DEVELOPMENT OF HIGH-TEMPERATURE OIL-RESISTANT RUBBER

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FOREWORD

This report was prepared by Battelle Memorial Institute under Supplemental Agreement No. S2(54-1068) to USAF Contract No. AF 33(616)-476. The original contract 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 Mr. D. L. Byerley acting as project engineer.

This report is the second part of WADC Technical Report 54-190. The first part of this report was published in June 1955 under the basic report number only; it should be considered as Part I although it was not so marked.

WADC TR 54-190 Part 2

This report describes additional research directed toward the development of rubber compounds that can be used in diester-type lubricating oil (Turbo Oil-15) for long exposures at 350 to 550 F. The evaluation of experimental compounds was confined largely to 350 F, although a few tests were made at 400 F. Studies at Battelle showed that poly-FBA (poly-1,1-dihydroperfluorobutyl acrylate) can be compounded to meet the minimum target requirements of this project. Further work with the acrylate-type rubbers, Hycar 4021 and Acrylon EA-5, showed no substantial gain for this class of rubbers. Hycar 4021 still misses the target requirements only because of a swell that is 6 per cent above the 30 per cent maximum desired. Acrylon EA-5 appears inferior to Hycar 4021 for this application because of its tendency to crack during hot-oil aging tests. This cracking tendency is unusual, inasmuch as the material is a saturated polymer.

Further attempts have been made to overcome the only real shortcoming of high-nitrile rubbers (such as Hycar 1001) for this application — cracking after hot-oil aging. Although some short-term improvements have been effected by employing thiols and amines as antioxidants, all such gains have been lost by the end of 500 hours' aging. Methods for eliminating a portion of the polymer double bonds by hydrogenation are under consideration. The double bonds or hydrogen atoms on carbons alpha to the double bond are presumed to be focal points for oxidative degradation.
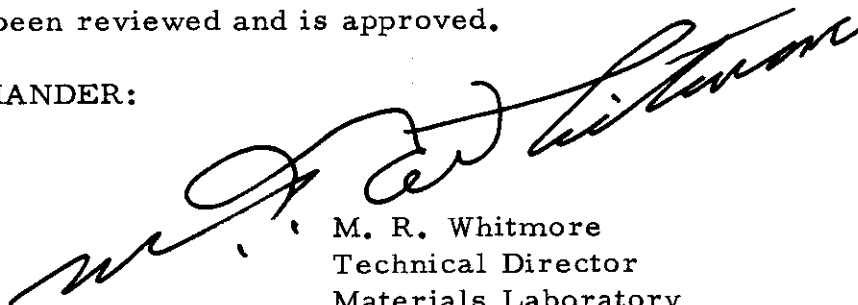
Two aluminum-block heaters for test-tube aging of rubber samples in oil at temperatures up to 500 F and 800 F, respectively, have been designed for WADC under this contract.

In future work on this basic problem, the emphasis will be shifted toward developing rubber compositions for use in synthetic hydraulic oils, such as the silicate esters.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

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

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INTRODUCTION

During recent years, the operating temperatures of aircraft have increased greatly because of increased speed and power requirements. This has necessitated a change in the type of lubricant and hydraulic oil used. It has been found necessary to use ester-type lubricants of the MIL-L-7808 type (such as Turbo Oil-15) and silicate ester-type hydraulic fluids (such as California Research No. 52742R). Unfortunately, both these types of fluid have been found to be very harmful to rubber-component parts at high temperatures.

This project was set up to develop rubber compositions that would be suitable for extended service at high temperature in synthetic lubricants and hydraulic fluids. The first objective was to develop a rubber composition that would withstand MIL-L-7808 lubricants (such as Turbo Oil-15) for at least 500 hours at 400 F. The second objective was to develop a rubber that would withstand silicate ester-type hydraulic fluids at 400 to 550 F. Minimum target properties for these two applications are given in Appendix I.

In working toward the first objective of this project, several approaches were possible. The most obvious approaches, and those receiving the major effort, were complete evaluations of commercial polymers and a Government-furnished experimental polymer, poly-FBA (i.e., poly-1,1-dihydroperfluorobutyl acrylate). Limited work also was done with a few experimental polymers, including some prepared at Battelle in connection with other research work.

Work on the first objective has been done in the past both at WADC and at Battelle. Battelle's previous work was reported in WADC Technical Report 54-190, dated April, 1954. Two commercial polymer types were found to be the most effective for use in Turbo Oil-15 at 350 F — the acrylate rubbers (Hycar 4021 and Acrylon EA-5) and the high-nitrile rubbers (such as Hycar 1001). The acrylate rubbers were found to retain physical properties very well after aging in hot oil, but swelled excessively. On the other hand, the nitrile rubbers displayed a higher degree of oil resistance, but were very susceptible to cracking when similarly aged.

Poly-FBA was developed at Minnesota Mining and Manufacturing Company for WADC. This polymer has been reported to meet all minimum target specifications for use in Turbo Oil-15 at 350 F. Because of limited availability and cost, this polymer has not been studied previously as extensively as the commercial polymers. Brief studies with this polymer, however, appear in this report.

Attention is just now shifting toward work with rubbers for resistance to synthetic hydraulic fluids. Although no work on this phase of the problem appears in this report, plans for future work in this direction are presented.

EXPERIMENTAL SECTION

Equipment and Procedures

Equipment

Aging of all samples was carried out in an aluminum block heater drilled to fit 38 x 200-mm test tubes. Details of these heaters were given in Appendix II to WADC TR 54-190. An improved design is also presented in Appendix IV of this report. Heat is supplied by a combination of continuous and intermittent heaters, so that a temperature of 350 or 400 ± 2 F was attained.

Procedures

Sample Preparation. Except for work with poly-FBA, ASTM Methods D15-52T and D412-51T were followed in the mixing, curing, and preparation of individual dumbbell-type specimens used for testing. For poly-FBA, mixing was carried out on a 2-1/2 x 7-inch mill fitted with adjustable guides. Curing was done in four-cavity molds, with individual cavities being 2 x 3 x 0.040 inch.

Hot-Oil Aging. The aging procedure consisted of suspending the rubber samples in 38 x 200-mm test tubes, adding Turbo Oil-15, and placing the test tubes in the aluminum-block heater for the desired length of time. At the beginning of this work, glass hangers that overlapped the top of the test tubes were used. Because of excessive breakage, these were replaced with 304 stainless steel (0.0475-inch diameter) wire, which has proven much more satisfactory. For each sample to be tested, two test tubes were used. One contained three dumbbell specimens for the stress-strain and hardness tests, and the second contained two samples approximately 1 x 1 inch for swelling and cracking determinations. In

evaluating poly-FBA, the dumbbell samples were used for all tests because of the limited amount of polymer. After the samples were suspended in the test tubes, 140 ml of Turbo Oil-15 was added and the tube covered with an inverted Petri dish. The cover fitted loosely and permitted the free entrance of air into the test tube. At the end of a given aging period, the tubes were removed from the aluminum block and allowed to cool for 1 hour. The rubber samples were then removed from the oil, dipped quickly in acetone to remove oil from the surface, and blotted dry before being tested.

Determination of Physical Properties. The stress-strain properties of all rubber specimens except poly-FBA were determined with a Scott Tensile Tester, Model L6, run at 20 inches per minute. Dumbbell samples were cut with Die C, ASTM Method D412-51T. To conserve material, poly-FBA dumbbell specimens were cut with a midget die having an overall length of 2 inches and a restricted section of 1/8 inch. This die is identical to a die currently in use at WADC. The loading required to test these small samples was below the range of the Scott Tester, so a Dillon Low-Range Tester was used at a speed of 20 inches per minute.

Hardness was determined by a Shore A2 Durometer, according to ASTM Method D314-52T.

Swelling was determined with a Kraus-Jolly balance. Per cent swelling was calculated by determining the relative volumes before and after aging.

The degree of cracking was rated by visual observation of 1 x 1-inch specimens flexed 180 degrees, using an arbitrary scale of (1) cracking, (2) crazing, and (3) no cracking.

Compression set was determined in both hot air and hot Turbo Oil-15. Set in hot air was determined according to ASTM Method D395-52T, Method B (Compression Set Under Constant Deflection). Details of the test method employed for determining compression set in Turbo Oil-15 at 350 F were given in pages 3 to 5 of WADC TR 54-190. In carrying out this test, one questionable feature developed. The test samples had a height of 0.225 inch (three plies from a standard 0.075-inch tensile sheet) and a diameter of 0.500 inch. Because of considerable swelling, it was found that the samples swelled around the spacers. To reduce this tendency, a smaller sample was investigated. Keeping the ratio of height to diameter constant, a sample of height 0.150 inch (two plies from a 0.075-inch tensile sheet) and a diameter of 0.333 inch was tried. Data are being accumulated on samples of both sizes, in order to decide which method should become standard. The test procedure employed is as follows:

(1) Disks are cut from 0.075-inch tensile sheet, either 0.333- or 0.500-inch diameter, and plied up, using either two or three disks. The total thickness of the sample is determined to the nearest 0.001 inch.

(2) The test specimens are placed between the plates of the compression device, along with spacers, and the assembly is tightened. The amount of initial compression on the sample depends upon the hardness, and is determined from the following table (from ASTM Method D395-52T):

Durometer Hardness	Deflection, per cent of original thickness
1 through 44	40
45 through 64	30
65 through 84	25
85 and over	20

(3) The assembly is then placed in a test tube. The tube is filled with Turbo Oil-15 and placed in the aluminum-block heater for the desired heating period.

(4) At the end of the test period, the test tubes are removed from the aluminum block and the samples removed from the test assembly. They are allowed to cool in air for 30 minutes.

(5) Final thickness is determined to the nearest 0.001 inch, and the compression set is determined from the following formula:

$$\text{Per cent compression set} = \frac{t_o - t_1}{t_o - t_s} \times 100 \; ,$$

where

t_o = initial thickness
t_1 = final thickness after cooling for 30 minutes
t_s = thickness of the spacers.

Low-temperature retraction tests were made according to ASTM Method D1329-54T, using an initial stretch of 55 per cent of the breaking elongation.

Compounding Studies With Nitrile-Type Rubber

In the period covered by the first annual report, extensive compounding studies with nitrile-type rubber were undertaken. The basic work was concerned with studies of polymers, fillers, curing systems, and curing conditions. It was found that under the test conditions used (350 F in Turbo Oil-15), the nitrile rubbers high in acrylonitrile content provided the best aging properties, and magnesia-filled compounds were superior to those

containing other fillers. Work with the better compounds was extended to include investigations of antioxidants, plasticizers, and processing oils, and studies were made of the effect of previously aged Turbo Oil-15 on the aging of the rubber.

Although the magnesia-filled compounds showed a marked improvement over the more common black-filled compounds, all of the acrylonitrile-type compositions evaluated were subject to surface hardening during aging. Then this hardened surface would crack when the samples were given a 180-degree flat bend. A few plasticizers and a number of the best commercial antioxidants were investigated in an effort to reduce this type of cracking, but with very little success. Some antioxidants even appeared to promote, rather than to retard, surface hardening.

At the beginning of the present report period, it was decided that the experimental effort with acrylonitrile-butadiene copolymers should be reduced, unless a novel method for preventing cracking could be found. Accordingly, a search was made of the literature to uncover unique methods for protecting an unsaturated rubber at far above the usual operating temperatures for rubbers of this type. Details concerning this literature survey can be found in Appendix II of this report. Some of the work with nitrile rubbers that appears in this report is the direct result of this survey. It will be observed that the base rubber composition employed for compounding studies with nitrile rubber is the previously developed magnesia-filled composition (Compound A-23). Although the compounding work with nitrile rubbers was confined largely to Hycar 1001, there is no intention to imply that other high-nitrile rubbers would not give comparable results.

The Effects of Thiols on Aging

It was recognized during this research project that the residual unsaturation in nitrile-rubber compounds probably is responsible for their cracking after hot-oil aging. One possible method for reducing this unsaturation (as discussed in the appended literature survey) is to add thiols to the double bonds. The results for several thiols investigated in this connection are shown in Table 1.* A few thiols produced undercures so serious that the compounding work necessary for their full evaluation was not justified.

In the early screening of thiols in nitrile rubber, small amounts of thiomalic acid and thiosalicylic acid improved the aging characteristics of the composition. This was evidenced in compounds aged 72 hours in Turbo Oil-15 at 350 F. For this short time period, "cracking" was reduced to "crazing", and the cracking that was found after 168 hours' aging appears to be somewhat less severe. Further improvement could not be obtained by employing larger amounts of these two thiols. When thiomalic acid was

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* The tables are grouped in Appendix III.

employed in amounts of approximately 50 per cent of that theoretically necessary to saturate the double bonds of the rubber, the compounds were extremely hard and cracking became worse, rather than better. Another equally futile attempt to prevent cracking of the rubber by using thiols is described in a subsequent section of this report.

Because of the limited gains made by thiols, it was not considered worthwhile to investigate the degree of saturation produced by this type of additive. Although the tendency for rubbers treated in this manner to stiffen may indicate the loss of some rubberlike properties through addition to double bonds along polymer chains, it is more probable that an objectionable amount of cross linking occurred, leaving an excess of double bonds still vulnerable to oxidation. For exposure to milder aging conditions, thiol treatment may offer some real advantage. However, under the conditions being studied here, the crack resistance imparted was far from adequate.

The Effects of Hot Milling on Hycar 1001

An attempt was made to improve the aging of Hycar 1001 by adding a thiol to the rubber during hot milling. For a control, the rubber was milled at 325 F for 15- and 30-minute periods with no compounding ingredients present. The same procedure was repeated with thiomalic acid added. The amount of this thiol employed was 50 per cent of that theoretically necessary to saturate the rubber. A reaction between the rubber and the thiol was quite apparent after only a few minutes of milling. After about 20 minutes, the mixture became so hard and dry that it would not stay on the mill.

The hot-milling experiment also demonstrated that Hycar 1001 alone tends to cure or cross link during such treatment, as the control likewise became dry and hard, though to a less extent. Although it was possible to keep the rubber alone on the mill for the full 30-minute period, it was rendered unprocessable by this treatment. It became too highly gelled to dissolve in solvent for an iodine-number determination.

The Effects of Sulfur- and Selenium-Containing Compounds on Aging

Some of the theories for antioxidant action described in the survey of literature (Appendix II) suggested that aromatic mercaptans and selenols, similar in structure to the phenolic-type antioxidants, might perform well at high temperatures. Unfortunately, mercaptans and selenols with such structures have extremely offensive odors. Aromatic disulfides, however, have been known to split at the S-S bond, thus forming free radicals identical

to those formed by the removal of hydrogen from the corresponding mercaptans. The disulfides have odors that are tolerable, and such materials are considerably easier to obtain commercially. A number of disulfides and one diselenide were evaluated as possible antioxidants in the magnesia-filled A-23 compound (Table 2). None of the materials tried appeared to improve aging. Cracking appeared to be accelerated, rather than retarded.

The Effects of Amine-Thiol Combinations on Aging

Further evidence of the antioxidant properties of thiols may be found in U.S. Patents 2,657,982 and 2,657,983. These patents claim that anti-oxidant systems containing combinations of amines and mercaptans (thiols) are much more effective as oxidation inhibitors for gasoline than are amines when used alone. Several combinations of an amine antioxidant (Flectol H) with mercaptans and disulfides were evaluated (Table 3). Combinations of Flectol H with thiomalic acid and thioglycolic acid produced the best retention of physical properties obtained thus far with nitrile-type rubber. However, none of the systems tried provided an adequate reduction in cracking.

The Effect of Chemical Compounds Related to Mercaptans and Amines

The trials given several compounds of selenium, phosphorus, arsenic, antimony, and bismuth were suggested by work done at Battelle for the United States Quartermaster Corps.* These elements belong to the same groups of the periodic table as oxygen and sulfur or nitrogen, and their chemical compounds usually have many similar properties. At temperatures of 194 to 302 F (90 to 150 C), selenols proved to be better modifiers for emulsion polymerization than the mercaptans. Although triphenyl phosphine did not modify polymerization as well as the selenols or mercaptans, it was thought possible that, in rubber at still higher temperatures, it might demonstrate a protective action. The results in Table 4 show that, although the aged physical properties of the rubber may have been improved slightly by these chemicals, cracking was not reduced.

The Effects of High Sulfur Level on an Unfilled Compound

The effects of various amounts of sulfur on the aging of unfilled Hycar 1001 are shown in Table 5. This most obvious method for saturating nitrile rubber had been neglected thus far. It is generally known that, during vulcanization, sulfur reacts in other ways besides the direct cross linking of

* "Synthetic Diene Elastomers Prepared at High Temperature", a Battelle Memorial Institute research project for the United States Quartermaster Corps under Contract No. DA-44-109-QM-708.

polymer chains. Various numbers of sulfur atoms can form ring structures across double bonds, or perhaps link two double bonds in the same polymer chain. Filler was omitted from the compounds in this series to compensate for the higher hardness, lower elongation, and higher modulus common to rubber compounds containing large amounts of sulfur. None of the compounds of this series showed improvement in crack resistance. The lack of filler, of course, contributed to a poor balance of physical properties, although this was a matter of secondary importance to cracking. It is interesting to note, however, that the best compound was obtained with a sulfur level intermediate between those used for so-called soft and hard rubbers (i.e., at about 10 phr), and that this amount of sulfur produced a rubber with essentially no swell. Perhaps reducing the penetration of oil into the rubber is one method for improving aging.

A Preliminary Evaluation of Hycar 1041

It was suggested by the B. F. Goodrich Chemical Company that Hycar 1041 might be more crack resistant than Hycar 1001 in compounds for high-temperature service. Hycar 1041 is a butadiene-acrylonitrile copolymer prepared at 5 C. The above suggestion was based on evidence that Hycar 1001 can be cured by heating without the addition of curatives (see section entitled "The Effects of Hot Milling on Hycar 1001"), whereas Hycar 1041 cannot. It was assumed that better high-temperature aging could be expected from a rubber that would not continue to cure when all of the added curatives had been depleted. A series of magnesia-filled compounds very similar to the A-23 formulation was prepared, using Hycar 1041 in place of Hycar 1001 (see Table 6). The most effective age-resisting agents found thus far for Hycar 1001 were used as additives in some of the compounds. Although the acrylonitrile-rubber contents of the two polymers are approximately the same, and both raw polymers contain the same commercial antioxidant, their cured compounds in this instance aged quite differently at the elevated test temperature. The control Hycar 1041 compound, i.e., without added antioxidant, was considerably inferior to a similar compound made with Hycar 1001. Flectol H appeared to have no effect whatever on the aging of Hycar 1041. The beneficial effects of thiomalic acid and thioglycolic acid, however, were much more pronounced in Hycar 1041 than they were in Hycar 1001 (Table 1). Even though all of the compounds cracked after 168 hours' exposure, limited additional work with Hycar 1041 was indicated.

Mercaptobenzimidazole as an Antioxidant

The improvements in age resistance produced by the use of mercapto-benzimidazole in rubber have been described by the French rubber chemist,

Jean Le Bras, in various patents and publications. This compound, similar in structure to mercaptobenzothiazole (the well-known rubber accelerator), was claimed to be most effective when used in combination with amine antioxidants in natural rubber.

One theory advanced for the antioxygenic behavior of mercaptobenzimidazole is that it slowly vulcanizes rubber during aging and thus partially neutralizes degradation reactions.* In the A-23 compound and under the test conditions employed at Battelle, the effects of mercaptobenzimidazole appear to be in disagreement with this theory. The results shown in Table 7 indicate that reversion of cure or depolymerization, or perhaps both, took place, even when small amounts of the compound were used. Cracking was not reduced.

The Effects of Aging Hycar 1001 in Di-(2-Ethylhexyl) Sebacate Obtained From Several Suppliers

The effects of di-(2-ethylhexyl) sebacate obtained from several different sources on the aging of the A-23 compound of Hycar 1001 are shown in Table 8. The variation in results obtained was also evident in similar studies with the acrylate polymers. For example, the Harchem dioctyl sebacate caused excessive swelling of both types of rubber. The data indicate that the swell of nitrile rubber in the Rohm and Haas and Pittsburgh Coke and Chemical diesters was lower than it was in Turbo Oil-15. Apparently the standard grades of dioctyl sebacate vary considerably among different manufacturers.

For convenience of comparison, the results of two other 500-hour tests on compounds of Hycar 1001 are also included in Table 8. These compounds contained two of the better antioxidant systems found to date. Because of the cracking failure of nitrile-rubber compounds relatively early in the aging process, very few compounds have been aged for the full 500-hour period. It is interesting to note that, with this compound, the early benefits derived from the mercaptan-amine combination of antioxidants appear to be completely lost after 500 hours of aging.

Compounding Studies With Acrylate Polymers

In the preceding annual report (WADC TR 54-190), it was shown that acrylate polymers, Hycar 4021 (a copolymer of ethyl acrylate and chloroethyl vinyl ether) and Acrylon EA-5 (a copolymer of ethyl acrylate and acrylonitrile), had considerable resistance to Turbo Oil-15 at 350 F. The best

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*Le Foll, J., Rubber Chem. Tech., 27, 157 (1954).

compositions were able to meet all of the target specifications after aging for 500 hours except the 30 per cent maximum swell. Therefore, during the current work, emphasis was placed on reducing the swelling.

Work was divided between Hycar 4021 and Acrylon EA-5, as earlier work had shown that each of these polymers had its merits. Hycar 4021 was usually excellent in its resistance to cracking, whereas Acrylon EA-5 was much more prone to cracking after hot-oil aging. On the other hand, Acrylon EA-5 had better swell resistance than Hycar 4021, though both were 5 to 10 per cent above the desired maximum.

This earlier work indicated fillers to be one of the important compounding variables, with Silene EF found to be the best of those evaluated. Consequently, considerable additional work was done with fillers, including more work with Silene EF, both alone and in combination with the Calcenes. Further work was done also with vulcanizing systems, nonextractable plasticizers, and experimental acrylate polymers, in following up leads obtained during the previous work. Some of the special studies presented in this report were done at the request of WADC, and others were made to clarify questionable points that arose concerning the testing procedures employed.

Compounding Studies With Hycar 4021

Silene EF as a Filler. It was indicated above that Silene EF was the most promising filler found for Hycar 4021. Consequently, work with this filler was extended, the current data being shown in Table 9. The results show that optimum aged properties were developed with a filler loading of about 70 phr of Silene EF. Increasing this loading to 81.7 phr produced a stock that had appreciably lower swell, but which cracked after aging. Attempts to reduce the original hardness and to lower the volume swell by employing an extractable plasticizer (10 phr Flexal R2H) were ineffective.

Attention is directed to the data for Recipe PA-95. This composition, containing 70 phr of Silene EF, was developed during the first year of research on this project. It is still the best produced on this program, as indicated by the results obtained after 500 hours' aging at 350 F. The minimum target specifications were met for all properties, with the exception of swell, which was about 6 per cent above the allowable maximum of 30 per cent.

Silene EF — Calcene Combinations as Fillers. The data in Table 9 indicate that Silene EF-filled Hycar 4021 stocks are quite promising, but failed to meet the target requirements because of excessive swelling. Application of the common method of reducing swell by increasing the filler content was not practical in the case of Silene EF, as an excessive hardness would be obtained. Therefore, an attempt was made to accomplish higher

loadings by replacing a part of the Silene EF with a less reinforcing filler material.

In Tables 10 and 11 are shown the results obtained by employing various combinations of two types of Calcene with Silene EF in the same base composition of Hycar 4021. Calcene TM is a coated calcium carbonate, and Calcene NC is an uncoated material of similar composition. It can be seen that higher loadings of the filler mixtures were needed to obtain the same initial hardness as was produced by Silene EF alone. However, the data in Table 10 show that, after aging, the stocks containing combinations of Silene EF with Calcene TM and Calcene TM alone were no better in swell resistance than stocks containing Silene EF alone. Since the increased filler loading did not reduce swelling, it is apparent that the polymer itself must have swelled more in the presence of the mixed filler, probably because of the lower degree of reinforcement.

The limited data on Calcene NC combinations, shown in Table 11, appear to indicate that mixtures of Silene EF and Calcene TM will perform no better than Silene EF alone.

Experimental Hi-Sil as a Filler. Work reported in WADC TR 54-190 indicated that Hi-Sil also was a promising filler for Hycar 4021. Vulcanizates containing this filler also met all minimum target specifications, except for swelling (which was about 15 per cent above the maximum allowable). Because of this, three experimental samples of Hi-Sil submitted by Columbia-Southern Chemical Corporation were evaluated. In this study, regular-production Hi-Sil was used as a control. In the first set of samples, a loading of 65 phr of Hi-Sil was used with no plasticizer. Since the original hardness of these stocks was higher than permitted, a second set of samples was prepared by using 10 phr of Flexol R2H along with the 65 phr of Hi-Sil.

Results in Table 12 show that none of the experimental Hi-Sil was superior to the regular Hi-Sil. Stocks containing the experimental Hi-Sil were somewhat more susceptible to cracking than those containing the regular Hi-Sil, although no significant differences could be noted in the results for other properties.

Miscellaneous Fillers. Several other fillers were evaluated in Hycar 4021, with the results shown in Table 13. The data for Recipes PA-208 and PA-209 show the result of using Du Pont Fine Silica as a filler. It was found that, for a given filler volume, Du Pont Fine Silica gave stocks with higher initial hardness and lower swell than did any other filler tried to date. For example, the data for stocks aged 168 hours at 350 F show that equivalent volumes of Hi-Sil or Silene EF produced stocks swelling about 40 to 45 per cent, compared with about 30 per cent for Du Pont Fine Silica. Even after the aging period was extended to 500 hours, the swell obtained for Du Pont Fine Silica stocks was about 20 per cent less than that obtained

for the Silene EF stocks, based on equivalent volumes of filler. However, there are two factors working against each other. The excessive stiffening contributed by the Du Pont Fine Silica dictates that the volume loading be kept low. The result is that a Du Pont Fine Silica stock will have a swell about equal to that of a Silene EF stock, compared on the basis of equal initial hardnesses. The Du Pont Fine Silica stocks of about 80 hardness also have the disadvantages that, after aging, they show excessively high hardness and low elongation, and are more prone to cracking.

Limited work is suggested with lower volume loadings of this filler in the future, as well as the auxiliary use of a nonextractable plasticizer, such as liquid nitrile or acrylate polymer. Although it is likely that initial hardness can be reduced in this manner, the extent to which swell and other aged properties will be affected by such changes in composition is not known.

Two samples were evaluated when using Calcene NC (a fine calcium carbonate) as the sole filler for Hycar 4021. Large volume loadings of this filler were accomplished without exceeding the allowable hardness of 80. Recipe PA-225 shows promise after aging for 168 hours, but no definite conclusions can be drawn until the aging has been extended to 500 hours.

Complete or partial substitution of thermal black, P-33, for a furnace black (Philblack A) also gave some interesting results. These data in Table 13 for Recipes PA-210, PA-211, and PA-212 show that high filler loadings of P-33 were tolerated without exceeding the initial hardness. After 500 hours' aging at 350 F, these stocks displayed somewhat low tensile strength and high swell, but the elongation was better than found for any other filler in Hycar 4021.

Further work is warranted with P-33 carbon black at higher filler loadings. Limited work also is advised for medium thermal black, a still less reinforcing carbon black.

Indulin A also was tested as a filler, as it is reported to contribute high elongation, low modulus, and good aging properties to unsaturated-type rubbers. In two of these batches, Indulin A was coprecipitated with Hycar 4521 Latex (a latex form of Hycar 4021). In the third batch, the Indulin A was dry milled into Hycar 4021 polymer. The data presented in Table 13 indicate that a high degree of polymer reversion occurred during aging when using the coprecipitated material. For these stocks, the aged tensile strength and hardness were low, and swelling was high. When the Indulin A was milled into the polymer, the aged properties of the rubber were even poorer. No further work seems desirable with Indulin A.

The data for Recipe PA-133 show the effect of using Hi-Sil C coated with 8 ppm of LM-3 silicone oil. After aging, the vulcanizates evidenced low tensile strength and were very soft. Further work with this filler is not indicated.

DPR N-27 as a Plasticizer. Past experience has shown that conventional plasticizers offered no advantage. This may be because they were readily extracted by the hot Turbo Oil-15, and their value lost as plasticizers. However, it was thought that a covulcanizable plasticizer might become an integral part of the polymer structure and resist extraction. There are very few nonextractable plasticizers known, but DPR N-27 (a depolymerized copolymer of butadiene/acrylonitrile containing 27 per cent acrylonitrile) was considered as possibly representative of this type. The results in Table 14 show several advantages for the use of DPR N-27. For the base stock containing 70 phr of Silene EF, the plasticizer produced a large decrease in initial hardness and an increase in elongation. After 500 hours' aging, the untempered stocks containing DPR N-27 had higher tensile strength, 5 to 10 per cent lower swell, and equal elongation and freedom from cracking, compared with the control stock containing only 70 phr Silene EF. For the stocks containing 81.7 phr Silene EF, the DPR N-27 decreased the cracking somewhat.

These results indicate two further approaches to the problem. First, the DPR N-27 was prepared from a copolymer containing 27 per cent acrylonitrile. Past work has shown that swelling of 35 per cent acrylonitrile stocks is much lower than that of 26 per cent acrylonitrile stocks (Tables 23 and 24, WADC TR 54-190). Therefore, a DPR from 35 per cent acrylonitrile rubber might contribute toward further reduction in swell. The second approach would be to evaluate a liquid acrylate polymer, such as Hycar 4001X11, as a plasticizer.

Sodium Metasilicate as a Vulcanizing Agent. B. F. Goodrich Chemical Company has recommended sodium metasilicate as a vulcanizing agent for Hycar PA (Service Bulletin H-6, August, 1950). Hycar PA is a polyethylacrylate, whereas Hycar 4021 contains about 95 per cent ethyl acrylate and 5 per cent chloroethyl vinyl ether. Thus, a system that works for Hycar PA might be expected to work with Hycar 4021, though the reverse might not be true.

To investigate this vulcanizing agent, two recipes recommended by the B. F. Goodrich Chemical Company for Hycar PA were employed for Hycar 4021. The data shown in Table 15 indicate that good original physical properties were obtained in this manner. However, Recipe PA-174, containing 9 phr of sodium metasilicate, was found to crack badly after only 72 hours' aging. Although Recipe PA-175, containing a much smaller amount of sodium metasilicate, along with Trimene Base, did not crack after 168 hours' aging, its tensile strength was very low and its swelling excessive. These limited tests indicate no promise for sodium metasilicate as a vulcanizing agent.

<u>Cracking of Philblack A-Filled Compositions.</u> Until recently, no difficulty had been encountered with the cracking of Hycar 4021 vulcanizates. Recently, however, cracking has been found in vulcanizates containing Philblack A after 500 hours' aging. There is some evidence to indicate that this cracking may be the result of factors other than those causing the cracking of Acrylon EA-5.

Recipe PA-52 was reported previously as having the best balance of vulcanizing agents for Hycar 4021 stocks containing Philblack A. As a consequence, this particular recipe has been repeated several times as a control. The data in Table 16 show the result of mixing and testing the same composition five times and a similar composition once. The first two times the recipe was used, encouraging results were obtained, with no cracking evident after 500 hours' hot-oil aging. The next four times, samples of an identical formulation cracked when aged under the same conditions. The samples that cracked had both lower tensile strength and hardness and higher swell than those that did not crack. The data for the 72- and 168-hour tests were fairly reproducible, with the exception of hardness.

It was first thought that the variability of results might be due to the Turbo Oil-15. Although, during this work, the different drums of Turbo Oil-15 were from the same batch, the difficulty appeared to coincide with the change in drums. However, Recipes PA-154 and PA-162 were evaluated later in Turbo Oil-15 from both drums and showed no difference.

Consideration was then given to the possibility that the difficulty might be caused by the particular batch of Hycar 4021 or Philblack A being used. New supplies of each were ordered and evaluated. The data in Table 17 show no established trend in a comparison of the four combinations of the old and new Hycar 4021 and Philblack A. The difficulty also coincided with the arrival of hot and humid summer weather, so it was thought that the carbon black might have picked up appreciable moisture. To investigate this, a fifth sample was evaluated in which the Philblack A was heated for 1 hour at 105 C to remove volatile matter prior to incorporation into the rubber. The results obtained under these conditions (Table 17) still indicated cracking. Swelling was much less for the rubber containing the new supply of carbon black and still better for the samples containing the oven-treated carbon black.

The evidence suggests that the filler material might be the cause of the cracking and poor physical properties. This is indicated by (1) the variation in swell results obtained for comparable carbon black samples with different histories and (2) the almost complete lack of cracking observed with rubber samples containing nonblack fillers, such as Hi-Sil and Silene EF. Satisfactory performance under all conditions might be obtained for other types of carbon black, such as those coarser in particle size.

Acrylon EA-5

Carbon Black as a Filler. In Table 43 of WADC TR 54-190, preliminary data were given on the evaluation of Acrylon EA-5 vulcanizates filled with carbon black. Because many of the 500-hour tests were not complete, results were not conclusive.

The initial work has been expanded considerably, with complete results shown in Table 18 of this report. Early work with Acrylon EA-5 was done using about 1.5 phr of triethylene tetramine as one of the vulcanizing agents. Vulcanizates of this type have one important advantage and one disadvantage, compared with similar vulcanizates prepared from Hycar 4021. Their advantage is that they do not continue to increase in swell with aging time. Their disadvantage is that, with only one exception (Recipe PA-83), all carbon black-filled vulcanizates of this polymer have cracked after 500 hours' aging. A rerun on Recipe PA-83 (PA-155) also was found to crack.

It was first thought that lower levels of triethylene tetramine to produce less cross linking might tend to reduce cracking. Results showed, however, that, even with as little as 0.8 phr of triethylene tetramine, cracking was still persistent. With this lower amount of triethylene tetramine, it was necessary to use at least 2.0 phr of sulfur to obtain a satisfactory cure in 120 minutes.

General conclusions that can be reached from an examination of the data in Table 18 are as follows:

(1) As the aging period increased, tensile strength, elongation, and swelling decreased. Hardness decreased to a minimum value and then started to increase.

(2) Tempering did not show any advantage.

(3) There was no significant difference between Philblack A and Philblack O.

(4) All the carbon black compositions shown tended to crack after 500 hours' aging.

The strong tendency toward cracking remains the major obstacle to the use of Acrylon EA-5. It is believed that this cracking is associated with a very low aged elongation (see a later section of this report). When using a 180-degree bend to evaluate cracking, the breaking elongation probably is exceeded. Therefore, in any future work with Acrylon EA-5, consideration should be directed toward increasing the aged elongation. Some progress already has been made in this direction by using nonblack fillers, such

as Silene EF, Hi-Sil, and Calcene NC. Vulcanizing systems such as Trimene Base, Monex, and Polyac should be evaluated, as these have been found to give high aged elongation with Hycar 4021. The use of an antioxidant, such as Agerite Resin D, may also offer some advantage.

Hi-Sil as a Filler. It was indicated earlier in this report that Hi-Sil was one of the more promising fillers for Hycar 4021. Because of this, limited work was done to evaluate this filler in Acrylon EA-5, with the results as shown in Table 19. Recipe PA-140 shows the effect of 54.2 phr of Hi-Sil with the usual level of vulcanizing agent. After 500 hours' aging, this composition had a low tensile strength and a high elongation, but displayed no cracking. In an effort to obtain better aged physical properties, better initial properties were sought by using additional curing agents. Adjustments in this direction, as indicated by results for Recipes PA-181, PA-216, and PA-217, did furnish some improvement in aged tensile strength and swelling. This was nullified, however, by a large loss in elongation and the introduction of cracking. The use of a larger amount of Hi-Sil (65 phr) appeared not to be the answer, as the original hardness was increased to above 90. Thus, in these limited tests, Hi-Sil did not appear to be a promising filler for Acrylon EA-5.

Miscellaneous Fillers. Since Silene EF has been found to be the best filler for Hycar 4021, a limited evaluation was made of it in Acrylon EA-5. The results, presented in Table 20, indicate that the aged properties of the vulcanizate improved as the filler loading was increased. Recipe PA-180, containing 70 phr of Silene EF, was the first polyacrylate rubber evaluated on this program that met the swelling specification during the entire aging period. However, this sample cracked after 500 hours' aging in the manner typical of carbon black-filled Acrylon EA-5 vulcanizates. It is interesting to note that Silene EF stocks reached an essentially constant swell early in the aging period. By contrast, similar carbon black stocks reached a maximum swell in about 2 days, and then the swell started to decrease.

Recipes PA-204 and PA-205 show initial work using P-33 thermal black, alone and in a mixture with Philblack A. Thermal black did not harden the stock as much as Philblack A, and therefore permitted a higher carbon black loading. However, after stocks filled with P-33 black were aged, their properties were essentially equal to those of similar stocks filled with Philblack A. They displayed the same typical cracking after 500 hours' aging.

Limited additional work is in progress with Silene EF to try to eliminate cracking. If this cracking can be eliminated, this type of stock stands a good chance of meeting the minimum target requirements of the project. It is hoped that Agerite Resin D will be of assistance in this direction.

Effect of Mercaptobenzimidazole as an Antioxidant. The use of mer-captobenzimidazole as an antioxidant for natural rubber was discussed in the section of this report on the compounding of nitrile rubber. It will be recalled that this material was claimed to be both a mild vulcanizing agent and a nonextractable antioxidant.

An evaluation of this material in Acrylon EA-5 is shown in Table 21. The data indicate that mercaptobenzimidazole increased the original ten-sile strength and hardness and lowered the elongation of this type of stock. Similar effects were noted in the properties of the aged stocks. This strongly implies that this additive acted as a vulcanizing agent, rather than as an antioxidant. This is further substantiated by the observation that the stock containing 5 phr of mercaptobenzimidazole was more prone to crack than the one containing only 1 phr of this additive.

Hycar 4021X26

An experimental polymer, Hycar 4021X26, supplied by B. F. Goodrich Chemical Company, was evaluated, with the results shown in Table 22. It can be seen that the aging behavior of this polymer was similar to that of Acrylon EA-5. The vulcanizates decreased in swell during aging, but cracked after 500 hours' aging. No further work is suggested for this polymer.

Acrylon EA-9

This polymer, supplied by American Monomer Corporation, is simi-lar to Acrylon EA-5, except that the EA-9 contains 9 per cent acrylonitrile, compared with 5 per cent in Acrylon EA-5. The results in Table 22 show this material to retain very good tensile strength after aging and to have very low swell. Cracking was evident, however, after only 168 hours' aging.

Because of the limited availability of this polymer, no further work is planned at the present time. However, if means are found for substan-tially reducing the cracking of Hycar 4021 or Acrylon EA-5, these same techniques should be applied to Acrylon EA-9.

Special Studies

Comparison of Oven and Press Curing. Because of the very long cures necessary to obtain maximum properties with acrylate polymers, only a limited number of samples can be cured in a working day. It was thought that the press output could be greatly increased if a short press cure followed by an oven cure at the same temperature could be used. By

using the normal curing temperature, instead of the higher temperature employed for "tempering", it was hoped that less curative would be lost during the oven cure. To investigate this idea, two typical compositions were prepared and evaluated (Table 23). Recipe PA-164 was identical to the best Hycar 4021 compound containing Silene EF, and Recipe PA-165 was an Acrylon EA-5 compound containing Philblack O. The samples that were given a short press cure followed by an oven cure were softer and had higher elongations originally than those cured entirely in the press. After short aging periods, these same samples had greater swell in Turbo Oil-15, and in some cases there was a marked difference in tensile strength and elongation. After 500 hours' aging, these differences were greatly minimized.

The results of this work show that satisfactory vulcanizates can be obtained by either method (press cure or press cure plus oven cure).

Aging Time Between Compounding and Curing. The standard practice on this project has been to follow a method of compounding one day, allowing the batch to rest overnight, and then curing it on the following day. It has been suggested to Battelle that vulcanizate properties might be improved by increasing the aging time between compounding and curing. In order to determine the importance of raw-stock aging time, a batch was compounded and portions of this batch cured after various time intervals. The aging periods for the raw stock were varied from 4 hours to 1 week.

The results, presented in Table 24, indicate that aging before curing was beneficial. Although tensile strength and hardness were the only two properties affected, the extent of improvement was significant for both original and aged properties. In original properties, the gain in tensile strength was about 300 psi, and hardness was increased by several points. It is possible that similar results could be obtained by mild oven aging before curing.

The Effect of Aging Acrylate-Type Rubber in Di-(2-Ethylhexyl) Sebacate Obtained From Several Suppliers. The use of Turbo Oil-15 as a test fluid for specification testing of rubber compounds is considered undesirable by WADC for two main reasons: first, Turbo Oil-15 is in short supply; and second, WADC has found Turbo Oil-15 to vary in its effect on rubber from batch to batch. Since the base stock of Turbo Oil-15 was presumed to be a diester (since reported to be essentially di-isoctyl sebacate, Chemical Week, November 27, 1954), it was thought that the use of a pure sebacate as the test fluid might be desirable. At the request of WADC, a limited study was made of (1) the relative effects of aging rubber in Turbo Oil-15 and in di-(2-ethylhexyl) sebacate, and (2) the reproducibility of results obtained by aging rubber in di-(2-ethylhexyl) sebacates from various sources.

The results of this work are shown in Table 25. When using Recipe PA-162, a carbon black-loaded stock, all samples aged in di-(2-ethylhexyl)

sebacate fell apart after 500 hours' aging. The sample aged in Penola Turbo Oil-15 was much better, although very much below target specifications. The poor behavior of this stock in Turbo Oil-15 is associated with difficulties encountered recently with the cracking of all carbon black-loaded Hycar 4021 vulcanizates. Further details on the performance of carbon black stocks are given in another section of this report. Recipe PA-162 was prepared at the same time as the other samples and, therefore, might have been expected to crack.

Somewhat different results were obtained with Recipe PA-163, a Silene EF-loaded stock. This stock gave satisfactory results even after 500 hours' aging in Turbo Oil-15. When di-(2-ethylhexyl) sebacate was used as the aging fluid, the rubber samples intended for use in swelling measurements began to disintegrate after 168 hours, and they were completely disintegrated after 500 hours. However, the tensile dumbbells were still intact after this same aging time. The usual testing procedure is to age three dumbbells in one test tube for stress-strain tests and to age two small samples in a second test tube for swelling measurements. The only obvious difference in aging conditions between the dumbbells and swell specimens is that the ratio of oil to rubber was greater for the swell specimens (i.e., the ones that disintegrated). This is the first time there has been any indication that this ratio might be important.

The data in Table 25 show that the di-(2-ethylhexyl) sebacates from the various manufacturers did not have the same effects on rubber. The sebacate supplied by Harchem (W. C. Hardesty Company, Incorporated, New York, New York) gave results distinctly different from those for sebacates from all of the other sources of this material.

On the basis of the results obtained, it does not appear advisable to use just any di-(2-ethylhexyl) sebacate as a standard test liquid for specification tests. It is understood that further work on test procedures will be carried out by a round-robin testing program for the AMS Sealing Ring Subcommittee.

Comparison of Open- and Closed-Tube Aging. In Table 26 of WADC TR 54-190, it was shown that a Hycar 1001 stock aged for 500 hours at 350 F in Turbo Oil-15 swelled approximately 60 per cent in a stoppered tube, compared with 15 per cent in an open tube where there was free access to air during aging. Because of the possibility of using similar rubbers in both hydraulic systems (where air is excluded) and lubricating systems (where considerable air might be present), this large difference in swelling was considered very significant.

At the request of WADC, the properties of two acrylate stocks were evaluated in both open and closed tubes (Table 26). Both compounds in the closed tubes swelled less and had higher hardness ratings than those in the

open tubes. In addition, Recipe PA-162, which had been prone to crack when aged in an open tube, did show this deficiency when aged in a closed tube. Only minor differences in other rubber physical properties were obtained for these two methods of aging.

The very high swelling found for aging Hycar 1001 stocks in a closed tube was not evident in the case of aging stocks of Hycar 4021. Whereas nitrile-type rubber tends to cross link when aged in contact with air, acrylate rubber appears to undergo only a slight depolymerization under the same conditions.

Comparison of Aging at 350 F and at 400 F. Although the work on this program has been confined almost entirely to an evaluation of vulcanizates at 350 F, evaluations at 400 F have been made on a few compounds. Those tested at this higher temperature include several vulcanizates that have come very close to meeting the specifications at 350 F. In Table 27 are shown the results for aging several different stocks at both 350 F and 400 F. The indications are that aging these rubber compositions for 500 hours at 350 F was approximately equal in severity to aging them for 168 hours at 400 F.

In future work, greater emphasis will be placed on the testing of promising rubber stocks at 400 F.

Blends of Hycar 1001 and Hycar 4021. It was known from previous work that vulcanizates of Hycar 1001 swelled much less than those of Hycar 4021 in Turbo Oil-15. Hycar 1001, however, had the serious disadvantage of cracking badly when aged in hot oil. In order to take advantage of both the oil resistance of Hycar 1001 and the crack resistance of Hycar 4021, several blends of these two materials were prepared and evaluated.

In the annual report WADC TR 54-190, the effects of adding up to 20 parts of Hycar 1001 to 100 phr of Hycar 4021 were presented. This work has been extended to 30 and 40 phr of Hycar 1001. It is evident from Table 28 that up to 20 phr of Hycar 1001 was beneficial in increasing the hardness, but contributed no other worthwhile effect. Both 30 and 40 phr of Hycar 1001 reduced swelling, but also caused cracking. Thus, there appears to be no real advantage in adding Hycar 1001 to Hycar 4021.

Blends of Acrylon EA-5 and Hycar 4021. It has been mentioned previously that, during aging, the swell of Hycar 4021 stocks increased, whereas that of Acrylon EA-5 stocks declined. An attempt was made, therefore, to maintain a constant amount of swelling during the aging period by making blends of these two polymers. The data in Table 29 show that a mixture of 60 per cent Hycar 4021 and 40 per cent Acrylon EA-5 did maintain constant swelling during aging, but even 20 per cent of Acrylon EA-5 in the mixture induced cracking.

Although cracking was encountered in these studies for mixtures of Acrylon EA-5 and Hycar 4021, it is quite possible that the use of nonblack filler materials would greatly reduce this tendency toward cracking. When more crack-resistant Acrylon EA-5 compositions are obtained, an investigation should be made of their blends with Hycar 4021.

Compression-Set Tests. In Table 46 of WADC TR 54-190, data were given on the compression set of two of the best stocks. Since that time, work has continued to determine compression set on stocks that closely approached the minimum specifications of this project.

Several tentative conclusions can be drawn from the limited data available in Table 30:

(1) The use of the thinner samples for aging in Turbo Oil-15 gave 5 to 15 per cent lower compression set.

(2) The compression set of samples aged in hot air was comparable with that of samples aged in hot Turbo Oil-15.

(3) The tempering of samples reduced their compression set considerably.

(4) Differences in compression set among the various samples tested were not considered significant.

(5) The compression set of samples aged in Turbo Oil-15 continued to increase after 72 hours' aging.

Low-Temperature-Retraction Tests. Although no low-temperature requirements were included in the target specifications of this project, WADC requested that low-temperature-retraction tests be made on some of the most promising compounds. Table 31 shows the results for nine Hycar 4021 stocks and one Acrylon EA-5 stock. The single stock from Acrylon EA-5 (Recipe PA-83) had the poorest TR-70 value of those tested (in the range of 2 to 4 C). All the Hycar 4021 stocks had TR-70 values in the range of -7 to 2 C, with no significant differences being noted among the various Hycar 4021 compounds. In each case, the tempered samples were slightly poorer in low-temperature properties than the untempered samples.

<u>Explanation of Cracking.</u> Analysis of the data obtained during this project points toward a fairly definite pattern for the cracking of samples during the 180-degree bend test following hot-oil aging. The general conclusion has been reached that cracking under these conditions is directly associated with loss in elongation during aging.

With Acrylon EA-5, this cracking has been a constant problem for samples aged 500 hours, but it has not been critical for shorter aging periods. With Hycar 4021, cracking has occurred under only two particular conditions: (1) when the stocks were heavily loaded with nonblack fillers, and (2) when using 40 phr of Philblack A as the filler material. The difficulty with Philblack A has been discussed earlier in this report.

It was first observed that cracking was most prevalent in samples having very low elongation after hot-oil aging. This appeared logical, since a portion of the sample is stretched or elongated during the 180-degree flat bend. Information received from WADC indicated that a 0.075-inch sample would elongate 100 to 110 per cent and a 0.040-inch sample about 60 per cent during a 180-degree flat bend. To verify this, two samples were ruled in 0.1-inch spacings, bent 180 degrees, and then measured to determine the elongation at various distances from the bend. The results are shown in Figure 1.* A maximum elongation of 50 per cent was noted right at the bend, and the elongation had dropped to 0 per cent at a distance of 0.3 inch from the bend. It is probable that the actual maximum elongation at the bend is considerably greater than that measurable between bench marks 0.1 inch apart. It is recognized that the distribution of the elongation during this bend will be a function of the stress-strain curve, and therefore will vary for different stocks. However, indications are that samples that elongate less than 50 per cent will be very prone to crack because of low elongation.

Two very interesting graphs were obtained (Figures 2 and 3) by plotting data obtained in the past on elongation and cracking. Results with Hycar 4021 and with Acrylon EA-5 were found to be very different. With Hycar 4021, most samples were found to retain an elongation in the range of 50 to 100 per cent after 72 and 168 hours' aging, with a few of the shortest samples cracking. After 500 hours' aging, the average elongation was actually a little higher, with no cracking noted. It should be pointed out, however, that samples that cracked after 168 hours were not tested after 500 hours' aging. Therefore, the samples in the 500-hour plot are only the best

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* Figures 1, 2, and 3 appear in Appendix III.

of those shown in the 168-hour plot. The tendency of this polymer to increase in swell with aging time also may have contributed toward increased plasticization for longer aging periods.

With Acrylon EA-5, a drastically different pattern was found. After aging for 72 and 168 hours, the elongation was generally over 200 per cent, with little tendency toward cracking. After 500 hours' aging, the elongation was generally reduced to less than 50 per cent, with most samples cracking. The observable trend for Acrylon EA-5 stocks to shrink during longer periods of hot-oil aging probably indicates reduced plasticization by the oil. This may be a factor in the very low elongation of these stocks after 500 hours' aging.

The tendency for saturated polymers, such as the acrylates, to crack might be due to chain scission or increased cross linking during aging and still be evident in low aged elongation. Regardless of the cause, it is believed that, if aged elongation can be maintained, cracking will be largely eliminated.

It is interesting to note that the target specifications for this project require a minimum aged elongation of 100 per cent. Evidence to date indicates that, if this elongation can be retained, cracking will be largely eliminated.

Compounding Studies With Poly-FBA

During this report period, a limited amount of compounding work was done with a 5-pound sample of poly-FBA (i.e., poly-1,1-dihydroperfluorobutyl acrylate, developed by Minnesota Mining and Manufacturing Company for WADC). Literature and information made available to Battelle during the course of this investigation, though limited, have pointed to certain advantages and disadvantages of this material. It does possess an outstanding combination of heat and oil resistance for use in Turbo Oil-15. Compounds of this polymer have been reported by WADC as meeting the minimum target requirements of this project for use in this synthetic lubricating oil at 350 F. Although the original physical properties of the best poly-FBA compounds are relatively weak, these compounds exhibit little loss of properties during hot-oil aging. Thus, there remains a real need to improve the original properties still further and to develop special product properties, such as low permanent set.

The best poly-FBA compounds reported by others have been reinforced with furnace black and cured with combinations of sulfur and polyfunctional amines. The state of cure obtained with compositions of this type has been very sensitive to the amount of curative, a situation that is not corrected easily by the simple expedient of adjusting curing time. This problem is

particularly acute in laboratory operations, where the limited availability of the polymer forces one to mix a few measured drops of curative into about 20 grams of polymer. In larger factory operations, the amounts of curative should be less critical, but there may be more difficulty in obtaining a good dispersion of compounding ingredients into this soft rubber.

One of the shortcomings of poly-FBA compounds is that improvements in compression set have been attained only by going to softer stocks. Thus, those having a hardness preferred for packings and seals (65 to 75 Shore A) are poor in compression set. In conventional rubbers, reasonably good set characteristics are not nearly so dependent on the type and amount of pigment loading.

The low-temperature properties of poly-FBA are relatively poor. Brittle points of the uncompounded and compounded polymer are usually above 0 F. The highly oil-resistant nature of the polymer precludes the use of ordinary plasticizers to attain better low-temperature properties.

The limited availability and relatively high cost (current price is about $25 per pound) have retarded extensive compounding studies with FBA-polymer. At least in initial compounding studies, the batch size of experimental compositions usually has been quite small. More work may need to be done with larger size experimental batches (200 to 500 grams) to bridge the gap to commercial production.

Most of the work done at Battelle has been aimed at making a general improvement in all of the physical properties of poly-FBA. One exception was an attempt to improve its low-temperature properties by blending another polymer into poly-FBA.

The Effects of Magnesia on Poly-FBA

A poly-FBA composition containing magnesia, Polyac, and lead peroxide was reported in the literature to have good physical properties after aging in air for 70 hours at 350 F. A number of compositions of this type were evaluated, since other compositions of poly-FBA usually are lacking in this property. The results of aging these compositions in Turbo Oil are shown in Table 32. These data indicate that magnesia exerted a slow curing action, which continued during aging and gave poor aging properties. It is worth noting, however, that the original physical properties of these batches were, in many cases, markedly superior to those of batches that were black filled and amine cured (Table 33). A method for controlling the rate of cure of magnesia-containing compositions could conceivably upgrade the aging properties of the resulting product.

The effects of several different types of filler on poly-FBA are shown in Table 33. Most of these compositions met the minimum target requirements for this research project after aging for 500 hours at 350 F. Of particular note is the low swell of compositions of this type, compared with the nonfluorinated acrylates previously discussed. The maintenance of other physical properties, however, is essentially in the same range for both fluorinated and nonfluorinated acrylates.

The aging results shown for work done outside Battelle are superior to those obtained at Battelle for an identical composition. This can be interpreted as indicating that the aging conditions employed at Battelle probably were more severe than those used by others. It will be recalled that considerable difference in results was reported previously for the oil aging of nitrile rubber in the absence of air and for oil aging in an oven compared with aging in an aluminum-block heater. The present results would imply that less air might have contacted the samples aged elsewhere. For the purpose of this report, it is suggested that fillers be compared on the basis of those samples aged at Battelle, all of which were aged in an aluminum-block heater under identical conditions.

The results for several different types of carbon black indicate that, for the same amount of loading, the finest particle-size black (Philblack E) gave the highest hardness, though it produced about the same tensile strength and elongation as slightly coarser blacks (Philblacks A and O). An appreciably coarser black (P-33) did not appear promising in this limited comparison. Increasing the amounts of curative in the P-33 stocks did little to improve the performance of this filler. The basic recipe in which these blacks were compared was taken from a paper presented by Major Hamlin, WADC. The results reported by him met the minimum target requirements of this project for use in Turbo Oil-15 at 350 F, as also do the results obtained at Battelle.

The material identified as "Ludox" was specially prepared at Battelle prior to compounding. It is sold as an aqueous colloidal solution of a hydrated silica. Before use, it was dried and then ball milled for 48 hours. This dry product did not appear to be an outstanding filler material for poly-FBA.

Encouraging results were obtained with Aerosil (now known as Cab-O-Sil) as a filler material. Although the initial and aged elongations were lower for stocks containing this filler than for those containing carbon black, the retention of tensile strength appears to be better than for the black stocks. Evidence of the higher reinforcing properties of Aerosil, compared with carbon black, may be found in the higher hardness values obtained for the Aerosil stocks. Of particular interest is the lower sensitivity of Aerosil stocks to the amount of triethylene tetramine curative, compared with the

carbon black stocks. Several of the Aerosil stocks, like the carbon black stocks, were found to meet the minimum requirements of this project for use in Turbo Oil-15 at 350 F.

The studies thus far with Aerosil have been limited to 25 and 35 phr of this material. It is quite possible that still better results could be obtained by investigating the effects of other levels of this filler on poly-FBA. Comparisons might then be made with black-filled stocks of similar hardnesses. It will be of particular interest to determine whether lower compression set can be obtained with Aerosil than with carbon black, when compared in stocks of the same hardness.

Blending Other Polymers With Poly-FBA

A series of copolymers of butadiene with acrylic acid and with methacrylic acid, prepared at Battelle, have been found to be very effective in improving the low-temperature properties of Thiokol-base fuel-tank sealants. It was hoped that blending such a copolymer with poly-FBA would result in substantial improvement in low-temperature properties, with a modest sacrifice in oil resistance. The two polymers appeared to be compatible in the uncured state, even though the butadiene copolymer, as added, was very hard. The results in Table 34 show that the vulcanizates of the blends did not have physical properties as good as those of poly-FBA alone. The improvement sought in the low-temperature properties by polymer blending was not achieved.

Compounding Miscellaneous Polymers

Neoprene Q

Neoprene type Q is a copolymer of chloroprene and acrylonitrile, reported to give excellent oil resistance. The data in Table 35 for one batch of this polymer show that its aged tensile strength and swell compared favorably with those of the acrylate vulcanizates. However, the aged elongation of this batch became negligible after 168 hours, and cracking was evident after only 72 hours' aging. Since results appear to be no better than those for nitrile rubber, no further work is suggested with this polymer.

Butyl Rubber

Because of the very low amount of unsaturation in butyl rubber, this material should exhibit a high degree of resistance to aging. To investigate this, two batches of butyl rubber were compounded and evaluated. The first was a conventional stock containing an SRF black. The second batch

was prepared to investigate the claims of a recent United States patent (United States Patent 2,666,753). This patent claims a substantial increase in the solvent resistance of butyl by the addition of a peroxide such as calcium peroxide.

The results in Table 35 show that the butyl disintegrated in 72 hours, whether used with or without calcium peroxide. Thus, it was not possible to determine whether the calcium peroxide had any effect at all.

Design, Construction, and Operation of Aluminum-Block-Type Heaters for Test-Tube Aging of Rubber

The demand by the aircraft and other industries for rubber components that will give satisfactory service at 350 F and above in contact with synthetic lubricating and hydraulic fluids has presented a problem in the laboratory aging of experimental rubber compositions under simulated service conditions. Fire and explosion hazards become pronounced when hot-air aging cabinets are used for this purpose. The high-temperature aging of rubber in any fluid, unless both are completely sealed in a suitable pressure vessel, will result in volatilization of some of the fluid into the oven. Such vapors may be ignited by the hot resistance wires used for heating the cabinet. In addition to the fire hazard, experience has indicated that rubbers aged alone contaminate each other when aged together in an oven.

Starting with advice from the B. F. Goodrich Chemical Company that solid aluminum blocks, drilled to hold test tubes, could be heated safely for this purpose, Battelle undertook to design and construct two heaters of this type. These were needed at Battelle in connection with the current program on the development of high-temperature, oil-resistant rubber. Their construction was reported in the first annual report on this project, WADC TR 54-190. These units now have given over a year of satisfactory service at 350 F, with closer temperature control than is believed practical for the usual air aging ovens. Higher operating temperatures are possible for these block heaters than for the customary air ovens, with considerably less fire and contamination hazard.

Battelle was requested to design and construct two units of this type for WADC, one to operate at temperatures up to 500 F and the other to operate at temperatures up to 800 F. Calculations that were made indicated that the same basic design could be employed for both temperatures, with an obvious advantage of having standardized parts. Details on the construction and operation of this equipment are shown in Appendix IV. These block heaters consist of an aluminum ingot drilled to hold a number of 38-mm test tubes in which the rubber samples can be placed. Strip heaters mounted

on the front and back of the block, shown in Figure 4*, provide the necessary heat. This block is completely encased in high-temperature insulation and enclosed in an aluminum cabinet, as shown in Figure 5. Selector switches and Variacs included in the electrical circuits permit close adjustment of the heat input required to maintain the desired block temperature. For close control on these units, it is desirable to balance the continuous heat input as closely as possible with the heat loss. A Fenwal temperature regulator is used with one set of heaters to provide automatic temperature control over the balance of the heat input. It is desirable to keep the ratio of the controlled input to total heat input as small as possible, to minimize temperature deviation of the block. It is also desirable to keep the exposed length of sample tube as short as possible to lessen heat losses.

A safety Fenwal controller was installed to prevent overheating. This is normally set to open just above the desired block temperature. In case of overheating to the selected safety temperature, the main relay is held open until the block cools below the set temperature. Heating then is resumed until the set temperature is reached again. It is possible, but not advisable, to control the block temperature with the safety Fenwal. This gives a total on-off heating and will produce temperature deviation in the block.

Figure 5 shows the front view and panel of the aluminum-block heater. Figure 6 shows the panel plan of the control board. Figure 7 gives the complete wiring diagram for the aluminum-block heaters. Figures 8 and 9 show details for the aluminum block and the frame, respectively.

The function of each of the control units, reading from left to right on the control panel, can be found in Appendix IV. Also in this appendix may be found a parts list and specifications for various parts.

SUMMARY

Work has been continued to develop a compound that will be suitable for use in Turbo Oil-15 at temperatures of 350 to 550 F. The most promising compounds are reported in Table 36. Two general types of poly-FBA stock have been found to meet all of the minimum target requirements. One of these types, previously reported by WADC, is reinforced with a fine furnace carbon black. The other type, developed at Battelle, is reinforced with Acrosil, a fine-particle-size silica. The best Hycar 4021 stocks (Recipes PA-95 and PA-184) failed to meet the target requirements only because of 5 to 10 per cent excessive swelling.

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* Figures 4 through 9 appear in Appendix III.

During the extensive compounding work described in the first annual report (WADC TR 54-190), it was found that nitrile rubbers aged in Turbo Oil-15 at 350 F stood up best when the reinforcing filler was magnesia. A compound of this type (such as Compound A-23, containing Hycar 1001) was one of the best aging compounds developed. Its critical weakness, however, was that it surface hardened during aging in hot Turbo Oil-15. Then the hardened surface would crack when the samples were flexed after aging. Variations in the curing system and antioxidants (added both to the rubber and to the oil) were ineffective in eliminating this cracking.

At the beginning of the present report period, a literature survey was made, searching for novel methods of improving aging. Most of the nitrile-rubber work described in this report attempts to employ such novel methods for preventing the cracking of the magnesia-filled Hycar 1001 compound (Compound A-23).

The Effects of Thiols and Related Compounds on Aging

It is believed that the unsaturation in nitrile-type rubber compounds is responsible for their cracking after aging in hot oil. On this basis, any means of reducing unsaturation should reduce cracking. One method for reducing unsaturation is to employ thiols (such as thiomalic acid and thioglycolic acid) as saturating agents. At low levels, some of these were found to improve the aged physical properties of the rubber and appeared to retard cracking slightly. At higher levels of the thiols, such as amounts sufficient to reduce unsaturation by 50 per cent, the aged compounds became extremely hard and cracking was accelerated.

An attempt to obtain better results by hot milling the thiol into Hycar 1001 was unsuccessful. During the milling, the stock became hard and impossible to process.

Since the previous work had suggested that low levels of thiols might have had some antioxidant action, several related compounds (most disulfides and diselenides) were evaluated as possible antioxidants. The results showed that aging was not improved and that cracking may have been accelerated.

It has been reported that amines and thiols can be used together to obtain more than additive antioxidant protection through synergism. Several combinations of these two classes of materials were tried. Although combinations of Flectol H with thiomalic acid and thioglycolic acid produced good retention of physical properties, there was no significant reduction in cracking. Gains made with these antioxidants were largely lost after 500 hours' aging in Turbo Oil-15.

Several chemical compounds related to the mercaptans and amines, i.e., compounds of selenium, phosphorus, arsenic, antimony, and bismuth, were examined for antioxidant action. None of these showed promise.

The Effects of High Sulfur Levels on an Unfilled Compound

One of the methods used for reducing unsaturation was to employ more than the usual amount of sulfur for vulcanization. A series of unfilled Hycar 1001 stocks was made with sulfur levels varied from 0.5 phr to 20 phr. All of the compounds in this series displayed a poor balance of physical properties and were generally inferior to filled compounds. However, the best results were obtained with a sulfur loading of 10 phr, which, incidentally, also gave a stock with substantially no swell.

A Preliminary Evaluation of Hycar 1041

Hycar 1041 (containing the same ratio of butadiene to acrylonitrile as Hycar 1001) was suggested by the B. F. Goodrich Chemical Company as possibly being more crack resistant than Hycar 1001. A comparison of these two rubbers in similar stocks, however, showed that stocks from Hycar 1001 were generally superior for aging in Turbo Oil-15.

Mercaptobenzimidazole as an Antioxidant

Several patents and publications have reported that mercaptobenzimidazole improves the aging of natural rubber. A recent theory for the behavior of this material is that it slowly vulcanizes rubber during aging, and thus partly neutralizes degradation reactions. An evaluation of mercaptobenzimidazole in Hycar 1001 indicated that, during the aging, there was a reversion of the cure, without a reduction in cracking.

Aging of Hycar 1001 in Di-(2-Ethylhexyl) Sebacates From Several Suppliers

Aging Hycar 1001 stocks for 500 hours in di-(2-ethylhexyl) sebacates from several different sources resulted in a large variation in the aged properties of the stock. This indicated that there was a considerable variation in di-(2-ethylhexyl) sebacate from various sources.

Compounding Studies With Acrylate Polymers

In the development work toward improving the hot-oil resistance of acrylate-type rubber polymers, most of the effort has been divided between

Hycar 4021 and Acrylon EA-5. Recipe PA-95 was reported as the best compound in the last annual report (WADC TR 54-190). This is still the best compound, although Recipe PA-184 compares very favorably with it. Except for the cracking of Recipe PA-180, it could be rated as the best compound of all. Although no significant improvement was made in the aged properties of acrylate polymers during the period covered by this report, several promising leads were uncovered. Vulcanizates of Hycar 4021 are now showing more promise than Acrylon EA-5, because of the persistent cracking of Acrylon EA-5 after 500-hour aging tests. It is evident that all of the best stocks developed were compounded with Silene EF.

Compounding Studies With Hycar 4021

The major emphasis of this phase of the work has been on the evaluation of a series of fillers. The most promising, as stated previously, was Silene EF. This was true both when it was used alone and when it was used in combination with 10 phr of DPR N-27. Attempts to increase the filler loading by employing Calcene in combination with the Silene EF were found to offer no advantage. Three experimental types of Hi-Sil were found to be poorer than regular Hi-Sil. For a given volume loading, Du Pont Fine Silica was found to give vulcanizates that were harder and swelled less than compositions containing any other filler evaluated. Within an acceptable hardness range, however, the Du Pont Fine Silica appeared to offer no outstanding advantage. Preliminary tests on Calcene NC and P-33 thermal black were encouraging, but results with Indulin A and silicone-coated Hi-Sil C do not indicate further work.

Cracking has been encountered with carbon black-filled stocks after they were aged for 500 hours.

Sodium metasilicate was found unsatisfactory as a vulcanizing agent.

Compounding Studies With Acrylon EA-5

The outstanding property of this material continues to be that it has better resistance to swelling than does Hycar 4021. Carbon black-filled stocks were found to reach a maximum swell of 30 to 40 per cent in about 2 days, and then to decrease in swell with time. Nonblack stocks reached a maximum swell of about 30 per cent and then maintained this swell during the entire aging period. The major shortcoming of the polymer continues to be cracking after 500 hours' aging, with this deficiency being found in both carbon black- and Silene EF-filled stocks. The use of Hi-Sil as a filler increased the resistance to cracking somewhat, but at a considerable sacrifice in tensile strength. The use of mercaptobenzimidazole as an antioxidant in a Silene EF-filled vulcanizate increased the aged tensile strength, but did not decrease cracking.

Unless a means is found to eliminate cracking of Acrylon EA-5 stocks after prolonged aging (500 hours), this material may be considered as showing little promise for use in Turbo Oil-15. On the other hand, if cracking could be eliminated, Acrylon EA-5 probably would be even more satisfactory than Hycar 4021, because of its lower swelling.

Compounding Studies With Hycar 4021X26

This experimental polymer did not show promise for use in hot Turbo Oil-15.

Compounding Studies With Acrylon EA-9

Although this polymer cracked badly after aging, its swell was only 15 to 20 per cent, compared with a minimum of about 30 to 35 per cent for the other acrylate polymers (Hycar 4021 and Acrylon EA-5).

Special Studies

Because of excessively long cures, the effect of a long press cure, compared with a short press cure followed by a long oven cure at the same temperature, was investigated. For the properties evaluated, both methods produced comparable vulcanizates. Aging for as much as 1 week between compounding and curing was found to give some improvement in aged properties.

The use of pure di-(2-ethylhexyl) sebacate as a testing fluid was found inadvisable. Vulcanizates of Hycar 4021 and Acrylon EA-5 disintegrated after aging in this fluid for 500 hours. When the aging took place in closed tubes, the hardness increased and swell was lowered, compared with aging in open tubes. No effect was noted on other properties.

Limited data were obtained to compare the severity of aging of stocks at 400 F and at 350 F. As an approximation, 168 hours at 400 F was found to be equal to 500 hours at 350 F.

Blends of both Hycar 1001 and Acrylon EA-5 with Hycar 4021 were prepared and evaluated. No advantage was found over using Hycar 4021 alone.

Compression-set tests were continued. Most of the vulcanizates were found to have a set of about 90 per cent after aging in Turbo Oil-15 at 350 F for 168 hours.

Limited data on low-temperature-retraction tests indicated that all Hycar 4021 stocks had TR-70 values of -7 to +2 C. One sample of Acrylon EA-5 was close to this, having a TR-70 value of 2 to 4 C.

An explanation of the cracking of Acrylon EA-5 vulcanizates has been suggested. It is believed that most of the cracking failures are actually elongation failures, as the aged samples do not retain sufficient elongation to be bent 180 degrees.

During this report period, a limited amount of work was done with FBA polymer (poly-1,1-dihydroperfluorobutyl acrylate) supplied by Minnesota Mining and Manufacturing Company. This polymer was reported to have an excellent combination of oil and heat resistances, although the original properties are rather poor.

Work at Battelle has been aimed at a general upgrading of the physical properties and an effort to improve low-temperature properties. To accomplish this, a number of fillers have been studied. Magnesia has been reported as giving poly-FBA stocks that have very good physical properties after air aging for 70 hours at 350 F. However, when stocks of this type were aged in hot Turbo Oil-15, magnesia acted as a slow curative and the stocks were found to crack. Furnace black-filled stocks, made and tested at Battelle, were found to have poorer properties than similar stocks reported by other laboratories, though they did meet the minimum target requirements of this project for use in Turbo Oil-15 at 350 F. It is evident that the testing done at Battelle does not correlate with that done elsewhere, and further standardization is necessary.

Experiments with Du Pont's "Ludox" and fine-thermal black showed that these fillers did not provide sufficient reinforcement, and work with them was discontinued.

The most promising filler found for poly-FBA is Aerosil. Compounds containing this filler have also met the minimum target requirements of this project for use in Turbo Oil-15 at 350 F. Although the original physical properties of Aerosil-filled stocks are, in some cases, slightly inferior to those of furnace black-filled stocks, their retention of properties during aging is better. There is a possibility that Aerosil stocks will have better compression set at 350 F than do furnace black-filled compositions of the same hardness.

A copolymer of acrylic acid, prepared at Battelle and found very successful in improving the low-temperature properties of Thiokol sealants, was blended with poly-FBA. No improvement in properties was obtained.

Compounding Miscellaneous Polymers

Two polymers, Neoprene Q and butyl rubber, were found unsatisfactory after limited evaluation. Neoprene Q acted as a typical unsaturated polymer, cracking and losing elongation after aging, although swelling was approximately 35 per cent. Butyl rubber vulcanizates disintegrated in the hot Turbo Oil-15.

Details are given in this report for the design, construction, and operation of aluminum-block-type heaters for the test-tube aging of rubber specimens.

PLANS FOR FUTURE WORK

Evaluation of Polymers in Hydraulic Oils

At the request of WADC, the emphasis on this project will be changed. To date, the effort has been directed toward developing a rubber composition that can be used at 350 F in a synthetic lubricant, such as Turbo Oil-15. The emphasis now will shift toward the development of a rubber composition for use at -65 F to 400 F in hydraulic oil. As no decision has been reached about the type of hydraulic fluid that will be adopted by the Air Force ultimately, it will be necessary to evaluate vulcanizates in three oils. These are Monsanto OS-45, Oronite MLO 8200, and a modified MLO 8200 containing 15 parts of dioctyl sebacate.

Screening work by WADC has indicated that neoprene is the most promising material for this application. Therefore, rubbers of this type will receive first consideration. Because of the great stress placed on the low-temperature performance of rubber for use in hydraulic systems, the present work with the acrylate polymers, such as Hycar 4021 and poly-FBA, probably will be greatly curtailed. Other polymers that will be given screening-type tests for this application are the following:

(1) Philprene VP (Phillips Chemical Company)
(2) DC 410 Gum (Dow Corning)
(3) Carboxylic polymers (B. F. Goodrich)
(4) Butyl (Enjay Company)
(5) Brominated butyl (B. F. Goodrich)
(6) Nitrile-type rubbers

A number of the above rubbers are unsaturated to some degree. Inasmuch as hydraulic systems operate more in the absence of air than do lubricating systems, the tendency for unsaturated rubbers to fail from surface hardening and cracking should be much less pronounced in hydraulic systems. The details of the laboratory aging of rubbers in hot hydraulic oil will need to be worked out with WADC to reflect the operating conditions actually to be encountered in aircraft.

Future Studies With Nitrile-Type Rubber

The surface cracking of nitrile rubber after aging in hot Turbo Oil appears to be an inherent deficiency of this type of polymer. During the latter part of this report period, the experimental effort with this type of rubber was reduced to investigations of promising leads for preventing this cracking. The future work described here applies essentially to the completion of such work now in progress.

Thiomalic acid and mercaptobenzimidazole have been shown to retard and invert cure, respectively, when they are used in magnesia-filled, nitrile rubber compounds. Since the cracking failure of nitrile rubber usually is confined to the surface of the aged specimen, it was reasoned that these additives might be more effective in the oil than in the rubber. Tests in progress include the use of thiomalic acid and mercaptobenzimidazole in the oil alone and in both the rubber and the oil.

Ionol (di-tertiarybutyl p-cresol) and Thiokol ZL 190 are being investigated as additives for Hycar 1041, the low-temperature nitrile rubber similar to Hycar 1001.

United States Patent 2,678,892 reports a simple method for hydrogenating the surface of finished rubber articles. This hydrogenated surface is claimed to have improved resistance to ozone and to aging. Several attempts have been made to follow these simple procedures for the hydrogenation of black-filled and magnesia-filled compounds of Hycar 1001. In some cases, the treatment has effected considerable stiffening of the rubber, indicative of hydrogen addition. Aging tests will be conducted on such products in Turbo Oil-15.

Future Studies With Acrylate Polymers

Although the target specifications for a rubber to be used in Turbo Oil-15 have not been met completely by vulcanizates of this type prepared to date, the extensive work with Hycar 4021 and Acrylon EA-5 may lead to improved aged properties. It is understood that WADC desires some of this work to continue.

The reduction of swell continues to be the major problem with the acrylates. Although initial compounding work with the acrylates greatly reduced the swell, further improvement in this property during the current work has been more difficult to obtain.

Research with fillers continues to be the most promising approach to this problem. Although Silene EF is the best filler evaluated to date, Calcene NC, thermal black, and Du Pont Fine Silica have shown some interesting properties in preliminary tests. These should be considered for further study. Other fillers may also deserve consideration.

Past work has shown some improvement in aged properties by using increased amounts of vulcanizing agent and increased curing temperature, and by extending the shelf aging time between compounding and curing. Further work should be done on all of these leads.

Several samples are being tested to determine whether an antioxidant, Agerite Resin D, will eliminate the tendency for some of the acrylate compositions to crack during hot-oil aging. There also is a possibility that a suitable plasticizer can be found to solve this cracking problem, as well as to reduce the swell of the acrylates.

Substitution of paraffin for stearic acid as a lubricant is under investigation.

Future Studies With Poly-FBA

All of the immediate work planned with poly-FBA is already in progress. Compounds filled with 15 and 20 phr of Aerosil and cured with sulfur and triethylene tetramine are undergoing aging tests in Turbo Oil-15. Compression-set tests are also being run at 350 F in Turbo Oil. The original physical properties of poly-FBA containing 20 phr of Aerosil are comparable with those of compounds containing 35 phr of furnace black.

Du Pont Fine Silica is also being investigated as a filler for poly-FBA. This silica has an organic coating that can be decomposed and driven off at 250 F. A sample of this silica, preheated to remove approximately 50 per cent of this organic coating, has been used in the compounding of poly-FBA. The compounds containing the preheated silica were much softer than those containing the untreated material. When aging tests are completed, it may be possible to determine whether higher loadings of preheated silica should be tried or whether silica in this form has a harmful retarding effect on the cure.

Additional work with poly-FBA in Turbo Oil-15 will depend largely on the outcome of the work in progress. Aerosil already has shown sufficient promise as a filler to merit a rigorous examination. The possibilities of Du Pont Fine Silica are undetermined as yet.

EXHIBIT B TO SUPPLEMENTAL AGREEMENT NO. S2(54-1068)
TO CONTRACT NO. AF 33(616)-476

I. DETAILED TECHNICAL REQUIREMENTS

A. The Contractor shall further investigate commercially available elastomers to determine if compound development can produce a more readily available rubber compound which will be suitable for use in synthetic oils at high temperatures. To date, an elastomer which retains good properties after immersion in synthetic oil at 350°F for periods of 500 hours has been produced, but this elastomer, poly-1,1-dihydroperfluorobutyl acrylate, is in the experimental stage and will probably not be available commercially for some time. Approaches to the problem shall include but not be limited to the following:

1. Extensive evaluation of commercially available polymers and polymer blends, particularly the acrylate polymers such as Hycar PA-21 and the Acrylons. Acrylonitrile-type copolymers will be given a limited amount of additional research.

2. Further study of unique curing systems especially designed for high-temperature applications.

3. Further compounding and evaluation of commercially developed experimental polymers.

4. Compounding and evaluation of Government-furnished experimental polymers. Particular attention should be given to poly-1,1-dihydroperfluorobutyl acrylate when this material becomes available for distribution. Since this material shows great promise at 350°F, it should be evaluated under the more strenuous conditions of 400°F.

B. Desired properties and test methods are as follows:

1. The primary objective is the development of a rubber which will retain satisfactory physical properties after use in synthetic oils, such as sebacates, adipates, etc., for 500-1000 hours at 400°F.

2. Test fluids for this work will include Esso Turbo No. 15 and other MIL-L-7808 oils as they may become available.

3. As a secondary objective, it is desired that a rubber compound be developed which will be resistant to the action of high-temperature

hydraulic fluids when immersed in a test fluid at 400°F to 550°F. Test fluids will be as designated by the Materials Laboratory.

4. Target properties for compounds to be used in synthetic lubricants are as follows:

Original Properties
Tensile   1200 psi min.
Elongation   250% min.
Shore "A" Hardness   50-80

Low temperature flexibility — should be determined on selected compounds with the ultimate requirement of flexibility at -65°F kept in mind.

Properties after Oil Immersion for 500-1000 hours at 400°F
Tensile 750 psi min.
Elongation 150% min.
Shore "A" 50-90
Volume Change -2 to +30%
Appearance — No evidence of checking or cracking after 180° flat bend.

5. Target properties for compounds intended for use in synthetic hydraulic fluids.

Original Properties
Tensile   1500 psi min.
Elongation   200% min.
Shore "A"   60-80

Low temperature flexibility — Not brittle at -65°F (may be sacrificed for exceptional high temperature properties)

Properties after Immersion in Fluid at 400°F for 100 Hours and/or 550°F for 10 Hours
Tensile   1000 psi min.
Elongation   100% min.
Shore "A"   60-90
Volume Change   0-10%

When applicable, testing shall be conducted in accordance with Federal Specification ZZ-R-601 or ASTM Standards on Rubber Products.

II. The Contractor shall develop, fabricate, and perform trial tests on two experimental aluminum-block heaters. One of these shall be capable of

continuous operation from ambient to 500°F. The other shall be capable of continuous operation between ambient and 800°F.

A. Requirements common to the two heaters are as follows:

    1. Must operate on 220V, 60 cycle AC.

    2. The electrical circuit shall include but not be limited to the following items:

        a. Double-pole single-throw master switch.

        b. Three sets of strip heaters to be used as heat sources. Each set should consist of two heaters, one on each side of the aluminum block, wired to operate in series for low heat and in parallel for high heat.

        c. An on-off switch and indicator light should be provided for each set of heaters.

        d. Two sets of heaters should be equipped with Variacs for heat-level control.

        e. Over-temperature protection should be included in the circuit.

    3. The aluminum block should be drilled to provide space for 60 test tubes (38 mm diameter by 200 mm length). The lip of the test tube should rest on the top surface of the heater when the test tube is inserted in the heater.

    4. The aluminum shall withstand the highest design temperature of the heater without sufficient weakening to cause damage.

    5. Sufficient insulation should be included in the construction to provide for minimum heat loss and temperature instability.

    6. The over-all dimensions of the aluminum block heaters should be approximately as follows:

        Length 41"
        Height 24"
        Depth 23".

LITERATURE SURVEY OF METHODS FOR IMPROVING
HEAT AND OIL RESISTANCE OF RUBBER

Introduction

A literature survey has been made of various rubbers and methods of compounding them to obtain a combination of high-temperature and oil resistance. Since a survey was made previously of the acrylate-type polymers, this class of rubbers is not included in the present survey. A special object of this survey was to uncover unusual methods for treating other rubbers, particularly nitrile-type polymers, to improve their hot-oil aging characteristics. Experience to date has shown that the latter type of rubber cracks badly when aged in Turbo Oil-15 for only a few days at 350 F.

Choice of Polymer

Considerable work has been done with natural and synthetic elastomers in attempts to improve their oil, heat, and oxidation resistance. Materials have been developed that swell only slightly in a variety of oils and others that do not deteriorate rapidly at temperatures of 350 F and higher. However, there is a scarcity of information in the literature on materials claimed to have a reasonably high degree of resistance to both heat and oils.

In considering heat and oxidation resistance alone, the saturated rubbers (such as butyl, the silicones, and the polyacrylates) have been found to be most outstanding. The silicones are used at temperatures up to about 500 F.[5, 19, 34] Butyl rubber and combinations of butyl with other elastomers are commonly used for tire bags[6, 31], which must endure repeated cures for tires at steam temperatures sometimes approaching 400 F.

Although the silicones show fair resistance to some oils[19], the resistance of some of the best silicone compounds to diester-type lubricants at temperatures in the range of 350 F is marginal. Butyl rubber is affected even more adversely by hot oils, as has been shown by comparative tests made on a number of rubbers at WADC[1]. Further gains might be made with these rubbers, since both are affected greatly by reinforcing agents and by other compounding variations. Du Pont hydrophobic silica, GS-199S, greatly improves the physical properties of silicones.[35] Carbon black has

a marked influence on the swell of butyl rubber.[39] Recent work with the heat treatment of butyl-carbon black masterbatches[10, 11, 30, 38] indicates that butyl may have some application. Because of its poor resistance to diesters, however, butyl might best serve in a combination with a more oil-resistant rubber. The aging of the one blend tried thus far (20 parts of butyl per 100 parts of Hycar 1001) showed no improvement over the control (100 per cent Hycar 1001). A blend of 20 to 50 parts (base stock) of a heat-treated butyl-carbon black masterbatch with 50 to 80 parts of Hycar 1000-X70 (60 per cent acrylonitrile in the base polymer) might be interesting. The acrylonitrile content of the blend would remain high, and unsaturation would be greatly reduced.

The neoprenes and the acrylonitrile-butadiene copolymers generally are used for oil-resistant applications. Although the neoprenes resist most hydrocarbon oils, their reported performance in diester-type lubricants is much poorer than that of the acrylonitrile copolymers.[1, 13, 14] At the beginning of this project, WADC suggested that work with commercial elastomers should emphasize compounding with the polyacrylates and butadiene-acrylonitrile copolymers.

Antioxidants

High temperatures greatly accelerate the reaction between organic elastomers and oxygen. Harrison and Cole[15] found that the rate of change in the stress-strain properties of GR-S is doubled for every 10 C rise in temperature. Elastomers that contain double-bonds after curing generally are more susceptible to oxidative degradation than are saturated elastomers. At the high temperatures of target specifications, it has been found that the differences in the aging of saturated and unsaturated rubbers are magnified. For this reason, the properties of two saturated rubbers were mentioned earlier in this report. It is felt that the residual unsaturation in vulcanizates of butadiene-acrylonitrile copolymers is mainly responsible for their aging failures. A thorough literature search for antioxidants particularly suited for high temperatures was considered necessary. The many antioxidants that are used in natural and synthetic rubbers can be classified, almost without exception, as amines or phenolics, or compounds that have these characteristic groups. The phenolic antioxidants grew from the need for nondiscoloring antioxidants for the synthetic rubbers, and are usually considered less effective than those that are not nondiscoloring. Recent work by Kitchen, Albert, and Smith[18], however, showed several phenolics that compared favorably with the amine controls. Both nitrile and GR-S were used and the phenolics tested were alkyl- and alkoxy-substituted phenols. Several suggestions have been made concerning requirements for and modes of antioxidant action:

(1) The phenolic group must be hindered by large ortho-substituted groups.

(2) Ortho hydrogen plays an important part (in amine antioxidants)[12].

(3) Phenolics are scavengers for free radicals, as in the stopping of emulsion polymerization.

(4) Oxidation-reduction potentials of phenolics and aromatic amines must be in the proper range.[3, 8, 9, 26]

(5) Phenyl-2-naphthylamine[7] and phenols[3] react with RO_2 radicals to break the oxidation chain.

The last two suggestions are backed by considerable evidence. Kitchen[18] and his collaborators feel that semiquinone-type nitrogen and oxygen radicals are mainly responsible for antioxidant activity and that the amines and phenolics function in essentially the same manner. The stereochemistry of the formation of these radicals[4] is in agreement with the structures that have been found in actual practice to give the most effective antioxidant action. Whereas para substitution is desirable in both aromatic secondary amines and in the phenols, ortho substitution is desirable only in the phenols. These substitutions not only bring the oxidation-reduction potentials into the proper range for the formation of these radicals, but also contribute to their stability once they are formed.

The above-mentioned semiquinone-type nitrogen and oxygen radicals are, theoretically, sufficiently resonance-stabilized to retain their structure long enough for an antioxidant function. Perhaps the resonance energies of such radicals formed from the common, commercial antioxidants are insufficient at very high temperatures. There may be related radicals that require these high temperatures for their formation. The chemistry of the formation of this semiquinone-type radical should be examined thoroughly.

The use of combinations of antioxidants rather than a single one is not new.[29] LeBras[20] described a two-component antioxidant system that was much more effective than either component when used alone. LeBras[21, 22, 23] and LeFoll[24] describe the "inactive making" effect of mercaptobenzimidazole as a deactivation of absorbed oxygen. Oxygen absorption is not reduced, but the physical properties of the rubber are maintained. A later paper by LeFoll[25] states that the competing reactions[36, 37] taking place during the aging of rubber may explain the "deactivation" mechanism as an actual acceleration effect. Although mercaptobenzimidazole apparently does not reduce chain scission, it may promote the formation of cross linking, and thus bring about the retention of physical properties. To encourage such a reaction in nitrile rubbers to be used at 350 F would be a step in the wrong direction. Shelton and Cox[33],

however, suggest that mercaptobenzimidazole affects the vulcanization process, rather than the oxidation process.

The work with combinations of antioxidants done thus far on this project has not been successful. Although some combination of commercial antioxidants might show moderate improvement, the time required for such a study is not warranted.

In the experimental work on this project thus far, no antioxidant or combination of antioxidants has prevented cracking of nitrile-rubber vul- canizates that have been exposed to diester oils at 350 F. It is generally felt that the residual unsaturation is largely responsible for this poor aging. Therefore, methods of saturating rubbers have been included in this liter- ature search.

Saturants

The saturation of natural rubber by chlorination results in the loss of its elastic properties. Chlorination of acrylonitrile-butadiene polymers probably would produce the same result. Kharasch, May, and Mayo suc- ceeded in thiocyanating[17] isoprene at room temperature. Naylor[28] suc- ceeded in destroying 7 to 100 per cent of the unsaturation in natural rubber by thiocyanation. Although previous work with thiocyanation of rubber was mentioned by Naylor, no references were cited. Neither does the author mention the effects of such treatment on the physical properties of the rub- ber. Thiocyanation of nitrile polymers, of course, could bring about the loss of elastic properties, as does the chlorination of natural rubber. The similarity of the thiocyanate group to the nitrile group could induce an un- wanted high degree of crystallinity in the polymer. The reaction takes place slowly, however, and a partial saturation may be possible.

The work of Jones, Moberly, and Reynolds[16] included a cursory evaluation of elastomers in which they had reduced unsaturation by hydro- genation. When the unsaturation in polybutadiene was reduced to about 20 per cent of its original value, changes in properties resulted. Improve- ments in freeze and age resistance were accompanied by a tendency toward plastic, rather than elastomeric, qualities. Permanent set increased and the recovery from tensile strain became very poor. The authors did not suggest that this tendency toward permanent deformation might be overcome through compounding, though this might be possible.

Recent work has shown that mercaptans will add to the double bonds in several elastomers. Marvel[27] and collaborators introduced side groups in polybutadiene by using small amounts of mercaptans. Their aim, how- ever, was to improve the oil resistance, rather than to reduce the unsatura- tion. Sernink, Banes, and Swaney[32], by mercaptan addition, reduced the

unsaturation in several elastomers, including Perbunan, by 30 to 50 per cent. They obtained similar results from three different methods of addition: (1) addition to rubber latex, (2) addition to rubber in solution, and (3) mass addition. Their work did not include an evaluation of the physical properties of the treated polymers. It may be that a 90 to 100 per cent reduction in unsaturation will be necessary before a nitrile-type rubber will resist age cracking at high temperatures.

The similarity of the mercapto group to the hydroxyl group and phenolics may point to another avenue of attack. There may be mercaptans that perform an antioxidant action as well as a saturating action. There are compounds related to the commercial antioxidants and to the mercaptans, namely, selenols and phosphines, that exhibit a modifying action similar to that of mercaptans at high polymerization temperatures.[2] The superiority of selenols over mercaptans in butadiene-acrylonitrile copolymerization may point to superior mercaptan and phosphine antioxidants for high-temperature applications. Not enough work has been done with compounds of the amine, phosphine, stibine, and bismuthine groups and the alcohol, mercaptan, selenol, and tellurol groups to establish relationships in and between these groups concerning antioxidant action. In some preliminary work on this project, several mercaptans show mild promise. The mercaptans, which are similar in structure to the most effective phenolic antioxidants, are not commercially available, however. The compounds of the groups mentioned above deserve careful consideration. Whether they perform as saturating agents or as antioxidants, or both, they appear to be the most promising lead for improving the crack resistance of nitrile-type elastomers.

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APPENDIX III

TABLES AND ILLUSTRATIONS

TABLE 1. THE EFFECTS OF VARIOUS THIOLS ON THE AGING OF HYCAR 1001

Recipe	Additive	Mole Per Mole of Unsaturation	Parts Per 89.3 Parts of Rubber	Original Physical Properties			Physical Properties After Aging in Penola Turbo Oil-15 72 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F				
				Tensile Strength, psi	Elongation, %	Hardness, Shore A	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking
A-23-J	None	—	—	2960	570	70	1320	320	72	20.7	Crazed	990	150	79	17.5	Cracked
A-263	Thioacetic acid	0.05	3.8	2540	660	74	1300	250	77	20.8	Cracked	950	130	83	21.5	Cracked
A-264	Thiodiacetic acid	0.05	5.9	2820	700	73	1300	460	71	24.3	Cracked	800	210	83	27.5	Cracked
A-265	Thioglycolic acid	0.05	4.6	2660	770	74	2580	350	75	19.0	Cracked	1440	180	80	19.3	Cracked
A-267	Thiosalicylic acid	0.05	7.7	2440	770	68	1250	440	68	24.8	Crazed	740	180	75	21.9	Cracked
A-268	o-Thiocresol	0.05	6.2	570	1030	67	1510	330	72	17.4	Cracked	870	150	71	21.2	Cracked
A-269	β-Thiodiglycol	0.05	6.1	2750	740	69	1100	430	75	25.6	Crazed	980	250	82	27.9	Cracked
A-256	Thioacetic acid	0.1	7.6	3100	620	77	1420	270	78	20.2	Cracked	760	100	87	17.6	Cracked
A-257	Thiodiacetic acid	0.1	11.8	1890	670	75	740	430	75	16.0	Cracked	540	170	81	14.7	Cracked
A-258	Thioglycolic acid	0.1	9.2	1310	850	69	1840	400	73	17.2	Cracked	1210	170	79	13.8	Cracked
A-259	Thiodiglycolic acid	0.1	18.2	1760	710	81	750	430	78	15.6	Cracked	600	160	85	14.2	Cracked
A-260	Thiosalicylic acid	0.1	15.4	3220	490	87	3310	260	90	17.0	Crazed	2370	90	92	14.5	Cracked
A-261	o-Thiocresol	0.1	12.4	150	1330	54	Undercure--not tested				—	—	—	—	—	—
A-262	β-Thiodiglycol	0.1	12.2	2450	720	68	1350	430	77	24.9	Cracked	760	160	87	22.0	Cracked
A-276	Di-isooctyl thiomalate	0.1	35.0	Additive was incompatible			—	—	—	—	Cracked	—	—	—	—	—
A-275	t-Dodecyl mercaptan	0.2	42.2	430	1220	47	250	130	71	13.9	Cracked	—	—	—	—	—
A-277	Thiomalic acid	0.1	15.0	2190	300	78	1750	120	83	10.3	Crazed	1000	80	85	9.0	Cracked
A-278	Thiomalic acid	0.3	45.0	1320	260	96	1180	50	97	5.3	Cracked	1010	30	98	3.7	Cracked
A-279	Thiomalic acid	0.5	75.0	1160	210	98	1230	40	98	4.3	Cracked	1120	20	97	3.7	Cracked

Note:

Base recipe:

Ingredients	Parts by Weight
Hycar 1001	89.3
Magnesia	89.3
Zinc oxide	4.5
Stearic acid	1.3
Sulfur	0.5
Methyl Tuads	0.2
Additive	As shown

Cure: 60 minutes at 298 F.

Recipe	Additive	Parts Per 100 Parts of Rubber	Original Physical Properties			Physical Properties After Aging in Turbo Oil-15 72 Hours at 350 F					Physical Properties After Aging in Turbo Oil-15 168 Hours at 350 F				
			Tensile Strength, psi	Elonga-tion, %	Hardness, Shore A	Tensile Strength, psi	Elonga-tion, %	Hardness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elonga-tion, %	Hardness, Shore A	Swell, %	Cracking
A-23-J	None	–	2960	570	70	1320	320	72	20.7	Crazed	990	150	79	17.5	Cracked
A-289	Di-p-tolyl disulfide	3	1640	780	63	1150	280	67	19.0	Cracked	870	190	70	18.8	Cracked
A-290	Phenyl diselenide	3	2040	580	67	1210	300	67	18.0	Cracked	880	160	75	18.9	Cracked
A-291	Phenyl disulfide	3	1360	630	67	1070	190	68	22.6	Cracked	780	180	69	22.2	Cracked
A-292	Benzyl disulfide	3	1850	550	78	1320	160	82	19.1	Cracked	930	140	82	18.2	Cracked
A-293	4,4'-Diamino diphenyl disulfide	3	1540	790	63	1060	420	64	20.0	Cracked	650	210	69	19.6	Cracked
A-294	2-Amino benzene thiol	3	1110	600	60	760	520	61	21.2	Cracked	490	270	65	21.9	Cracked
A-295	t-Octyl disulfide	3	2070	630	62	1260	400	65	23.0	Cracked	690	230	66	24.6	Cracked
A-296	Phenyl ethyl sulfide	3	1750	570	66	1030	320	67	22.8	Cracked	600	180	70	22.6	Cracked

Note:

Base recipe:

Ingredients	Parts by Weight
Hycar 1001	100
Zinc oxide	5
Magnesia	100
Stearic acid	1.5
Sulfur	0.5
Methyl Tuads	0.25
Additive	As shown

Cure: 60 minutes at 298 F.

TABLE 3. THE EFFECTS OF COMBINING AMINE WITH SULFUR COMPOUNDS ON THE AGING OF HYCAR 1001

Recipe	Additives	Parts Per 100 Parts of Rubber	Original Physical Properties			Physical Properties After Aging in Penola Turbo Oil-15 72 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F				
			Tensile Strength, psi	Elongation, %	Hardness, Shore A	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking
A-307	Control	–	2420	650	73	1680	350	70	23.4	Slight cracking	810	130	80	24.0	Cracked
A-308	Flectol H	3	2320	670	74	1520	350	69	19.6	Slight cracking	1080	200	78	16.9	Cracked
A-309	Thioglycolic acid	5.2	2450	670	73	2230	310	71	19.9	Slight cracking	1510	180	76	21.6	Cracked
A-311	Thiomalic acid	8.4	1720	670	65	1590	350	61	17.3	Slight cracking	1100	200	70	15.9	Cracked
A-310(a)	Flectol H / Thioglycolic acid	3 / 5.2	1730	490	80	1620	230	75	17.3	Slight cracking	1400	190	79	15.0	Cracked
A-312(a)	Flectol H / Thiomalic acid	3 / 8.4	1930	580	67	1860	300	65	13.7	Slight cracking	1480	210	73	12.4	Cracked
A-313(b)	Flectol H / 4,4'-Diamino diphenyl disulfide	3 / 3	1730	860	68	1240	460	61	19.5	Cracked	950	260	74	17.5	Cracked
A-314(b)	Flectol H / Di-p-tolyl disulfide	3 / 3	1300	830	67	1230	290	68	19.0	Slight cracking	860	160	75	17.9	Cracked
A-315(b)	Flectol H / Diphenyl diselenide	3 / 3	2060	740	68	1280	390	66	20.7	Slight cracking	1020	230	74	17.9	Cracked
A-316(b)	Flectol H / Phenyl disulfide	3 / 3	1190	1050	66	1190	280	67	18.8	Slight cracking	800	160	73	17.6	Cracked
A-317(b)	Flectol H / Benzyl disulfide	3 / 3	1490	780	67	1360	340	67	21.7	Slight cracking	1030	230	75	19.1	Cracked

Base recipe:

Ingredients	Parts by Weight
Hycar 1001	100
Magnesia ELC	100
Zinc oxide	5
Stearic acid	1.5
Sulfur	0.5
Methyl Tuads	0.25
Additives	As shown

Cure: 60 minutes at 298 F.

Note:

(a) Results for aging a similar compound for 500 hours are shown in Table 8.

(b) In the base recipe, Methyl Tuads was increased to 0.5 part per 100 parts of rubber.

TABLE 4. THE EFFECTS OF CHEMICAL COMPOUNDS RELATED TO MERCAPTANS AND AMINES ON THE AGING OF HYCAR 1001

| Recipe | Additive | Parts Per 100 Parts of Rubber | Original Physical Properties | | | Physical Properties After Aging in Penola Turbo Oil-15 72 Hours at 350 F | | | | |
			Tensile Strength, psi	Elonga-tion, %	Hardness, Shore A	Tensile Strength, psi	Elonga-tion, %	Hardness, Shore A	Swell, %	Cracking
A-23-H	None	—	2420	650	73	1680	350	70	23.4	Slight cracking
A-283	Phenyl propyl selenol	4.0	2050	760	67	1660	350	72	16.3	Cracked
A-284	Sec-octyl selenol	4.0	2040	730	68	1580	300	74	13.5	Cracked
A-285	Triphenyl phosphine	4.0	1940	670	69	1230	400	67	21.6	Cracked
A-286	Triphenyl arsine	4.0	3180	510	73	1640	290	77	14.3	Cracked
A-287	Triphenyl stibine	4.0	3050	450	75	1330	300	62	26.7	Cracked
A-288	Triphenyl bismuthine	4.0	2600	440	77	1080	180	77	15.3	Cracked

Note:

Base recipe:	Ingredients	Parts by Weight
	Hycar 1001	100
	Zinc oxide	5
	Magnesia	100
	Stearic acid	1.5
	Sulfur	0.5
	Methyl Tuads	0.5
	Additive	As shown

Cure: 60 minutes at 298 F.

TABLE 5. EVALUATION OF HIGH-SULFUR COMPOUNDS IN THE AGING OF HYCAR 1001

Recipe	Parts of Sulfur Per 100 Parts of Rubber	Original Physical Properties			Physical Properties After Aging in Penola Turbo Oil-15 72 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F				
		Tensile Strength, psi	Elongation, %	Hardness, Shore A	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking
A-297	0.5	290	540	38	20	120	41	19.7	Cracked	110	10	44	17	Cracked
A-298	5	550	200	55	200	30	64	7.1	Cracked	340	10	68	5.8	Cracked
A-299	10	800	140	63	1050	50	94	0.3	Cracked	1420	30	97	-0.2	Cracked
A-300	20	1520	130	76	6000+	-	100+	-5.8	Cracked	6000+	-	100+	-6.0	Cracked

Note:

Base Recipe:

Ingredients	Parts by Weight
Hycar 1001	100
Zinc oxide	5
Stearic acid	1.5
Sulfur	As shown
Methyl Tuads	0.25

Cure: 60 minutes at 298 F.

TABLE 6. PRELIMINARY EVALUATION OF HYCAR 1041

Recipe	Additives	Parts Per 100 Parts of Rubber	Original Physical Properties			Physical Properties After Aging in Penola Turbo Oil-15 72 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F				
			Tensile Strength, psi	Elongation, %	Hardness, Shore A	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking
A-23-H	Hycar 1001 control	None	2420	650	73	1680	350	70	23.4	Slight cracking	810	130	80	24.0	Cracked
41A-c	Control	None	2800	580	70	770	360	70	26.3	Cracked	580	190	81	25.1	Cracked
41A-1	Thiomalic acid	8.4	960	600	72	1620	320	74	17.3	Crazed	1290	180	80	18.4	Cracked
41A-2	Thioglycolic acid	5.2	1620	780	71	1750	330	76	24.0	Cracked	1150	190	81	23.2	Cracked
41A-3	Flectol H	3	2990	580	70	710	320	67	24.0	Cracked	560	170	80	20.4	Cracked
41A-4	Thiomalic acid / Flectol H	8.4 / 3	1730	590	73	1670	230	77	13.5	Crazed	1280	140	82	12.9	Cracked
41A-5	Thioglycolic acid / Flectol H	5.2 / 3	1740	850	73	1770	350	77	17.6	Cracked	1230	210	81	16.5	Cracked
41A-6	Thiomalic acid	16.8	1300	400	80	1650	180	84	13.1	Crazed	1170	110	85	12.9	Cracked
41A-7	Flectol H	6	2970	590	71	830	330	68	19.6	Cracked	590	170	80	18.7	Cracked
41A-8	Thiomalic acid / Flectol H	16.8 / 6	1180	540	80	730	220	83	9.7	Crazed	1400	140	85	9.2	Cracked

Note:

Base recipe:

Ingredients	Parts by Weight
Hycar 1041	100
Magnesia	100
Zinc oxide	5
Stearic acid	1.5
Sulfur	0.75
Methyl Tuads	0.5
Additives	As shown

Cure: 60 minutes at 298 F.

TABLE 7. MISCELLANEOUS RUNS, INCLUDING THOSE WITH MERCAPTOBENZIMIDAZOLE

Recipe	Additives	Parts Per 100 Parts of Rubber	Original Physical Properties			Physical Properties After Aging in Penola Turbo Oil-15 72 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F				
			Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking
A-307 (A-23 H)	Control	—	2420	650	73	1680	350	70	23.4	Slight cracking	810	130	80	24.0	Cracked
A-308	Flectol H	3	2320	670	74	1520	350	69	19.6	Slight cracking	1080	200	78	16.9	Cracked
A-306	Mercaptobenzimidazole	3	1900	570	66	890	470	60	18.8	Cracked	670	290	67	19.2	Cracked
A-301	Flectol H Mercaptobenzimidazole	3 3	1920	610	64	840	460	60	18.1	Slight cracking	560	260	64	16.9	Cracked
A-302	Flectol H Mercaptobenzimidazole	10 10	1950	640	63	730	560	50	11.2	Slight cracking	450	370	55	10.3	Cracked
A-303	Flectol H	3	1840	610	57	130	200	39	49.5	Cracked	150	120	51	50.1	Cracked
A-304	Flectol H	2	1940	570	58	200	80	52	63.2	Cracked	250	40	63	99.1	Cracked

Note:

Base Recipe:

Parts by Weight

Ingredients	A-301, A-302, A-306, A-307, A-308	A-303	A-304
Hycar 1001	100	60	70
Smoked sheet	—	40	—
Hypalon S-2	—	—	30
Magnesia	100	100	100
Zinc oxide	5	5	5
Stearic acid	1.5	1.5	1.5
Sulfur	0.5	0.5	0.5
Methyl Tuads	0.25	0.25	0.5
Additives	As shown	As shown	As shown

Cure: 60 minutes at 298 F.

TABLE 8. AGING OF HYCAR 1001 VULCANIZATES IN

Recipe	Aging Medium	Manufacturer	Original Physical Properties			Physical Properties 72 Hours		
			Tensile Strength, psi	Elongation, %	Hardness, Shore A	Tensile Strength, psi	Elongation, %	Hardness, Shore A
A-23L	Turbo Oil-15	—	2000	680	70	1270	370	71
A-23L	DEHS[a]	Harchem	—	—	—	640	220	76
A-23L	DEHS[a]	Pittsburgh Coke and Chemical	—	—	—	830	260	78
A-23L	DEHS[a]	Rohm and Haas	—	—	—	820	280	78
A-318	Turbo Oil-15	—	3310	440	75	1770	180	75
A-319	Turbo Oil-15	—	2460	610	77	1940	280	77

Note:

Recipes used:

Ingredients	Parts by Weight		
	A23L	A-318	A-319
Hycar 1001	100	100	100
Zinc oxide	5	5	5
Magnesia ELC	100	100	100
Stearic acid	1.5	1.5	1.5
Sulfur	0.5	0.5	0.5
Methyl Tuads	0.25	0.5	0.5
Thiomalic acid	—	8.4	—
Thioglycolic acid	—	—	5.2
Flectol H	—	3	3

Cure: 60 minutes at 298 F.

(a) DEHS = di-(2-ethylhexyl) sebacate.

TURBO OIL-15 AND DI-(2-ETHYLHEXYL) SEBACATE

After Aging at 350 F		Physical Properties After Aging 168 Hours at 350 F					Physical Properties After Aging 500 Hours at 350 F				
Swell, %	Cracking	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking
27.1	Cracked	840	160	78	26.8	Cracked	650	90	80	24.0	Cracked
40.3	Cracked	670	150	80	95.3	Cracked	510	90	81		Partially disintegrated
10.6	Cracked	610	150	82	10.6	Cracked	570	70	85	9.4	Cracked
9.1	Cracked	610	150	82	8.5	Cracked	550	60	85	8.3	Cracked
12.8	Cracked	860	90	82	12.1	Cracked	700	50	82	11.6	Cracked
17.9	Cracked	1300	190	80	17.2	Cracked	750	80	85	16.1	Cracked

TABLE 9.   EFFECT OF SILENE EF ON THE AGING

Recipe	Silene EF Phr by Weight	Phr by Volume	Plasticizer, phr by weight	Cure, minutes at 310 F	Treatment After Cure	Original Physical Properties Tensile Strength, psi	Elongation, %	Hardness, Shore A	Physical Properties Penola Turbo Oil-15 Tensile Strength, psi	Elongation, %
PA-60	46.6	24.4	0	30	None	1080	630	63	390	100
				30	Tempered[a]	1030	140	67	570	90
PA-94	58.3	30.6	0	30	None	1070	490	75	550	90
				30	Tempered	1180	120	85	760	80
PA-130	58.3	30.6	10	30	None	1020	640	59	450	110
				30	Tempered	1080	190	63	620	90
PA-95	70.0	36.7	0	30	None	1080	430	85	760	80
				30	Tempered	1260	100	93	900	80
PA-145	70.0	36.7	10	60	None	730	420	66	610	90
				120	None	840	350	68	610	70
				60	Tempered	1020	130	78	730	80
				120	Tempered	1160	120	78	770	70
PA-169	81.7	42.9	0	60	None	1010	140	82	1080	50
				120	None	990	120	85	1120	50
				60	Tempered	1100	60	88	1160	40
				120	Tempered	1150	50	89	1190	40
PA-176	81.7	42.9	10	60	None	1110	340	92	1030	60
				120	None	1180	240	92	1180	60
				60	Tempered	1360	120	94	1010	60
				120	Tempered	1370	100	94	1230	50

Note:

Base recipe:	Ingredients	Parts by Weight
	Hycar 4021	100
	Silene EF	As shown
	Plasticizer (Flexol R2H)	As shown
	Stearic acid	1
	Methyl Tuads	2
	Triethylene tetramine	1.5

(a) All tempering was for 7 hours at 350 F.

PROPERTIES OF HYCAR 4021 VULCANIZATES

After Aging in 72 Hours at 350 F			Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 500 Hours at 350 F				
Hardness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking
48	47.1	None	450	120	45	50.4	None	500	150	49	63.6	None
57	38.1	None	570	100	57	45.4	None	430	100	56	58.1	None
64	39.7	None	580	90	64	40.9	None	920	110	57	46.9	None
71	32.7	None	840	90	70	33.9	None	970	90	65	42.8	None
47	45.1	None	450	100	44	47.3	None	—	—	—	—	—
52	34.4	None	530	80	54	37.0	None	—	—	—	—	—
74	35.4	None	800	90	71	37.6	None	1000	100	64	42.7	None
80	28.6	None	940	80	79	30.8	None	1240	90	72	35.7	None
55	34.7	None	710	90	56	36.0	None	—	—	—	—	—
58	29.7	None	760	90	59	31.8	None	—	—	—	—	—
60	28.6	None	760	80	59	32.8	None	—	—	—	—	—
63	26.4	None	790	80	61	32.1	None	—	—	—	—	—
81	22.1	Cracked	1060	50	79	24.8	Cracked	—	—	—	—	—
81	20.9	Cracked	1030	50	82	24.1	Cracked	—	—	—	—	—
83	19.1	Cracked	1260	50	82	22.9	Cracked	—	—	—	—	—
84	20.6	Cracked	1180	50	83	22.5	Cracked	—	—	—	—	—
81	23.8	None	1000	60	84	26.0	None	—	—	—	—	—
83	18.5	Cracked	1120	60	85	23.4	Cracked	—	—	—	—	—
85	20.8	Cracked	1000	40	87	24.9	Cracked	—	—	—	—	—
88	19.9	Cracked	1170	50	88	23.3	Cracked	—	—	—	—	—

TABLE 10. EFFECTS OF SILENE EF-CALCENE TM

Recipe	Filler	Filler Phr by Weight	Filler Phr by Volume	Cure, minutes at 310 F	Treatment After Cure	Original Physical Properties Tensile Strength, psi	Original Physical Properties Elongation, %	Original Physical Properties Hardness, Shore A	Physical Properties Penola Turbo Oil-15 Tensile Strength, psi	Physical Properties Penola Turbo Oil-15 Elongation, %	Physical Properties Penola Turbo Oil-15 Hardness, Shore A
PA-128	Silene EF	58.3	30.6	30	None	1080	400	68	530	70	59
				60	None	1060	280	72	470	60	63
				120	None	1000	220	70	560	60	65
				30	Tempered[a]	1020	110	76	680	70	66
				60	Tempered	1190	110	79	600	60	69
				120	Tempered	1200	100	78	560	60	70
PA-131	Silene EF	46.6	30.6	60	None	890	490	54	270	80	43
	Calcene TM	13.9		120	None	900	380	56	350	80	47
				60	Tempered	910	140	65	480	80	52
				120	Tempered	840	120	66	550	80	54
PA-95	Silene EF	70	36.7	30	None	1080	430	85	760	80	74
				30	Tempered	1260	100	93	900	80	80
PA-146	Silene EF	58.3	36.7	60	None	920	310	68	600	80	57
	Calcene TM	13.9		120	None	920	290	66	660	70	58
				60	Tempered	1080	110	78	690	70	62
				120	Tempered	1120	120	77	760	60	66
PA-169	Silene EF	81.7	42.7	60	None	1010	140	82	1080	50	81
				120	None	990	120	85	1120	50	81
				60	Tempered	1100	60	88	1160	40	83
				120	Tempered	1150	50	89	1190	40	84
PA-147	Silene EF	58.3	42.7	60	None	920	400	71	540	90	54
	Calcene TM	27.8		120	None	930	260	77	690	80	62
				60	Tempered	1120	130	82	830	70	67
				120	Tempered	1180	110	83	800	70	69
PA-148	Silene EF	46.6	48.9	60	None	740	380	70	480	100	51
	Calcene TM	55.6		120	None	760	300	72	520	90	53
				60	Tempered	940	120	82	700	80	61
				120	Tempered	940	110	83	750	80	64
PA-149	Silene EF	46.6	61.2	60	None	670	310	77	550	90	57
	Calcene TM	83.4		120	None	720	280	81	630	80	62
				60	Tempered	900	90	87	740	70	68
				120	Tempered	960	90	89	840	70	70

Note:

Basic recipe:

Ingredients	Parts by Weight
Hycar 4021	100
Filler	As shown
Stearic acid	1
Methyl Tuads	2
Triethylene tetramine	1.5

(a) All tempering was for 7 hours at 350 F.

After Aging in 72 Hours at 350 F		Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 500 Hours at 350 F				
Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking
38.0	None	650	80	60	38.3	None	–	–	–	–	–
34.8	None	560	60	62	37.7	None	–	–	–	–	–
33.4	None	610	70	65	36.2	None	–	–	–	–	–
31.9	None	560	60	66	37.8	None	–	–	–	–	–
31.4	None	660	60	67	37.0	None	–	–	–	–	–
32.7	None	720	60	69	35.7	None	–	–	–	–	–
42.1	None	390	90	42	43.1	None	–	–	–	–	–
38.8	None	450	80	46	43.7	None	–	–	–	–	–
35.9	None	440	70	51	42.2	None	–	–	–	–	–
35.6	None	500	80	52	40.1	None	–	–	–	–	–
35.4	None	800	90	71	37.6	None	1000	100	64	42.7	None
28.6	None	940	80	79	30.8	None	1240	90	72	35.7	None
37.6	None	550	90	54	41.2	None	–	–	–	–	–
33.2	None	540	80	59	35.3	None	–	–	–	–	–
31.7	None	630	70	61	33.2	None	–	–	–	–	–
29.4	None	520	60	63	35.2	None	–	–	–	–	–
22.1	Cracked	1060	50	79	24.8	Cracked	–	–	–	–	–
20.9	Cracked	1030	50	82	24.1	Cracked	–	–	–	–	–
19.1	Cracked	1260	50	82	22.9	Cracked	–	–	–	–	–
20.6	Cracked	1180	50	83	22.5	Cracked	–	–	–	–	–
36.6	None	510	80	58	36.7	None	760	120	53	55.5	None
31.8	None	610	70	64	35.5	None	750	90	58	50.9	None
29.8	None	560	70	64	35.3	None	730	90	59	48.6	None
28.5	None	600	60	67	33.0	None	740	80	63	45.7	Edge cracking
37.1	None	490	100	53	40.1	None	–	–	–	–	–
34.8	None	590	90	56	37.3	None	–	–	–	–	–
29.9	None	660	80	61	35.0	None	–	–	–	–	–
29.9	None	690	80	61	32.4	None	–	–	–	–	–
31.3	None	560	90	58	35.3	None	740	100	58	48.4	None
29.9	None	620	80	63	36.2	None	820	90	63	42.8	None
26.2	None	660	70	66	29.3	None	910	80	64	42.9	Cracked
24.4	None	730	70	69	29.0	None	800	70	66	40.6	Cracked

TABLE 11. EFFECTS OF SILENE EF – CALCENE NC COMBINATIONS ON THE AGING PROPERTIES OF HYCAR 4021

Recipe	Filler	Filler Phr by Weight	Filler Phr by Volume	Cure, minutes at 310 F	Original Tensile Strength, psi	Original Elongation, %	Original Hardness, Shore A	168 Hr Tensile Strength, psi	168 Hr Elongation, %	168 Hr Hardness, Shore A	168 Hr Swell, %	168 Hr Cracking	500 Hr Tensile Strength, psi	500 Hr Elongation, %	500 Hr Hardness, Shore A	500 Hr Swell, %	500 Hr Cracking
PA-60	Silene EF	46.6	24.4	30	1080	630	63	450	120	45	50.4	None	500	150	49	63.6	None
PA-221	Silene EF	46.6	48.7	30	960	380	84	920	70	74	32.0	None					
	Calcene NC	60		60	1000	300	87	950	60	77	29.9	None	Tests in progress				
PA-222	Silene EF	46.6	52.9	30	930	330	90	1030	70	77	29.6	None	—	—	—	—	—
	Calcene NC	70		60	990	290	92	1180	60	80	27.4	None	—	—	—	—	—
PA-223	Silene EF	46.6	57.0	30	910	300	93	1170	60	81	27.4	None	—	—	—	—	—
	Calcene NC	80		60	950	210	93	1310	60	81	27.4	None	—	—	—	—	—
PA-189	Silene EF	46.6	61.2	60	1000	160	98	1670	30	97	17.3	Cracked	—	—	—	—	—
	Calcene NC	90		120	1130	100	98	1910	30	97	16.9	Cracked	—	—	—	—	—
PA-94	Silene EF	58.3	30.6	30	1070	490	75	580	90	64	40.9	None	920	110	57	46.9	None
PA-218	Silene EF	58.3	42.7	30	890	530	75	690	100	61	41.8	None					
	Calene NC	30		60	930	200	84	1040	70	76	30.1	None	Tests in progress				
PA-219	Silene EF	58.3	46.9	30	900	270	87	900	60	77	29.7	None	—	—	—	—	—
	Calcene NC	40		60	920	200	87	1060	70	78	28.4	None	—	—	—	—	—
PA-220	Silene EF	58.3	50.9	30	920	310	91	1100	80	79	30.8	None	—	—	—	—	—
	Calcene NC	50		60	920	290	90	960	70	78	33.2	None	—	—	—	—	—
PA-95	Silene EF	70.0	36.7	30	1080	430	85	800	90	71	37.6	None	1000	100	64	42.7	None

Note:

Base recipe:

Ingredients	Parts by Weight
Hycar 4021	100
Filler	As shown
Stearic acid	1
Methyl Tuads	2
Triethylene tetramine	1.5

All samples untempered.

TABLE 12. EFFECT OF EXPERIMENTAL HI-SIL ON THE

Recipe	Filler	Flexol R2H, parts by weight	Cure, minutes at 310 F	Treatment After Cure	Original Physical Properties			Physical Properties After Penola Turbo Oil-15 72 Hours			
					Tensile Strength, psi	Elongation, %	Hardness, Shore A	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %
PA-170	Hi-Sil	0	30	None	1500	510	92	1060	90	72	38.3
			60	None	1670	450	93	1330	90	78	34.0
			120	None	1360	240	95	1260	80	80	30.5
			30	Tempered[a]	1810	160	95	1310	80	76	30.6
			60	Tempered	1910	150	96	1340	70	82	29.3
			120	Tempered	1740	120	96	1470	80	83	27.3
PA-171	Hi-Sil X303	0	30	None	1950	640	91	1010	120	71	40.5
			60	None	1880	670	91	1050	130	71	43.8
			120	None	1800	390	94	1040	100	79	32.8
			30	Tempered	2050	240	93	1150	130	74	37.6
			60	Tempered	2110	260	94	940	110	73	40.6
			120	Tempered	1870	190	95	1240	100	78	36.4
PA-172	Hi-Sil 53AK199	0	30	None	1870	630	95	1260	110	82	37.3
			60	None	1790	470	97	1280	80	84	31.2
			120	None	1640	340	98	1360	90	84	29.3
			30	Tempered	1780	170	97	1310	100	81	33.5
			60	Tempered	1780	130	98	1250	80	86	30.1
			120	Tempered	1650	110	98	1260	80	85	30.4
PA-173	Hi-Sil 53SMK108	0	30	None	1840	650	97	1050	100	85	36.2
			60	None	1640	460	98	1260	110	83	35.0
			120	None	1350	310	98	950	70	87	31.6
			30	Tempered	1470	130	98	920	80	83	36.3
			60	Tempered	1760	150	98	1080	100	80	36.0
			120	Tempered	1540	120	98	1130	80	86	33.0
PA-197	Hi-Sil	10	60	None	1300	550	90	1080	110	77	31.5
			120	None	1330	400	93	910	70	80	26.5
			60	Tempered	1530	170	93	1160	100	81	25.8
			120	Tempered	1660	150	95	1140	80	83	23.4
PA-198	Hi-Sil X303	10	60	None	1510	570	96	990	70	86	26.9
			120	None	1280	450	96	930	50	89	24.4
			60	Tempered	960	50	98	960	60	87	25.7
			120	Tempered	1170	40	98	790	60	87	24.2
PA-199	Hi-Sil 53AK199	10	60	None	1610	560	90	1000	100	77	28.1
			120	None	1460	450	91	970	80	82	27.4
			60	Tempered	1650	180	95	1000	80	78	27.6
			120	Tempered	1600	160	95	940	80	82	23.4
PA-200	Hi-Sil 53SMK108	10	60	None	1610	530	96	1090	80	87	26.9
			120	None	1620	460	96	950	50	89	24.0
			60	Tempered	1170	90	97	1070	80	86	26.3
			120	Tempered	1300	80	97	1110	60	89	24.0

Footnotes appear on page 67.

AGING PROPERTIES OF HYCAR 4021 VULCANIZATES

Aging in at 350 F	Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 500 Hours at 350 F				
Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking
None	1000	80	77	38.6	None	1160	120	77	50.8	None
None	1210	80	80	37.5	None	790	120	78	53.4	Cracked
None	1270	80	81	32.6	None	680	110	77	47.5	Cracked on edge
None	1300	80	82	33.6	None	1090	100	79	52.1	None
None	1200	70	83	37.5	None	1020	80	80	47.3	None
None	1260	70	81	31.8	None	1130	80	80	48.9	Cracked on edge
None	1110	120	73	46.3	None	650	120	70	60.0	Cracked
None	990	120	73	45.8	None	740	90	77	56.5	Cracked
None	910	80	78	39.7	None	–	–	–	–	–
None	840	130	69	46.3	None	490	120	70	59.5	Cracked
None	920	110	72	43.3	None	820	80	74	45.5	Cracked
None	1060	90	79	44.0	None	–	–	–	–	–
None	1150	100	83	37.2	None	950	110	80	52.8	Cracked
None	1250	80	83	35.0	None	920	70	85	46.1	Cracked
None	1330	80	85	32.8	None	910	80	85	45.3	Cracked
None	980	70	83	35.8	None	500	80	79	50.8	Cracked
None	1280	80	85	33.8	None	990	100	81	43.6	Cracked
None	1320	70	86	31.8	None	970	80	83	44.1	Cracked
None	910	70	83	40.2	None	840	70	84	54.7	Cracked
None	840	70	84	37.9	None	570	50	84	51.9	Cracked
None	1110	70	84	32.5	None	520	60	84	46.5	Cracked
None	740	80	82	41.4	None	600	60	85	48.3	Cracked
None	1100	90	83	42.3	None	500	60	81	48.9	Cracked
None	940	60	85	34.0	Cracked	420	40	84	46.3	Cracked
None	990	110	79	33.4	None	690	110	78	46.5	None
None	890	80	81	29.9	None	980	110	82	37.5	None
None	1030	80	82	31.4	None	720	120	80	39.8	None
None	1070	80	84	29.0	None	880	120	83	36.9	None
Cracked	910	60	86	30.0	Cracked	–	–	–	–	–
Cracked	910	50	88	25.7	Cracked	–	–	–	–	–
Cracked	840	70	86	32.0	Cracked	–	–	–	–	–
Cracked	970	60	87	28.7	Cracked	–	–	–	–	–
None	900	90	80	32.6	None	–	–	–	–	–
None	980	80	81	30.4	None	–	–	–	–	–
None	960	90	80	32.9	None	–	–	–	–	–
Cracked	860	70	81	30.1	None	–	–	–	–	–
None	1020	60	86	28.6	None	–	–	–	–	–
Cracked	980	60	88	27.9	Cracked	–	–	–	–	–
Cracked	810	60	89	30.2	Cracked	–	–	–	–	–
Cracked	1110	60	89	26.7	Cracked	–	–	–	–	–

Note:

Base recipe:

Ingredients	Parts by Weight
Hycar 4021	100
Filler	65
Flexol R2H	As shown
Stearic acid	1
Methyl Tuads	2
Triethylene tetramine	1.5

(a) All tempering was for 7 hours at 350 F.

TABLE 13. EFFECTS OF MISCELLANEOUS FILLERS ON THE

Recipe	Type	Phr by Weight	Phr by Volume	Cure, minutes at 310 F	Treatment After Cure	Original Physical Properties — Tensile Strength, psi	Original Physical Properties — Elongation, %	Original Physical Properties — Hardness, Shore A	After Penola Turbo Oil-15 72 Hours — Tensile Strength, psi	After Penola Turbo Oil-15 72 Hours — Elongation, %	After Penola Turbo Oil-15 72 Hours — Hardness, Shore A
PA-208	Du Pont Fine Silica	41.3	24.4	30	None	1490	690	78	1310	90	86
				60	None	1690	610	83	1320	100	87
				120	None	1480	510	83	1330	90	87
				30	Tempered[a]	1720	310	91	1310	80	83
				60	Tempered	1620	290	93	1260	70	86
				120	Tempered	1350	240	93	1260	70	87
PA-209	Du Pont Fine Silica	51.7	30.6	30	None	1470	640	89	1510	50	94
				60	None	1540	550	89	1520	50	95
				120	None	1410	500	90	1670	50	95
				30	Tempered	1800	280	96	1400	40	93
				60	Tempered	1040	120	96	1340	40	93
				120	Tempered	1730	250	96	1440	40	93
PA-224	Calcene NC	90	36.7	30	None	830	510	51	420	80	53
				60	None	870	450	54	460	80	54
				120	None	800	360	58	570	80	58
				30	Tempered	760	120	74	600	80	60
				60	Tempered	840	140	78	720	80	61
				120	Tempered	840	150	78	670	80	62
PA-225	Calcene NC	120	48.8	30	None	810	530	63	800	70	63
				60	None	770	390	69	840	70	65
				120	None	730	310	71	840	70	65
				30	Tempered	1020	120	84	920	70	70
				60	Tempered	1080	110	85	920	60	71
				120	Tempered	1010	100	85	990	70	71
PA-210	P-33 carbon black	60	36.7	60	None	840	210	56	550	130	48
				120	None	930	210	58	600	140	50
				60	Tempered	1000	180	66	600	120	50
				120	Tempered	930	160	67	580	130	50
PA-211	P-33 carbon black	80	48.8	60	None	890	200	65	780	140	54
				120	None	960	170	66	740	130	57
				60	Tempered	1000	170	75	750	130	56
				120	Tempered	1000	150	76	700	120	58
PA-212	Philblack A	30	30.6	60	None	1630	200	68	800	110	48
	P-33 carbon black	20		120	None	1680	170	69	740	100	50
				60	Tempered	1390	120	74	810	110	49
				120	Tempered	1590	130	74	750	100	52
PA-167	Indulin A	28.9[b]	24.4	120	None	840	630	45	330	150	32
				120	Tempered	1120	300	60	410	150	35
PA-168	Indulin A	28.9[b]	24.4	120	None	240	800	24	60	160	17
				120	Tempered	400	360	32	80	170	20
PA-201	Indulin A	43.4[b]	36.7	120	None	570	550	39	–	–	–
				120	Tempered	870	220	48	–	–	–
PA-133	LM-3-coated Hi-Sil C	43.3	24.4	60	None	1510	550	54	290	110	32
				120	None	1210	450	54	350	110	37
				60	Tempered	1110	240	61	400	110	39
				120	Tempered	1070	200	64	450	100	42

Footnotes appear on page 71.

AGING PROPERTIES OF HYCAR 4021 VULCANIZATES

Aging in at 350 F		Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 500 Hours at 350 F				
Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking
27.5	None	1150	60	87	28.2	None	860	20	91	33.1	Cracked
26.6	None	1210	50	88	28.2	None	820	20	92	35.6	Cracked
25.8	None	1180	50	88	26.5	None	850	20	92	34.0	Cracked
31.1	None	1200	80	86	34.3	None	830	20	93	31.8	Cracked
30.3	None	1260	60	88	33.5	None	900	20	93	35.4	Cracked
30.2	None	1160	50	89	34.7	None	950	20	93	36.4	Cracked
19.6	None	1510	40	95	20.6	Cracked	–	–	–	–	–
18.9	None	1370	30	95	21.5	Cracked	–	–	–	–	–
19.0	None	1330	30	95	19.5	Cracked	–	–	–	–	–
24.6	None	1430	40	94	26.8	None	–	–	–	–	–
24.2	None	1400	40	95	27.6	Cracked	–	–	–	–	–
23.2	None	1670	40	96	25.0	Cracked	–	–	–	–	–
33.1	None	510	80	55	43.7	None	–	–	–	–	–
34.6	None	560	80	55	43.0	None	–	–	–	–	–
32.8	None	650	90	56	39.8	None	–	–	–	–	–
29.7	None	640	70	58	42.1	None	–	–	–	–	–
30.7	None	610	70	59	37.8	None	–	–	–	–	–
30.8	None	680	80	60	36.6	None	–				
28.9	None	710	70	60	36.3	None					
27.7	None	880	70	61	34.2	None					
27.2	None	860	70	65	34.5	None	Tests in progress				
26.0	None	880	70	65	32.4	None					
25.3	None	880	70	67	32.5	None					
25.7	None	860	60	68	31.5	None					
36.0	None	570	120	44	42.3	None	–	–	–	–	–
35.0	None	550	110	50	40.7	None	–	–	–	–	–
39.5	None	580	120	49	42.7	None	–	–	–	–	–
38.7	None	610	120	50	43.5	None	–	–	–	–	–
32.9	None	770	120	54	38.9	None	370	100	45	47.3	None
30.8	None	740	110	54	33.3	None	410	100	52	47.1	None
36.1	None	580	100	54	38.0	None	340	100	47	54.1	None
33.8	None	650	110	57	39.2	None	420	100	53	49.7	None
38.1	None	600	90	46	43.3	None	–	–	–	–	–
36.6	None	600	80	49	40.7	None	–	–	–	–	–
40.3	None	590	110	44	54.4	None	–	–	–	–	–
39.7	None	720	100	49	51.8	None	–	–	–	–	–
58.5	None	410	140	34	58.6	None	–	–	–	–	–
53.2	None	390	140	37	56.7	None	–	–	–	–	–
61.0	None	60	160	15	67.2	None	–	–	–	–	–
60.1	None	70	180	13	71.1	None	–	–	–	–	–
–	–	270	120	31	57.4	None	–	–	–	–	–
–	–	210	110	30	54.0	None	–	–	–	–	–
49.5	None	330	120	32	61.6	None	–	–	–	–	–
47.2	None	360	100	37	51.5	None	–	–	–	–	–
46.1	None	350	110	38	55.4	None	–	–	–	–	–
46.7	None	450	110	40	54.0	None	–	–	–	–	–

Footnotes for Table 13

Note:

Recipes used:

Ingredients	Parts by Weight	
	All Except Recipes PA-210, PA-211, and PA-212	Recipes PA-210, PA-211, and PA-212
Hycar 4021	100	100
Filler	As shown	As shown
Stearic acid	1	1
Sulfur	–	0.9
Methyl Tuads	2	1
Triethylene tetramine	1.5	2.1

(a) All tempering was for 7 hours at 350 F.

(b) Recipes PA-167 and PA-201 were prepared by coprecipitating Hycar 4521 Latex with Indulin A. Recipe PA-168 was prepared by dry milling Indulin A into Hycar 4021.

TABLE 14. EFFECT OF ADDING DPR N-27 TO

Recipe	Silene EF, phr by weight	DPR N-27, phr by weight	Cure, minutes at 310 F	Treat-ment After Cure	Original Physical Properties			Physical Properties in Penola Turbo Oil-15		
					Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A
PA-144	70	0	60	None	990	260	72	720	70	61
			120	None	1050	230	76	910	70	65
			60	Tempered[a]	1390	100	80	870	70	67
			120	Tempered	1440	90	85	870	60	69
PA-184	70	10	60	None	800	330	77	860	60	73
			120	None	890	210	80	910	50	75
			60	Tempered	1210	100	86	980	50	80
			120	Tempered	1320	90	88	1010	50	80
PA-185	70	20	60	None	840	630	71	710	70	66
			120	None	960	480	71	680	50	70
			60	Tempered	1210	150	80	880	60	74
			120	Tempered	1260	140	80	790	50	75
PA-169	81.7	0	60	None	1010	140	82	1080	50	81
			120	None	990	120	85	1120	50	81
			60	Tempered	1100	60	88	1160	40	83
			120	Tempered	1150	50	89	1190	40	84
PA-186	81.7	10	60	None	1000	470	84	990	50	82
			120	None	1030	300	86	1110	50	83
			60	Tempered	1570	120	91	1200	50	85
			120	Tempered	1540	100	93	1310	50	85
PA-187	81.7	20	60	None	950	640	80	900	70	77
			120	None	980	440	81	880	70	81
			60	Tempered	1240	130	89	1050	60	85
			120	Tempered	1200	100	92	1090	50	86

Note:

Base recipe:

Ingredients	Parts by Weight
Hycar 4021	100
Silene EF	As shown
DPR N-27	As shown
Stearic acid	1
Methyl Tuads	2
Triethylene tetramine	1.5

(a) All tempering was for 7 hours at 350 F.

SILENE EF-LOADED HYCAR 4021 VULCANIZATES

After Aging 72 Hours at 350 F		Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 500 Hours at 350 F				
Swell, %	Cracking	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking
35.2	None	820	80	60	36.5	None	670	90	57	49.3	None
30.5	None	860	70	67	32.8	None	880	90	62	41.8	None
27.7	None	820	60	66	33.1	None	820	80	65	42.4	None
28.0	None	850	60	68	32.3	None	780	70	67	42.4	None
28.2	None	800	50	72	29.4	None	1100	70	75	37.6	None
25.7	None	1060	40	76	28.4	None	1150	60	78	34.1	None
24.0	Cracked	1030	50	78	28.5	None	1130	60	80	34.3	None
24.0	Cracked	1010	40	79	28.8	None	1240	60	83	33.3	Cracked
30.0	None	750	70	72	31.7	None	980	70	80	39.8	None
29.6	None	740	60	73	30.7	None	930	70	77	36.4	None
30.0	None	880	50	76	30.8	None	1230	60	85	36.3	Cracked
27.3	None	890	50	77	32.4	None	1240	60	83	37.4	Cracked
22.1	Cracked	1060	50	79	24.8	Cracked	–	–	–	–	–
20.9	Cracked	1030	50	82	24.1	Cracked	–	–	–	–	–
19.1	Cracked	1260	50	82	22.9	Cracked	–	–	–	–	–
20.6	Cracked	1180	50	83	22.5	Cracked	–	–	–	–	–
25.7	None	1190	60	83	29.2	None	–	–	–	–	–
24.5	None	1270	50	86	26.5	None	–	–	–	–	–
24.5	Cracked	1210	50	84	29.4	Cracked	–	–	–	–	–
24.0	Cracked	1340	50	90	27.0	Cracked	–	–	–	–	–
26.3	None	990	70	81	30.3	None	–	–	–	–	–
24.5	None	1250	50	89	25.1	Cracked	–	–	–	–	–
25.1	None	1350	50	91	26.3	Cracked	–	–	–	–	–
23.3	Cracked	1330	40	92	23.8	Cracked	–	–	–	–	–

TABLE 15. EFFECT OF SODIUM METASILICATE AS A VULCANIZING AGENT IN HYCAR 4021

Recipe	Cure, minutes at 310 F	Treatment After Cure	Original Physical Properties			Physical Properties After Aging in Penola Turbo Oil-15 72 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F				
			Tensile Strength, psi	Elongation, %	Hardness, Shore A	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking
PA-174	30	None	2520	220	72	980	40	72	26.4	Cracked	870	40	72	37.8	Cracked
	60	None	2050	200	74	720	40	77	25.2	Cracked	900	30	74	29.1	Cracked
	120	None	2140	80	83	1150	40	78	25.9	Cracked	870	30	79	33.9	Cracked
	30	Tempered(a)	1880	80	80	1080	40	78	24.6	Cracked	880	40	73	34.3	Cracked
	60	Tempered	1790	80	82	1020	40	80	22.3	Cracked	950	40	77	30.6	Cracked
	120	Tempered	1920	60	87	980	40	80	22.9	Cracked	1070	40	80	28.2	Cracked
PA-175	30	None	1610	480	52	300	70	47	48.3	None	220	60	49	54.7	None
	60	None	1730	330	55	280	60	52	46.0	None	230	50	51	49.0	None
	120	None	1640	270	55	350	60	55	43.7	None	230	50	53	49.2	None
	30	Tempered	1630	160	63	390	60	51	45.2	None	270	60	51	53.1	None
	60	Tempered	1560	150	65	310	60	53	44.3	None	200	50	55	48.6	None
	120	Tempered	1350	130	66	340	50	58	42.7	None	230	50	57	50.6	None

Note:

Recipes used:

Ingredients	Parts by Weight	
	PA-174	PA-175
Hycar 4021	100	100
Philblack A	40	40
Stearic acid	1	1
Sodium metasilicate	9	1.5
Trimene base	–	2

(a) All tempering was for 7 hours at 350 F.

TABLE 16. CRACKING OF PHILBLACK A-FILLED

Recipe	Cure, minutes at 310 F	Original Physical Properties			Physical Properties After Aging in Turbo Oil-15 72 Hours at 350 F					
		Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking	Type of Turbo Oil
PA-52	30	1590	300	67	670	130	52	43.4	None	Penola-1
PA-82	15	1920	260	70	750	110	52	42.1	None	Penola-1
	30	1820	230	72	870	110	55	39.7	None	Penola-1
	60	1940	200	73	860	100	55	39.1	None	Penola-1
	120	1920	160	74	820	100	58	36.1	None	Penola-1
PA-150	30	1540	180	65	600	100	42	43.4	None	Penola-2
	60	1540	140	68	460	80	47	39.6	None	Penola-2
	120	1520	130	69	460	80	49	36.4	None	Penola-2
PA-151	30	1720	180	70	710	90	48	41.8	None	Penola-2
	60	1680	130	74	790	80	50	37.6	None	Penola-2
	120	1920	110	75	770	80	53	37.9	None	Penola-2
PA-154	120	1810	150	67	650	80	46	39.1	None	Penola-1
PA-162	60	2010	220	66	590	110	42	42.1	None	Penola-1
					620	110	42	42.3	None	Penola-2

Note:

Recipes used:

Ingredients	Parts by Weight	
	PA-151	All Others
Hycar 4021	100	100
Philblack A	50	40
Stearic acid	1	1
Sulfur	0.9	0.9
Methyl Tuads	1	1
Triethylene tetramine	2.1	2.1

Penola-1 indicates Penola Turbo Oil-15, Code LF-1294-11, Drum 1.
Penola-2 indicates Penola Turbo Oil-15, Code LF-1294-11, Drum 2.

All samples untempered.

HYCAR 4021 VULCANIZATES AFTER AGING

Physical Properties After Aging in Turbo Oil-15 168 Hours at 350 F						Physical Properties After Aging in Turbo Oil-15 500 Hours at 350 F					
Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Type of Turbo Oil	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Type of Turbo Oil
760	130	52	46.2	None	Penola-1	820	150	46	55.4	None	Penola-1
400	80	56	43.1	None	Penola-1	550	100	47	53.2	None	Penola-1
460	80	58	41.2	None	Penola-1	620	110	50	53.5	None	Penola-1
560	70	59	39.8	None	Penola-1	600	90	53	52.2	None	Penola-1
690	70	60	40.3	None	Penola-1	790	110	53	46.5	None	Penola-1
540	80	50	45.0	None	Penola-2	400	80	39	75.1	Cracked	Penola-2
520	80	50	40.0	None	Penola-2	240	60	40	85.9	Cracked	Penola-2
510	80	53	39.9	None	Penola-2	230	60	43	76.4	Cracked	Penola-2
350	60	53	41.1	None	Penola-2	330	70	44	67.6	Cracked	Penola-2
690	70	55	40.6	None	Penola-2	150	60	44	60.5	Cracked	Penola-2
710	70	57	40.2	None	Penola-2	160	60	44	64.5	Cracked	Penola-2
660	80	48	45.0	None	Penola-1	210	70	32	131.9	Cracked	Penola-1
						230	80	32	68.3	Cracked	Penola-1
						240	70	33	76.1	Cracked	Penola-2
510	90	42	52.8	None	Penola-1	0	90	20	90.0	Cracked	Penola-1
470	90	43	49.1	None	Penola-2	90	100	23	90.3	Cracked	Penola-2

TABLE 17. CRACKING OF PHILBLACK A-FILLED

| Recipe | Cure, minutes at 310 F | Original Physical Properties | | | Physical Properties After Aging in Turbo Oil-15 72 Hours at 350 F | | | | | Type of Turbo Oil |
		Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking	
PA-191	60	1940	220	69	670	90	48	42.6	None	Penola-2
PA-192	60	1690	230	65	640	90	47	40.4	None	Penola-2
PA-193	60	1620	120	78	430	110	41	30.7	None	Penola-2
PA-194	60	1580	290	64	650	130	35	51.8	None	Penola-2
PA-195	60	1550	190	68	670	120	41	45.6	None	Penola-2

Note:

Recipes used:

| Ingredients | Parts by Weight | | | | |
	PA-191	PA-192	PA-193	PA-194	PA-195
Hycar 4021 (old, Lot 207)	100	100	100	–	–
Hycar 4021 (new, Lot 241)	–	–	–	100	100
Philblack A (old)	40	–	–	40	–
Philblack A (old, heated)	–	–	40	–	–
Philblack A (new)	–	40	–	–	40
Stearic acid	1	1	1	1	1
Sulfur	0.9	0.9	0.9	0.9	0.9
Methyl Tuads	1	1	·1	1	1
Triethylene tetramine	2.1	2.1	2.1	2.1	2.1

Penola-2 indicates Penola Turbo Oil-15, Code LF-1294-11, Drum 2.

All samples untempered.

HYCAR 4021 VULCANIZATES AFTER AGING

Physical Properties After Aging in Turbo Oil-15 168 Hours at 350 F						Physical Properties After Aging in Turbo Oil-15 500 Hours at 350 F					
Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking	Type of Turbo Oil	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking	Type of Turbo Oil
690	120	41	48.3	None	Penola-2	290	90	32	116.9	Cracked	Penola-2
570	100	42	48.9	None	Penola-2	250	100	33	88.3	Cracked	Penola-2
280	100	29	41.7	None	Penola-2	250	50	46	52.7	Cracked	Penola-2
640	130	36	60.4	None	Penola-2	160	120	28	130.9	Cracked	Penola-2
490	120	42	54.1	None	Penola-2	110	90	28	83.4	Edge cracking	Penola-2

TABLE 18. PROPERTIES OF CARBON BLACK-LOADED

Recipe	Vulcanizing Agent, phr			Filler		Cure, minutes at 310 F	Treatment After Cure	Original Physical Properties			Physical Properties After Turbo Oil-15 72 Hours		
	TETA	Sulfur	Methyl Tuads	Type	Amount, phr			Tensile Strength, psi	Elongation, %	Hardness, Shore A	Tensile Strength, psi	Elongation, %	Hardness, Shore A
PA-161	0.8	–	2.0	Philblack O	50	–	–	–	–	–	–	Undercured at 120	
PA-110	0.8	0.8	2.4	Philblack O	40	–	–	–	–	–	–	Undercured at 120	
PA-160	0.8	1.0	2.0	Philblack O	50	–	–	–	–	–	–	Undercured at 120	
PA-159	0.8	2.0	–	Philblack O	50	60	None	660	970	62	450	810	28
						120	None	860	810	64	530	710	28
PA-158	0.8	2.0	1.0	Philblack O	50	60	None	690	1270	53	80	>1200	23
						120	None	1150	970	55	220	1030	23
PA-109	0.8	2.4	0.8	Philblack O	40	60	None	1770	790	63	350	1380	35
PA-57	1.5	–	2.0	Philblack O	40	–	–	–	–	–	–	Undercured at 120	
PA-58	1.5	1.0	–	Philblack O	40	30	None	1790	670	70	1150	520	38
PA-83	1.5	1.0	–	Philblack O	50	15	None	2370	520	81	1180	430	43
						30	None	2410	470	82	1200	330	48
						60	None	2320	450	84	1120	360	50
PA-155	1.5	1.0	–	Philblack O	50	60	None	2020	450	68	1450	380	43
						120	None	2270	380	71	1500	280	47
PA-157	1.5	1.0	2.0	Philblack O	50	60	None	1950	540	62	1060	620	29
						120	None	2060	520	63	1210	470	32
PA-84	1.5	2.0	–	Philblack O	50	30	None	2270	490	84	1080	390	47
						60	None	2310	410	87	1260	330	53
PA-156	1.5	2.0	–	Philblack O	50	60	None	2030	410	70	1400	350	46
						120	None	2040	380	74	1470	290	53
PA-108	2.1	0.9	1.0	Philblack O	40	30	None	2430	450	71	1220	310	46
PA-122	2.1	0.9	1.0	Philblack O	50	30	None	2370	400	82	1280	250	55
						60	None	2400	310	82	1310	180	60
						120	None	2380	250	85	1300	150	69
						30	Tempered[a]	2150	260	84	1470	220	62
						60	Tempered	2020	180	86	1410	170	67
						120	Tempered	1850	130	90	1100	120	72
PA-123	2.1	0.9	1.0	Philblack O	40	30	None	2290	440	73	1360	230	54
						60	None	2450	360	74	1270	180	57
						120	None	2500	310	75	1130	150	60
						30	Tempered	2290	290	77	1200	190	55
						60	Tempered	2290	200	81	1110	150	60
						120	Tempered	1970	160	81	1160	140	64
PA-141	4.2	1.8	2.0	Philblack O	40	30	None	2280	190	79	1250	100	67
						60	None	2120	140	83	1380	70	75
						120	None	2370	120	87	1370	70	81
						30	Tempered	2040	110	87	1240	80	69
						60	Tempered	1870	80	92	1100	60	76
						120	Tempered	2480	80	95	1230	60	83
PA-106	0.8	0.8	2.4	Philblack A	40	–	–	–	–	–	–	Undercured at 120	
PA-105	0.8	2.4	0.8	Philblack A	40	–	–	–	–	–	–	Undercured at 120	

Aging in ___ at 350 F			Physical Properties After Aging in Turbo Oil-15 168 Hours at 350 F						Physical Properties After Aging in Turbo Oil-15 500 Hours at 350 F					
Swell, %	Cracking	Type of Turbo Oil	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Type of Turbo Oil	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Type of Turbo Oil
minutes	–	–	–	–	–	–	–	–	–	–	–	–	–	–
minutes	–	–	–	–	–	–	–	–	–	–	–	–	–	–
minutes	–	–	–	–	–	–	–	–	–	–	–	–	–	–
52.2	None	Penola	210	470	27	46.3	None	Penola	–	–	–	–	Cracked	Penola
53.8	None	Penola	340	370	32	43.9	None	Penola	–	–	–	–	Cracked	Penola
60.4	None	Penola	210	470	37	50.3	None	Penola	–	–	–	–	Cracked	Penola
56.9	None	Penola	110	420	31	47.2	None	Penola	–	–	–	–	Cracked	Penola
49.9	None	Penola	400	1150	34	58.1	None	Penola	–	–	–	–	Cracked	Penola
minutes	–	–	–	–	–	–	–	–	–	–	–	–	–	–
43.4	None	Esso	520	330	40	39.5	None	Esso	–	–	–	–	–	–
48.1	None	Esso	520	300	41	42.4	None	Esso	–	–	–	–	–	–
45.3	None	Esso	500	230	49	42.6	None	Esso	360	110	71	27.2	None	Penola
47.1	None	Esso	450	210	52	40.9	None	Esso	–	–	–	–	–	–
45.2	None	Penola	890	340	42	43.6	None	Penola	640	20	85	20.7	Cracked	Penola
38.1	None	Penola	630	190	48	32.2	Cracked	Penola	540	20	87	19.1	Cracked	Penola
48.6	None	Penola	300	210	42	40.0	None	Penola	–	–	–	–	Cracked	Penola
43.3	None	Penola	370	160	45	31.5	None	Penola	–	–	–	–	Cracked	Penola
47.4	None	Esso	540	290	48	42.6	None	Esso	–	–	–	–	–	–
42.4	None	Esso	350	140	55	36.3	None	Esso	–	–	–	–	–	–
39.5	None	Penola	690	230	47	40.1	None	Penola	600	30	87	16.9	Cracked	Penola
40.0	None	Penola	580	150	55	37.2	None	Penola	810	20	91	20.6	Cracked	Penola
42.0	None	Penola	1200	300	46	44.1	None	Penola	460	100	68	24.1	Cracked	Penola
39.0	None	Penola	1320	200	60	39.0	None	Penola	700	40	89	18.1	Cracked	Penola
34.8	None	Penola	1080	160	63	35.6	None	Penola	550	40	87	16.7	Cracked	Penola
30.2	None	Penola	830	100	70	32.3	None	Penola	530	30	88	16.9	Cracked	Penola
37.3	None	Penola	1120	230	60	38.5	None	Penola	–	–	–	–	–	–
37.5	None	Penola	770	110	67	37.9	None	Penola	400	50	79	26.3	Cracked	Penola
33.0	None	Penola	660	80	72	34.4	None	Penola	700	30	90	23.2	Cracked	Penola
37.7	None	Penola	940	170	55	38.8	None	Penola	400	40	80	17.4	Cracked	Penola
33.9	None	Penola	820	140	60	32.5	None	Penola	230	50	70	20.0	Cracked	Penola
30.5	None	Penola	680	110	62	30.0	None	Penola	420	30	83	16.5	Cracked	Penola
39.5	None	Penola	950	170	55	40.8	None	Penola	340	40	76	26.7	Cracked	Penola
32.3	None	Penola	670	120	60	34.7	None	Penola	530	30	85	22.6	Cracked	Penola
33.1	None	Penola	620	90	67	31.7	None	Penola	530	30	85	20.3	Cracked	Penola
24.8	None	Penola	780	80	67	27.6	Cracked	Penola	–	–	–	–	–	–
20.5	None	Penola	640	50	79	23.2	Cracked	Penola	–	–	–	–	–	–
18.0	None	Penola	–	– Insufficient sample		–		–	–	–	–	–	–	–
27.1	None	Penola	810	70	75	28.2	Cracked	Penola	–	–	–	–	–	–
23.4	None	Penola	780	50	82	25.2	Cracked	Penola	–	–	–	–	–	–
20.8	Cracked	Penola	1010	40	89	22.6	Cracked	Penola	–	–	–	–	–	–
minutes	–	–	–	–	–	–	–	–	–	–	–	–	–	–
minutes	–	–	–	–	–	–	–	–	–	–	–	–	–	–

TABLE 18.

Recipe	TETA	Sulfur	Methyl Tuads	Filler Type	Amount, phr	Cure, minutes at 310 F	Treatment After Cure	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Tensile Strength, psi	Elongation, %	Hardness, Shore A
PA-5	1.5	–	2.0	Philblack A	40	30	None	1310	820	66	750	800	32
						30	Tempered	1690	450	71	850	600	34
PA-107	1.8	0.4	1.8	Philblack A	40	60	None	1640	620	63	720	920	34
PA-104	2.1	0.9	1.0	Philblack A	40	30	None	1920	450	71	980	300	43
PA-120	2.1	0.9	1.0	Philblack A	40	30	None	2120	300	75	1200	150	55
						60	None	2120	230	78	1150	130	64
						120	None	2110	190	82	1300	120	69
						30	Tempered	2180	200	82	1140	140	60
						60	Tempered	2150	150	86	1060	120	67
						120	Tempered	1980	130	88	1050	90	73
PA-121	2.1	0.9	1.0	Philblack A	50	30	None	1930	250	80	1170	150	63
						60	None	1980	200	82	1220	120	72
						120	None	2180	180	84	1350	110	73
						30	Tempered	1840	170	86	1110	140	64
						60	Tempered	1970	130	86	1260	110	73
						120	Tempered	2020	130	86	1120	90	76

Note:

Base recipe:

Ingredients	Parts by Weight
Acrylon EA-5	100
Filler	As shown
Stearic acid	1
Vulcanizing agent	As shown

(a) All tempering was for 7 hours at 350 F.

(Continued)

Aging in at 350 F			Physical Properties After Aging in Turbo Oil-15 168 Hours at 350 F						Physical Properties After Aging in Turbo Oil-15 500 Hours at 350 F					
Swell, %	Cracking	Type of Turbo Oil	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking	Type of Turbo Oil	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking	Type of Turbo Oil
52.1	None	Esso	340	450	32	43.6	None	Esso	–	–	–	–	Cracked	Penola
48.7	None	Esso	410	400	34	40.4	None	Esso	–	–	–	–	–	–
54.6	None	Penola	800	890	33	54.4	None	Penola	–	–	–	–	–	–
39.0	None	Penola	1140	260	47	37.6	None	Penola	480	50	83	24.9	Cracked	Penola
32.3	None	Penola	540	90	63	31.8	None	Penola	320	40	70	19.2	Cracked	Penola
30.1	None	Penola	710	90	67	28.9	Cracked	Penola	–	–	–	–	–	–
25.8	None	Penola	610	60	75	25.6	Cracked	Penola	–	–	–	–	–	–
35.3	None	Penola	910	110	64	35.4	None	Penola	290	40	75	18.7	Cracked	Penola
31.8	None	Penola	540	70	68	31.3	Cracked	Penola	–	–	–	–	–	–
28.8	None	Penola	600	60	77	29.9	Cracked	Penola	–	–	–	–	–	–
32.6	None	Penola	960	110	67	34.2	None	Penola	620	30	90	14.5	Cracked	Penola
28.9	None	Penola	720	80	71	28.3	None	Penola	680	30	92	15.0	Cracked	Penola
27.3	None	Penola	750	80	74	26.9	None	Penola	690	30	91	15.3	Cracked	Penola
36.0	None	Penola	930	100	69	38.9	None	Penola	610	30	87	20.7	Cracked	Penola
30.2	None	Penola	730	70	75	30.6	Cracked	Penola	–	–	–	–	–	–
28.1	None	Penola	700	60	77	28.3	Cracked	Penola	–	–	–	–	–	–

TABLE 19. PROPERTIES OF HI-SIL-LOADED

	Vulcanizing Agent, phr			Hi-Sil, phr	Cure, minutes at 310 F	Treat-ment After Cure	Original Physical Properties			Physical Properties in Penola Turbo Oil-15		
Recipe	TETA	Sulfur	Methyl Tuads				Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A
PA-139	2.1	0.9	1.0	43.3	30	None	1360	860	63	560	750	41
					60	None	1050	750	70	560	650	47
					120	None	1500	690	67	940	580	47
					30	Tempered[a]	1600	790	72	380	670	33
					60	Tempered	1360	630	72	560	660	42
					120	Tempered	1680	600	74	790	570	45
PA-140	2.1	0.9	1.0	54.2	30	None	1190	780	80	430	580	57
					60	None	1340	780	82	600	610	63
					120	None	1370	650	83	850	520	65
					30	Tempered	1610	780	83	540	570	55
					60	Tempered	1510	700	87	520	580	60
					120	Tempered	1630	560	87	780	470	65
PA-216	2.8	1.2	1.3	54.2	60	None	1320	550	90	–	–	–
					120	None	1710	480	90	–	–	–
PA-217	3.5	1.5	1.7	54.2	60	None	1820	440	90	–	–	–
					120	None	1820	310	91	–	–	–
PA-181	4.2	1.8	2.0	54.2	60	None	1970	310	90	1640	140	82
					120	None	2090	220	94	1720	110	85
					60	Tempered	1980	190	94	1540	130	84
					120	Tempered	1950	130	95	1540	90	88
PA-182	2.1	0.9	1.0	65.0	60	None	1080	580	94	460	230	80
					120	None	1200	460	96	770	300	85
					60	Tempered	1200	470	95	560	320	83
					120	Tempered	1380	420	97	680	220	82

Note:

Base recipe:	Ingredients	Parts by Weight
	Acrylon EA-5	100
	Hi-Sil	As shown
	Stearic acid	1
	Vulcanizing agents	As shown

(a) All tempering was for 7 hours at 350 F.

(b) Slight disintegration of sample.

ACRYLON EA-5 VULCANIZATES AFTER AGING

After Aging 72 Hours at 350 F		Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 500 Hours at 350 F				
Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking
48.0	None	440	720	41	53.7	None	–	–	–	–	–
47.9	None	640	680	48	58.1	None	–	–	–	–	–
43.0	None	830	510	51	50.4	None	–	–	–	–	–
49.8	None	280	680	42	53.6	None	–	–	–	–	–
45.9	None	380	710	42	52.1	None	–	–	–	–	–
46.1	None	520	590	42	48.3	None	–	–	–	–	–
40.3	None	470	640	54	47.2	None	130	240	45	44.8	None
39.1	None	540	600	62	47.8	None	270	420	52	43.0	None
36.2	None	730	480	63	40.9	None	390	250	60	38.7	Crack at edge
40.9	None	280	620	58	44.9	None	–	–	–	–	–
38.9	None	380	620	63	43.0	None	–	–	–	–	–
38.2	None	600	490	64	42.9	None	310	300	59	38.8	Crack at edge
–	–	770	260	73	36.6	None	370	50	84	26.8	Cracked
–	–	760	180	75	32.9	None	530	30	88	22.5	Cracked
–	–	1260	160	82	29.4	None	560	40	89	21.5	Cracked
–	–	1240	120	85	23.8	Crack at edge	890	20	95	17.9	Cracked
22.2	None	1110	70	87	23.0	None	–	–	–	–	–
18.9	None	1220	50	88	19.8	Cracked	–	–	–	–	–
25.1	None	1440	110	85	28.1	Cracked	–	–	–	–	–
21.0	None	1180	80	89	22.9	Cracked	–	–	–	–	–
30.5	None	340	30	74	41.1	None[b]	–	–	–	–	–
28.7	None	470	140	82	38.2	None[b]	–	–	–	–	–
32.7	None	480	300	79	37.6	None[b]	–	–	–	–	–
30.6	None	460	190	80	36.4	None[b]	–	–	–	–	–

Recipe	Filler Type	Amount, phr	Cure, minutes at 310 F	Treatment After Cure	Original Physical Properties Tensile Strength, psi	Elongation, %	Hardness, Shore A	Physical Properties in Penola Turbo Oil-15 Tensile Strength, psi	Elongation, %	Hardness, Shore A
PA-137	Silene EF	46.6	60	None	1190	730	56	800	480	27
			120	None	1330	540	60	1000	300	39
			60	Tempered[a]	1350	680	57	790	420	30
			120	Tempered	1420	450	64	970	300	42
PA-138	Silene EF	58.3	60	None	1300	750	69	900*	440	39
			120	None	1670	500	70	1370	260	53
			60	Tempered	1560	610	70	900	370	41
			120	Tempered	1810	400	71	1300	210	53
PA-180	Silene EF	70.0	60	None	1410	380	78	1170	200	68
			120	None	1670	270	84	1440	130	72
			60	Tempered	1590	380	81	1140	200	67
			120	Tempered	1750	240	87	1390	130	73
PA-204	P-33 carbon black	70.0	60	None	1040	320	76	–	–	Insufficient
			120	None	1090	290	78	910	190	56
PA-205	Philblack A	20.0	30	None	1570	370	67	1060	230	40
	P-33 carbon black	35.0	60	None	1530	280	70	990	170	47
			120	None	1460	220	73	950	160	53

Note:

Base recipe:

Ingredients	Parts by Weight
Acrylon EA-5	100
Filler	As shown
Stearic acid	1
Sulfur	0.9
Methyl Tuads	1.0
Triethylene tetramine	2.1

(a) All tempering was for 7 hours at 350 F.

PROPERTIES OF ACRYLON EA-5 VULCANIZATES AFTER AGING

After Aging 72 Hours at 350 F		Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 500 Hours at 350 F				
Swell, %	Cracking	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking
54.6	None	740	460	25	54.0	None	–	–	–	–	Crack at edge
42.4	None	1000	270	41	43.3	None	–	–	–	–	Cracked
46.2	None	720	450	30	51.5	None	–	–	–	–	–
38.8	None	900	280	41	43.3	None	–	–	–	–	–
44.0	None	860	450	42	47.6	None	–	–	–	–	–
32.1	None	1340	240	55	37.6	None	500	100	61	29.3	Cracked
42.7	None	720	360	38	46.9	None	–	–	–	–	–
35.0	None	1260	190	55	36.6	None	590	90	67	34.1	Cracked
33.2	None	1150	190	68	37.2	None	790	170	67	33.7	Cracked
28.7	None	1450	120	74	29.7	None	1080	80	77	31.8	Cracked
33.7	None	1080	190	66	37.6	None	850	160	70	38.1	Cracked
28.3	None	1460	110	75	30.7	None	910	70	79	29.0	Cracked
sample	–	540	110	57	25.8	None	–	–	–	–	–
23.6	None	420	70	67	18.4	Cracked	–	–	–	–	–
32.9	None	600	150	46	32.6	None	660	40	85	11.7	Cracked
29.0	None	480	110	52	29.1	None	840	30	89	14.1	Cracked
26.1	None	450	90	56	24.8	None	1000	20	96	16.5	Cracked

TABLE 21. EFFECT OF MERCAPTOBENZIMIDAZOLE ON AGING

Recipe	Mercaptobenz-imidazole, phr	Cure, minutes at 310 F	Treatment After Cure	Original Physical Properties			Physical Properties After Aging in Penola Turbo Oil-15 72 Hours at 350 F				
				Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking
PA-213	0	30	None	1430	570	72	1100	450	31	47.0	None
		60	None	1640	480	73	1230	460	33	45.5	None
		120	None	1750	410	76	1400	360	40	40.2	None
		30	Tempered[a]	1710	370	80	1250	370	40	42.1	None
		60	Tempered	1900	320	80	1130	410	41	42.6	None
		120	Tempered	1770	260	82	1270	330	46	38.8	None
PA-214	1	30	None	2060	370	80	1470	270	48	38.4	None
		60	None	2110	270	82	1530	230	53	34.9	None
		120	None	2210	290	82	1420	210	57	33.8	None
		30	Tempered	1910	230	83	1470	250	55	36.2	None
		60	Tempered	2160	190	84	1390	200	59	34.8	None
		120	Tempered	2020	180	86	1500	210	62	32.6	None
PA-215	5	30	None	1870	330	82	1440	220	58	31.2	None
		60	None	1950	290	83	1310	170	64	28.3	None
		120	None	1920	260	84	1500	160	70	28.1	None
		30	Tempered	1790	190	88	1280	190	62	30.8	None
		60	Tempered	1810	170	90	1440	170	68	26.7	None
		120	Tempered	1860	170	90	1480	150	73	25.9	None

Note:

Base recipe:

Ingredients	Parts by Weight
Acrylon E A-5	100
Philblack A	50
Stearic acid	1
Mercaptobenzimidazole	As shown
Sulfur	1
Triethylene tetramine	1.5

(a) All tempering was for 7 hours at 350 F.

PROPERTIES OF ACRYLON EA-5 VULCANIZATES

Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking
	Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 500 Hours at 350 F			
580	280	34	40.8	None	770	40	90	10.2	Cracked
610	250	38	35.0	None	730	30	92	12.5	Cracked
480	190	44	33.7	None	600	40	88	13.9	Cracked
560	250	38	37.0	None	350	40	82	23.4	Cracked
280	140	44	33.2	None	600	30	90	18.3	Cracked
460	170	47	33.6	None	490	30	89	14.8	Cracked
470	110	56	35.3	None	1030	30	93	13.9	Cracked
500	100	57	34.1	None	1110	20	96	15.3	Cracked
500	90	63	30.6	None	1210	20	96	13.5	Cracked
700	140	53	34.3	None	640	60	83	19.3	Cracked
480	90	59	33.3	None	840	30	92	19.5	Cracked
470	80	59	29.8	None	730	20	94	16.5	Cracked
590	90	74	28.2	None	1240	30	95	8.2	Cracked
820	120	71	27.9	None	1090	20	95	12.5	Cracked
820	90	81	23.2	Cracked	1430	20	98	8.6	Cracked
730	110	73	29.6	None	1090	20	95	14.4	Cracked
540	60	81	25.2	Cracked	1210	10	97	18.4	Cracked
540	60	81	24.1	Cracked	1490	10	99	16.2	Cracked

TABLE 22.   AGING PROPERTIES OF HYCAR 4021X26

Recipe	Polymer	Cure, minutes at 310 F	Treatment After Cure	Original Physical Properties			Physical Properties After Penola Turbo Oil-15 72 Hours		
				Tensile Strength, psi	Elonga- tion, %	Hard- ness, Shore A	Tensile Strength, psi	Elonga- tion, %	Hard- ness, Shore A
PA-143	Hycar 4021X26	60	None Tempered[a]	2050 1850	440 260	74 87	1110 1140	280 230	45 52
PA-142	Acrylon EA-9	60	None Tempered	2450 2800	140 90	86 95	1880 1590	90 70	82 84

Note:

Recipes used:

Ingredients	Parts by Weight	
	PA-143	PA-142
Hycar 4021X26	100	—
Acrylon EA-9	—	100
Philblack O	40	40
Stearic acid	1	1
Sulfur	0.9	0.9
Methyl Tuads	1	1
Triethylene tetramine	2.1	2.1

(a) All tempering was for 7 hours at 350 F.

AND ACRYLON EA-9 VULCANIZATES

Aging in at 350 F		Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 500 Hours at 350 F				
Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking
37.3	None	390	190	41	35.6	None	420	30	82	19.1	Cracked
39.0	None	470	130	52	36.4	None	580	20	87	22.1	Cracked
15.6	None	1090	60	89	13.4	Cracked	–	–	–	–	–
19.2	None	1350	50	91	18.5	Cracked	–	–	–	–	–

TABLE 23. EFFECT OF CURING CONDITIONS ON

| Recipe | Treatment After Cure | Original Physical Properties | | | Physical Properties After Aging in Penola Turbo Oil-15 72 Hours at 350 F | | | | |
		Tensile Strength, psi	Elonga- tion, %	Hard- ness, Shore A	Tensile Strength, psi	Elonga- tion, %	Hard- ness, Shore A	Swell, %	Cracking
PA-164A[a]	None	950	240	75	860	70	64	32.6	None
B	None	960	340	73	750	80	60	37.9	None
C	None	990	180	80	920	60	69	28.4	None
D	None	990	250	75	810	70	62	35.1	None
PA-165A[a]	None	2260	370	77	1350	290	45	41.6	None
B	None	2330	420	74	1150	310	43	42.4	None
C	None	2380	350	80	1340	230	50	37.9	None
D	None	2320	330	75	1240	290	44	46.6	None

Note:

| | | Parts by Weight | |
Recipes used:	Ingredients	PA-164	PA-165
	Hycar 4021	100	—
	Acrylon EA-5	—	100
	Silene EF	70	—
	Philblack O	—	50
	Stearic acid	1	1
	Methyl Tuads	2	—
	Triethylene tetramine	1.5	1.5
	Sulfur	—	1

(a) A = Cured 60 minutes in press in mold at 310 F.

B = Cured 30 minutes in press, removed from mold, and cured for 30 minutes in air oven at 310 F.

C = Cured 120 minutes in press in mold at 310 F.

D = Cured 30 minutes in press, removed from mold, and cured for 90 minutes in air oven at 310 F.

AGING PROPERTIES OF ACRYLATE VULCANIZATES

Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 500 Hours at 350 F				
Tensile Strength, psi	Elonga- tion, %	Hard- ness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elonga- tion, %	Hard- ness, Shore A	Swell, %	Cracking
900	70	64	34.3	None	960	90	62	44.6	None
710	70	62	39.3	None	840	90	60	52.8	None
900	60	68	30.4	None	950	80	64	42.7	None
850	70	63	36.8	None	890	90	61	46.7	None
630	190	49	39.3	None	670	20	89	21.0	Cracked
1200	310	41	43.9	None	720	30	87	23.3	Cracked
860	180	52	36.1	None	800	20	89	18.9	Cracked
1030	300	43	42.0	None	550	30	87	17.7	Cracked

TABLE 24. EFFECT OF AGING TIME BETWEEN COMPOUNDING

Recipe	Aging Time Between Compounding and Curing, hours	Original Physical Properties			Physical Properties After Aging in Penola Turbo Oil-15 72 Hours at 350 F				
		Tensile Strength, psi	Elongation, %	Hardness, Shore A	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking
PA-166	< 4	930	250	79	820	60	66	27.9	None
	24	970	200	85	970	60	74	26.7	None
	72	1100	190	83	1040	60	73	26.0	None
	168	1270	290	87	1080	60	75	27.8	None

Note:

Base recipe:

Ingredients	Parts by Weight
Hycar 4021	100
Silene EF	70
Stearic acid	1
Methyl Tuads	2
Triethylene tetramine	1.5

Cure: 60 minutes at 310 F.

All samples untempered.

AND CURING ON PROPERTIES OF HYCAR 4021 VULCANIZATES

Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 500 Hours at 350 F				
Tensile Strength, psi	Elonga- tion, %	Hard- ness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elonga- tion, %	Hard- ness, Shore A	Swell, %	Cracking
860	60	70	30.5	None	870	80	70	43.3	None
870	60	75	28.1	None	1010	80	71	39.2	None
970	60	75	27.8	None	1080	70	72	37.5	None
1030	60	76	29.7	None	1140	90	73	39.9	None

TABLE 25. AGING OF HYCAR 4021 VULCANIZATES

Recipe	Aging Medium	Manufacturer	Original Physical Properties			Physical Properties After Aging 72 Hours at 350 F				
			Tensile Strength, psi	Elonga- tion, %	Hard- ness, Shore A	Tensile Strength, psi	Elonga- tion, %	Hard- ness, Shore A	Swell, %	Cracking
PA-162	Turbo Oil-15	Penola Oil Co.	2010	220	66	620	110	42	42.3	None
	DEHS[a]	Rohm and Haas				590	110	42	47.8	None
	DEHS[a]	Pittsburgh Coke and Chemical				570	120	38	53.8	None
	DEHS[a]	Harchem				350	120	25	77.7	None
	DEHS[a]	Rubber Corporation of America				800	150	33	56.5	None
	DEHS[a]	Reichold				960	160	37	57.4	None
PA-163	Turbo Oil-15	Penola Oil Co.	930	290	71	700	70	60	35.8	None
	DEHS[a]	Rohm and Haas				750	90	58	28.6	None
	DEHS[a]	Pittsburgh Coke and Chemical				730	80	58	28.7	None
	DEHS[a]	Harchem				680	90	55	41.2	None
	DEHS[a]	Rubber Corporation of America				870	100	60	26.0	None
	DEHS[a]	Reichold				880	100	57	31.5	None

Note:

		Parts by Weight	
Recipes used:	Ingredients	PA-162	PA-163
	Hycar 4021	100	100
	Philblack A	40	—
	Silene EF	—	70
	Stearic acid	1	1
	Methyl Tuads	1	2
	Sulfur	0.9	—
	Triethylene tetramine	2.1	1.5

Cure: 60 minutes at 310 F.

All samples untempered.

(a) Di-(2-ethylhexyl) sebacate.
(b) Samples partially disintegrated.

Physical Properties After Aging 168 Hours at 350 F					Physical Properties After Aging 500 Hours at 350 F				
Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking
470	90	43	49.1	None	90	100	23	90.3	Cracked
630	130	40	48.6	None					
450	140	31	57.0	None					
260	160	20	64.0	None	Samples badly swollen, fell apart when handled; estimated swelling: 200%				
640	140	29	77.3	None					
430	140	26	60.5	None					
640	70	59	39.3	None	860	100	53	53.4	None
740	90	56	−18.7[b]	None	510	70	50	Disintegrated	None
670	80	58	−11.9[b]	None	300	50	52	Disintegrated	None
540	80	53	−4.1[b]	None	240	60	54	Disintegrated	None
690	90	56	11.3[b]	None	450	60	53	Disintegrated	None
640	80	58	5.4[b]	None	490	60	54	Disintegrated	None

TABLE 26. COMPARISON OF OPEN- AND CLOSED-

Recipe	Aging Conditions	Original Physical Properties			Physical Properties After Aging in Penola Turbo Oil-15 72 Hours at 350 F				
		Tensile Strength, psi	Elongation, %	Hardness, Shore A	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking
PA-162[a]	Open tube[b]	2010	220	66	620	110	42	42.3	None
	Closed tube[c]	–	–	–	430	110	33	41.4	None
PA-163[a]	Open tube[b]	930	290	71	700	70	60	35.8	None
	Closed tube[c]	–	–	–	690	70	59	36.9	None

(a) See Table 25 for recipes used.

(b) Open tube – test tube covered with loose-fitting Petri dish, allowing air to enter during aging.

(c) Closed tube – test tube fitted with ground glass stopper, allowing no fresh air to enter during aging.

TUBE AGING WITH HYCAR 4021 VULCANIZATES

Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 500 Hours at 350 F				
Tensile Strength psi	Elonga- tion, %	Hard- ness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elonga- tion, %	Hard- ness, Shore A	Swell, %	Cracking
470	90	43	49.1	None	90	100	23	90.3	Cracked
400	90	36	49.1	None	330	90	36	77.9	None
640	70	59	39.3	None	860	100	53	53.4	None
620	70	59	36.5	None	710	60	62	44.8	None

TABLE 27. COMPARISON OF

Recipe	Cure, minutes at 310 F	Original Physical Properties			Physical Properties After Aging in Penola Turbo Oil-15 72 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 72 Hours at 400 F				
		Tensile Strength, psi	Elongation, %	Hardness, Shore A	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking
PA-140	30	1190	780	80	430	580	57	40.3	None	230	540	53	40.1	None
	120	1370	650	83	850	520	65	36.2	None	410	510	53	42.2	None
PA-144	60	990	260	72	720	70	61	35.2	None	520	60	64	33.5	None
PA-148	120	760	300	72	520	90	53	34.8	None	610	60	61	35.0	None
PA-164	30	950	240	75	860	70	64	32.6	None	620	60	66	34.1	None
PA-170	30	1500	510	92	1060	90	72	38.3	None	970	70	76	40.1	None
	120	1360	240	95	1260	80	80	30.5	None	1070	70	77	34.6	None
PA-180	120	1670	270	84	1440	130	72	28.7	None	1360	110	75	27.4	None
PA-195	60	1550	190	68	670	120	41	45.6	None	400	80	46	50.4	None

Note:

Recipes used:

Ingredients	Parts by Weight						
	PA-140	PA-144	PA-148	PA-164	PA-170	PA-180	PA-195
Hycar 40 21	—	100	100	100	100	—	100
Acrylon EA-5	100	—	—	—	—	100	—
Philblack A	—	—	—	—	—	—	40
Hi-Sil	54.2	—	—	—	65	—	—
Silene EF	—	70	46.6	70	—	70	—
Calcene TM	—	—	55.6	—	—	—	—
Stearic acid	1	1	1	1	1	1	1
Sulfur	0.9	—	—	—	—	0.9	0.9
Methyl Tuads	1	2	2	2	2	1	1
Triethylene tetramine	2.1	1.5	1.5	1.5	1.5	2.1	2.1

All samples untempered.

Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 400 F					Physical Properties After Aging in Penola Turbo Oil-15 500 Hours at 350 F				
Tensile Strength, psi	Elonga- tion, %	Hard- ness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elonga- tion, %	Hard- ness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elonga- tion, %	Hard- ness, Shore A	Swell, %	Cracking
470	640	54	47.2	None	140	330	51	44.1	Cracked	130	240	45	44.8	None
730	480	63	40.9	None	260	320	55	43.5	Cracked	390	250	60	38.7	Edge cracking
820	80	60	36.5	None	500	50	64	41.3	None	670	90	57	49.3	None
590	90	56	37.3	None	550	60	61	44.3	None	–	–	–	–	–
900	70	64	34.3	None	490	50	63	39.1	None	960	90	62	44.6	None
1000	80	77	38.6	None	800	70	76	49.4	None	1160	120	77	50.8	None
1270	80	81	32.6	None	930	70	74	44.1	None	680	110	77	47.5	Edge cracking
1450	120	74	29.7	None	1020	90	75	26.8	None	1080	80	77	31.8	Cracked
490	120	42	54.1	None	190	70	43	68.4	None	110	90	28	83.4	Edge cracking

TABLE 28. THE EFFECT OF ADDING

Recipe	Hycar 1001, parts by weight	Treatment After Cure	Cure, minutes at 310 F	Original Physical Properties			Physical Properties After Turbo Oil-15 72 Hours			
				Tensile Strength, psi	Elongation, %	Hardness, Shore A	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %
PA-88	0	None	30	1560	490	64	510	90	55	41.1
PA-80	5	None	30	1750	500	64	690	110	53	44.3
PA-81	10	None	30	1590	590	63	800	120	52	44.8
PA-101	15	None	30	1450	510	64	590	90	60	39.7
		Tempered[a]	30	1340	130	85	880	80	75	35.1
PA-102	20	None	30	1380	560	64	780	110	57	40.6
		Tempered	30	1580	170	83	740	80	72	36.9
PA-134	30	None	30	1350	570	49	500	90	50	39.6
		None	60	1740	440	50	260	50	53	36.1
		None	120	1470	320	51	410	70	54 ·	36.3
		Tempered	30	740	70	75	740	70	68	32.7
		Tempered	60	770	70	75	540	40	70	32.9
		Tempered	120	760	70	76	560	40	70	31.1
PA-135	40	None	30	1410	550	44	210	60	49	38.7
		None	60	1510	450	46	220	60	51	35.3
		None	120	1540	360	47	240	50	53	34.5
		Tempered	30	730	70	76	400	30	70	31.7
		Tempered	60	620	60	77	450	30	70	31.4
		Tempered	120	670	60	78	420	20	71	31.4
PA-136	40	None	30	1180	270	54	290	60	60	34.6
		None	60	1220	230	56	280	50	60	32.1
		None	120	1330	190	57	300	40	63	29.2
		Tempered	30	790	60	80	660	30	75	30.2
		Tempered	60	740	50	81	560	30	75	29.5
		Tempered	120	800	40	81	500	20	76	27.5

Note·
Recipes used:

Ingredients	Parts by Weight	
	All Except Recipe PA-136	P A-136
Hycar 4021	100	100
Hycar 1001	As shown	As shown
Philblack A	40	40
Stearic acid	1	1
Methyl Tuads	2	1
Triethylene tetramine	1.5	2.1
Sulfur	–	0.9

(a) Tempering was for 7 hours at 350 F.

Aging in at 350 F		Physical Properties After Aging in Turbo Oil-15 168 Hours at 350 F						Physical Properties After Aging in Turbo Oil-15 500 Hours at 350 F					
Cracking	Type of Turbo Oil	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Type of Turbo Oil	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Type of Turbo Oil
None	Penola	390	80	56	44.4	None	Penola	610	120	48	58.7	None	Penola
None	Esso	560	100	58	48.3	None	Esso	860	160	59	53.4	None	Esso
None	Esso	630	100	62	52.5	None	Esso	930	170	68	57.3	None	Esso
None	Penola	860	90	68	41.0	None	Penola	970	80	85	54.4	None	Penola
None	Penola	900	70	77	37.7	None	Penola	1120	70	91	49.2	None	Penola
None	Penola	740	90	63	46.2	None	Penola	930	100	75	60.4	None	Penola
None	Penola	850	80	74	39.6	None	Penola	950	80	83	51.5	None	Penola
None	Penola	540	70	60	45.1	None	Penola	–	–	–	–	–	–
None	Penola	510	50	61	40.5	Cracked	Penola	–	–	–	–	–	–
None	Penola	380	40	62	38.4	Cracked	Penola	–	–	–	–	–	–
None	Penola	780	50	72	34.6	Cracked	Penola	–	–	–	–	–	–
None	Penola	540	40	72	38.4	Cracked	Penola	–	–	–	–	–	–
Cracked	Penola	580	30	73	33.1	Cracked	Penola	–	–	–	–	–	–
None	Penola	330	40	60	47.4	Cracked	Penola	–	–	–	–	–	–
None	Penola	250	30	63	42.7	Cracked	Penola	–	–	–	–	–	–
None	Penola	320	30	61	39.9	Cracked	Penola	–	–	–	–	–	–
Cracked	Penola	410	20	72	35.7	Cracked	Penola	–	–	–	–	–	–
Cracked	Penola	420	20	73	34.9	Cracked	Penola	–	–	–	–	–	–
Cracked	Penola	500	20	74	34.9	Cracked	Penola	–	–	–	–	–	–
None	Penola	610	40	71	35.7	Cracked	Penola	–	–	–	–	–	–
Cracked	Penola	430	40	73	37.6	Cracked	Penola	–	–	–	–	–	–
Cracked	Penola	530	30	73	32.3	Cracked	Penola	–	–	–	–	–	–
Cracked	Penola	750	30	78	36.6	Cracked	Penola	–	–	–	–	–	–
Cracked	Penola	720	30	78	31.8	Cracked	Penola	–	–	–	–	–	–
Cracked	Penola	660	20	78	29.9	Cracked	Penola	–	–	–	–	–	–

TABLE 29. EFFECT OF VARYING HYCAR 4021 AND

Recipe	Polymer Ratio		Treatment After Cure	Cure, minutes at 310 F	Original Physical Properties			Physical Properties After Aging in Turbo Oil-15 72 hours at 350 F					Type of Turbo Oil
	Hycar 4021	Acrylon EA-5			Tensile Strength, psi	Elongation, %	Hardness, Shore A	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	
PA-82	100	–	None	60	1940	200	73	860	100	55	39.1	None	Penola
			None	120	1920	160	74	820	100	58	36.1	None	Penola
PA-124	80	20	None	60	1940	210	70	670	100	51	43.0	None	Penola
			None	120	2000	200	70	820	110	51	40.8	None	Penola
			Tempered[a]	60	1640	130	70	930	110	53	42.7	None	Penola
			Tempered	120	1520	130	69	1010	120	54	43.3	None	Penola
PA-125	60	40	None	60	1920	230	71	860	120	50	42.1	None	Penola
			None	120	1860	190	70	810	110	50	37.7	None	Penola
			Tempered	60	1770	140	70	850	100	53	41.1	None	Penola
			Tempered	120	1660	130	70	780	100	55	40.4	None	Penola
PA-126	40	60	None	60	1940	330	65	1030	160	46	42.3	None	Penola
			None	120	1650	190	69	950	130	50	36.4	None	Penola
			Tempered	60	1810	190	71	1010	150	48	42.4	None	Penola
			Tempered	120	1630	150	71	1010	120	53	39.7	None	Penola
PA-127	20	80	None	60	2000	310	68	1140	170	44	37.9	None	Penola
			None	120	2160	230	69	1100	140	50	33.4	None	Penola
			Tempered	60	2010	190	74	1160	160	51	39.8	None	Penola
			Tempered	120	1920	150	76	1050	130	55	36.0	None	Penola
PA-120	–	100	None	30	2120	300	75	1200	150	55	32.3	None	Penola
			None	60	2120	230	78	1150	130	64	30.1	None	Penola
			None	120	2110	190	82	1300	120	69	25.8	None	Penola
			Tempered	30	2180	200	82	1140	140	60	35.3	None	Penola
			Tempered	60	2150	150	86	1060	120	67	31.8	None	Penola
			Tempered	120	1980	130	88	1050	90	73	28.8	None	Penola

Note:

Base recipe:	Ingredients	Parts by Weight
	Polymer	As shown
	Philblack A	40
	Stearic acid	1
	Methyl Tuads	1
	Sulfur	0.9
	Triethylene tetramine	2.1

(a) All tempering was for 7 hours at 350 F.

ACRYLON EA-5 RATIO ON AGING PROPERTIES

Physical Properties After Aging in Turbo Oil-15 168 Hours at 350 F						Physical Properties After Aging in Turbo Oil-15 500 Hours at 350 F					
Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking	Type of Turbo Oil	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking	Type of Turbo Oil
560	70	59	39.8	None	Penola	600	90	53	52.2	None	Penola
690	70	60	40.3	None	Penola	790	110	53	46.5	None	Penola
770	100	55	43.4	None	Penola	490	90	45	49.6	None	Penola
760	100	56	42.2	None	Penola	380	80	49	54.6	None	Penola
700	90	53	48.2	None	Penola	290	90	47	52.8	Cracked	Penola
940	110	53	46.4	None	Penola	470	100	48	53.7	Cracked	Penola
840	110	54	42.7	None	Penola	440	70	55	36.6	Cracked	Penola
770	90	55	38.5	None	Penola	260	60	55	39.4	Cracked	Penola
790	110	52	43.0	None	Penola	270	70	52	44.3	Cracked	Penola
790	100	51	41.9	None	Penola	370	70	54	40.3	Cracked	Penola
650	130	45	44.4	None	Penola	290	70	57	32.0	Cracked	Penola
960	130	50	38.6	None	Penola	280	70	67	30.1	Cracked	Penola
890	150	48	43.0	None	Penola	100	60	60	40.4	Cracked	Penola
700	120	52	42.5	None	Penola	140	60	60	35.8	Cracked	Penola
1110	170	45	38.0	None	Penola	380	50	77	24.0	Cracked	Penola
1130	140	51	33.7	None	Penola	410	40	79	24.3	Cracked	Penola
960	150	51	40.2	None	Penola	230	40	71	28.1	Cracked	Penola
630	110	55	35.6	None	Penola	330	40	71	24.5	Cracked	Penola
540	90	63	31.8	None	Penola	320	40	70	19.2	Cracked	Penola
710	90	67	28.9	Cracked	Penola	–	–	–	–	–	–
610	60	75	25.6	Cracked	Penola	–	–	–	–	–	–
910	110	64	35.4	None	Penola	290	40	75	18.7	Cracked	Penola
540	70	68	31.3	Cracked	Penola	–	–	–	–	–	–
600	60	77	29.9	Cracked	Penola	–	–	–	–	–	–

TABLE 30. SUMMARY OF COMPRESSION-SET TESTS

			Permanent Set, per cent				
			Aged in Penola Turbo Oil-15 at 350 F				Aged in Hot Air at 250 F 70 Hours,
			72 Hours		168 Hours		
Recipe	Cure, minutes at 310 F	Treatment After Cure	Sample 0.150 Inch Thick	Sample 0.225 Inch Thick	Sample 0.150 Inch Thick	Sample 0.225 Inch Thick	Sample 0.525 Inch Thick
PA-16	30	None	—	92	—	97	—
		Tempered	—	74	—	81	—
PA-52	30	None	—	94	—	96	—
		Tempered	—	64	—	69	—
PA-144	60	None	—	89	—	90	—
PA-147	60	None	—	91	92	86	—
	120	None	74	86	81	94	88
PA-149	60	None	86	94	93	97	93
	120	None	—	—	91	94	—
PA-184	60	None	—	—	89	96	—

Note:

Recipes used:

| Ingredients | Parts by Weight | | | | | |
	PA-16	PA-52	PA-144	PA-147	PA-149	PA-184
Hycar 4021	100	100	100	100	100	100
Philblack A	—	40	—	—	—	—
Hi-Sil	45.2	—	—	—	—	—
Silene EF	—	—	70	58.3	46.6	70
Calcene TM	—	—	—	27.8	83.4	—
DPR N-27	—	—	—	—	—	10
Stearic acid	1	1	1	1	1	1
Sulfur	—	0.9	—	—	—	—
Methyl Tuads	2	1	2	2	2	2
Triethylene tetramine	1.5	2.1	1.5	1.5	1.5	1.5

TABLE 31. LOW-TEMPERATURE-RETRACTION

	Recipe PA-52		Recipe PA-83		Recipe PA-88		Recipe PA-95	
	Untempered	Tempered	Untempered	Tempered	Untempered	Tempered	Untempered	Tempered
								Retraction
Low-Temperature-Retraction Value								
TR-10	−17.4	−15.4	−9.0	−9.4	−20.5	−15.0	−17.2	−14.4
TR-20	−15.1	−13.1	−6.2	−6.1	−16.9	−12.6	−14.8	−11.4
TR-50	−10.3	−8.7	−0.9	−0.2	−10.7	−8.0	−9.6	−7.3
TR-70	−7.7	−6.3	+2.8	+4.1	−6.6	−4.3	−6.0	−2.8
								Recipe, parts
Ingredients								
Hycar 4021	100		—		100		100	
Acrylon EA-5	—		100		—		—	
Philblack A	40		—		40		—	
Philblack O	—		50		—		—	
Silene EF	—		—		—		70	
Hi-Sil	—		—		—		—	
Calcene TM	—		—		—		—	
Stearic acid	1.0		1.0		1.0		1.0	
Sulfur	0.9		1.0		—		—	
Methyl Tuads	1.0		—		2.0		2.0	
Triethylene tetramine	2.1		1.5		1.5		1.5	
Flexol R2H	—		—		—		—	
DPR N-27	—		—		—		—	
Cure, minutes at 310 F	30		30		30		30	

TESTS ON ACRYLATE VULCANIZATES

	Recipe PA-98		Recipe PA-144	Recipe PA-147	Recipe PA-147	Recipe PA-149	Recipe PA-149	Recipe PA-184	Recipe PA-185	Recipe PA-185
	Untempered	Tempered	Untempered	Untempered	Untempered	Untempered	Untempered	Untempered	Untempered	Untempered
Temperature, C										
	−18.9	−15.8	−17.5	−16.3	−15.6	−15.5	−15.3	−18.9	−23.3	−18.7
	−15.0	−12.5	−15.1	−13.1	−11.8	−11.8	−11.1	−15.0	−18.1	−15.5
	−8.0	−6.3	−10.1	−8.8	−8.3	−8.3	−7.5	−10.5	−11.9	−10.2
	−3.2	+2.4	−7.9	−4.5	−3.9	−3.1	−2.9	−7.0	−7.5	−6.8

by weight

Recipe PA-98	Recipe PA-144	Recipe PA-147	Recipe PA-147	Recipe PA-149	Recipe PA-149	Recipe PA-184	Recipe PA-185	Recipe PA-185
100	100	100	100	100	100	100	100	100
–	–	–	–	–	–	–	–	–
–	–	–	–	–	–	–	–	–
–	–	–	–	–	–	–	–	–
–	70	58.3	58.3	46.6	46.6	70	70	70
75.8	–	–	–	–	–	–	–	–
–	–	27.8	27.8	83.4	83.4	–	–	–
1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
–	–	–	–	–	–	–	–	–
2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
10	–	–	–	–	–	–	–	–
–	–	–	–	–	–	10	20	20
30	60	60	120	60	120	60	60	120

Recipe	MgO, phr	Polyac, phr	PbO₂, phr	Temperature Cure (60 Minutes), F	Original Physical Properties			Physical Properties in Penola Turbo Oil-15		
					Tensile Strength, psi	Elongation, %	Hardness, Shore A	Tensile Strength, psi	Elongation, %	Hardness, Shore A
FBA-3	25	5	5	310	1160	670	37	1240	130	62
FBA-4	25	5	10	310	1350	590	43	910	60	83
FBA-5	40	5	10	310	1510	530	45	1350	50	85
FBA-6	60	5	10	310	1530	420	52	1540	40	90
FBA-7	80	5	10	310	1440	350	55	1650	40	92
FBA-8	20	3	3	425	–	–	–	–	–	Bubbled
FBA-9	40	3	3	400	1310	210	67	–	–	Not
FBA-10	60	3	3	400	1280	140	77	–	–	Not
FBA-11	20	3	1.5	400	–	–	–	–	–	Stuck
FBA-12	40	3	1.5	400	1240	260	53	900	150	62
FBA-13	60	3	1.5	400	1060	210	69	–	–	Not
FBA-18	20	3	3	400	–	–	–	–	–	Stuck
		Sulfur, phr	TETA[a], phr							
FBA-14	10	1	1	310	1050	370	36	300	120	55
FBA-15	20	1	1	310	1190	300	44	410	80	63
FBA-16	40	1	1	310	1840	130	69	900	40	89
FBA-17	60	1	1	310	1790	120	80	–	–	Not

(a) Triethylene tetramine.

MAGNESIA IN POLY-FBA COMPOUNDS

After Aging 72 Hours at 350 F		Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 500 Hours at 350 F				
Swell, %	Cracking	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking
9.3	None	880	60	72	20.5	None	350	20	74	100.4	Cracked
10.2	None	780	20	90	14.5	Cracked	410	0	85	101.6	Cracked
5.5	None	1570	20	92	10.5	Cracked	510	0	93	72.9	Cracked
7.5	Cracked	1800	20	92	9.0	Cracked	590	0	95	68.2	Cracked
4.5	Cracked	1870	20	96	14.5	Cracked	200	0	98	83.3	Cracked
and stuck in the mold	–	–	–	–	–	–	–	–	–	–	–
tested	–	–	–	–	–	–	–	–	–	–	–
tested	–	–	–	–	–	–	–	–	–	–	–
to the mold	–	–	–	–	–	–	–	–	–	–	–
5.3	None	640	80	70	40.6	None	350	20	79	56.9	Cracked
tested	–	–	–	–	–	–	–	–	–	–	–
to the molds badly	–	–	–	–	–	–	–	–	–	–	–
2.4	None	520	100	64	7.6	None	520	20	75	44.4	Cracked
1.5	None	490	60	71	5.8	None	240	0	82	39.8	Cracked
–5.5	Cracked	720	30	90	20.2	Cracked	–	Too brittle to be tested			–
tested	–	–	–	–	–	–	–	–	–	–	–

TABLE 33. THE EFFECTS OF SEVERAL

	Filler			Original Physical Properties			Physical Properties Penola Turbo Oil-15		
Recipe	Name	Amount, phr	TETA[a] phr	Tensile Strength, psi	Elonga- tion, %	Hard- ness, Shore A	Tensile Strength, psi	Elonga- tion, %	Hard- ness, Shore A
FBA[b]	Philblack O	35	1.0	1315	348	60	–	–	–
FBA[b]	Philblack O	40	1.1	1263	360	60	–	–	–
FBA-1	Philblack O	35	1.0	960	150	63	770	110	64
FBA-2	Philblack E	35	1.25	1110	370	66	730	200	57
FBA-19	Philblack O	35	1.0	1230	430	43	950	330	50
FBA-20	Philblack A	35	1.0	1340	410	44	1010	300	49
FBA-21	Philblack E	35	1.0	940	690	50	650	480	53
FBA-25	P-33	60	1.5	690	180	68	Stuck to the mold; sufficient cured		
FBA-26	P-33	60	1.75	870	120	72			
FBA-27	P-33	60	2.0	–	–	–	–	–	Stuck to
FBA-22	Ludox[c]	20	1.0	Stuck to the mold badly; could not be tested					
FBA-23	Ludox[c]	40	1.0						
FBA-24	Ludox[c]	60	1.0	420	170	49	460	140	59
FBA-28	Aerosil	35	1.5	1260	320	91	850	110	93
FBA-29	Aerosil	35	1.75	1170	100	95	Original hardness too high		
FBA-30	Aerosil	35	2.0	1320	80	95			
FBA-31	Aerosil	25	1.1	1230	200	72	930	120	72
FBA-32	Aerosil	25	1.2	1120	180	74	1050	110	75
FBA-33	Aerosil	25	1.3	1280	180	75	1060	110	80
FBA-34	Aerosil	25	1.4	–	–	–	–	–	Tore apart

Note:

Base recipe:

Ingredients	Parts by Weight
Poly-FBA	100
Paraffin	1
Sulfur	1
Filler	As shown
Triethylene tetramine	As shown

Cure: 60 minutes at 310 F.

(a) Triethylene tetramine.

(b) Reported in a paper by Major M. C. Hamlin, WADC, at the SAE Symposium on Aircraft High Temperature Static Seals for All Media; Cleveland, Ohio, May 18, 1954.

(c) Du Pont colloidal silica; air dried, then ball milled 48 hours.

FILLERS ON POLY-FBA COMPOUNDS

After Aging 72 Hours at 350 F		Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 500 Hours at 350 F				
Swell, %	Cracking	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elonga-tion, %	Hard-ness, Shore A	Swell, %	Cracking
–	–	–	–	–	–	–	749	295	53	–	None
–	–	–	–	–	–	–	911	190	66	–	None
6.9	None	740	100	64	6.2	None	590	120	65	6.2	None
6.6	None	880	280	62	6.3	None	440	200	76	10.5	None
5.0	None	800	310	56	9.1	None	490	360	53	8.5	None
9.1	None	870	320	51	4.6	None	500	370	53	9.0	None
9.6	None	470	460	59	11.6	None	410	80	76	16.3	None
sheets for original physical properties only											
the mold; could not be tested		–	–	–	–	–	–	–	–	–	–
4.1	None	330	90	64	3.3	None	370	100	55	3.7	None
5.3	None	910	150	93	6.8	None	950	20	95	10.8	Cracked
4.4	None	990	120	72	4.4	None	660	130	80	9.3	None
4.4	None	1040	120	79	4.8	None	750	110	83	7.2	None
4.3	None	640	80	82	2.3	None	810	100	85	6.4	None
when taken from the mold		–	–	–	–	–	–	–	–	–	–

TABLE 34. BLENDING EXPERIMENTS WITH POLY-FBA

Recipe	Bd/MAA(a), phr	TETA(b), phr	Original Physical Properties			Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F					Low-Temperature Retraction Test, Retraction Temperature, C			
			Tensile Strength, psi	Elongation, %	Hardness, Shore A	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	TR-10	TR-20	TR-50	TR-70
FBA-35	–	1.0	1310	360	55	650	240	64	5.8	None	–19	–16	–11	–1
FBA-36	5	1.0	770	190	87	Not tested	–	–	–	–	–13	–5	+22	–
FBA-37	5	1.2	840	150	85	920	60	93	5.6	Cracked	–16	–15	+13	–
FBA-38	5	1.4	860	80	87	Not tested	–	–	–	–	–	–	–	–
FBA-39	10	1.0	790	170	93	1030	50	96	9.8	Cracked	–18	–9	+17	–
FBA-40	10	1.2	920	80	93	Not tested	–	–	–	–	–	–	–	–
FBA-41	10	1.4	Tore apart when taken from the mold			–	–	–	–	–	–	–	–	–

Note:

Base recipe: Ingredients	Parts by Weight
FBA polymer	100
Philblack O	35
Paraffin	1
Bd/MAA copolymer	Varied
Sulfur	1
Triethylene tetramine	Varied

Cure: 60 minutes at 310 F.

(a) 80/20-Butadiene/methacrylic acid copolymer prepared at Battelle.

(b) Triethylene tetramine.

TABLE 35. AGING PROPERTIES OF NEOPRENE Q AND BUTYL VULCANIZATES

Recipe	Polymer	Cure, minutes at 310 F	Original Physical Properties			Physical Properties After Aging in Penola Turbo Oil-15 72 Hours at 350 F					Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F				
			Tensile Strength, psi	Elongation, %	Hardness, Shore A	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking	Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking
N-1	Neoprene Q	20	1950	270	77	480	50	74	34.4	Cracked	340	20	81	28.1	Cracked
		40	2000	240	79	520	50	76	35.7	Cracked	570	0	94	30.2	Cracked
		60	1920	240	81	410	50	76	35.8	Cracked	560	0	98	29.0	Cracked
Bu-1	Butyl	60	1300	260	59	Samples disintegrated					--	--	--	--	--
		120	1220	260	59						--	--	--	--	--
Bu-3	Butyl	60	1360	280	59						--	--	--	--	--
		120	1380	300	59						--	--	--	--	--

Note:
Recipes used:

Ingredients	Parts by Weight		
	N-1	Bu-1	Bu-3
Neoprene Q	100	-	-
Butyl	-	100	100
Philblack A	50	-	-
SRF Black	-	50	50
Stearic acid	1	0.5	0.5
Zinc oxide	5	5	5
Sulfur	-	2	2
Methyl Tuads	-	1	1
Plasticizer SC	10	-	-
ELC Magnesia	4	-	-
NA-22	0.5	-	-
Polyac	-	1	1
Altax	-	1	1
Calcium peroxide	-	-	10

All samples untempered.

TABLE 36. SUMMARY OF COMPOUNDS HAVING BEST AGED PROPERTIES

Recipe	Cure, minutes at 310 F	Treatment After Cure	Original Physical Properties Tensile Strength, psi	Elongation, %	Hardness, Shore A	Physical Properties After Aging in Turbo Oil-15 500 Hours at 350 F Tensile Strength, psi	Elongation, %	Hardness, Shore A	Swell, %	Cracking
PA-95	30	None	1080	430	85	1000	100	64	42.7	None
		Tempered(a)	1260	100	93	1240	90	72	35.7	None
PA-184	60	None	800	330	77	1100	70	75	37.6	None
	120	None	890	210	80	1150	60	78	34.1	None
	60	Tempered	1210	100	86	1130	60	80	34.3	Cracked
	120	Tempered	1320	90	88	1240	60	83	33.3	Cracked
PA-180	60	None	1410	380	78	790	170	67	33.7	Cracked
	120	None	1670	270	84	1080	80	77	31.8	Cracked
	60	Tempered	1590	380	81	850	160	70	38.1	Cracked
	120	Tempered	1750	240	87	910	70	79	29.0	Cracked
A-319	60	None	2460	610	77	750	80	85	16.1	Cracked
A-23L	60	None	2000	680	70	650	90	80	24.0	Cracked
FBA-1	60	None	960	150	63	590	120	65	6.2	None
FBA-20	60	None	1340	410	44	500	370	53	9.0	None
FBA-31	60	None	1230	200	72	660	130	80	9.3	None

Note:

Recipes used:

Ingredients	Parts by Weight PA-95	PA-184	PA-180
Hycar 4021	100	100	–
Acrylon EA-5	–	–	100
Silene EF	70	70	70
DPR N-27	–	10	–
Stearic acid	1	1	1
Sulfur	–	–	0.9
Methyl Tuads	2	2	1
Triethylene tetramine	1.5	1.5	2.1

Ingredients	Parts by Weight A-319	A-23L
Hycar 1001	100	100
Zinc oxide	5	5
Magnesia ELC	100	100
Stearic acid	1.5	1.5
Sulfur	0.5	0.5
Methyl Tuads	0.25	0.25
Thioglycolic acid	5.2	–
Flectol H	3	–

Ingredients	Parts by Weight FBA-1	FBA-20	FBA-31
FBA Polymer	100	100	100
Philblack O	35	–	–
Philblack A	–	35	25
Aerosil	–	–	1
Paraffin	1	1	1
Sulfur	1	1	1
Triethylene tetramine	1	1	1.1

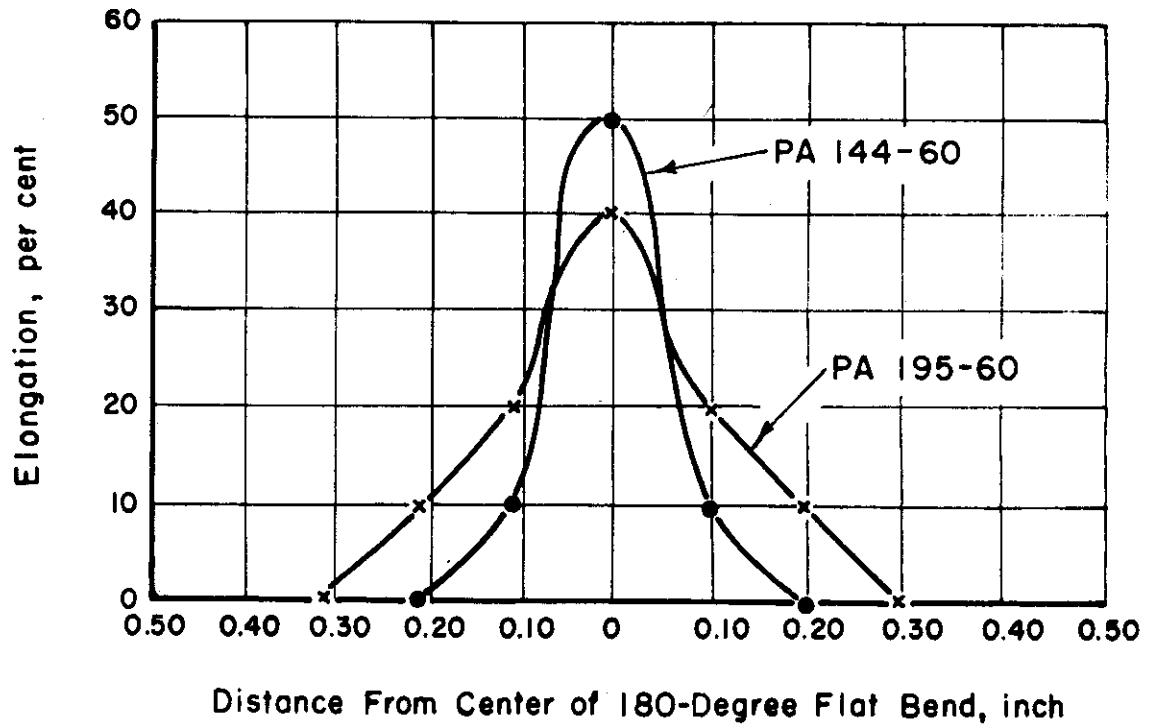(a) All tempering was for 7 hours at 350 F.

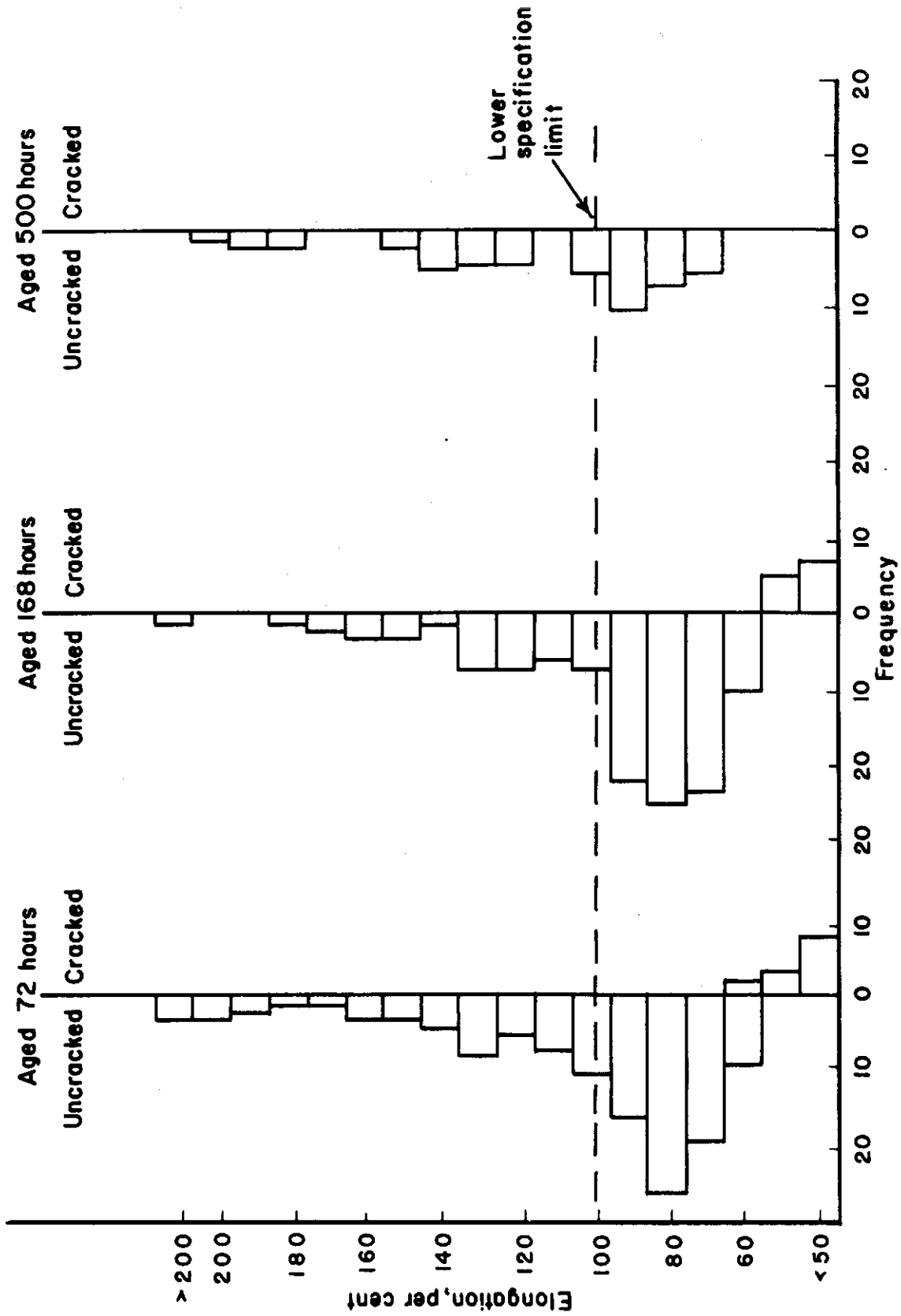FIGURE 1. ELONGATION OF 0.075-INCH-THICK VULCANIZATES
WHEN BENT 180 DEGREES

O-12021

FIGURE 2. RELATION BETWEEN CRACKING AND ELONGATION OF HYCAR 4021 VULCANIZATES
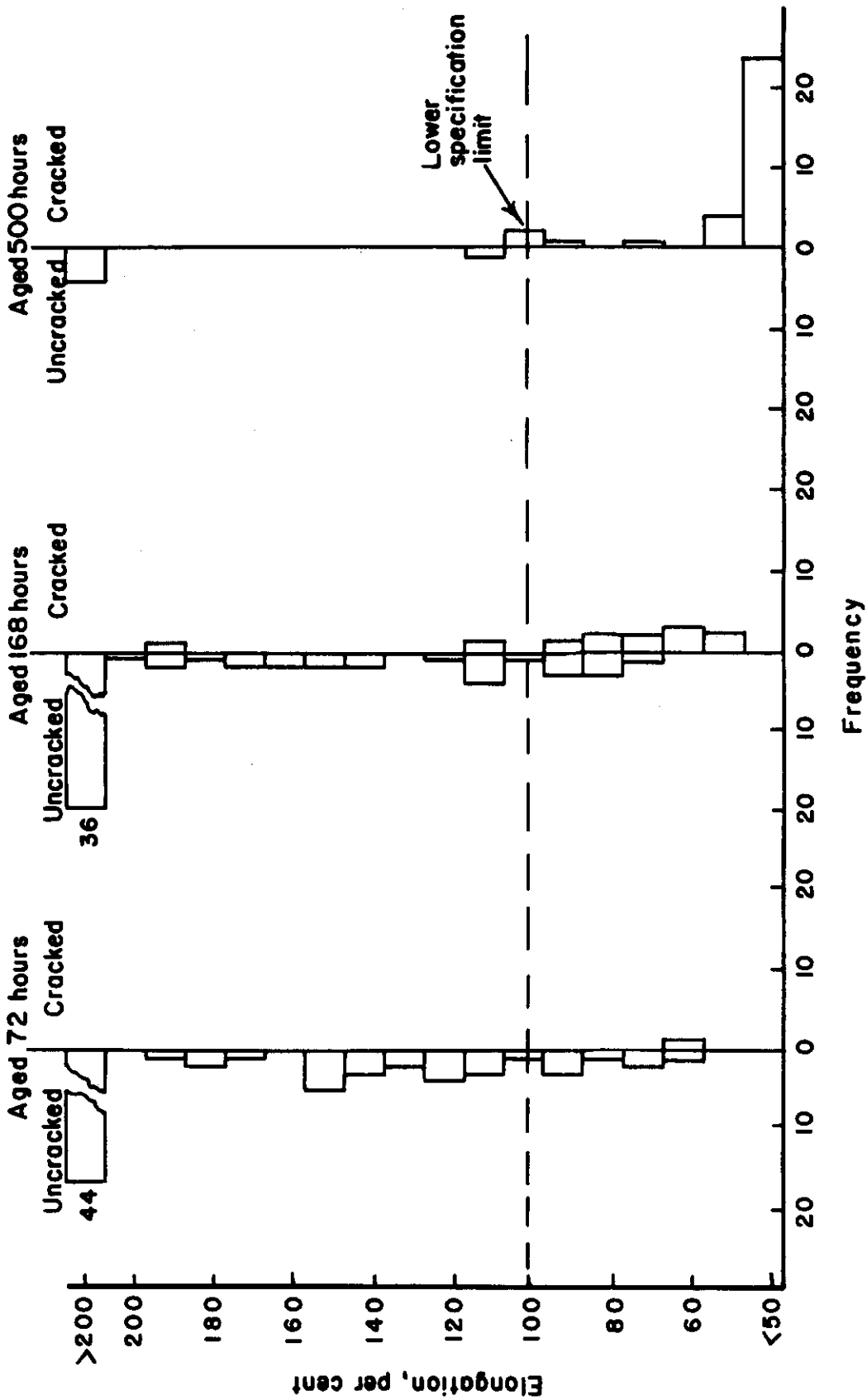AFTER AGING IN TURBO OIL-15 AT 350 F

FIGURE 3. RELATION BETWEEN CRACKING AND ELONGATION OF ACRYLON EA-5 VULCANIZATES AFTER AGING IN TURBO OIL-15 AT 350F
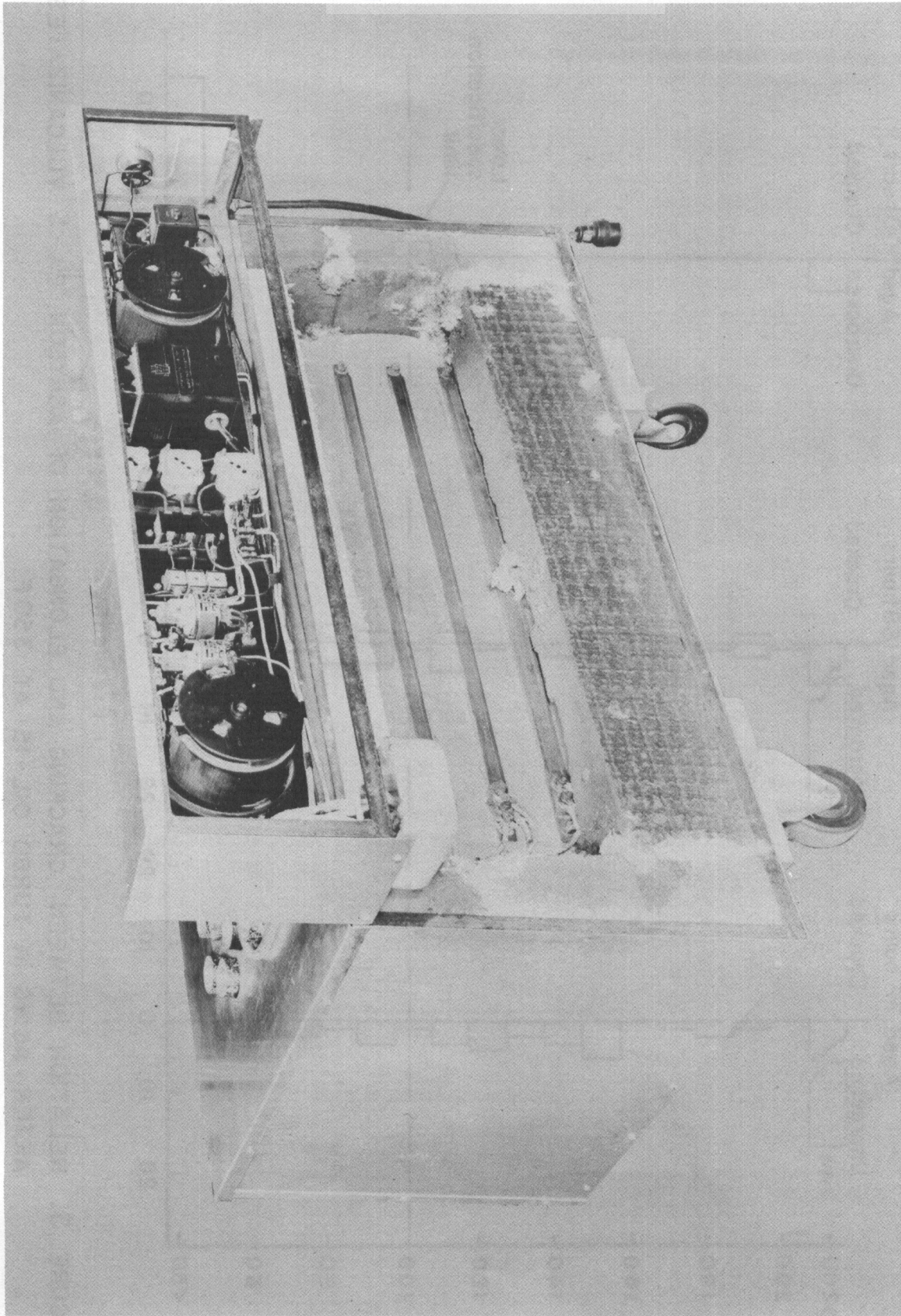
A-12019

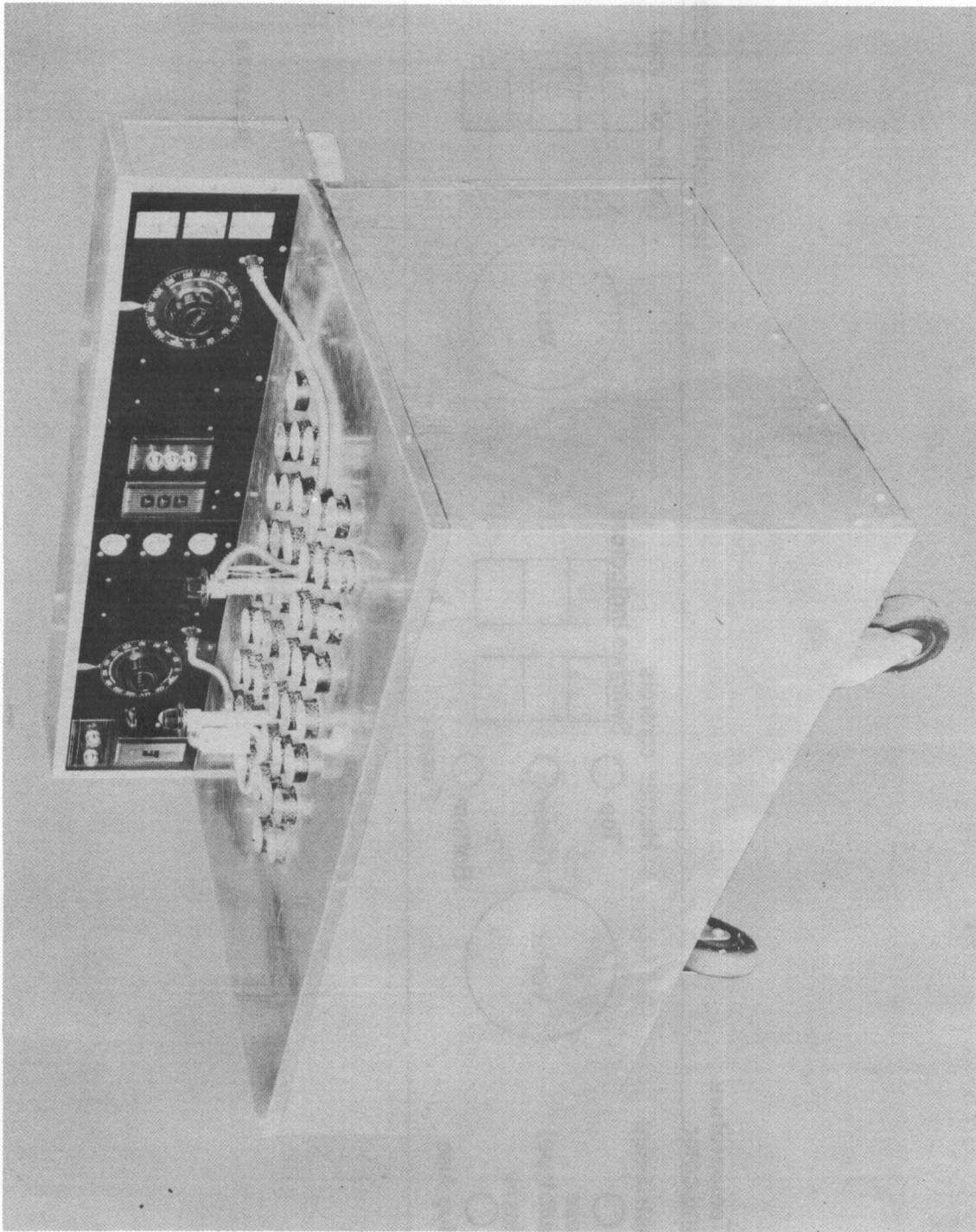FIGURE 4. REAR VIEW OF ALUMINUM-BLOCK HEATER, SHOWING CONSTRUCTION
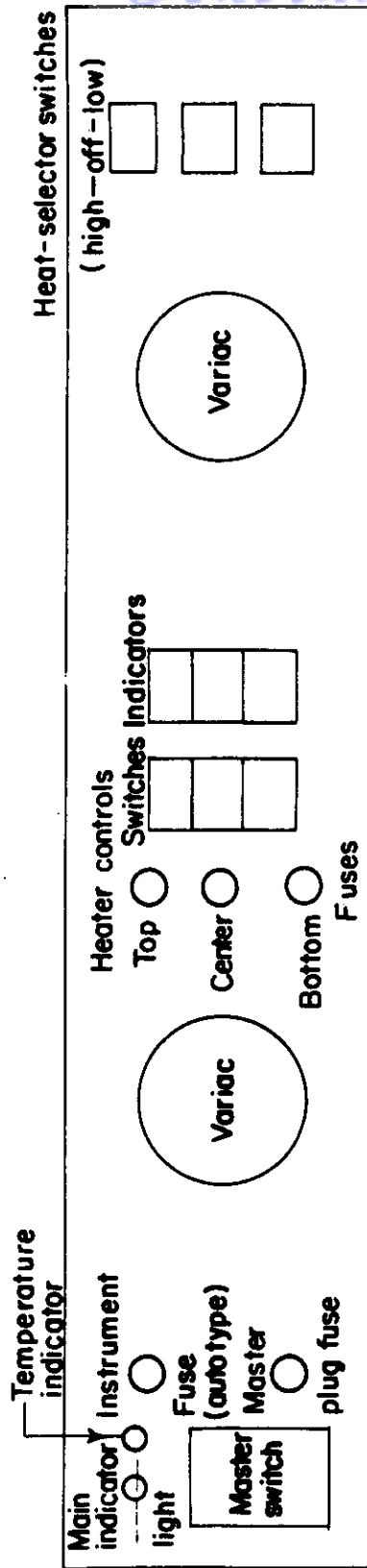
FIGURE 5. FRONT VIEW OF ALUMINUM-BLOCK HEATER

Heat-selector switches

(high—off—low)

0-22658

Variac

Switches Indicators

Heater controls

Top ◯

Center ◯

Bottom ◯

Fuses

Variac

Temperature indicator

Instrument

Fuse (auto type)

Master

plug fuse

Main indicator light

Master switch

FIGURE 6. CONTROL PANEL FOR ALUMINUM—BLOCK HEATER
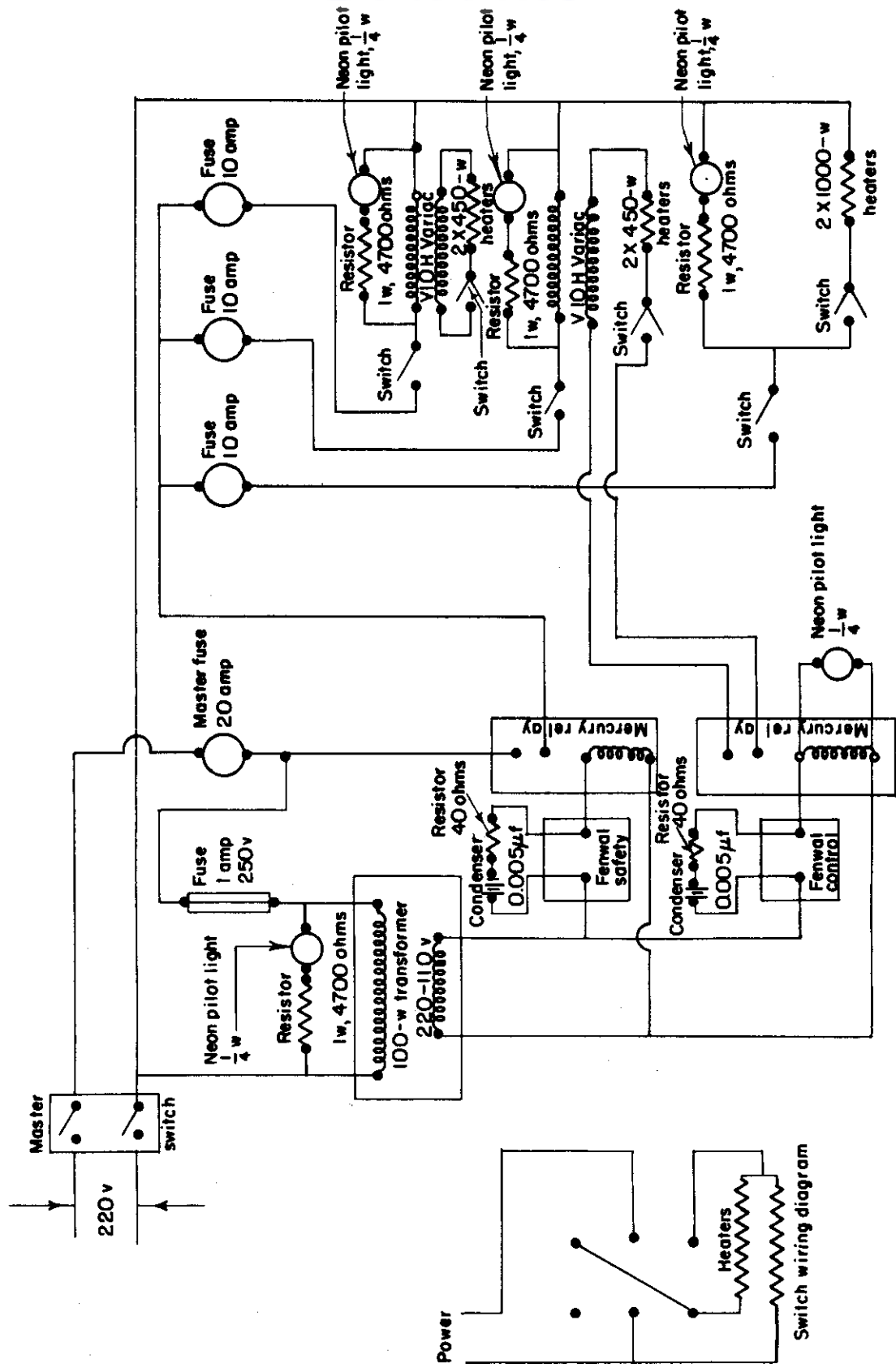
FIGURE 7. WIRING DIAGRAM FOR ALUMINUM-BLOCK HEATERS

O-22659
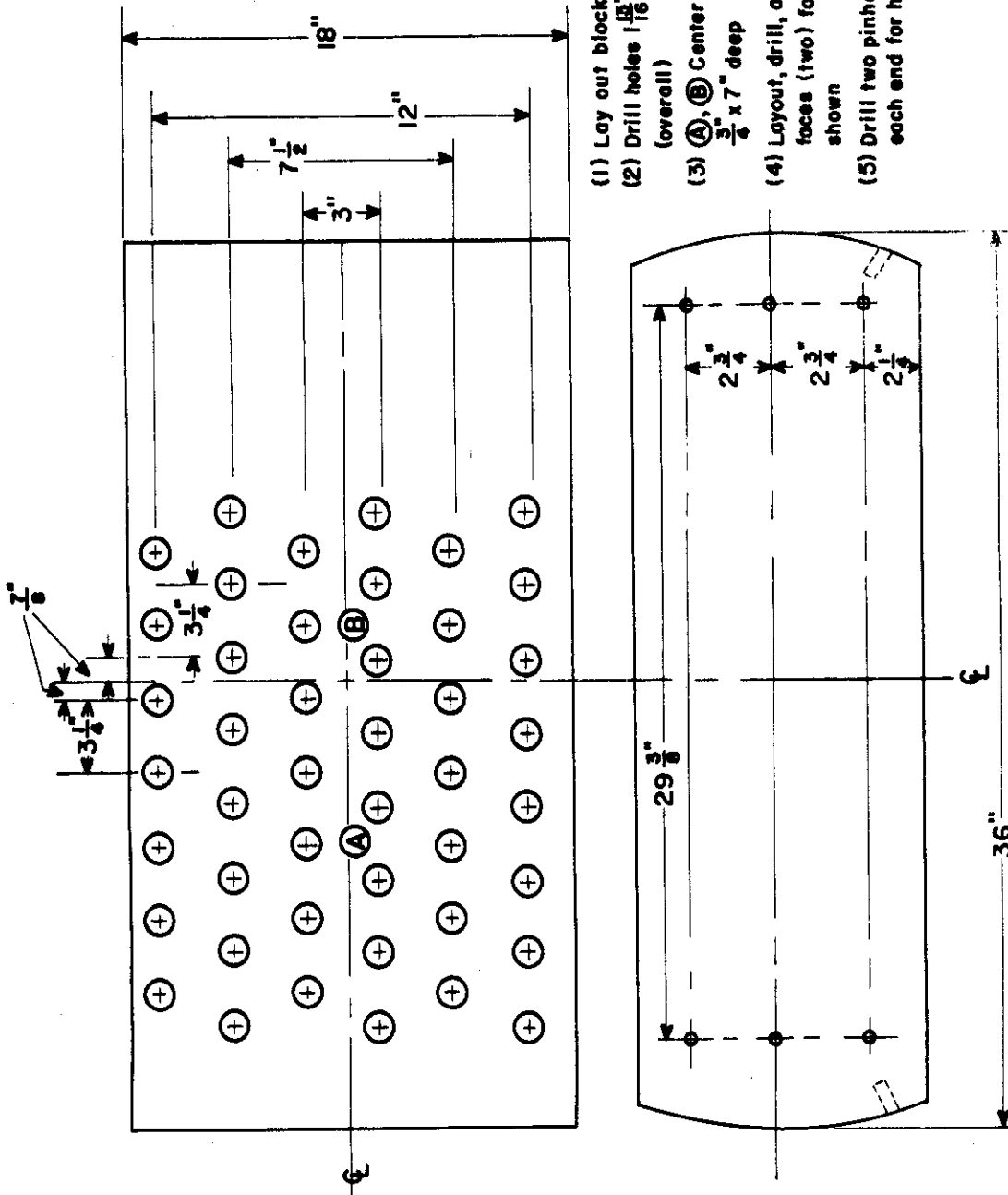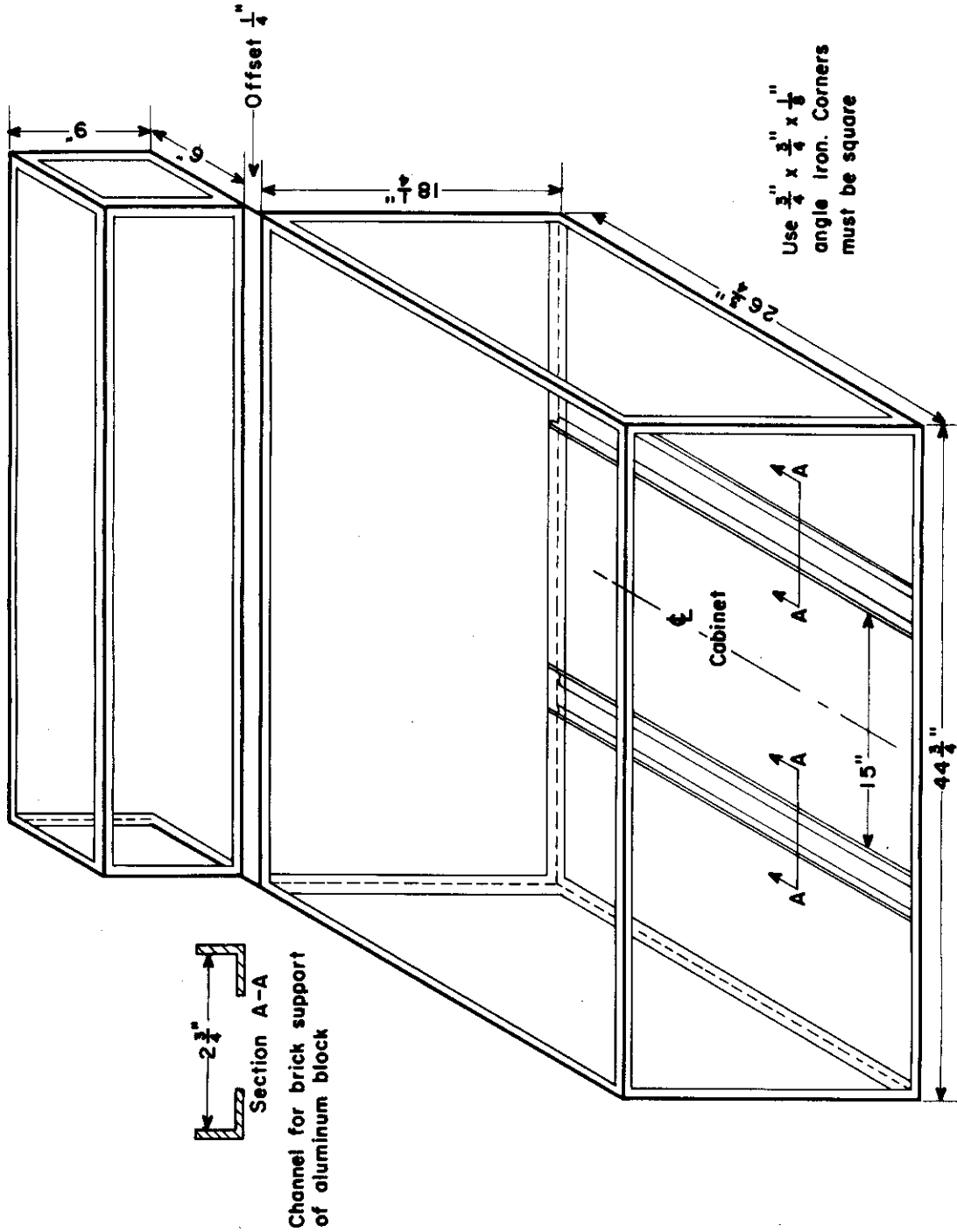
FIGURE 8. MACHINING SPECIFICATIONS FOR ALUMINUM BLOCK

(1) Lay out block from ₵ as shown
(2) Drill holes 1 13/16" diameter and 7" deep (overall)
(3) Ⓐ, Ⓑ Center holes in web. Drill 3/4" x 7" deep
(4) Layout, drill, and tap (1/4" x 20" x 1") faces (two) for strip heaters as shown
(5) Drill two pinholes, as shown, on each end and for hoisting cables

0-22705

FIGURE 9. CABINET FRAME FOR HEATING BLOCK

0-22706

DESIGN OF ALUMINUM-BLOCK HEATERS

Function of Control-Panel Components

Component	Comment
A. Master switch	This is a double-pole single-throw switch that disconnects both legs of the 230-volt supply.
B. Main indicator light	This light glows when power is on. Power failure or a burned-out instrument fuse or main fuse will prevent lighting.
C. Temperature-indicator light	This indicates block temperature relative to control-temperature setting of the Fenwal regulator on the right end of the block. Light burns when the block temperature is low and controller is calling for heat. Note: Circuit will not supply heat, however, unless heater switch center is on and selector switch is set for heat.
D. Instrument fuse	This is a low-amperage instrument fuse used as protection in the transformer circuit. Failure turns out all lights.
E. Master-plug fuse	This is a 20-ampere fuse used to protect against overload or major short in the unit. Failure cuts off all lights.
F. Left Variac	This governs the input voltage to the top pair of heaters. This pair of heaters is controlled manually and is continuous when operating.
G. Heater fuses	Fuse protection is provided for each set of heaters, i.e., the top, center, and bottom. The plug fuses are oriented in the same relative position as the heater sets protected. Failure of a fuse will turn off the corresponding indicator light.

Component	Comment
H. Heater-circuit switches	On-off switches are provided for each set of heaters and are positioned like the set controlled, i.e., top, center, bottom.
I. Heater-circuit indicators	An indicator light is provided for each heater circuit. Failure of the lights when the switch is on and the fuse intact indicates the power has been cut off by the safety Fenwal controller (left end of the block). If all lights on the panel are out, check the points listed for the main indicator light (B).
J. Right Variac	This governs the input voltage to the center (Fenwal controlled) pair of heaters.

K. Heat-selector switches	A selector switch is provided for each pair of heaters, i.e., top, center, and bottom. Orientation of the switch is the same as that of the heater set controlled. Each selector switch has three positions:
	    (1) Toggle up - high heat
	    (2) Toggle center - no heat
	    (3) Toggle down - low heat
	Note: It is advisable to switch off either the main power or the heater-circuit power before changing the selector switch. Heater-circuit indicator lights are not controlled by the selector switches. Lights may be burning, therefore, without heat's going to the block if the selector switches are in the "off" position.

Caution: The Fenwal controllers used in these blocks are tension operated and should not be inserted in a hot block without first setting the Fenwal to the approximate block temperature. Conversely, a Fenwal in a heated block should not be reset for a temperature over 200 F lower than the block temperature. In either case, the stress induced by the overheating may damage the controller.

Item	Part	Specification	Source[a]
1	Aluminum block	As-cast ingot, 10 x 36 x 19 inches Alloy 2S Machining details given in Figure 8	Aluminum Company of America
2	Aluminum tubing	1-3/4 x 0.058 inch, Type 61S	
3	Cabinet frame	3/4 x 3/4 x 1/8-inch angle iron Frame details given in Figure 9	Battelle shop
4	Cabinet	Sheet aluminum	Battelle shop
5	Insulation	B. H. Monoblock, 36 x 6 x 4 inches	Baldwin-Hill, Trenton, New Jersey
6	Strip heaters	4 - Type SN-3045 Strip Heaters, 230 volts, 450 watts, Monel sheath 2 - Type SNH-30 Strip Heaters, 230 volts, 1000 watts, Monel sheath	E. L. Wiegand Company, Pittsburgh 8, Pennsylvania
7	Temperature controllers	2 - No. 13150 Fenwal Controllers Modifications: (1) Length of temperature-sensitive shell, 11 inches to flange (2) Lead wire, 28 inches (3) Armored cable, 28 inches	Fenwal, Incorporated, Ashland, Massachusetts
8	Variacs	2 - Type V10H	General Radio Company, Cambridge 39, Massachusetts

Item	Part	Specification	Source[a]
9	Transformer	100 volt-amperes, Catalog No. 71G21 230 volts to 115 volts, GE	
10	Relays	2 - Mercury Relays, Catalog No. 7020, 110 volt coil, 30 amperes	H. B. Instrument Company, Philadelphia, Pennsylvania
11	Resistors	4 - 1 watt, 4700 ohms 2 - 40 ohms	
12	Condensers	2 - 0.005 microfarad	
13	Control jacks	2 - Cinch-Jones Plugs No. P302CCT 2 - Cinch-Jones Sockets S-302-FP	
14	Selector switches	3 - Cutler-Hammer Model C-2 DPDT 30 amperes, 125 volts, alternating current	

(a) Where items and sources are not specified, any general supplier of the desired equipment should be able to furnish the necessary item.

LIST OF MATERIALS AND THEIR SOURCES

Material	Composition	Source
Acrylon EA-5	Copolymer of ethyl acrylate and acrylonitrile	American Monomer Corporation
Acrylon EA-9	Copolymer of ethyl acrylate and acrylonitrile	American Monomer Corporation
Aerosil (now known as Cab-O-Sil)	Silica	Godfrey L. Cabot, Incorporated
Altax	Benzothiazyl disulfide	R. T. Vanderbilt Company
2-Amino benzene thiol	2-Amino benzene thiol	Eastman Kodak Company
Butyl rubber (GRI-25)	Copolymer of isobutylene and isoprene	Standard Oil Company of New Jersey
Cancene TM	Calcium carbonate	Columbia-Southern Chemical Corporation
Calcium peroxide	Calcium peroxide	Fischer Scientific Company
Continex SRF	SRF carbon black	Continental Carbon Company
4,4'-Diamino diphenyl disulfide	4,4'-Diamino diphenyl disulfide	Eastman Kodak Company
Di-iso-octyl thiomalate	Di-iso-octyl thiomalate	Evans Chemetics, Incorporated
Di-p-tolyl disulfide	Di-p-tolyl disulfide	Eastman Kodak Company

Material	Composition	Source
Tert-dodecyl mercaptan	Tert-dodecyl mercaptan	Phillips Petroleum Company
DPR N-27	Copolymer of 73% butadiene and 27% acrylonitrile	DPR, Incorporated
Du Pont Fine Silica (now known as Valron Estersil)	Organic-coated silica	Du Pont
FBA Polymer	Poly-1,1-dihydroperfluorobutyl acrylate	Minnesota Mining and Manufacturing Company
Flectol H	Condensation product of acetone and aniline	Monsanto Chemical Company
Flexol R2H	Polyester	Carbide and Carbon Chemicals Corporation
Hi-Sil	Hydrated silica	Columbia-Southern Chemical Corporation
Hi-Sil C	Hydrated silica	Ditto
Hi-Sil X303	Experimental Hi-Sil	"
Hi-Sil 53AK199	Ditto	"
Hi-Sil 53SMK108	"	"
Hycar 1001	Copolymer of butadiene and acrylonitrile	B. F. Goodrich Chemical Company
Hycar 1041	Ditto	Ditto
Hycar 4021	Copolymer of ethyl acrylate and chloroethylvinyl ether	"
Hycar 4021X26	Unknown	"
Indulin A	Lignin	West Virginia Pulp and Paper Company
Ludox	Colloidal silica	Du Pont

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Material	Composition	Source
Magnesia ELC	Magnesium oxide	Michigan Chemical Corporation
Mercaptobenzimi-dazole	Mercaptobenzimidazole	Monsanto Chemical Company
Methyl Tuads	Tetramethyl thiuram disulfide	R. T. Vanderbilt Company
NA-22	2-Mercaptoimidazoline	Du Pont
Tert-octyl disulfide	Tert-octyl disulfide	Phillips Chemical Company
Sec-octyl selenol	Sec-octyl selenol	Battelle preparation
Neoprene Q	Copolymer of chloro-prene and acrylonitrile	Du Pont
Phenyl diselenide	Phenyl diselenide	Battelle preparation
Phenyl disulfide	Phenyl disulfide	Eastman Kodak Company
Phenyl ethyl sulfide	Phenyl ethyl sulfide	Ditto
Phenyl propyl selenol	Phenyl propyl selenol	Battelle preparation
Philblack A	MAF carbon black	Phillips Chemical Company
Philblack E	SAF carbon black	Ditto
Philblack O	HAF carbon black	"
Plasticizer SC	Glycol ester of a fatty acid	Drew Chemical Company
Polyac	Polymer of paradinitro-sobenzene	Du Pont
P-33	Fine thermal carbon black	Thermatomic Carbon Company
Silene EF	Hydrated calcium silicate	Columbia-Southern Chemical Corpora-tion

Material	Composition	Source
Sodium metasilicate	Sodium metasilicate	Du Pont
Stearic acid	Stearic acid	Binney and Smith
Sulfur	Sulfur	Stauffer Chemical Company
Thioacetic acid	Thioacetic acid	Matheson, Coleman, and Bell, Incorporated
o-Thiocresol	o-Thiocresol	Eastman Kodak Company
Thiodiacetic acid	Thiodiacetic acid	Unknown
β-Thiodiglycol	β,β'-Dihydroxyethyl sulfide	Unknown
Thiodiglycolic acid	Thiodiglycolic acid	Eastman Kodak Company
Thiomalic acid	Mercapto succinic acid	Evans Chemetics, Incorporated
Thioglycolic acid	Thioglycolic acid	Eastman Kodak Company
Thiosalicylic acid	Thiosalicylic acid	Eastman Kodak Company
Triethylene tetramine	Triethylene tetramine	Carbide and Carbon Chemical Corporation
Trimene base	Reaction product of ethyl chloride, formaldehyde, and ammonia	Naugatuck Chemical
Triphenyl arsine	Triphenyl arsine	Eastman Kodak Company
Triphenyl bismuthine	Triphenyl bismuthine	Ditto
Triphenyl phosphine	Triphenyl phosphine	"

Material	Composition	Source
Triphenyl stibine	Triphenyl stibine	Eastman Kodak Company
Zinc oxide	Zinc oxide	New Jersey Zinc Sales Company