100% Modulus, Lbs/In^2	390	(Fails 180° Bend)	461	-
Tensile Strength, Lbs/In^2	580	844	646	-
Elongation, %	252	59	197	-
Set at Break, %	49	10	31	-
Hardness, Shore A	77	91	78	-

Table 11

Two Poly FBA Compounds - (1) Effect of Tetrone A
(2) A General Purpose Good Hardness Compound

	Recipes	
Compound No.	456-91-1	257-86-1
Poly FBA	100	100
Paraffin	1	1
Sulfur	-	1
Tetrone A	2	-
HAF Black	35	40
TETA	1	1.25
Cure	60' at 310°F	30' at 320°F
Original Properties		
100% Modulus, Lbs/In^2	253	417
Tensile Strength, Lbs/In^2	1175	1133
Elongation, %	470	249
Set at Break, %	30	15
Hardness, Shore A	-	67
Air Oven Aged 70 Hours at 350°F		
100% Modulus, Lbs/In^2	544	-
Tensile Strength, Lbs/In^2	897	785
Elongation, %	234	253
Set at Break, %	26	25
Hardness, Shore A	78	66
Aged at 350°F in MIL-L-7808 Synthetic Oil		
	250 Hours	100 Hours
100% Modulus, Lbs/In^2	897	-
Tensile Strength, Lbs/In^2	1021	1070
Elongation, %	138	158
Set at Break, %	13	08
Hardness, Shore A	78	68
	500 Hours	500 Hours
100% Modulus, Lbs/In^2	-	-
Tensile Strength, Lbs/In^2	871	718
Elongation, %	90	157
Set at Break, %	16	22
Hardness, Shore A	82	70

Table 12

Various Aliphatic Amines as Curing Agents for Poly FBA

Recipes

Compound No. 2707-	1-1	1-2	1-3	1-4	1-5
Poly FBA	100	100	100	100	100
Paraffin	1	1	1	1	1
Sulfur	1	1	1	1	1
HAF Black	35	35	35	35	35
DETA	1.25	-	-	-	-
TEPA	-	1.00	1.50	-	-
Poly Amine H	-	-	-	1.25	-
Poly Amine T	-	-	-	-	2
Cure - 30' at 320°F					

Physical Properties

	1-1	1-2	1-3	1-4	1-5
100% Modulus, Lbs/In^2	343	225	332	247	320
Tensile Strength, Lbs/In^2	1024	1082	1166	1127	1035
Elongation, %	212	390	244	404	362
Set at Break, %	06	14	09	15	36
Hardness, Shore A	58	48	53	55	55

Table 13

Highly Loaded FBA Compounds

Compound No. 257-	Recipes 43-1	43-2	43-3
Poly FBA	100	100	100
Paraffin	1	1	1
Sulfur	1	1	1
TETA	1.25	1.25	1.25
Thermax (MT) Black	60	80	100
Cure - 30' at 320°F			

Physical Properties

	43-1	43-2	43-3
100% Modulus, Lbs/In^2	431	515	638
Tensile Strength, Lbs/In^2	650	698	696
Elongation, %	195	239	168
Set at Break, %	03	05	04
Hardness, Shore A	56	63	73

Table 14

Effect of Two (2) Non Furnace Black Fillers on Poly FBA

Recipes

Compound No.	456-86-1	257-15-2
Poly FBA	100	100
Paraffin	-	1
Stearic Acid	1	-
Sulfur	1	1
TETA	1	1
Aerosil	-	35
Alkaline Channel Black	35	-
Cure - 60' at 310°F		

Original Properties

	456-86-1	257-15-2
100% Modulus, Lbs/In^2	377	639
Tensile Strength, Lbs/In^2	1343	1178
Elongation, %	377	196
Set at Break, %	33	10
Hardness, Shore A	63	73

Air Oven Aged 70 Hours at 350°F

	456-86-1	257-15-2
100% Modulus, Lbs/In^2	525	-
Tensile Strength, Lbs/In^2	562	995
Elongation, %	198	95
Set at Break, %	37	15
Hardness, Shore A	93	85

Aged in MIL-L-7808 Synthetic Oil at 350°F

	250 Hours	100 Hours
100% Modulus, Lbs/In^2	760	544
Tensile Strength, Lbs/In^2	1095	674
Elongation, %	181	140
Set at Break, %	17	02
Hardness, Shore A	75	53

	500 Hours	250 Hours
100% Modulus, Lbs/In^2	666	-
Tensile Strength, Lbs/In^2	861	578
Elongation, %	149	85
Set at Break, %	22	03
Hardness, Shore A	84	52

Table 15

Effect of Antioxidants on Properties of Poly FBA

Recipes

Compound No. 257-	61-1	86-2	67-2 (refer to Compound 49-2, Table 29 for control)
Poly FBA	100	100	100
Paraffin	1	1	1
Sulfur	1	1	1
FEF Black	45	45	45
TETA	1.25	1.25	1
Agerite Powder	2	-	-
Sulfasan R	-	1	2
Cure - 30' at 320°F			
Original Properties			
100% Modulus, Lbs/In^2	746*	438	469
Tensile Strength, Lbs/In^2	1092	1050	1102
Elongation, %	166	245	157
Set at Break, %	08	12	04
Hardness, Shore A	73	68	65
Air Oven Aged 70 Hours at 350°F			
100% Modulus, Lbs/In^2	461**	531	-
Tensile Strength, Lbs/In^2	580	855	965
Elongation, %	176	162	90
Set at Break, %	20	14	06
Hardness, Shore A	80	73	84
Aged at 350°F in MIL-L-7808 Synthetic Oil in Open Test Tubes			
100 Hours			
100% Modulus, Lbs/In^2	-	-	-
Tensile Strength, Lbs/In^2	993	1000	1283
Elongation, %	100	100	84
Set at Break, %	08	04	02
Hardness, Shore A	78	74	82
250 Hours			
100% Modulus, Lbs/In^2	-	-	-
Tensile Strength, Lbs/In^2	951	-	1137
Elongation, %	75	-	64
Set at Break, %	06	-	07
Hardness, Shore A	87	-	82
500 Hours			
100% Modulus, Lbs/In^2	-	-	-
Tensile Strength, Lbs/In^2	800	849	994
Elongation, %	74	92	60
Set at Break, %	11	09	04
Hardness, Shore A	85	75	88

* Compound 61-1 somewhat overcured.
** Compound 61-1 Aged 140 Hours.

Table 16*

Effect of Various Fluids on Typical Poly FBA Compounds
(Compound 456-30-1 - See Table 22)

	JP-4 Fuel 70 Hrs at 212°F	Hydrocarbon Reference Fuel 70/30 Isooctane/Toluene 70 Hrs at 212°F
Tensile Strength, Lbs/In^2	1070	560
Elongation, %	160	115
Set at Break, %	-	-
Hardness, Shore A	65	47
Volume Increase, %	10	28(14 at RT)

	Hydrocarbon Oil MIL-L-6081A 300 Hrs at 250°F	Synthetic Lubricant Poly Glycol base 300 Hrs at 350°F
Tensile Strength, Lbs/In^2	1114	655
Elongation, %	233	106
Set at Break, %	16	5
Hardness, Shore A	74	75
Volume Increase, %	3	14

* Tests run and recorded by W. W. Jackson, WADC, WCRTR-3.

Table 17

Effect of Precure Storage on Physical Properties of Poly FBA - Summer*

Compound No. 456-	1-1	1-2	1-3
Poly FBA	100	100	100
Stearic Acid	1	1	1
Sulfur	1	1	1
HAF Black	35	35	35
TETA	1	1.25	1.50

Extruded and Autoclave Cured 60' at 320°F

Physical Properties

	Immediate Cure			40 Hr Delay			72 Hr. Delay		
Compound No.	1-1	1-2	1-3	1-1	1-2	1-3	1-1	1-2	1-3
100% Modulus, Lbs/In^2	469	906	-	285	465	577	240	349	496
Tensile Strength, Lbs/In^2	1208	1083	1069	1114	1243	1162	1174	1187	1190
Elongation, %	225	128	83	357	262	195	407	309	217
Set at Break, %	14	09	10	22	13	12	19	15	15

* Test run in summer time in uncontrolled (non air conditioned) atmosphere.

Table 18

Effect of Precure Storage on Physical Properties of Poly FBA - Winter

Compound No. 257-	87-1	87-2	87-3
Poly FBA	100	100	100
Paraffin	1	1	1
Sulfur	1	1	1
HAF Black	35	35	35
TETA	1	1.35	1.70

Press Cured 30' at 320°F

Original Physical Properties

Conditions	24 Hrs Rest Before Cure			100 Hrs Rest Before Cure			200 Hrs Rest Before Cure		
Compound No.	87-1	87-2	87-3	87-1	87-2	87-3	87-1	87-2	87-3
100% Modulus, Lbs/In^2	303	468	750	240	389	730	300	414	648
Tensile Strength, Lbs/In^2	1407	1440	1244	1431	1518	1409	1379	1463	1598
Elongation, %	395	240	142	417	298	158	442	282	175
Set at Break, %	17	10	06	18	11	04	19	09	06
Hardness, Shore A	57	60	67	50	58	65	48	55	60

Table 19

Effect of Precure Storage on Air Oven Aging -
(for Recipes see Table 18)

Test Conditions	Aged in Forced Convection Oven 70 Hours at 350°F (Open Test Tubes)								
Compound No. 257-	87-1	87-2	87-3	87-1	87-2	87-3	87-1	87-2	87-3
Parts TETA	1	1.35	1.70	1	1.35	1.70	1	1.35	1.70
Precure Storage	24 Hours			100 Hours			200 Hours		
100% Modulus, Lbs/In^2	312	487	792	247	429	630	260	354	616
Tensile Strength, Lbs/In^2	765	1027	1045	693	987	1181	681	935	1167
Elongation, %	405	251	100	484	301	168	495	343	202
Set at Break, %	35	14	07	41	16	06	46	16	08
Hardness, Shore A	52	57	67	50	53	58	47	52	55

Table 20

Effect of Precure Storage on Synthetic Oil Aging
(for Recipes see Table 18)

Test Conditions			Aged in Open Test Tubes in Synthetic Oil (MIL-L-7808B) at 350°F for 100, 250, and 500 Hours								
Compound No. 257-			87-1	87-2	87-3	87-1	87-2	87-3	87-1	87-2	87-3
Parts TETA			1.0	1.35	1.7	1.0	1.35	1.7	1.0	1.35	1.7
Precure Storage			24 Hours			100 Hours			200 Hours		
100% Modulus, Lbs/In^2	AGING PERIOD - HOURS	100	422	653	-	348	614	-	364	617	-
		250	409	719	-	316	606	-	370	573	-
		500	331	628	180°Bend OK	296	542	180°Bend OK	266	542	180°Bend OK
Tensile Strength, Lbs/In^2		100	1177	1138	962	1267	1107	961	1222	1206	1102
		250	984	1002	832	1007	1135	996	1017	1143	1090
		500	741	879	814	660	900	874	628	933	968
Elongation, %		100	243	152	97	294	170	93	310	159	91
		250	236	131	76	281	166	79	302	174	88
		500	246	142	70	305	176	82	377	167	96
Set at Break, %		100	08	06	04	08	06	05	08	06	04
		250	15	06	04	08	10	04	13	-	04
		500	18	08	05	21	07	05	28	06	-
Hardness, Shore A		100	51	57	68	42	52	62	42	52	60
		250	49	61	73	46	56	67	45	52	65
		500	53	58	73	47	55	68	43	55	63

Table 21

Effect of Atmosphere during Precure Storage on Cured Properties

Recipe

Compound No.	257-90-1
Poly FBA	100
Paraffin	1
Sulfur	1
TETA	1.25
HAF Black	35

Cured 30' at 320°F

Physical Properties

Precure Storage	24 Hours in Air	180 Hours in Air	180 Hours Wrapped in Polyethylene
100% Modulus, Lbs/In^2	428	338	354
Tensile Strength, Lbs/In^2	1473	1484	1561
Elongation, %	301	344	315
Set at Break, %	16	15	15
Hardness, Shore A	53	40	43

Table 22

Aging Poly FBA in MIL-L-7808 Synthetic Oil at 350°F

Compound No. 456-	30-1	30-2	30-3
Poly FBA	100	100	100
Stearic Acid	1	1	1
Sulfur	1	1	1
HAF Black	35	-	-
SAF Black	-	35	35
TETA	1	1	1.25
Cured - 60' at 310°F			
Original Physical Properties			
100% Modulus, Lbs/In^2	315	245	352
Tensile Strength, Lbs/In^2	1256	1154	1296
Elongation, %	301	450	389
Set at Break, %	09	31	32
Hardness, Shore A	52	53	63
Aged at 350°F in MIL-L-7808 Synthetic Oil in Open Test Tubes			
250 Hours			
100% Modulus, Lbs/In^2	587	402	629
Tensile Strength, Lbs/In^2	1060	1017	1081
Elongation, %	186	302	221
Set at Break, %	02	05	13
Hardness, Shore A	63	61	74
500 Hours			
100% Modulus, Lbs/In^2	526	357	539
Tensile Strength, Lbs/In^2	929	935	1018
Elongation, %	167	375	251
Set at Break, %	06	18	18
Hardness, Shore A	63	63	75
1000 Hours			
100% Modulus, Lbs/In^2	440	295	466
Tensile Strength, Lbs/In^2	935	696	869
Elongation, %	221	494	303
Set at Break, %	04	59	42
Hardness, Shore A	63	57	70

Table 23

Effect of Aging Poly FBA in MIL-L-7808 Synthetic Lubricant at 400°F

Recipes

Compound No. 456-	13-1	13-2	30-3
Poly FBA	100	100	100
Stearic Acid	1	1	1
Sulfur	1	1	1
HAF Black	35	-	-
SAF Black	-	35	35
TETA	1	1	1.25

Cure - 60' at 320°F (Autoclave)

	13-1	13-2	30-3
Original Properties			
100% Modulus, Lbs/In^2	286	348	352
Tensile Strength, Lbs/In^2	1345	1312	1296
Elongation, %	297	395	395
Set at Break, %	08	22	32
Hardness, Shore A	56	63	63
Aged 100 Hours			
100% Modulus, Lbs/In^2	299	346	633
Tensile Strength, Lbs/In^2	511	564	805
Elongation, %	198	355	161
Set at Break, %	19	62	18
Hardness, Shore A	60	60	68
Aged 175 Hours			
100% Modulus, Lbs/In^2	232	277	602
Tensile Strength, Lbs/In^2	441	355	779
Elongation, %	217	366	172
Set at Break, %	19	98	20
Hardness, Shore A	56	58	73
Aged 265 Hours			
100% Modulus, Lbs/In^2	232	252	529
Tensile Strength, Lbs/In^2	348	315	620
Elongation, %	194	345	142
Set at Break, %	20	96	18
Hardness, Shore A	52	60	70

Table 24

Effect of Aging Poly FBA in Silicate Ester Fluid (MLO-5277) at 400°F*

Compound No. 456-	30-1	30-2	45-3
Recipes			
Poly FBA	See Table 22		100
Stearic Acid			1
Sulfur			1
SAF Black			35
TETA			1.5
Cure			60' at 310°F
Original Physical Properties			
100% Modulus, Lbs/In^2	See Table 22		771
Tensile Strength, Lbs/In^2			1293
Elongation, %			208
Set at Break, %			33
Hardness, Shore A			81
Aged 50 Hours			
100% Modulus, Lbs/In^2	240	501	-
Tensile Strength, Lbs/In^2	683	721	-
Elongation, %	230	211	-
Set at Break, %	13	18	-
Hardness, Shore A	57	69	-
Aged 100 Hours			
100% Modulus, Lbs/In^2	238	-	805
Tensile Strength, Lbs/In^2	583	511	1022
Elongation, %	244	148	190
Set at Break, %	19	19	32
Hardness, Shore A	52	73	85
Aged 175 Hours			
100% Modulus, Lbs/In^2	190	337	-
Tensile Strength, Lbs/In^2	307	397	-
Elongation, %	214	185	-
Set at Break, %	13	35	-
Hardness, Shore A	52	71	-

* Samples taken from molded and extruded stocks.

Table 25

Effect of Aging Poly FBA at 500°F in MIL-L-7808 Synthetic Oil and in Silicate Ester (MLO-5277) (Samples Taken From Extruded Stock)

Compound No. 456-45-3
(For Recipe and Properties - See Table 24)

Aged 8 Hours

	MIL-L-7808 Oil	MLO-5277 Fluid
100% Modulus, Lbs/In^2	715	569
Tensile Strength, Lbs/In^2	771	780
Elongation, %	145	181
Set at Break, %	18	32
Hardness, Shore A	75	75

Aged 16 Hours

	MIL-L-7808 Oil	MLO-5277 Fluid
100% Modulus, Lbs/In^2	498	296
Tensile Strength, Lbs/In^2	543	360
Elongation, %	153	191
Set at Break, %	38	30
Hardness, Shore A	75	64

Aged 24 Hours

	MIL-L-7808 Oil	MLO-5277 Fluid
100% Modulus, Lbs/In^2	448	225
Tensile Strength, Lbs/In^2	495	246
Elongation, %	141	157
Set at Break, %	38	15
Hardness, Shore A	72	57

Table 26

Effect of Aging Poly FBA at 550°F in Silicate Ester MLO-8200

Compound No. 456-45-3
(For Recipe and Properties - See Table 24)
(Samples taken from Molded Stock)

Aged 3 Hours*

100% Modulus, Lbs/In^2	549
Tensile Strength, Lbs/In^2	731
Elongation, %	218
Set at Break, %	54
Hardness, Shore A	83

Aged 5 Hours*

100% Modulus, Lbs/In^2	-
Tensile Strength, Lbs/In^2	320
Elongation, %	379
Set at Break, %	114
Hardness, Shore A	70

* Aged in lightly stoppered test tubes to reduce evaporation and fire hazard.

Table 27
(See Table 30 for further information)

Effect of Compounding Variations on Compression Set
(70 Hours at 250°F - 30% Compression)

Compound No. 257-	18-1	18-2	18-3	19-1
Poly FBA	100	100	100	100
Paraffin	1	1	1	1
Sulfur	1	1	1	1
TETA	1	1	1.25	1.50
HAF Black	25	25	25	25
Cured - 60' at 310°F	same day	one day rest	same day	same day
Physical Properties				
100% Modulus, Lbs/In^2	235	192	269	410
Tensile Strength, Lbs/In^2	1336	1181	1227	1094
Elongation, %	350	373	263	196
Set at Break, %	14	7	7	6
Hardness, Shore A	50	43	43	54
Compression Set, % (70 hrs at 250°F 30% Compression after 24 hours Temper at 300°F 1/2 size discs)	20.9 (14.6)	22.0 (16.1)	17.1 (13.5)	31.1 (17.2)
Hardness, Shore A	(50)	(41)	(49)	(53)

(Parenthetical figures represent additional 30' recovery at 300°F)

Table 28
(See Table 31 for further information)

Effect of Compounding Variations on Compression Set
(70 Hours at 250°F - 30% Compression)

Compound No. 257-	19-2	23-1	23-2	25-1	49-1
Poly FBA	100	100	100	100	100
Paraffin	1	1	1	1	1
Sulfur	1	0.5	0.5	-	1
TETA	1	1	1.25	1.25	-
Trimene Base	-	-	-	-	2.5
HAF Black	-	35	25	25	-
FEF Black	35	-	-	-	35
Cured	60' at 310°F after 1 Day Rest				30' at 320°F
Physical Properties					
100% Modulus, Lbs/In^2	235	373	485	692	418
Tensile Strength, Lbs/In^2	1224	1248	1342	1136	1188
Elongation, %	366	312	223	174	287
Set at Break, %	10	12	3	3	8
Hardness, Shore A	44	62	65	63	62
Compression Set, % (70 hrs at 250°F 30% Compression after 24 hours Temper at 300°F 1/2 size discs)	27.9 (14.8)	29.9 (20.7)	23.7 (13.9)	20.7 -	35.3 (22.5)
Hardness, Shore A	(44)	(57)	(54)	62	(71)

(Parenthetical figures represent additional 30' recovery at 300°F)

Table 29
(See Table 32 for further information)

Effect of Compounding Variations on Compression Set
(70 Hours at 250°F - 30% Compression)

Compound No. 257-	49-2	49-3	55-1	55-2	55-3
Poly FBA	100	100	100	100	100
Paraffin	1	1	1	1	1
Sulfur	1	1	1	1	1
TETA	1.25	1.25	-	1	1.25
Trimene Base	-	-	2	-	-
FEF Black	45	35	45	45	-
SAF Black	-	-	-	-	25
Agerite Powder	-	-	-	2	-
Cure	----30' at 320°F------		--------- 60' at 310°F -----------		

Physical Properties

	49-2	49-3	55-1	55-2	55-3
100% Modulus, Lbs/In^2	426	243	319	304	238
Tensile Strength, Lbs/In^2	1016	1125	813	1023	1145
Elongation, %	256	303	388	348	567
Set at Break, %	5	3	17	10	22
Hardness, Shore A	68	56	55	50	42
Compression Set, %	32.7	34.7	49.3	36.8	25.9
(70 hrs at 250°F	(25.4)	(26.1)			
30% Compression after 24 hours Temper at 300°F 1/2 size discs)	20.0% (Tested same as others except using standard **1/2"** thick discs)				
Hardness, Shore A	(69)	(59)	66	57	65

(Parenthetical figures represent an additional 30' recovery at 300°F)

Table 30

Aging Characteristics of Compression Set Studies

Compound No. 257-	18-1	18-2	18-3	19-1
Oven Aged 70 Hours at 350°F in Open Test Tubes				
100% Modulus, Lbs/In^2	253	150	321	506
Tensile Strength, Lbs/In^2	828	855	882	872
Elongation, %	325	450	267	177
Set at Break, %	15	16	12	06
Hardness, Shore A	48	40	52	56
Aged at 350°F in MIL-L-7808 Synthetic Oil in Open Test Tubes				
100 Hours				
100% Modulus, Lbs/In^2	378	281	586	-
Tensile Strength, Lbs/In^2	1051	1150	910	883
Elongation, %	190	260	164	110
Set at Break, %	05	05	05	05
Hardness, Shore A	53	45	54	58
250 Hours				
100% Modulus, Lbs/In^2	605	361	710	(180° Bend - OK)
Tensile Strength, Lbs/In^2	1059	1158	1010	916
Elongation, %	168	252	146	95
Set at Break, %	07	06	06	08
Hardness, Shore A	54	45	55	65
500 Hours				
100% Modulus, Lbs/In^2	470	363	586	(180° Bend - OK)
Tensile Strength, Lbs/In^2	912	912	894	838
Elongation, %	193	220	153	63
Set at Break, %	05	04	07	05
Hardness, Shore A	56	47	58	68

Table 31

Aging Characteristics of Compression Set Studies

Compound No. 257-	19-2	23-1	23-2	25-1	49-1
Oven Aged 70 Hours at 350°F in Open Test Tubes					
100% Modulus, Lbs/In^2	158	251	582	628	481
Tensile Strength, Lbs/In^2	654	517	970	881	888
Elongation, %	425	402	169	143	219
Set at Break, %	13	46	05	04	12
Hardness, Shore A	47	65	56	53	55
Aged at 350°F in MIL-L-7808 Synthetic Oil in Open Test Tubes					
100 Hours					
100% Modulus, Lbs/In^2	285	502	-	-	(180° Bend - OK)
Tensile Strength, Lbs/In^2	1145	1123	983	803	1067
Elongation, %	246	211	97	88	$<$ 100
Set at Break, %	06	06	03	02	-
Hardness, Shore A	47	61	63	66	82
250 Hours					
100% Modulus, Lbs/In^2	497	623	(Fails 180° Bend)	-	(Fails 180° Bend)
Tensile Strength, Lbs/In^2	1093	1088	927	880	1050
Elongation, %	228	190	$<$ 100	85	45
Set at Break, %	05	08	05	05	06
Hardness, Shore A	48	68	71	76	93
500 Hours					
100% Modulus, Lbs/In^2	327	531	(180° Bend - OK)	-	-
Tensile Strength, Lbs/In^2	942	845	865	773	817
Elongation, %	251	205	$<$ 100	70	$<$ 50
Set at Break, %	12	18	05	03	-
Hardness, Shore A	51	70	71	77	97

Table 32

Aging Characteristics of Compression Set Studies

Compound No. 257-	49-2	49-3	55-1	55-2	55-3
Oven Aged 70 Hours at 350°F in Open Test Tubes					
100% Modulus, Lbs/In^2	360	260	260	382	245
Tensile Strength, Lbs/In^2	804	890	429	742	575
Elongation, %	261	332	425	343	612
Set at Break, %	08	06	62	19	52
Hardness, Shore A	50	35	-	-	-
Aged at 350°F in MIL-L-7808 Synthetic Oil in Open Test Tubes					
100 Hours					
100% Modulus, Lbs/In^2	667	545	-	554	333
Tensile Strength, Lbs/In^2	979	870	850	1062	1108
Elongation, %	147	165	95	197	346
Set at Break, %	05	04	10	06	07
Hardness, Shore A	62	50	82	62	48
250 Hours					
100% Modulus, Lbs/In^2	771	599	-	516	367
Tensile Strength, Lbs/In^2	994	975	772	843	869
Elongation, %	145	147	84	185	230
Set at Break, %	04	03	08	06	11
Hardness, Shore A	70	55	85	62	52
500 Hours					
100% Modulus, Lbs/In^2	750	499	-	462	367
Tensile Strength, Lbs/In^2	862	802	787	759	760
Elongation, %	120	155	<100	217	348
Set at Break, %	06	05	12	16	21
Hardness, Shore A	70	53	86	62	56

Table 33

Poly FBA
Resistance to Red Fuming Nitric Acid

Compound No. 456-	30-1	45-3	74-4
Poly FBA	100	100	100
Stearic Acid	1	1	1
Sulfur	1	1	2
HAF Black	35	-	35
SAF Black	-	35	35
TETA	1	1.5	1.5

Cured - 60' at 310°F

Original Properties

100% Modulus, Lbs/In^2	315	771	820
Tensile Strength, Lbs/In^2	1256	1293	1307
Elongation, %	301	208	181
Set at Break, %	09	33	14
Hardness, Shore A	52	81	75

Aged 70 Hours in RFNA (Room Temp.)

100% Modulus, Lbs/In^2	149	130	81
Tensile Strength, Lbs/In^2	579	592	492
Elongation, %	514	542	510
Set at Break, %	35	50	37
Hardness, Shore A	-	-	-

Aged 200 Hours in RFNA (Room Temp.)
then rinsed 24 hours in running water

100% Modulus, Lbs/In^2	346	256	323
Tensile Strength, Lbs/In^2	1278	1007	1163
Elongation, %	329	448	345
Set at Break, %	20	55	27
Hardness, Shore A	50	-	-

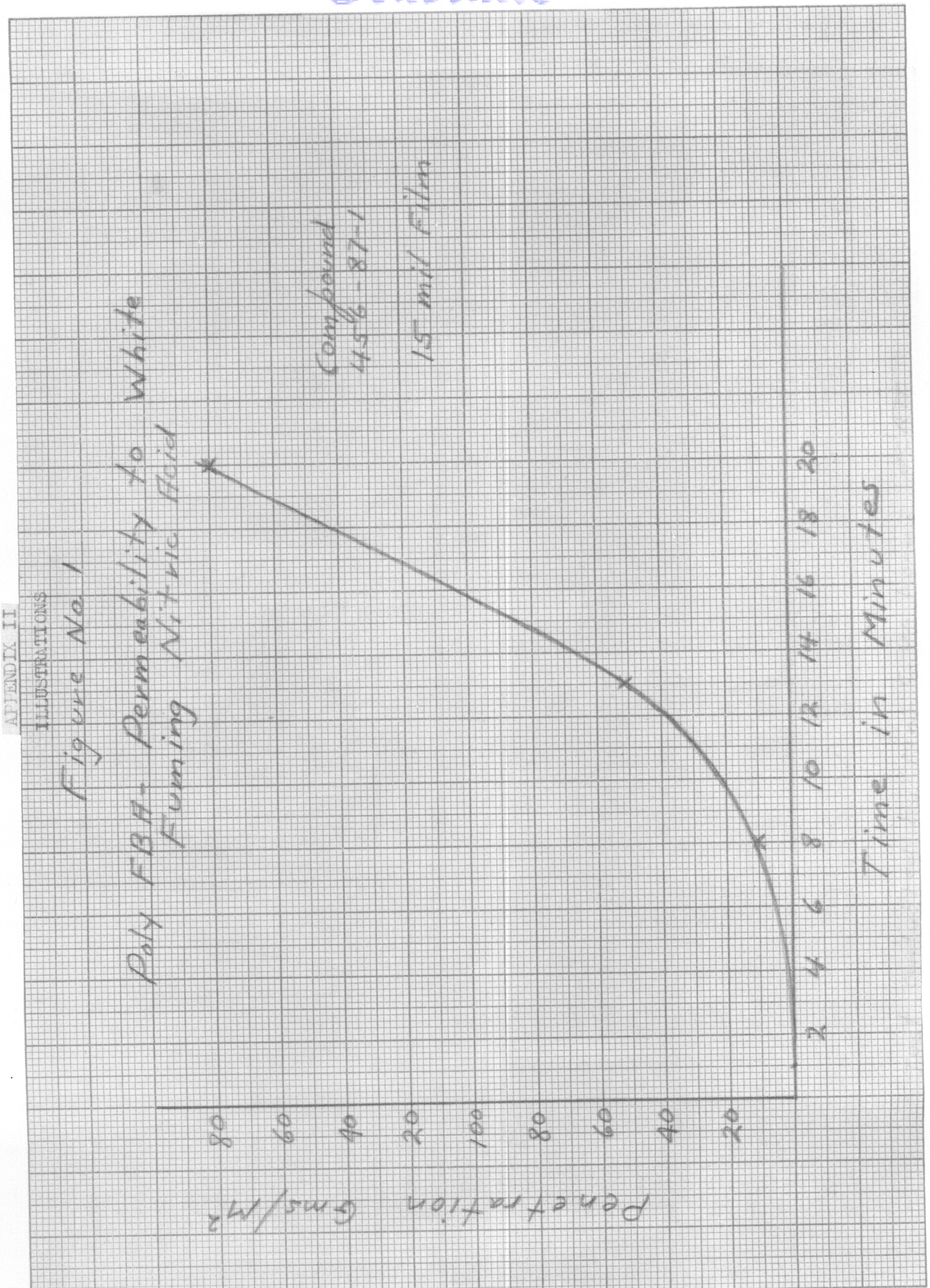
Figure No. 1
Poly FBA- Permeability to White Fuming Nitric Acid
Compound 4556-87-1
15 mil Film
2 4 6 8 10 12 14 16 18 20
Time in Minutes
20 40 60 80 100 20 40 60 80
Penetration Gms/M2

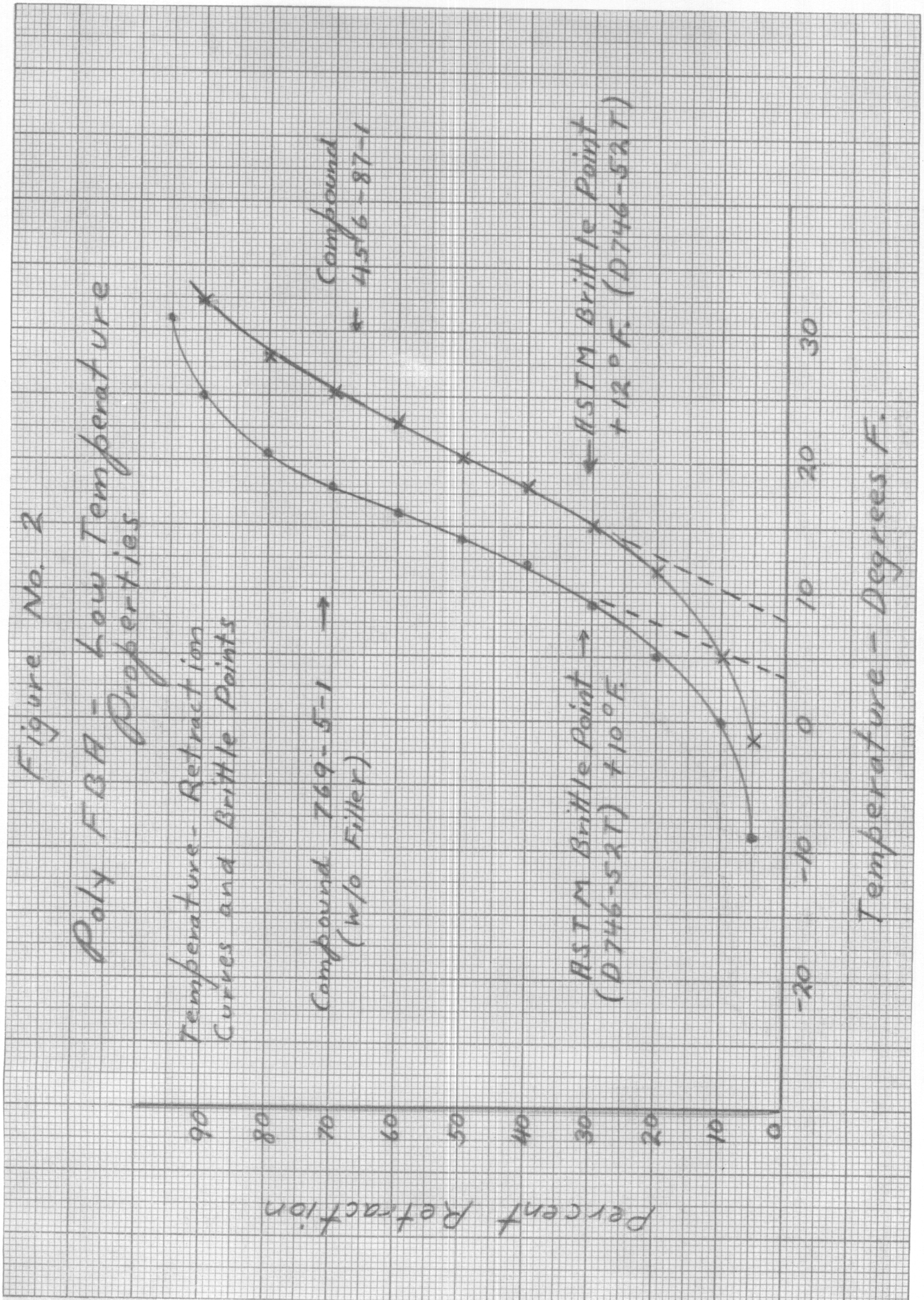

Figure No. 2
Poly FBA - Low Temperature Properties
Temperature - Retraction Curves and Brittle Points
Compound 769-5-1 (w/o Filler)
Compound 456-87-1
ASTM Brittle Point (D746-52T) +10°F.
ASTM Brittle Point +12°F. (D746-52T)
Percent Retraction
90
80
70
60
50
40
30
20
10
0
-20
-10
0
10
20
30
Temperature - Degrees F.

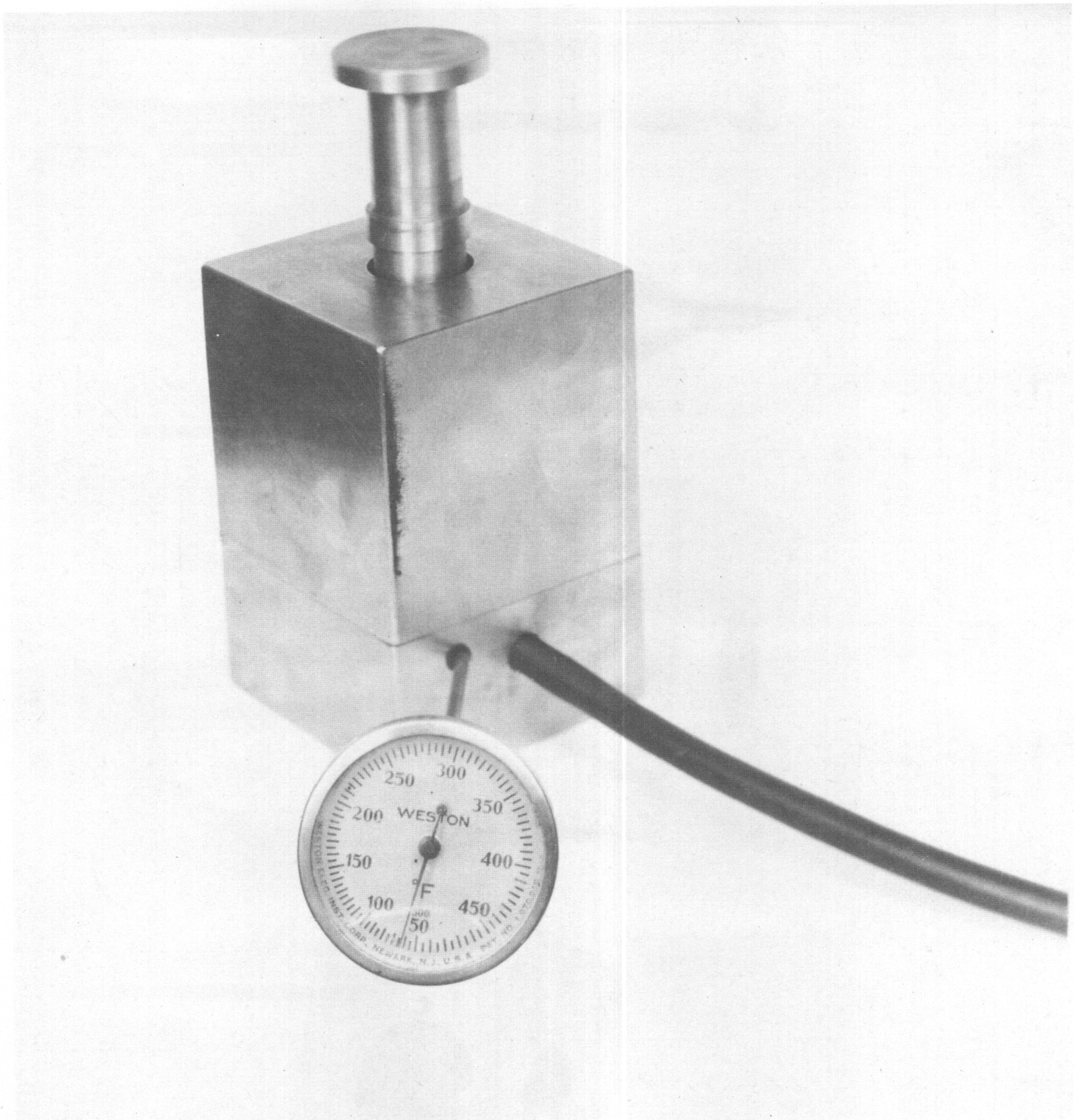

Figure 3 - Plunger Type Experimental Extruder - Assembled

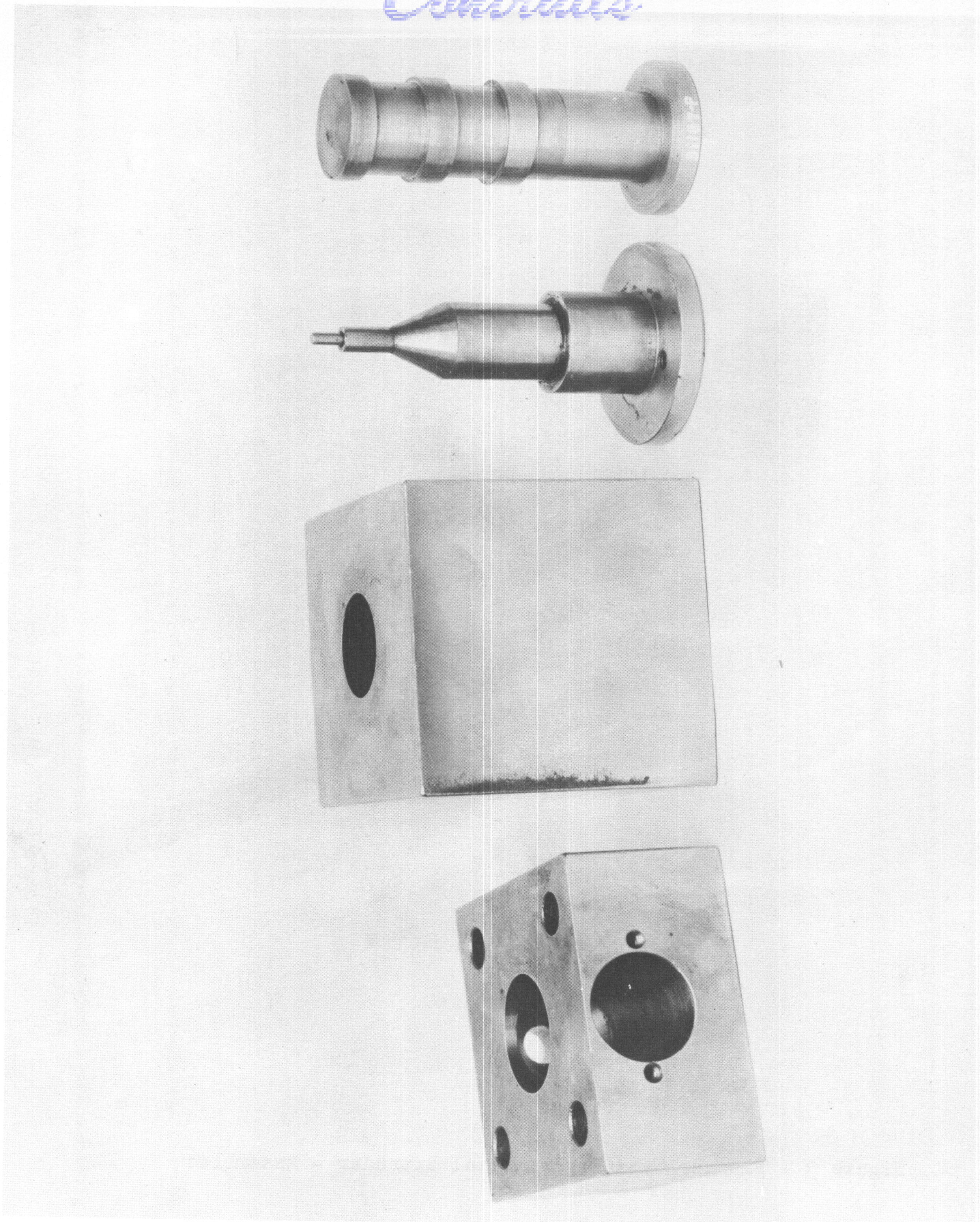

Figure 4 - Plunger Type Experimental Extruder - Disassembled

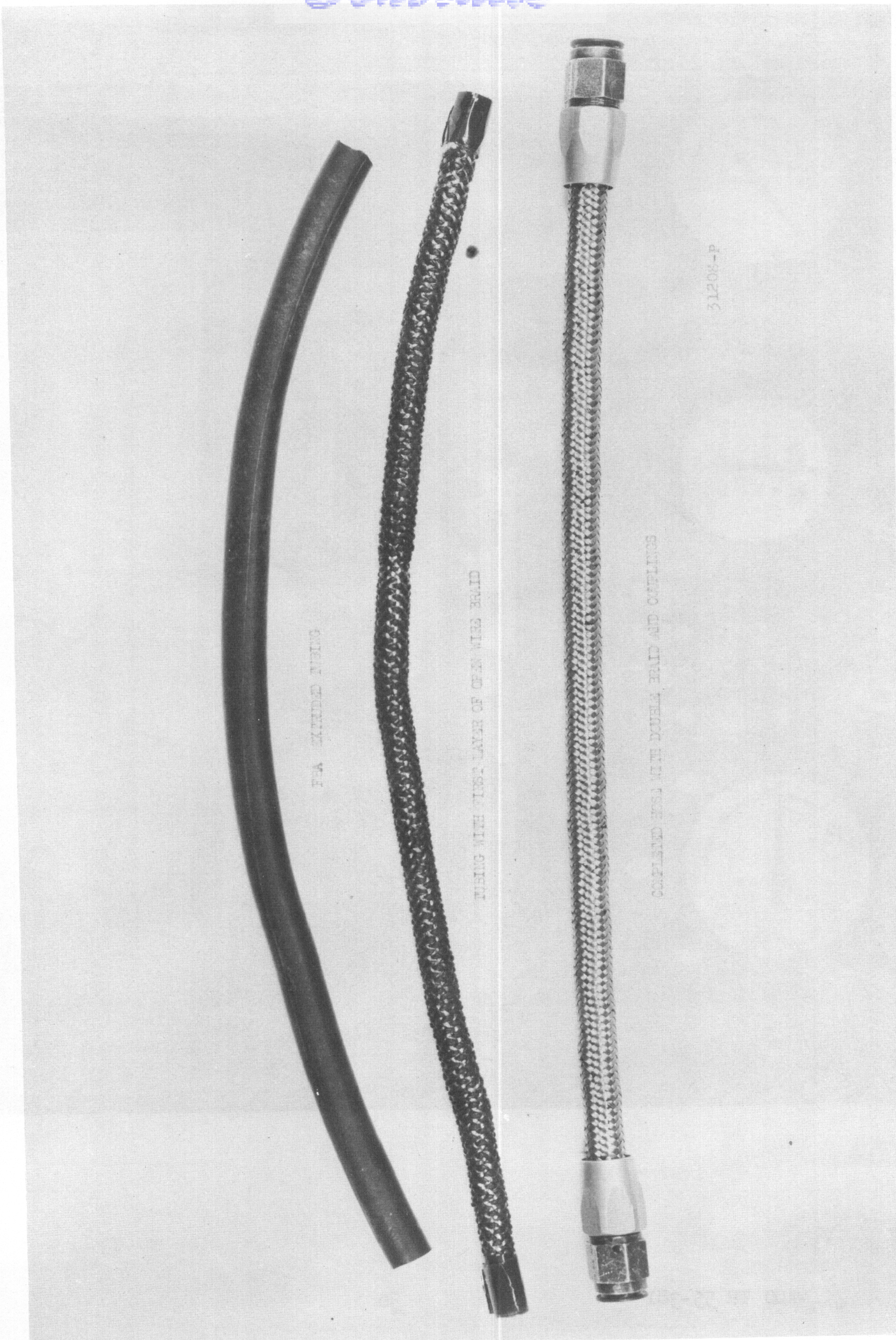

Figure 5 - Poly FBA Hose

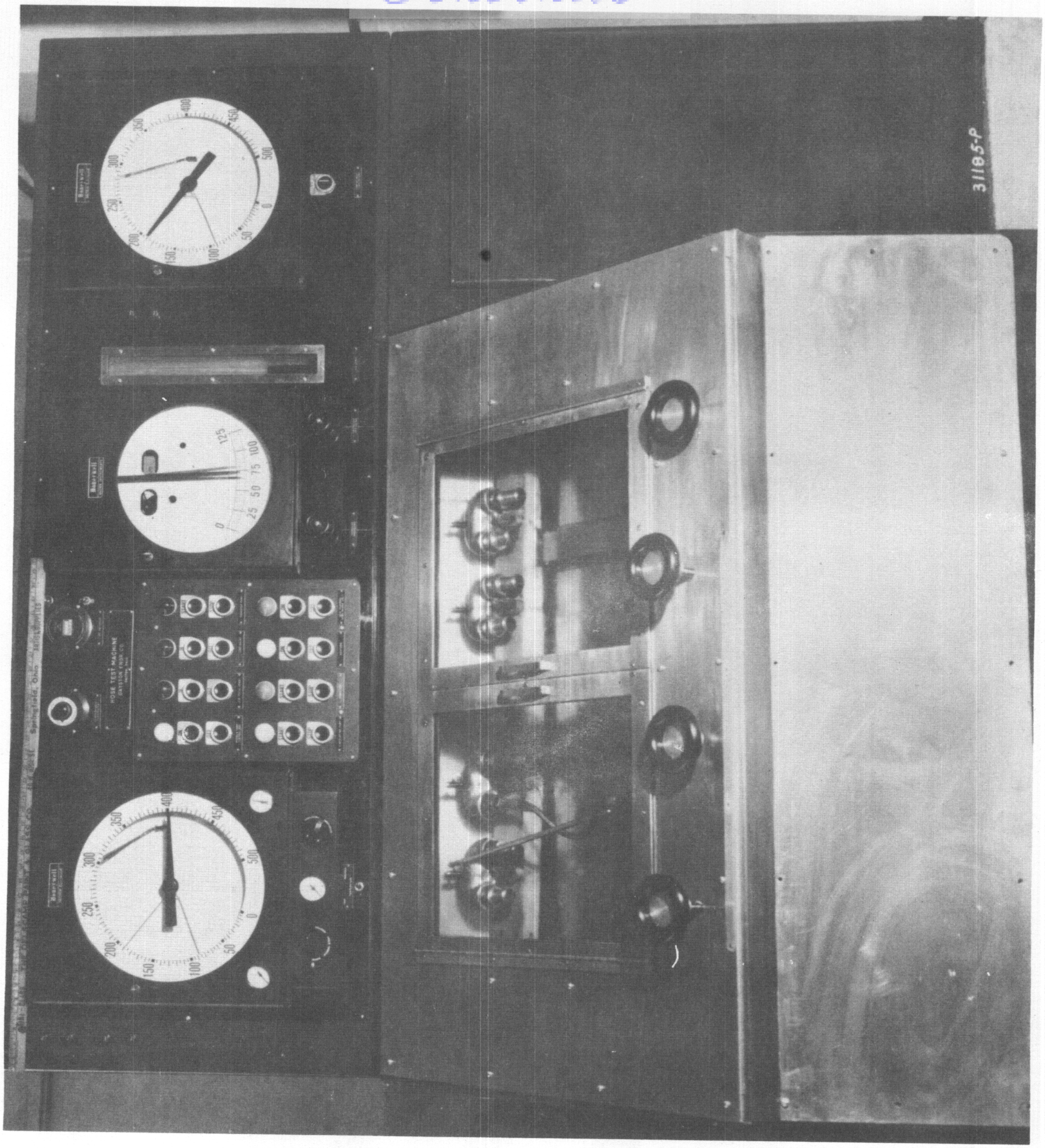

Figure 6 - Materials Laboratory Hose Tester

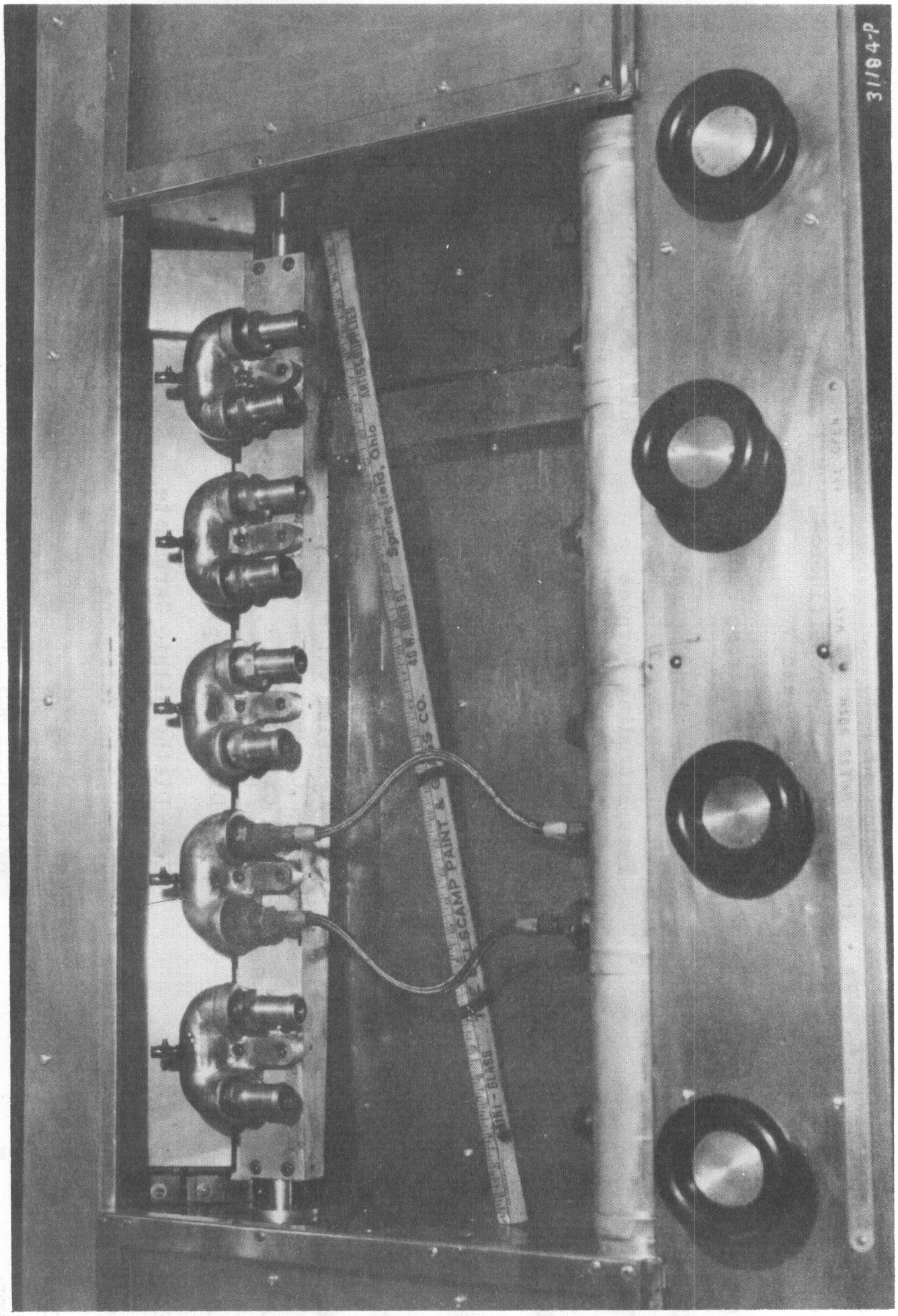

Figure 7 - Close Up View of Hose Test Chamber

APPENDIX III

List of Materials

Common Name or Symbol	Technical Name	Source
Poly FBA	Poly 1,1 dihydroperfluorobutyl Acrylate	Minnesota Mining & Mfg. Company
Polyac	Poly Para Dinitroso Benzene	E. I. duPont de Nemours & Co., Inc.
EPC Black	Easy Processing Channel Black	-
FEF Black	Fast Extruding Furnace Black (Philblack A)	Phillips Petroleum Company
HAF Black	High Abrasion Furnace Black (Philblack O)	Phillips Petroleum Company
SAF Black	Super Abrasion Furnace Black (Philblack E)	Phillips Petroleum Company
Texas 109 Black	Alkaline Channel Black	Sid Richardson Carbon Company
Magnesia	ELC Magnesium Oxide	-
Litharge	Lead Oxide (PbO)	-
Lead Peroxide	Lead Dioxide (PbO_2)	-
Ethyl Zimate	Zinc Diethyldithio Carbamate	R. T. Vanderbilt Company
TETA	Triethylene Tetramine	-
Trimene Base	-	Naugatuck Chemical Company
DETA	Diethylene Triamine	-

APPENDIX III

List of Materials (Cont'd)

Common Name or Symbol	Technical Name	Source
TEPA	Tetraethylene Pentamine	-
Polyamine H	-	Carbide & Carbon Chemical Company
Polyamine T	-	Carbide & Carbon Chemical Company
Agerite Powder	Phenyl Beta Naphthylamine	R. T. Vanderbilt Company
Sulfasan R	Morpholine Disulfide	Monsanto Chemical Company
Aerosil	Very Fine Silica	Godfrey L. Cabot Company

Note: The synthetic lubricant conforming to Specification MIL-L-7808 used in these tests was Esso Turbo Oil No. 15, a product of The Standard Oil Company of New Jersey.

The two "Silicate Esters" (MLO-5277 and MLO-8200) used in these tests were organo-silicon compounds developed under Air Force Contract by the California Research Corporation.