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DEVELOPMENT OF HIGH-TEMPERATURE OIL-RESISTANT RUBBER

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FOREWORD

This report was prepared by the Battelle Memorial Institute under USAF Contract No. AF 33(616)-476. The contract was initiated under Research and Development Order No. R-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.

ABSTRACT

Research toward the development of rubber compounds, to be used in connection with a diester-type lubricating oil (Turbo Oil-15) for long term exposure at 350 to 550 F, is described in this report. The evaluation of experimental compounds was confined to one temperature (350 F). At this temperature, the most promising results were obtained with compounds prepared from an acrylate polymer, Hycar 4021, compounded with Silene EF. The best composition of this type fell short of the target requirements only because of about 6 per cent excessive swelling. Another acrylate-type rubber, Acrylon EA-5, shows about equal promise for this application.

Compounds of a butadiene-acrylonitrile copolymer (Hycar 1001) showed promise, except that they cracked badly when aged in Turbo Oil-15 at 350 F. ELC Magnesia was the best reinforcing agent used with this polymer. Variations in the antioxidant and vulcanizing system provided only slight improvement in crack resistance. The chief weakness of this polymer is the vulnerability of its double bonds to oxidation.

The emphasis of future research on this project will be directed toward compounding acrylate-type rubbers, including poly-1, 1-dihydroperfluorobutyl acrylate, and in seeking methods for protecting the double bonds of acrylonitrile-type rubber.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. E. Sorte
Colonel, USAF
Chief, Materials Laboratory
Directorate of Laboratories

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INTRODUCTION

Because of increased speeds and power requirements, it has become necessary to increase the operating temperature in aircraft lubricating and hydraulic systems. With this temperature increase, present fluids are not satisfactory, and it has been found necessary to use ester-type lubricants of the MIL-L-7808 type (such as Turbo Oil-15) and ester-type hydraulic fluids (such as California Research No. 52742R silicate ester base fluid). These esters in combination with high temperatures have been found to be very harmful to rubber component parts.

This project has been set up with two objectives. The first is the development of a rubber composition which will withstand MIL-L-7808 lubricants for at least 500 hours at 350 F. The second objective is the development of a rubber which will withstand ester-type hydraulic fluids at 400 to 550 F. Minimum target properties for these compounds are given in Appendix A.

Several approaches to the problem also are listed in Appendix A. The most obvious one, which has received major effort, is a complete evaluation of commercially available polymers and blends of these polymers. Limited work also was done to evaluate high-temperature antioxidants, study unique curing systems especially designed for high-temperature applications, and determine the effect of oil additives which may inhibit rubber deterioration.

Earlier work by Wright Air Development Center and others narrowed the field of commercial polymers suitable for this application to three types. Acrylonitrile-butadiene copolymers (such as Hycar 1001) are known to have a high degree of oil resistance and appear promising, though susceptible to cracking when aged in oil at 350 F. Acrylate polymers (such as Hycar 4021) are reported to have outstanding resistance to dry heat, though they tend to swell excessively in hot oil.

In addition to these two commercial polymers, there was one experimental rubber which had shown promise for this application. This rubber is known as FBA polymer (poly-1, 1-dihydroperfluorobutyl acrylate). It was developed by the Minnesota Mining and Manufacturing Company for WADC and found to meet all the minimum target specifications for use in Turbo Oil-15 at 350 F. No work has yet been done with this polymer at Battelle because it is quite expensive and not readily available. However, a sample has just been obtained and it is planned to study it in some detail for comparative purposes.

EXPERIMENTAL SECTION

Equipment and Procedures

Equipment

At the beginning of this program, oil aging was conducted in a circulating-air oven operating at a temperature of 350 F. This method of aging was not too satisfactory, so an aluminum-block heating unit was constructed to provide a more uniform heat source and to increase safety and convenience. Sketches of the construction and control system employed may be found in Appendix B (Figures 1, 2, and 3). The holes shown in the block were drilled to fit 38 x 200-ml test tubes.

The block is heated by a combination of continuous and intermittent heaters so that the continuous heat supplied is just short of that required to attain the desired temperature. This unit is set to hold a temperature of 350 ± 2 F, but it has operated satisfactorily at 500 F. Another unit, essentially the same as the one described, except for slight modifications in the control system, was constructed and is in operation.

Procedures

Sample Preparation. ASTM Procedures D15-52T and D412-51T were followed in the mixing, curing, and preparation of individual dumbbell-type specimens used for testing.

Hot-Air Aging. The aging of dumbbell-type specimens was conducted in accordance with ASTM Procedure D573-52. Samples were aged at 350 F in a circulating-air oven. Specimens were suspended on a Chromel A wire, using glass-bead spacers.

Hot-Oil Aging. The original hot-oil-aging procedure consisted of suspending three dumbbell-type specimens from the same stock on a Chromel A wire in a 200-ml test tube containing 140 ml of Turbo Oil-15. The top of the test tube was closed with an aluminum-foil-covered cork stopper. The seal was made sufficiently tight to prevent excessive vapor loss, but insufficient to provide an air-tight seal. These test tubes were held upright in a metal rack and placed in a circulating-air oven.

Certain modifications of this procedure were necessary (1) to provide more realistic aging conditions, (2) to provide a more uniform heat source, and (3) to increase safety and convenience.

The modified aging procedure consisted of suspending three dumbbell specimens from the same stock on a glass-rod hanger which overlapped the top of a 38 x 200-ml test tube. Then 140 ml of Turbo Oil-15 was added and the tube covered with an inverted Petri dish. The cover fitted loosely and permitted the entrance of air into the test tube. The tube then was positioned in a hole in the aluminum block and heated at 350 F for the specified time. At the end of the aging period, the tubes were removed from the aluminum block and allowed to cool for one hour. The rubber samples were then removed from the oil, dipped quickly in acetone to remove oil from the surface, and tested.

Aging tests have shown that a high degree of reproducibility of results is obtained by this modified procedure.

Determination of Physical Properties. Hardness was determined by a Shore Durometer (A2) according to ASTM Procedure D314-52T.

The stress-strain properties of rubber specimens were determined with a Scott Tensile Tester, Model L6, run at a speed of 20 inches per minute. Specimens were cut with Die C, ASTM Procedure D412-51T.

Swell was determined by the formula

$$V = \frac{(W_3-W_4) - (W_1-W_2)}{(W_1-W_2)} \times 100,$$

where

W_1 = initial weight in air
W_2 = initial weight in water
W_3 = weight in air after immersion test
W_4 = weight in water after immersion test
V = percentage change in volume.

For the major part of this project, weighing of samples for swell determinations was done on an analytical balance. Since this was quite slow and time consuming, a Kraus-Jolly balance was obtained, which saved about half the time required to determine the amount of swell. A comparison of the two methods showed that swelling was about one per cent lower when determined by the Kraus-Jolly balance than when determined by the analytical balance. Thus, the Kraus-Jolly balance was considered to be sufficiently accurate for preliminary screening tests on this program.

The degree of cracking was rated by visual observation of 1 x 1-inch specimens flexed 180 degrees. Degree of cracking was reported as (1) no cracking, (2) crazing, or (3) cracking.

Compression Set in Turbo Oil-15. Compression set of vulcanized rubber is determined by ASTM Method D395-52T. In this method, the test sample is placed in a compression device and compressed under constant load or constant deflection. The assembly is then placed in a circulating-air oven for a suitable time and temperature. At the end of the heating period, the sample is removed and compression set determined.

This project, however, requires that the compressed sample be aged in hot Turbo Oil. Therefore, some deviations from the ASTM test are necessary. These are discussed in the following paragraphs.

Since the compression set is being determined in hot Turbo Oil, a compression device was designed (Figure 4) which fits inside the 75 x 200-ml test tubes used for aging. By this means, the aluminum-block heaters can be used for the compression-set test. For simplicity, the device was designed for constant deflection. Six samples can be tested simultaneously.

The ASTM test is made on a cylindrical disk 1.129 inches in diameter and 0.5 inch thick. If a solid piece is not available, a maximum of seven thin disks can be plied together to obtain the required thickness. As this diameter was too great for use in the prescribed test tube, a smaller test specimen had to be used. It was decided that the ratio of the height to diameter should be the same as that of the ASTM test specimen. Consequently, a test specimen 0.50 inch in diameter and 0.225 inch in thickness was used. The proper thickness was obtained by plying up three disks cut from the standard 0.075-inch tensile sheets. The test procedure is as follows:

(1) Disks are cut from 0.075-inch tensile sheets and plied up, using a total of three disks. Total thickness of the sample is determined to the nearest 0.001 inch.

(2) The test specimens are then placed between the plates of the compression device along with spacers and the assembly tightened. The amount of initial compression on the samples depends upon 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. Preliminary work includes aging the samples for 72 and 168 hours at 350 F.

(4) At the end of the test period, the samples 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:

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

where

t_o = initial thickness
t_i = final thickness after cooling for 30 minutes.

Compounding Studies With Nitrile-Type Rubber

Preliminary Compounding Studies

Initial screening studies were conducted with Hycar 1001, a copolymer of acrylonitrile and butadiene, to explore some of the factors which might influence the stability of this rubber at 350 F.

The results of these preliminary tests, shown in Tables 2 and 3, disclosed that both carbon black and plasticizers impair the aging qualities of Hycar 1001. In addition, it was observed that AgeRite Powder, a conventional antioxidant, was totally ineffective in improving the aging of this rubber at 350 F.

Efforts at Battelle to reproduce the outstanding aging results obtained by the United States Rubber Company on their Recipe No. 40513-B were unsuccessful. Failure to duplicate these results was believed to be due to differences in aging procedure, rather than to possible differences in compounding techniques. The aging at United States Rubber Company was conducted in air-tight glass containers, in contrast to containers at Battelle which permitted air to enter. The deleterious effect of air on the aging of rubber has been well established, and is discussed in a later section of this report.

It was evident from data obtained early in this research program that both the compounding of rubber and the method of aging were of primary concern. Since it was apparent that all of the compounding ingredients incorporated into the rubber could be expected to influence its performance at 350 F, an extensive search was initiated to find materials which would retard the deterioration of the rubber at elevated temperatures. The problem of establishing a realistic and reproducible aging procedure was solved satisfactorily by the construction and installation of an aluminum-block heater, which was described in detail earlier in this report.

Effect of Various Fillers on Nitrile-Type Rubber

Carbon Black. Numerous studies have confirmed that carbon black does not appear suitable for use in Hycar 1001 compounds designed for high-temperature applications.

Initial investigations of the effect of carbon black indicated that normal amounts (e.g., HAF black in a concentration of 40 to 80 phr, where phr is an abbreviation for parts per 100 parts rubber) produced vulcanizates possessing poor oil-aging properties. The strong tendency for these compositions to crack during the early stages of oil aging was attributed to carbon black. This was borne out in a later study (Table 4), in which the carbon black level was reduced to 25 phr. The crack resistance of these compounds was improved, but at the expense of an appreciable loss in tensile strength, both before and after aging.

Further evidence of the undesirability of employing carbon black in heat-resistant Hycar 1001 stocks was shown in a study in which both magnesia and carbon black were employed as fillers. The results of this study, shown in Table 5, reveal that the tensile strength, elongation, hardness, and crack resistance of these vulcanizates were progressively poorer as the percentage of carbon black was increased.

In addition to the unsatisfactory performance of stocks containing carbon black, there are other factors which indicate that carbon black is not suitable for reinforcing rubber compounds exposed to high temperatures. It is known that carbon blacks adsorb oxygen, have catalytic properties, and partially volatilize at temperatures as low as 250 F. These factors suggest that, at 350 F, carbon black might prove decidedly detrimental when used as a reinforcing agent for rubber. The general view that carbon black does not appear desirable for use in rubber compositions exposed to elevated temperatures is shared with others who have conducted investigations along this same line.

Zinc Oxide and Magnesia. Since results obtained with carbon black were not encouraging, consideration was given to the possible use of non-black fillers. This general class of fillers is usually considered to have poorer reinforcement properties in rubber. However, in the present application, considerable sacrifice can be made in initial properties if better aged properties can be attained.

The results obtained by employing unusually high levels of zinc oxide and magnesia, which are commonly used only in nominal amounts as activators, are shown in Table 6. Zinc oxide has been used in rubber at loading levels up to 100 phr to produce stocks with good air-aging properties (New Jersey Zinc Oxide Company, The Activator, Vol 9, No. 1, March, 1949), but its use at this high level in stocks designed for both heat and oil resistance does not appear promising. Neither of these pigments at the activation level of 5 phr in a nonreinforced stock was effective, either in producing satisfactory initial properties or in retaining these properties after aging. However, when magnesia was employed at a level of 100 phr, a remarkable improvement in both original and oil-aged properties was obtained. Additional compounding with this filler (Table 6) disclosed that optimum aging characteristics are attained at the 100 phr loading level (Compound A-23).

Although Compound A-23 evidenced cracking after aging for 7 days in Turbo Oil-15 at 350 F, it met all of the other minimum target specifications at the end of this aging period. On this basis, it was selected as the standard recipe for future compounding studies. Unfortunately, this composition possesses poor processing characteristics. Slight modifications in the formulation afford easier processing, but at the expense of hot-oil-aging qualities.

Other Nonblack Fillers. On the basis of the excellent aging results obtained with magnesia, exploratory studies were made of several other nonblack fillers. These studies were of a screening nature, and no attempt was made to determine the optimum loading level or to adjust the basic recipe to show these pigments to best advantage. Although these adjustments would probably enhance the aging characteristics, improvements which can be made in this direction appear to be rather limited, even for the more promising of these materials.

As indicated in Table 8, none of these pigments imparted the excellent balance of oil-aged properties exhibited by magnesia-filled compounds. In general, these fillers produced vulcanizates which lacked a proper balance of tensile strength and elongation, and displayed poor crack resistance.

Effect of Curing System on Nitrile-Type Rubber

The emphasis of this phase of the compounding program has been on low-sulfur and nonsulfur curing systems, since stocks containing low levels of available sulfur frequently have been reported in the literature as possessing good aging characteristics. Although curing studies to date have not exhausted all of the various types of vulcanizing agents, it is evident from the data that none has been found which produces outstanding aging properties. In most cases, the aged physical properties were in the same range, regardless of the curing agents employed.

An investigation of several accelerators of the thiuram type (Table 9), a class reported to give excellent aging characteristics, disclosed a distinct difference between thiuram monosulfides (Monex and Pentex) and thiuram polysulfides (Tetrone and Tuex). The initial properties of the monosulfides were distinctly lower than those of the polysulfides, presumably because (1) more sulfur is available for vulcanization from the latter, and (2) the rate of vulcanization should be faster in the presence of the greater amount of sulfur. The apparent undercures obtained with the monosulfides account for the illusion that stocks vulcanized with them show better retention of tensile strength but suffer a greater loss in elongation upon aging. It can be assumed that, at the aging temperature of 350 F, the monosulfide stocks continue to vulcanize and pass through a maximum in tensile strength around 2000 psi or greater, and that this is accompanied by a decline in elongation to about the same range as that of the polysulfide stocks. Among the cures obtained solely with a thiuram, the poorest aging results were obtained with the higher loading of the tetrasulfide (Tetrone), as judged by the product's greater loss in elongation after 168 hours of hot-oil aging.

It is interesting to note that, even though the thiuram monosulfide stocks appear greatly undercured, they swelled no more than more fully cured vulcanizates obtained with thiuram polysulfides. However, this should not be taken as an indication that a good method of prolonging the useful life of stocks for this application is to grossly undercure them. It is well known that when an insufficient number of cross links are formed during vulcanization, the vulcanizate has less resistance to swelling. Since the aging temperature is above the curing temperatures commonly employed, and with vulcanization certainly continuing during aging, there is little hope of extending the life of the rubber for a period longer than the time the cure was shortened, an insignificant gain for the risk involved.

The results of evaluating several other low-sulfur and nonsulfur curing systems in a magnesia-filled stock are shown in Table 10.

In the sulfur-containing systems, a tighter cure (as evidenced by a greater tensile strength) was obtained with higher concentrations of available sulfur. However, this had no beneficial effect on the physical properties of these vulcanizates after they were hot-oil aged for 7 days.

The incorporation of sulfurless curatives (such as calcium oxide, cadmium oxide, litharge, and dinitrobenzene) produced vulcanizates with aging properties similar to those obtained with sulfur, but none that were better.

The examination of several peroxide-cured compounds, supplied by the B. F. Goodrich Chemical Company, revealed that these vulcanizates were unsatisfactory for use at 350 F. The physical properties of these compositions were extremely poor after only 3 days' hot-oil aging, as can be seen in Table 11.

From the data obtained from these curative studies, it appears unlikely that any significant improvement in the aging characteristics of butadiene-acrylonitrile copolymers can be achieved by selective choice of type and amount of curing agent. This is in direct contrast to curative studies with acrylate polymers, which show that the curatives have a direct influence on the aging qualities of this type of rubber.

Effect of Antioxidants on Nitrile-Type Rubber

Extensive efforts have been directed toward finding a means of retarding or preventing the oxidation of nitrile rubber, which results in cracking. Studies in this area have included (1) an evaluation of commercial and experimental antioxidants, (2) an investigation of the effect of high levels of antioxidants, and (3) a study of the desirability of adding anitioxidant to rubber, oil, and both. Only very meager improvements in crack resistance were obtained as a result of these studies.

Evaluation of Normal Amounts of Various Antioxidants. The effectiveness of several commercial and experimental antioxidants in a carbon black-filled Hycar 1001 formulation is illustrated in Table 12. Inspection of the aging data reveals that none of the conventional antioxidants imparted significant improvement in aging characteristics, although Parazone, Flectol H, and PDA-10 appear slightly more effective than the others in retarding cracking.

The experimental antioxidants included materials used as (1) short-stopping agents in emulsion polymerization and (2) sequestering agents. Since a considerable number of undesirable cross links are formed during the aging of rubber, as demonstrated most dramatically by the decline in elongation, it was thought that materials which tend to limit and/or stop polymer growth might likewise block the formation of undesirable cross links during aging. A number of the compounds selected for this study were phenolic in nature. Some commercial antioxidants fit into this general class of organic compounds, and a number of derivatives of this type have been used as antioxidants for oil. In fact, some have been used as antioxidants for diester oils [Atkins, Baker, Murphy, and Zisman, Ind. Eng. Chem., 39, 491 (1947)], and might be present in Turbo Oil-15. Aging results disclosed that, although these experimental materials were not effective antioxidants at 350 F, they were as efficient as the conventional antioxidants. Pyrogallol and t-butyl catechol appeared more efficient than the others with respect to improving crack resistance.

Similar results (Table 13) were obtained with phenolic-type compounds in the standard magnesia-filled stock used in this investigation, i.e., aging properties were not significantly influenced by the inclusion of these antioxidants in the formulation.

It is interesting to note that the only property apparently affected by any of the antioxidants in these compounds was an improvement in crack resistance, but even this was too slight to be significant.

The effect of possible synergistic combinations of antioxidants on the aging qualities of a magnesia-containing compound was investigated. It was hoped that greater-than-additive protection would be afforded by employing more than one type of antioxidant. However, as indicated in Table 13, no additional protection against degradation was provided by the two combinations of antioxidants examined.

The results of evaluating several types of vinyl stabilizers as antioxidants in a Hycar 1001 formulation are shown in Table 14. Stabilizers are used for many purposes in polyvinyl chloride. Some act as mild acceptors for hydrogen chloride, others prevent polyene formation by a Diels-Alder reaction with the polymer chain, and still others reduce free radical formation (which otherwise would transfer energy for oxidation of polymer chains at double bonds). Although the heat degradation of nitrile rubber occurs in a different fashion than that of polyvinyl chloride, it was considered possible that some of the stabilizers for PVC might be effective high-temperature antioxidants for rubber through some of their unique properties.

Only one stabilizer, known as Stabilizer A-5 (an epoxy-type compound), imparted any significant improvement in aged physical properties. The composition containing this stabilizer possessed better tensile strength and elongation than the control containing no antioxidant. However, this material, like the others, contributed little to crack resistance.

High Concentration of Antioxidants. A study was conducted to determine if larger amounts (10 and 15 phr) of antioxidant than are usually employed would supply added protection for the rubber against deterioration. Three of the more effective antioxidants were selected for this study (pyrogallol, o-cresol, and AgeRite Resin D).

As indicated in Table 15, the only significant improvement in hot-oil aging properties was obtained when large amounts of AgeRite Resin D were employed. Elongation and tensile strength were improved in this case, but cracking was not retarded.

These data indicate that it is doubtful if satisfactory improvement in aging properties can be attained solely by adding large concentrations of conventional antioxidants to the rubber.

Adding Antioxidant to Rubber, Oil, and Both. Attention was given to the problem of whether more protection of the rubber could be obtained by adding antioxidant to the rubber, oil, or both. Although it is customary to add antioxidant only to rubber, it was thought that certain advantages might be gained by adding antioxidant to the oil as well. Antioxidant added to the oil might be effective in inhibiting oxidation at the oil-rubber interface. It also was thought possible that antioxidants might retard the formation of degradation products from the oil and thus prevent their possible harmful effects on the rubber.

Table 16 presents data obtained from hot-oil aging tests in which antioxidant was added to both the rubber and oil. Four antioxidants (Flectol H, Parazone, pyrogallol, and o-cresol) were incorporated in a Hycar 1001 recipe at 3 and 20.1 phr. These same antioxidants also were added to the oil in similar amounts, giving concentrations based on the oil of approximately 0.15 and 1 per cent, respectively.

The compounds containing Flectol H evidenced greater elongation and slightly higher tensile strength when the antioxidant concentration was increased, and cracking was reduced somewhat when the same amount of Flectol H also was added to the oil.

Increasing the amount of pyrogallol in the rubber, and the addition of a similar amount of this material to the oil, imparted greater tensile strength, though at the expense of hardness and elongation. There was no apparent advantage in adding o-cresol or Parazone to the oil or increasing their amounts in the rubber.

In the series of oil-aging tests, in which antioxidant was added only to the oil, 0.15 and 1 per cent of phenothiazine, Parapoid C, and Flectol H were used. The results, in Table 17, show that the rubber possessed slightly higher elongation and tensile strength when aged in oil containing 1 per cent Flectol H than when aged in oils containing the other two materials. However, the crack resistance of this composition was not improved as much as when this antioxidant was added to both the rubber and oil.

In summarizing this phase of the antioxidant work, it appears that better protection against deterioration of the rubber is provided when the antioxidant is added to both the rubber and oil, although this still does not impart sufficient crack resistance to make the rubber suitable for use at 350 F. There is, of course, the possibility that other antioxidants might be found which will be more effective.

Effect of Processing Aids

Initial compounding and aging tests indicated that zinc stearate, and possibly zinc oxide and stearic acid, might be contributing toward inferior aging qualities. As pointed out earlier in this report, high levels of zinc oxide were injurious to hot-oil-aged properties. The results of a brief study of the effect of stearic acid in a nonreinforced stock, shown in Table 18, disclosed that large amounts (5 phr) of this ingredient also were detrimental to aging characteristics. Consequently, additional studies were conducted to determine the effect of low concentrations of these ingredients.

These data (Table 19) revealed that reducing the zinc oxide and stearic acid levels below 5 and 1.5 phr, respectively, imparted only a slight decline in aged properties. Omitting the zinc oxide slightly reduced aged properties, while omitting the stearic acid, or both stearic acid and zinc oxide, gave decidedly poorer properties. The optimum concentrations appear to be 2.5 to 5 phr zinc oxide and 0.75 to 1.5 phr stearic acid.

The poor processing characteristics of Compound A-23, previously mentioned, were markedly improved by the addition of 5 phr of zinc stearate. However, this improvement was attained at the expense of an appreciable loss in other oil-aged properties. Magnesium stearate, talc, and

Acrawax CT were less efficient in improving processability and also were detrimental to the aging characteristics (Table 20).

Effect of Nonextractible Plasticizers, Softeners, and Other Additives

The effects of plasticizers, softeners, and similar materials on the aging characteristics of a magnesia-filled stock (Compound A-23) were investigated. Although it was expected that these additives would reduce tensile strength, it was hoped that this loss would be counterbalanced by gains in elongation and crack resistance.

As shown in Tables 21 and 22, none of these materials retarded cracking. Two nitrile-type plasticizers (Hycar 1012 x 41 and ODN) enhanced elongation at the expense of tensile strength. Other additives, in general, impaired both tensile strength and elongation.

No evidence was obtained which indicated that any of these softening-type additives were completely extracted. The fact that the compositions containing the various additives swelled more than the control containing no additive indicates that residual additive remained in all these compositions after hot-oil aging. Among all the compositions containing these additives the lowest swell values were obtained for the compositions containing Hycar liquid polymers. This is attributed to the strong polar nature of the Hycar liquid polymers, in contrast to materials such as Vistanex and butyl rubber, which are nonpolar and swell excessively in ester-type fluids.

The results of this work suggest that plasticizers and softener-type materials are not beneficial in nitrile-rubber compounds designed for use at elevated temperatures.

Effect of Acrylonitrile Content of Copolymer

A brief study was conducted to determine the effect of the acrylonitrile content of the base copolymer on the aging properties of magnesia- and carbon black-filled stocks. Several rubbers, ranging in acrylonitrile content from 18 to 60 per cent, were included in this study.

Although the rubbers with lower acrylonitrile content were known to possess poor oil resistance, it was hoped that plasticization from swelling would improve crack resistance. As shown in Tables 23 and 24, these copolymers (Hycar 1002, Paracril B, and Paracril AJ) softened, swelled excessively, and exhibited poor retention of physical properties after oil aging. The improvement sought in crack resistance, by permitting more swelling, did not materialize.

Chemigum N3NS, which is slightly higher in acrylonitrile content than Hycar 1001, displayed hot-oil-aging properties similar to those obtained with Hycar 1001. Presumably, these two rubbers could be used interchangeably for many applications.

Two higher acrylonitrile rubbers, Hycar 1000 x 70 (60 per cent acrylonitrile) and an experimental copolymer (1457-60, containing 55 per cent acrylonitrile and prepared at Battelle), were evaluated to determine if a reduction in the butadiene content, which would reduce the number of double bonds available for oxidation, would improve crack resistance. The results of aging these compositions showed no improvement in crack resistance, although, as expected, they did display low swell and good retention of tensile strength. However, the hardness was too high and elongation was too low after oil aging at 350 F for only 72 hours

These data confirm the belief that, in the nitrile rubber class, those rubbers containing 40 to 45 per cent acrylonitrile offer the best hot-oil-aging properties.

Effect of Curing Conditions

A curing cycle of 60 minutes at 298 F was arbitrarily selected at the beginning of this project, in order to expedite the evaluation program. Although it was obvious that this was not the optimum curing cycle for all compositions, it was believed that the curing conditions would have comparatively little effect on the physical properties of rubber after it is aged at 350 F for long periods of time.

A study, in which Compound A-23 was cured at 298, 350, and 400 F, supported this contention. The results of this study, shown in Table 25, reveal that the vulcanizates cured at elevated temperatures exhibited no advantage over those cured at our standard curing temperature, either in original or oil-aged properties.

Effect of Aging Conditions

Effect of Air. It has been mentioned that air plays an important role in the rate of aging of rubber in oil. The data in Table 26 illustrate the relative effects of "limited" and "unlimited" air on the aging properties of Compound A-23. As might be expected, the sample exposed to excess air (loosely covered with a Petri dish) swelled less, increased more in hardness, and suffered greater loss in elongation and tensile strength—presumably due to the greater amount of cross linking occurring in this sample from oxidation.

Further evidence of the greater severity of the unlimited-air system was found in the considerably greater amount of sludge resulting after oil aging by this method, compared with that of the limited-air (ground-glass stoppered bottle) system. It is understood that aircraft lubrication systems pump a considerable amount of air with the oil, and that it is virtually impossible to totally exclude air. Therefore, contact of rubber with aerated oil seems probable and points toward the need for oil aging rubber under conditions where free access to air is permitted. The difference in aging results reported among different laboratories is probably due to the amount of air permitted to contact the oil during aging. It appears essential, therefore, that standardization of test procedures among the various laboratories is not only desirable but mandatory.

Effect of Metals. A study was made of the effect of metals on the oil aging of rubber at 350 F. Small pieces of various metals (steel, copper, aluminum, magnesium, and silver) were placed in the bottom of the containers, where they were not in direct contact with the rubber specimens. No special prec .utions were taken to exclude air from contact with the oil during aging. The fact that no marked trends were noted in the results (Table 27), including those for a control containing no metal, suggest that metals contacting the oil have no appreciable influence on the aging of rubber, at least in systems exposed to unlimited air. Any limited influence the metals may have exerted on the aging was undoubtedly masked by the gross effects of air.

Reproducibility of Results

Studies were conducted to determine whether reproducible results could be obtained (1) with our test equipment and procedures and (2) by testing a series of batches of the same composition.

Table 28 illustrates the degree of reproducibility that can be achieved with our test equipment and procedures. These tests, conducted on specimens from the same batch of compounded stock, demonstrated that experimental work can be repeated with a high degree of accuracy.

A separate study was made to determine if different batches of the same formulation would yield duplicate results. These stocks were processed in an identical fashion and aged simultaneously. As shown in Table 29, the physical properties of the three stocks, both before and after aging, were well within the limits allowed for experimental error.

Degradation of Turbo Oil-15

Although air is the dominant factor in the degradation of rubber at high temperatures, consideration was given to the possibility that the oil also might be a contributing factor.

Extended oil-aging tests were performed in Turbo Oil-15 and in di-(2-ethylhexyl) sebacate to determine if used oil was more harmful than fresh oil to new rubber. It was anticipated that degradation products from the oil, and the accumulation of impurities in the oil from previously aged rubber specimens, might make the oil progressively more harmful to new rubber.

Table 30 illustrates the comparative effect of the two diester-type oils on Compounds A-23 and A-97. Seven sets of fresh rubber samples from each of these compositions were aged consecutively for 72 hours in each of these oils at 350 F. After 21 days (500 hours) at 350 F, neither aged fluid showed any indication of deteriorating new rubber more rapidly than when it was fresh. Thus, these limited data indicate that the diester fluids do not become increasingly harmful to rubber, at least within this time limit. Results of these tests showed that identical rubber specimens aged in di-(2-ethylhexyl) sebacate swelled somewhat less than those aged in Turbo Oil-15, but other rubber properties were little affected by the choice of the aging oil. The similarity of results obtained by aging rubber in a pure diester oil and in a compounded oil (Turbo Oil-15) is striking. The difference in hot-oil-aging properties exhibited by Compounds A-23 and A-97, as discussed previously, is attributed to the harmful effect of zinc stearate in the A-97 compound.

Tests also were conducted to determine the degree of degradation of Turbo Oil-15 and di-(2-ethylhexyl) sebacate when individually heated at 350 F. Tests were performed in both closed and open (regular) containers. Oil samples were removed after heating for 24, 72, and 168 hours, and the per cent active oxygen was determined according to the procedure described in Protective and Decorative Coatings (Mattiello). Essentially, this method involves the addition of potassium iodide to the oil and titrating liberated iodine with sodium thiosulfate solution.

The results of these tests, shown in Table 31, reveal that the amount of degradation of both oils was negligible in closed systems. As anticipated, deterioration was much greater in the regular containers, in which the oils were exposed to an unlimited supply of air. In this system, degradation reached a maximum in from 24 to 72 hours and then rapidly declined. One possible explanation for this phenomenon is that peroxides are formed from only a small portion of these oils. Since peroxides may be considered as intermediate products in degradation reactions, a decline in peroxide content may indicate that more peroxides are being converted to other products than are being formed.

It is interesting to note that Turbo Oil-15 degraded more than did di-(2-ethylhexyl) sebacate, which contains no protective agents.

Comparison of Esso and Penola Turbo Oil-15

During the course of this research, the original 55-gallon drum of Esso Turbo Oil-15, supplied by the Materials Laboratory, WPAFB, was depleted and it was necessary to order additional oil. The new supply of Turbo Oil-15 was ordered from the local agent for this oil, the Penola Oil Company, Detroit, Michigan. To determine whether the new oil (Penola) would give results identical to those obtained with the oil in the first drum (Esso), four rubber stocks were aged in oil from both these sources. Results, shown in Table 32, disclosed that the rubbers aged in Esso Oil were softer and swelled more than those aged in Penola Oil. Although distinct differences in these properties were observed, these discrepancies were not considered critical since most Hycar 1001 compounds have displayed satisfactory swell resistance and hardness. Other rubber properties were little affected by the choice of oil.

Compounding Studies With Hycar 4021

A large amount of work has been done to determine the effect of several compounding variables on the properties of Hycar 4021 vulcanizates after aging in Turbo Oil-15 at 350 F. Variables studied included vulcanizing systems, fillers, and lubricants. A literature survey was made on the compounding of acrylate-type rubber, as a background for this study. This survey is presented in Appendix E to this report.

Effect of Vulcanizing Systems on Hycar 4021

Several vulcanizing systems were studied in detail to determine which one gave optimum aged properties to vulcanizates. Data in the literature indicate that good aging properties are obtained when a mixture of vulcanizing agents is used. This mixture should contain an amine and sulfur, with or without a sulfur-liberating material. In order to verify this, four different systems were studied. These were triethylene tetramine - sulfur - Tuads, Trimene base - sulfur - Tuads, triethylene tetramine - sulfur - Monex, and triethylene tetramine - sulfur - Polyac.

Triethylene Tetramine - Sulfur - Tuads. The combination of triethylene tetramine, sulfur, and Tuads has been recommended as a vulcanizing system which imparts good heat resistance to Hycar 4021 vulcanizates. Since the particular combination of these three materials was considered important, a number of ratios were explored, with results shown in Table 33, for Batches PA-2 through PA-39. In order to help correlate these data, triangular graphs were prepared (Figures 5 through 8), which aid in explaining the effect of each of these vulcanizing agents. These data demonstrate that a compromise needs to be made to obtain the best balance of the four properties desired. After aging for 168 hours in Turbo Oil-15 at 350 F, compositions having the highest tensile strength and hardness, and lowest swell, are found in the area of high amounts of triethylene tetramine and low amounts of sulfur and Tuads. However, the best aged elongation is found in the areas of low triethylene tetramine and about one-half part sulfur and one-half part Tuads. The optimum balance of all four properties appears to be in the area of 40 to 65 per cent triethylene tetramine, 15 to 30 per cent sulfur, and 10 to 40 per cent Tuads.

In order to narrow this range, nine additional batches (PA-48 through PA-56) were prepared, with ratios of the three vulcanizing agents within the optimum range mentioned above. It was found that five of these batches (PA-50, -52, -53, -55, and -56) had very good oil-aged properties, even after the 500-hour test (see Table 33). The 500-hour aging test demonstrated that Batch PA-52 was the best of this group of compositions, with PA-53 being a close second. The other batches in this group were not aged beyond 168 hours, because of low hardness and/or high swelling.

It is interesting to note that, after 500 hours of aging, Batch PA-52 had properties comparable to PA-2, although the latter contained no sulfur in its vulcanizing system. Because of this similarity, these two batches were recompounded and tested. Results of this work are shown in Table 34. It can be seen that there was a rather wide variation in some of the properties between duplicate samples. Part of this can be attributed to variations in compounding, curing, testing, etc., between the duplicate batches. On the basis of these results no significant difference can be detected between the two vulcanizing systems. For the purpose of future work, both of these ratios will be considered optimum.

It should be noted that only one curing time was used to test each batch reported in Tables 33 and 34. In all cases, the shortest curing time was used which did not cause noticeable permanent set when the sample was stretched by hand. Limited work was, therefore, undertaken to determine the effect of long curing times on the hot-oil-aged properties. Data in Table 35 show that long curing times have a beneficial effect on aged properties. Tensile strength and hardness are increased, swelling decreased, and the elongation is unaffected. In view of the long heating time at a high temperature involved in aging the rubber, it previously has been assumed that initial cure conditions were not very important, so long as an adequate cure was obtained. However, these data indicate that the initial cure is more important than was first thought.

After it was determined that a longer curing time improved the aged properties, presumably because of a tighter cure, it was thought that the use of additional vulcanizing agent might be a better method of accomplishing the same result, since long curing times are not practical for factory production. By comparing Batch PA-112 and PA-113 (see Table 35), it can be seen that doubling the amount of vulcanizing agent decreased swelling about 10 per cent, with only a slightly harmful effect on the aged elongation.

More work is planned to determine the effects of long curing times and increased vulcanizing agent. In addition, a study will be made of the effect of raising the curing temperature in order to get tighter cures in a more reasonable curing time.

Trimene Base - Sulfur - Tuads. Trimene base, sulfur, and Tuads have been recommended as another vulcanizing system which gives heat-resistant vulcanizates with Hycar 4021. Several batches were selected to evaluate this system, with results shown in Table 36. With one exception, the vulcanizates had low hardness, high swell, and, frequently, high elongation, after aging in Turbo Oil-15. The one exception, Batch PA-119, showed that greatly improved aging properties can be obtained by doubling the amount of vulcanizing agent. Longer cures also improve the aged properties.

The results with mixtures of Trimene base, sulfur, and Tuads were found to be consistent with earlier work with triethylene tetramine, sulfur, and Tuads. That is, in both cases, increased amounts of vulcanizing agent and longer cures improved the hot-oil-aged properties. However, the increased vulcanizing agent was much more beneficial than the increased curing time, although the two effects supplemented each other. For comparable ranges of concentration, the curing system containing Trimene base, sulfur, and Tuads gave softer aged vulcanizates which swelled more than those containing triethylene tetramine, sulfur, and Tuads. It is planned to investigate further the use of high loadings of Trimene base, in order to follow this encouraging lead.

Triethylene Tetramine - Sulfur - Monex and Triethylene Tetramine - Sulfur - Polyac. Monex and Polyac, alone or in combination with sulfur, have been recommended as giving vulcanizates with very good heat resistance [Mast, W. C., Dietz, T. J., Dean, R. L., and Fisher, C. H., India Rubber World, 116, 355 (1947)]. The results of some preliminary work done to verify this are shown in Table 37. Batches cured with little or no triethylene tetramine swelled excessively and became very soft during hot-oil aging. However, when a better balance of the three vulcanizing agents was used, results were more encouraging. Batches PA-79 with Monex and PA-87 with Polyac are much improved but are still inferior to similar compounds containing Tuads as a vulcanizing agent. More work is planned with Monex and Polyac, in an effort to develop optimum aged properties.

Effect of Various Fillers on Hycar 4021

It has been reported (B. F. Goodrich Chemical Company, "Polyacrylic Rubber", Service Bulletin H-11, March, 1953) that the choice of fillers is very important in compounding Hycar 4021 stocks. Because of this, several fillers were evaluated to determine their relative merit.

Hi-Sil. Hi-Sil is one of the best nonblack fillers for Hycar 4021. In the study of Hi-Sil as a reinforcing agent, a series of compounds was made in which the amounts of this material were varied. The data in Table 38 indicate that optimum hot-oil-aged properties were obtained by the use of high loadings of Hi-Sil (65 or 75.8 phr) with 10 phr plasticizer with the stocks being tempered before aging. Lower loadings of Hi-Sil, elimination of the plasticizer, or elimination of the tempering increased the swelling. Use of 20 phr of plasticizer was harmful to the aged tensile strength.

The one disadvantage of the high Hi-Sil loadings was that the hardness was in excess of 80 before aging. However, it was possible to reduce this hardness to a satisfactory level by using a plasticizer.

Fillers Other Than Hi-Sil. Several fillers other than Hi-Sil were also evaluated in Hycar 4021. The data shown in Table 39 indicate that Silene EF has the most promise of those tested. Batch PA-95, with 70 phr Silene EF, more nearly meets the target specifications than any stock evaluated on this program. After 500 hours' aging at 350 F, it failed by only a narrow margin on three of the requirements. These were as follows:

Property	PA-95	Specification
Unaged hardness	85	80 max
Aged swelling	35.7	30.0 max
Aged elongation	90	100 min

At the beginning of our work, B. F. Goodrich Chemical Company recommended 40 phr of Philblack A in Hycar 4021 as a standard stock. Because of our results with high loadings of Hi-Sil and Silene EF, high loadings of this carbon black also were investigated. Table 39 shows that varying the loading of Philblack A from 40 to 80 phr produced no consistent improvement in the aged physical properties. The same data also show that the aged vulcanizates with this black were inferior to the aged vulcanizates prepared with Hi-Sil or Silene EF. Substitution of Calcene TM for part of the Philblack A decreased the aged swelling, but at a sacrifice to the aged tensile strength.

ELC magnesia vulcanizates also were evaluated, because of the promising results found for this material in Hycar 1001. However, excessive swelling and even cracking were found for Hycar 4021 compositions containing this filler.

Preliminary evaluations with Hi-Sil C, Philblack O, and Aerosil gave rather mediocre results and appear to merit no further work.

As a result of work to date, it is planned to continue the studies of Hi-Sil and Silene EF as reinforcing agents for Hycar 4021.

Effect of Lubricants on Hycar 4021

Stocks containing Hycar 4021 are difficult to process, because of sticking or splitting during milling. To overcome this, a small amount of stearic acid has usually been added to recipes containing this rubber. While this reduced the problem, it did not eliminate it. Therefore, a short study was made of the effect of increasing the stearic acid content and trying other lubricants.

The results in Table 40 indicate that sticking was reduced by the use of additional stearic acid, but at the expense of the hot-oil-aged physical properties. The two lubricants tried, other than stearic acid (Acrawax CT and lanolin), had a similar adverse effect on Hycar 4021. In view of these results, it was decided to continue the use of one part of stearic acid in all future work. Since beginning this study, it has been found that careful control of the mill temperature is a satisfactory method for obtaining better processing.

Comparison of Effects of Esso and Penola Turbo Oil on Hycar 4021

In an earlier section of this report, a comparison was made between the effects of Esso and Penola Turbo Oil-15 on compositions from Hycar 1001. A similar comparison was made on compositions from Hycar 4021, with results given in Table 41. The samples aged in the Penola oil for 72 hours had about 200 psi less tensile strength than those aged in the Esso oil. However, when the oil aging was extended to 500 hours, the rubber samples aged in both oils gave practically identical results. No other significant differences were noted between the effects of the two types of oil. It is understood that the Materials Laboratory, WPAFB, has had similar experience in the aging of rubber in different lots of Esso Turbo Oil-15.

Blends of Hycar 1001 and Hycar 4021

Previous work has shown that Hycar 1001 vulcanizates swell much less than Hycar 4021 in Turbo Oil-15. Hycar 1001, however, has the serious disadvantage of cracking badly when hot-oil aged. In order to take advantage of the oil resistance of the nitrile rubber, some studies were made in which it was blended with relatively large amounts of the acrylate rubber, Hycar 4021.

The data in Table 42 show that up to 20 parts of Hycar 1001 was beneficial in increasing the hardness, but contributed no other beneficial effects to Hycar 4021. It is very interesting to observe that there was no cracking in any of the blends of these two rubbers in the ratios explored. Tempering was found to lower the aged swelling.

This work will be extended to 30 and 40 parts of Hycar 1001, to determine whether swelling can be reduced without inducing cracking during extended hot-oil aging.

Compounding Acrylon EA-5

Acrylon EA-5 is a copolymer of 95 per cent ethyl acrylate and 5 per cent acrylonitrile, produced by the American Monomer Corporation. This material has been evaluated to determine vulcanizate properties after aging in Turbo Oil-15 at 350 F. Complete data are shown in Table 41. Because a large number of 500-hour tests are in progress, most of the conclusions must be drawn on the basis of only 168 hours' aging. Conclusions which appear evident at this time are as follows:

(1) Small amounts of triethylene tetramine (0.8 part) are insufficient to form a suitable cure. Larger amounts of amine correct this.

(2) Long cures result in lower aged tensile strength and elongation (which is undesirable), but decrease the aged swelling (which is very desirable).

(3) Data are insufficient to show whether Philblack A or Philblack O is the better black to use. However, results with Philblack E indicate that this type of carbon black retards the cure.

(4) Data are insufficient to determine whether 40 or 50 parts of carbon black are optimum.

(5) Tempering showed no advantage.

(6) As the aging period increased, tensile strength, elongation, and swelling all decreased. Hardness reached a minimum and then increased.

(7) Cracking was noted in several samples. However, data are insufficient to determine the cause of this.

In comparing the results for Acrylon EA-5 with those for Hycar 4021, previously discussed, it is believed that Acrylon EA-5 shows promise equal to Hycar 4021. The strongest point in favor of Acrylon EA-5 is that it evidences decreased swell during prolonged hot-oil aging. Excessive swelling is the one shortcoming of the Hycar 4021, as it swells continually throughout the aging period. When all of the 500-hour-aging tests are completed (Table 43), a much better picture on Acrylon EA-5 will be available.

Work with Acrylon EA-5 will continue. This polymer will be used alone and in blends with Hycar 4021, to determine how this polymer can best be used. In view of the encouraging results obtained with Silene EF and Hi-Sil as reinforcing agents for Hycar 4021, studies will be made with these and other nonblack pigments in Acrylon EA-5. However, carbon blacks still show promise in acrylate-type rubbers, in contrast to their poor showing in nitrile-type rubber.

Compounding Miscellaneous Polymers

Four polymers were given a brief evaluation to see how their aged properties compare with Hycar 4021.

Hycar PA (Polyethylacrylate)

This material was compounded in a recipe recommended by B. F. Goodrich Chemical Company. The data in Table 44 show Hycar PA has very good resistance to swelling, but very poor elongation and cracking after aging.

Acrylon BA-12 (Copolymer of Butyl Acrylate and Acrylonitrile)

Acrylon BA-12 was compounded in a recipe recommended by American Monomer Corporation. After aging in Turbo Oil-15 (Table 44), the vulcanizates became very soft and swelled excessively.

Philprene VP (Copolymer of Vinyl Pyridine and Butadiene

Philprene VP was compounded according to the recommendations of Phillips Chemical Company. Data in Table 44 indicate a negligible tensile strength and elongation after aging. In addition, it showed the typical cracking of an unsaturated polymer.

Silicone

A number of cured silicone-rubber stocks were obtained from commercial sources for a preliminary evaluation of the resistance of this type of rubber to hot Turbo Oil-15. As indicated in Table 45, these compositions displayed poor physical properties after oil aging at 350 F.

Further research with the silicones was deemed inadvisable, since previous experience with this type of rubber at WADC revealed that it tends to dissolve when a critical temperature is reached. Even though this critical temperature may be considerably higher than 350 F, there is always the possibility that an overheat will occur which will destroy the rubber. While excessive temperatures also are harmful to "organic-type" rubbers, these do not tend to disintegrate in contact with hot oil as do the silicones.

Compression-Set Tests

Because of the possibility of using acrylate polymers in seals such as 0-rings, compression-set measurements were made on several promising stocks. An apparatus was specially designed so that the tests could be made in the aluminum-block heater. Complete details of this test have been given previously in this report.

Table 46 shows the results of compression-set tests on two of the most promising stocks, one a carbon black stock and one a nonblack stock. Results indicate a compression set of about 95 per cent, although tempering decreased this to about 75 per cent.

It is planned to limit hot compression-set tests to only those samples which closely approach the target specifications.

SUMMARY

Compounding Nitrile-Type Rubber

The main effort of this phase of the research program has been concentrated on the selection and evaluation of conventional and experimental compounding ingredients which impart oil- and heat-resistant qualities to butadiene-acrylonitrile copolymers, principally Hycar 1001. The importance of proper types and concentrations of rubber ingredients was established early in the program, when preliminary aging data disclosed that any material added to the rubber influenced its performance at elevated temperatures. Therefore, a search was initiated to find compounding ingredients which neither decompose nor promote degradation of the rubber at 350 F.

Fillers

Early filler studies showed that loadings of 40 to 80 parts of carbon black per 100 parts rubber (phr) induced cracking and contributed toward other inferior aging characteristics. Reducing the carbon black level to 25 phr improved the crack resistance, but at the expense of other aging properties.

Attention was given to nonblack fillers, and aging data revealed that this type of reinforcing agent was superior to carbon black in compounds exposed to diester oils at 350 F. The best results to date with nitrile-type rubber have been obtained with compositions containing 100 phr of magnesia (Compound A-23). It exhibited low swell and excellent retention of physical properties after aging in Turbo Oil-15 at 350 F for 168 hours. However, it lacked adequate crack resistance. Endeavors to provide better crack resistance by means of selected nonextractible plasticizers, softeners, antioxidants, and other compounding materials were unsuccessful. In fact, in most instances, the incorporation of other ingredients in this formulation impaired all hot-oil aging properties.

Curing Systems

An investigation of low-sulfur and nonsulfur curing systems disclosed that, while the original physical properties were influenced by the choice of curatives, the properties after aging in Turbo Oil-15 at 350 F for 168 hours were affected only slightly by differences in curing systems. In most instances, the aged properties were quite similar, regardless of the curing system employed.

It was observed that tighter cures were obtained with larger amounts of available sulfur, but this had no beneficial effect on the physical properties of the vulcanizates after hot-oil aging.

Nonsulfur curatives produced vulcanizates which displayed properties similar to those obtained with sulfur, but none which were better. Aging data failed to reveal any advantage attained with nonsulfur curing agents which could not be achieved with low-sulfur systems.

Antioxidants

Extensive antioxidant studies were conducted in an effort to find a method of providing adequate protection for the rubber against degradation at 350 F. These included studies to determine (1) the effectiveness of conventional and experimental antioxidants, (2) the effect of large amounts of antioxidants, and (3) the desirability of adding antioxidants to the rubber, oil, or both.

An evaluation of commercial and experimental antioxidants disclosed that none afforded sufficient protection to render the rubber suitable for use at 350 F. Some contributed toward slightly better crack resistance, but gains in this direction were meager. Combinations of some of the more effective antioxidants did not exhibit any synergistic action or evidence greater-than-additive protection.

It was thought that, perhaps, larger amounts of antioxidant than are commonly used might provide greater protection for the rubber against oxidation. However, aging results illustrated that, in most instances, large concentrations (10 to 20 phr) of antioxidant were no more effective than normal amounts.

Data obtained from tests in which antioxidants were added to the rubber, oil, and both, revealed that limited improvement was obtained when the agent was added to both.

Processing Aids

Compounding studies with zinc oxide and stearic acid disclosed that the optimum concentrations of these ingredients were 2.5 to 5 and 0.75 to 1.5 phr, respectively. Increasing or decreasing the amounts beyond these limits impaired aging properties.

The processability of some of the more heavily loaded stocks, such as Compound A-23, was markedly improved by the incorporation of zinc stearate in these compositions. However, even small amounts of this material drastically reduced hot-oil-aged properties. Other processing aids produced similar results.

Nonextractible Plasticizers, Softeners, and Other Additives

No significant improvement in aging properties was observed in compositions containing nonextractible plasticizers and softeners. Although it was expected that these materials would reduce tensile strength, it was felt that this loss could be tolerated if sufficient improvements were obtained in crack resistance and elongation.

Nitrile-type plasticizers enhanced aged elongation, but at the sacrifice of tensile strength. These plasticizers did not contribute toward crack resistance. Other plasticizers and softeners, such as Paraplex G-25, Factice, and hydrocarbon resins, were detrimental to the aging characteristics, indicating that the use of such ingredients is not desirable in oil-resistant compounds for high-temperature service.

Effect of Acrylonitrile Content of Copolymer

A brief study of acrylonitrile-butadiene copolymers revealed that those containing 40 to 45 per cent acrylonitrile (Hycar 1001, Chemigum N3NS) displayed the best aging qualities. Lower acrylonitrile rubbers (Hycar 1002, Paracril B, and Paracril AJ) softened, swelled excessively, and showed poor retention of initial properties. High acrylonitrile rubbers (55 to 60 per cent acrylonitrile) exhibited unsatisfactory hardness and elongation.

Effect of Curing Conditions

Initial data obtained on the influence of high curing temperatures (350 to 400 F) on Hycar 1001 stocks indicated that these higher-than-usual curing temperatures have little effect on aging qualities. Since vulcanization certainly continues during aging, there was considered to be only a remote possibility that better aging characteristics could be achieved by the use of more stringent curing conditions.

Effect of Aging Conditions

The results of hot-oil-aging tests showed that air is the major factor in promoting degradation of the rubber. Compositions aged in oil exposed to only a limited amount of air displayed far better retention of physical properties than those aged in oil exposed to unlimited air.

A study of the effect of metals on oil aging of rubber indicated that contact of oil with metals does not appear to influence the oil-aged properties of the rubber, at least in systems exposed to unlimited air.

Reproducibility of Results

Tests were performed to determine the degree of reproducibility of results for oil aging of rubber. The data indicated that results could be duplicated, both for the testing of a series of batches of the same composition and for a series of tests on the same batch of stocks.

Degradation of Turbo Oil-15

Aging tests conducted on rubber samples immersed in Turbo Oil-15 and in di-(2-ethylhexyl) sebacate at 350 F demonstrated that, after 500 hours, neither of the aged fluids deteriorated new rubber more rapidly than when it was fresh. Very similar results were obtained on rubbers aged in these two diester-type oils.

Tests performed to determine the increase in peroxide content of these diester oils at 350 F indicated that the deterioration of both these oils occurs only in the presence of air.

Compounding Hycar 4021

Hycar 4021 vulcanizates were developed on this program that displayed improved resistance to aging in Turbo Oil-15 at 350 F. The best compositions possessed properties which met all of the minimum target specifications, except that of swelling. The lowest swelling results obtained were about 6 per cent higher than the target maximum of 30 per cent. Table 47 shows data for the best compounds arranged in order of increasing swelling after 500 hours' aging.

Fillers were found to be one of the most important compounding variables. The most promising stocks were prepared with Silene EF and Hi-Sil (Batches PA-94, -95, and -98). Philblack A stocks were somewhat poorer in performance. An increase in the Philblack A content from 40 to 80 parts showed no consistent advantage. When using ELC Magnesia, Hi-Sil C, Philblack O, and Aerosil as a filler, hot-oil-aged properties were about equal to or poorer than those that were found with the use of Philblack A.

Several vulcanizing systems were evaluated, the best ones containing triethylene tetramine and Tuads, with or without sulfur. Two optimum ratios of these components were determined, as shown in Batches PA-2 and PA-52. When the vulcanizing systems Trimene base - sulfur - Tuads, triethylene tetramine - sulfur - Polyac, or triethylene tetramine - sulfur - Monex were used, inferior aging properties resulted. Long cures and large amounts of vulcanizing agent were also found to improve the aged properties.

The use of up to 20 parts of Hycar 1001 with Hycar 4021 increased the aged hardness, but otherwise had little effect on properties.

One part of stearic acid was found to be the optimum amount to use as a lubricant. Acrawax CT and lanolin were found to be less desirable as lubricants.

Compression set tests on the best stocks indicate a set of about 95 per cent before tempering and 75 per cent after tempering.

Compounding Acrylon EA-5

Acrylon EA-5 vulcanizates show considerable promise as can be seen by the results on Batch PA-83. This was the only sample tested which met the specification for a maximum of 30 per cent swelling after 500 hours. However, for shorter periods of aging this sample exhibited swell in excess of this amount.

A proper balance of vulcanizing agent was found to be very important with this polymer. Unless a correct balance was used, vulcanizates were badly undercured, even after 120 minutes of curing time.

Compounding Miscellaneous Polymers

Limited evaluations were made on Hycar PA, Acrylon BA-12, Philprene VP, and silicones. Results indicate that these polymers are completely unsuited for this application.

PLANS FOR FUTURE WORK

This project has been extended to December 31, 1954, to provide for additional research on the development of rubber compositions that will be suitable for use in hot oils. The emphasis is to shift to temperatures higher than 350 F, with the first step being 400 F.

Future work on this project will involve primarily (1) a limited amount of research with nitrile-type rubber, aimed at improving the crack resistance of this rubber, (2) a continuation of the compounding studies with the acrylate-type rubbers, with special effort directed toward reducing the swell of this type rubber, and (3) initiation of a compounding study on a new experimental polymer, known as FBA (poly-1, 1-dihydroperfluoro-butyl acrylate). Specific details of the work to be done are presented in the following sections.

Nitrile-Type Rubber

Extensive compounding studies with nitrile-type polymers have disclosed that although stocks can be compounded which display low swell and good retention of physical properties after prolonged exposure to diester-type oils at 350 F, the cracking of this type rubber has not been satisfactorily eliminated. Only meager gains in this direction have been accomplished by variations in fillers, antioxidants, curing agents, and other compounding ingredients.

The cracking is believed to be an inherent characteristic of the rubber, stemming from the vulnerability of the double bonds in the butadiene portion of the rubber molecule to oxidation. This belief is supported by aging data which indicate that cross linking and chain scission, both presumably the result of oxidation, occur during aging of the rubber. The gradual increase in hardness and reduction in elongation and swell is evidence of cross linking; the reduction in tensile strength and cracking indicate oxidative chain scission. When hot-oil aging was conducted in air-tight containers, there was less degradation of both these types. The higher amount of swell and the tendency for surface crazing of compounds aged in closed containers are interpreted as evidence of chain scission occurring, even in the limited amount of air present. The more rapid degradation of nitrile rubber in air than in oil for 72-hour-aging tests also indicates that air may well play a dominant role in the deterioration of this rubber.

Further evidence that the unsaturation of nitrile rubbers promotes cracking is illustrated by the fact that saturated polymers, such as the acrylates, do not tend to crack after aging at 350 F.

Thus, it appears that the elimination of cracking in nitrile rubber compositions would involve some means of saturating the double bonds of the rubber without cross linking. One possible method of accomplishing this is to employ a material which can be compounded into rubber and perform this function. Several materials of this type are under investigation at this time, to determine if this is a feasible approach to the problem.

Other possible methods of reducing unsaturation include (1) hydrogenation and (2) polymerization of an entirely new acrylonitrile-containing polymer, which has a low amount of unsaturation. Hydrogenation of butadiene and other polymers by the Phillips Petroleum Company [Jones, Moberly, and Reynolds, Ind. Eng. Chem., 45, 1117, (May, 1953)] led to the development of much more oxidation-resistant polymers. While the successful hydrogenation of nitrile-type rubber has not been reported, it seems possible that such a polymer might be developed.

The polymerization of a unique acrylonitrile-containing polymer having a low amount of unsaturation might possibly be achieved by replacing a portion of the butadiene in a butadiene-acrylonitrile polymer with a monomer which will contribute no double bonds to the polymer. The product should be suitable for vulcanization by conventional methods employed for butyl rubber or other diene-containing polymers. Cross linking, through vulcanization, will be necessary to produce a three-dimensional-type structure. The latter is required to produce rubber-like properties.

While all these approaches to the problem of reducing unsaturation in nitrile rubber would be too extensive to be included in the present program, it is felt that studies of this type are needed before this type rubber can successfully be used in hot oils.

Acrylate Polymers

Several promising leads have been uncovered and will be further developed in order to obtain the best possible aged properties in Hycar 4021 and Acrylon EA-5. Particular emphasis will be placed on reducing swell, as this is the one property which does not meet minimum specifications. Past work has shown that use of the proper ratios of vulcanizing agents in high levels and employing long curing times results in tighter cures and reduces swell. These leads will be followed further, along with a study of the effect of increasing the curing temperature.

Further work with fillers also will be undertaken. Hi-Sil and Silene EF are the most promising to date and will receive further attention. Several experimental types of Hi-Sil and Indulin will also be evaluated.

Work will be divided about equally between Hycar 4021 and Acrylon EA-5 as both of these polymers show about equal promise. Furthermore, blends of these two polymers and additional blends of Hycar 1001 with Hycar 4021 are now under test. Samples of experimental polymers Hycar 4021 x 26 and Acrylon EA-9 are on hand to be tested.

Limited work is also planned on plasticizer evaluation using both the extractible and nonextractible types.

FBA Polymer

A limited experimental program is planned with the FBA polymer (poly-1-1-dihydroperfluorobutyl acrylate), produced for WADC by the Minnesota Mining and Manufacturing Company. Preliminary tests at WADC indicate this polymer meets all the minimum requirements for use in Turbo Oil-15 at 350 F. With their work as a background, an effort will be made to improve the performance of this polymer and to extend its usefulness to higher temperatures. A limitation on this work at Battelle will be the amounts of this polymer that will be made available for study.

APPENDIX A

EXHIBIT A TO CONTRACT NO. AF 33(616)-476

APPENDIX A

EXHIBIT A TO CONTRACT NO. AF 33(616)-476

I General Description.

A. The Contractor shall exert his best efforts toward the development and physical testing of a rubber compound suitable for fabrication into seals, gaskets, hose or other rubber items which may be required to withstand the action of synthetic lubricants and/or hydraulic fluids at elevated temperatures.

B. Modification of the requirements or procedures may be permitted if such changes are agreed upon by the Materials Laboratory, Wright Air Development Center, and the Contractor.

II Detailed Description.

A. The rapid introduction of synthetic base lubricants and hydraulic fluids into aircraft applications has resulted in a growing demand for elastomeric materials suitable for retaining these lubricants and fluids, particularly at elevated temperatures. Known work to date has resulted in compounds exhibiting reasonable rubber-like characteristics for periods only slightly in excess of 100 hours under simulated service conditions. Compounds should therefore be developed with the end products in mind so as to produce an elastomer with properties equaling or surpassing the values set forth in the following paragraph. Suggested approaches to the problem may include but not be limited to the following:

1. Extensive evaluation of commercially available polymers and polymer blends
2. Evaluation and, if necessary, development of high-temperature antioxidants
3. Study of unique curing systems especially designed for high-temperature applications
4. Preliminary compounding and evaluation of commercially developed experimental polymers
5. Compounding and evaluation of Government-furnished experimental polymers
6. Limited-scale polymer development, if feasible
7. Consideration of oil additives which may inhibit rubber deterioration.

B. Desired properties and test methods are as follows:

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 1000 hours at 350 F. This is the minimum temperature, and although a duration of 1000 hours at this temperature may not be reached, efforts will continually be directed toward obtaining a rubber which will retain satisfactory properties for as long as possible.

Test fluids for this work will include Esso Turbo Oil-15, and other MIL-L-7808 oils as they may become available.

As a secondary objective it is desired that a rubber compound be developed which will be resistant to the action of synthetic hydraulic fluids when immersed in a test fluid at 400 F to 550 F. Test fluids will be California Research No. 52742R silicate ester base fluid and such others as may be developed.

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

Original Properties

Tensile	1000 psi min
Elongation	200% min
Shore "A" Hardness	50-80

Low-temperature flexibility - may be ignored during initial development. Ultimate requirement of flexibility at -65 F should be given consideration if possible.

Properties After Oil Immersion for 500-1000 Hours at 350 F

Tensile	500 psi min
Elongation	100% min
Shore "A"	50-90
Volume change	-2 to +30%

Appearance - No evidence of checking or cracking after 180-degree flat bend.

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 500 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. Due to a lack of standardized test procedures for extreme high-temperature evaluation, consideration should be given to developing same.

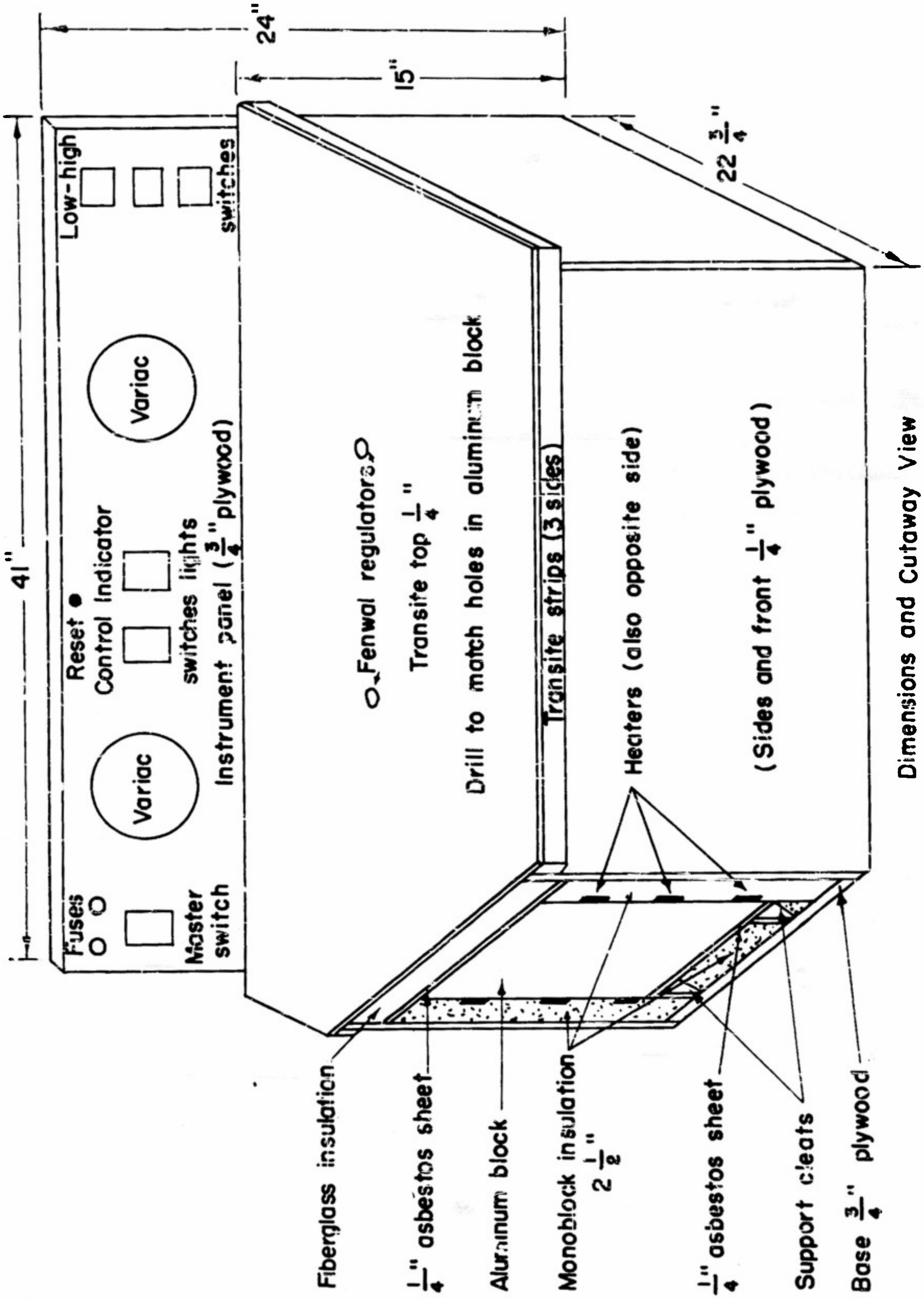
C. The materials and laboratory equipment required in the performance of this contract shall normally be furnished by the Contractor. However, in the event that a required item of equipment, not readily available to the Contractor, is available at the Materials Laboratory, portions of the work requiring use of that equipment may be conducted at the Materials Laboratory by mutual agreement between the Materials Laboratory and the Contractor. In addition, if such materials as are necessary to the performance of this contract are not readily available to the Contractor (e.g., synthetic oils), an attempt will be made by the Materials Laboratory to provide a source of these materials for the Contractor.

D. Compounding and processing of the various batches shall be as considered necessary or desirable to produce optimum properties, with the exception of low-temperature properties as noted in II-B above. Complete formulation, compounding, and processing data shall be included in reports submitted.

E. Samples of promising developments, in the form of three (3) standard tensile slabs, together with necessary formulations, compounding, and processing information in letter form shall be submitted to the Materials Laboratory, Wright Air Development Center, as they become available throughout the life of the contract.

APPENDIX B

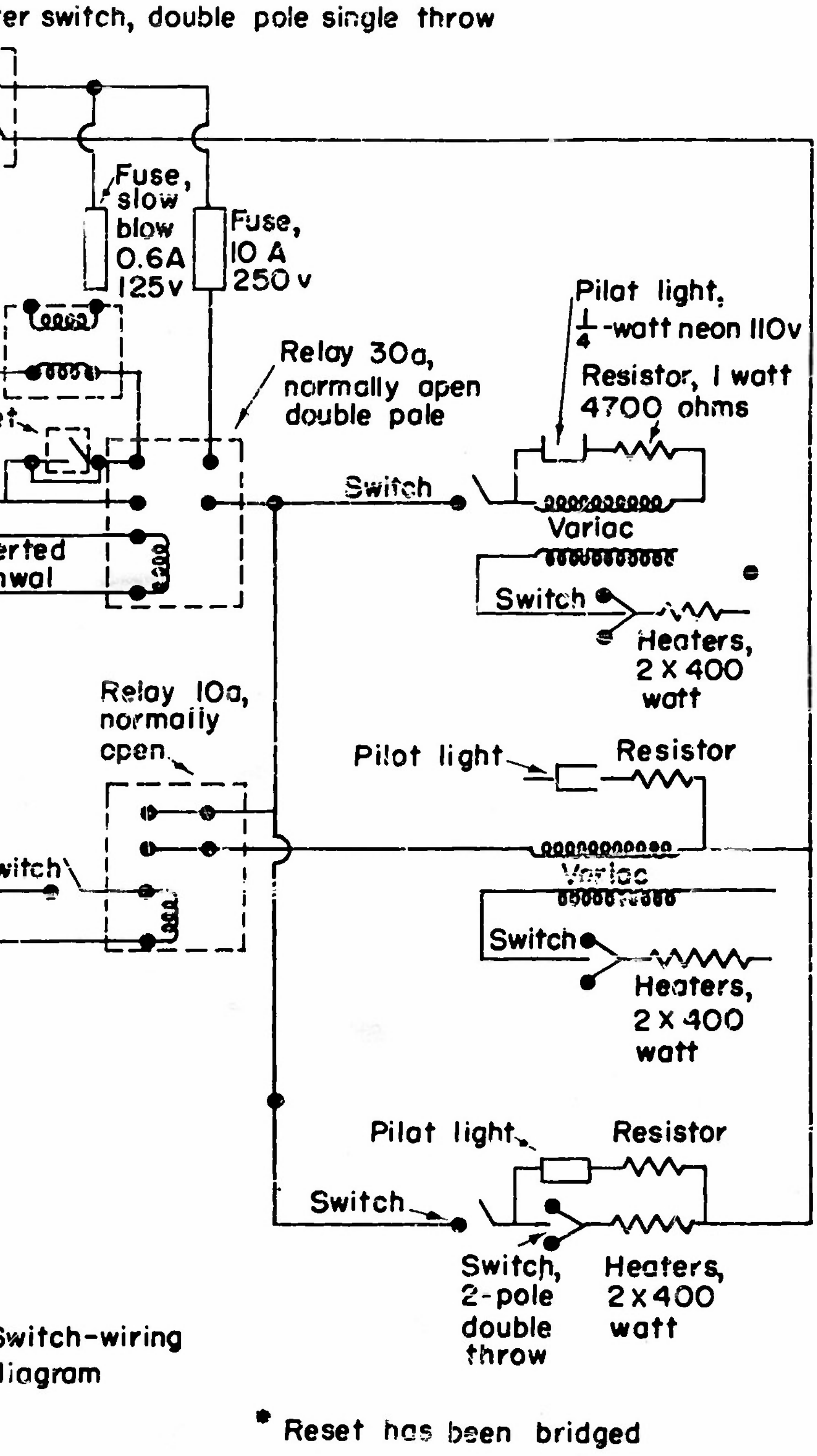
DRAWING OF ALUMINUM-BLOCK HEATING UNIT

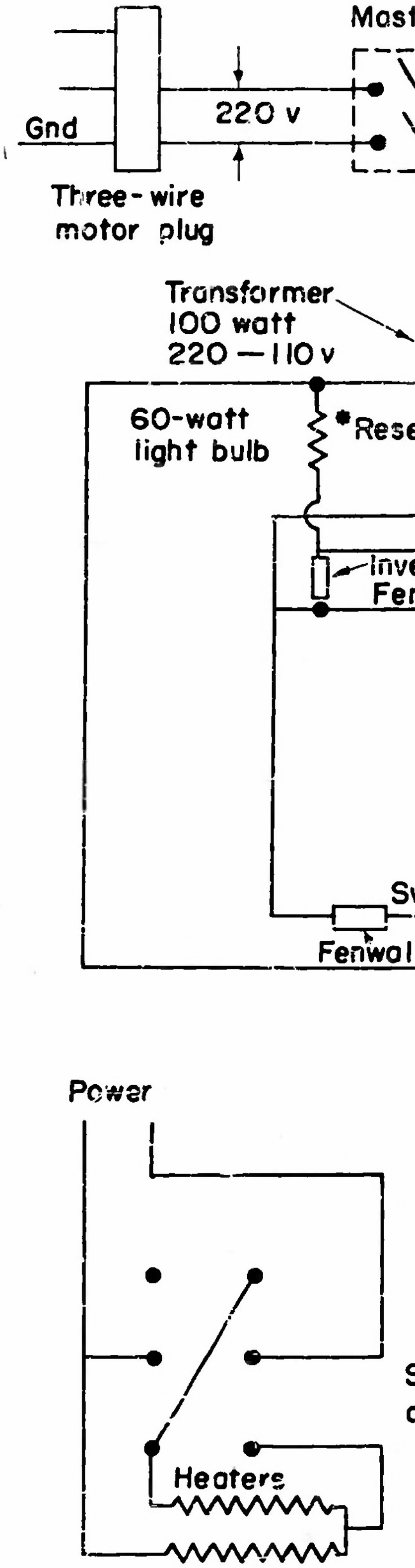


Dimensions and Cutaway View

FIGURE I. ALUMINUM - BLOCK HEATER

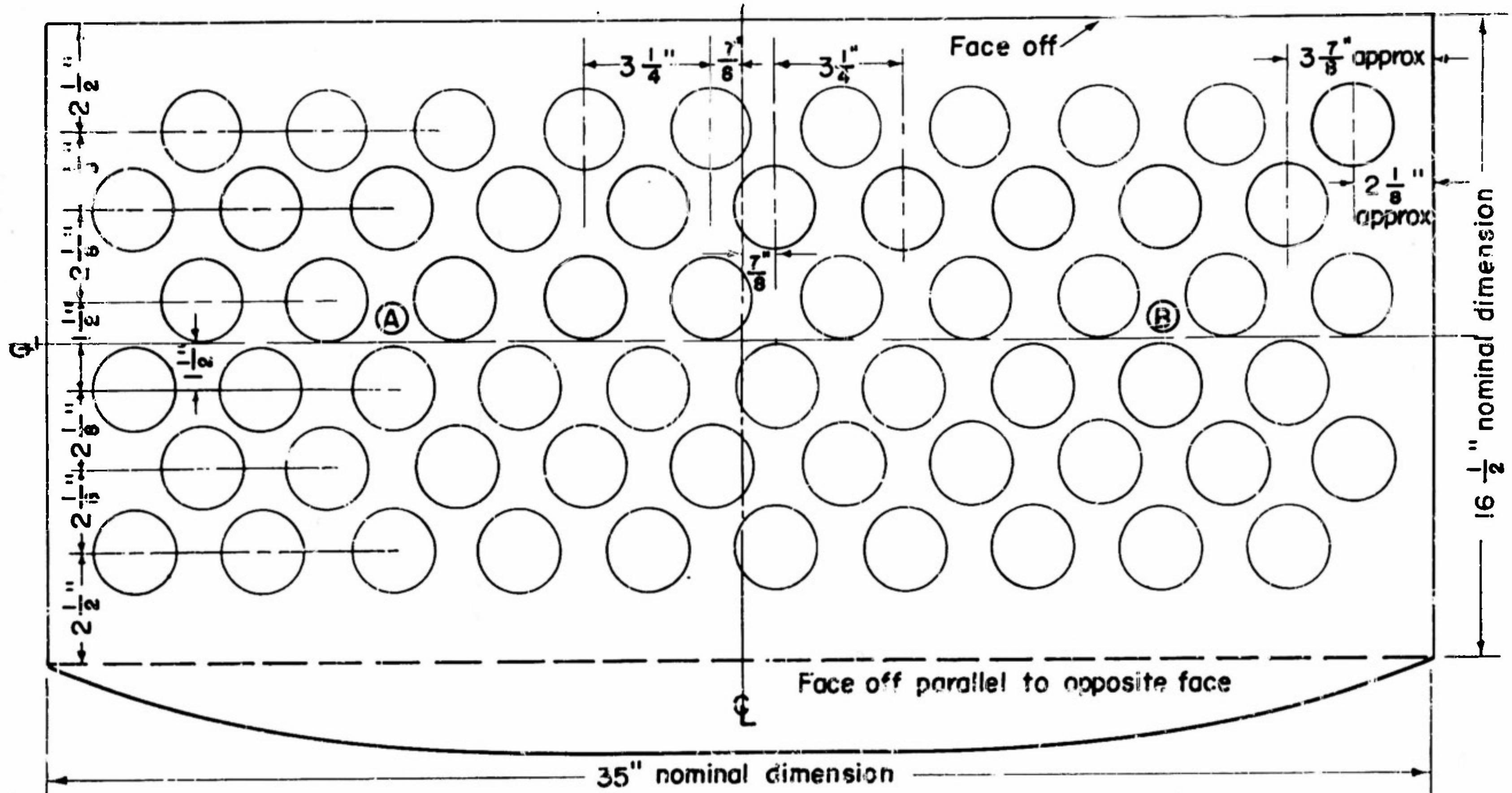
0-21493





Switch-wiring diagram

FIGURE 2. WIRING DIAGRAM



(1) Lay out block from ℄ as shown
(2) Drill holes (60) $1\frac{3}{4}$" diameter and 7" deep (over-all)
(3) AB center line drill holes for $\frac{1}{2}$-14NPT, 4" deep; tap, $\frac{3}{4}$" deep
(4) Lay out, drill, and tap faces for three strip heaters per face

FIGURE 3. ALUMINUM AGING BLOCK FABRICATED FROM SHEET-METAL INGOT CASTING

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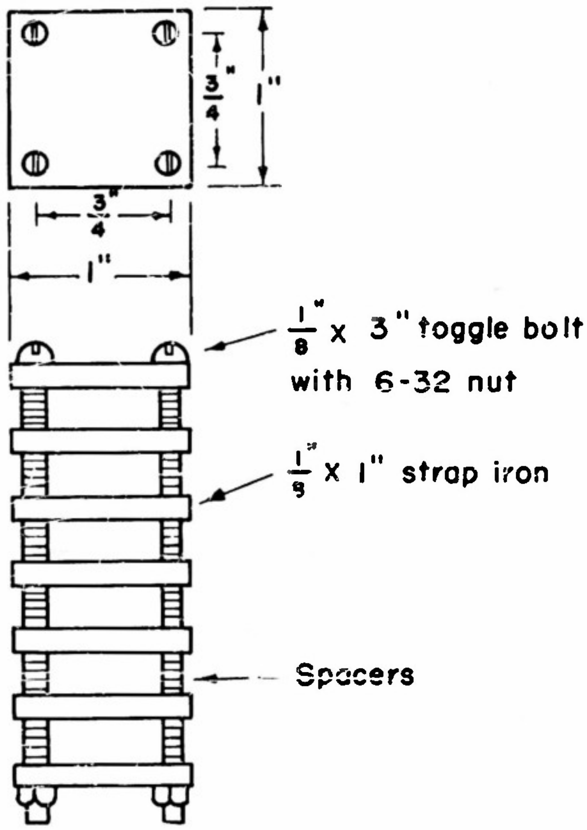
APPENDIX C

DRAWING OF JIG FOR DETERMINING COMPRESSION SET IN HOT OIL



Cross-hatched area indicates samples

Full scale

FIGURE 4. JIG FOR DETERMINING COMPRESSION SET IN HOT OIL

APPENDIX D

LIST OF MATERIALS AND THEIR SOURCE

APPENDIX D

LIST OF MATERIALS AND THEIR SOURCE

Material	Composition	Source
Acrawax CT	Not disclosed	Glyco Products Company, Incorporated
Acrylon BA-12	Copolymer of butyl acrylate and acrylonitrile	American Monomer Corporation
Acrylon EA-5	Copolymer of ethyl acrylate and acrylonitrile	American Monomer Corporation
Aerosil	Silica	Godfrey L. Cabot, Inc.
AgeRite Alba	Hydroquinone monobenzyl ether	R. T. Vanderbilt Company
AgeRite Hipar	Mixture of phenyl-beta-naphthyl-amine, p-isopropoxy diphenylamine, and diphenyl-p-phenylene diamine	Ditto
AgeRite Powder	Phenyl-beta-naphthyl-amine	"
AgeRite Resin D	Polymerized trimethyl-dihydroquinone	"
Altax	Benzothiazyl disulfide	"
Alumina hydrate	Aluminum oxide, hydrated	Westvaco Chlorine Products Corporation
Aluminum oxide	Aluminum oxide	J. T. Baker Chemical Company
Aluminum silicate	Aluminum silicate	Kraft Chemical Company
Aminox	Reaction product of diphenylamine and acetone	Naugatuck Chemical Division, United States Rubber Company

Material	Composition	Source
Aniline	Aniline	Distillation Products Industries, Division of Eastman Kodak Company
Antox	Condensation product of butyraldehyde and aniline	Rubber Chemicals Division, E. I. du Pont de Nemours & Company, Incorporated
Barytes	Barium sulfate	Thompson, Weinman and Company, Incorporated
Benzal chloride	Benzal chloride	Fisher Scientific Company
p-Benzylaminophenol	p-Benzylaminophenol	Distillation Products Industries, Division of Eastman Kodak Company
B.L.E.	Mixture of a complex diarylamine-ketone-aldehyde reaction product and n, n'-diphenyl-p-phenylendiamine	Naugatuck Chemical Division, United States Rubber Company
t-Butyl catechol	t-Butyl catechol	Distillation Products Industries, Division of Eastman Kodak Company
Cadmium oxide	Cadmium oxide	J. T. Baker Chemical Company
Calcene TM	Calcium carbonate	Columbia-Southern Chemical Corporation
Calcium oxide	Calcium oxide	J. T. Baker Chemical Company
Catechol	Catechol	Battelle
Chemigum N3NS	Copolymer of butadiene and acrylonitrile	The Goodyear Tire & Rubber Company
o-Cresol	o-Cresol	Battelle
Dibutyl sebacate	Dibutyl sebacate	Rohm & Haas Company

Material	Composition	Source
Dibutyl tin maleate	Dibutyl tin maleate	Metal & Thermit Corporation
2, 5-Dicyclohexyl hydroquinone	2, 5-Dicyclohexyl hydroquinone	Monsanto Chemical Company
Dinitrobenzene	Dinitrobenzene	J. T. Baker Chemical Company
Disodium lead versenate	Disodium lead versenate	Bersworth Chemical Company
Di-tert-butyl-para-cresol	Di-tert-butyl-para-cresol	Battelle
Dixie Clay	Kaolin	R. T. Vanderbilt Company
DPR Synthetic N-27	Not disclosed	DPR, Incorporated
Dyphos	Dibasic lead phosphite	National Lead Company
Ethyl tellurac	Tellurium diethyldithiocarbamate	R. T. Vanderbilt Company
Ethyl Tuads	Tetraethylthiuram disulfide	Ditto
Ferro 903	Cadmium stabilizer	Ferro Chemical Corporation
Ferro 1820	Barium stabilizer	Ditto
Flectol H	Condensation product of acetone and aniline	Monsanto Chemical Company
Flexol R2H	Polyester	Carbide & Carbon Chemicals Company
Gilsonite	Asphaltic hydrocarbon	Allied Asphalt & Mineral Corporation
Glyptal Plasticizer 2557	Polymeric-type plasticizer	General Electric Company

Material	Composition	Source
GMF	p-Quinonedioxime	Naugatuck Chemical Division, United States Rubber Company
GR-I 18	Copolymer of isobutylene and isoprene	Rubber Reserve Company
Hi-Sil	Hydrated silica	Columbia-Southern Chemical Corporation
Hi-Sil C	Hydrated silica	Ditto
Hycar 1000 x 70	Copolymer of butadiene and acrylonitrile	B. F. Goodrich Chemical Company
Hycar 1001	Ditto	Ditto
Hycar 1002	"	"
Hycar 1011 x 15	"	"
Hycar 1012 x 41	"	"
Hycar 4021	Copolymer of ethyl acryiate and chloroethyl vinyl ether	"
Hycar PA	Polyethyl acrylate	"
Hydroquinone	Hydroquinone	Mallinckrodt Chemical Works
2-Hydroxyquinoline	2-Hydroxyquinoline	Distillation Products Industries, Division of Eastman Kodak Company
Lanolin	Refined wool fat	Lanatex Products Sales Company
Light precipitated chalk	Calcium carbonate	Thompson, Weinman and Company, Incorporated
Litharge	Lead monoxide	National Lead Company
Magnesia (ELC)	Magnesium oxide	Michigan Chemical Corporation

Material	Composition	Source
Magnesium carbonate	Magnesium carbonate	Michigan Chemical Corporation
Magnesium stearate	Magnesium stearate	Witco Chemical Company
Manganese dioxide	Manganese dioxide	J. T. Baker Chemical Company
Mark XI	Cadmium-barium salt	Argus Chemical Company
Mark XX	Epoxy material	Ditto
Methyl Tuads	Tetramethylthiuram disulfide	R. T. Vanderbilt Company
Mica	Mica	Diamond Alkali Company
Micronex	Medium processing channel black	Binney & Smith Company
Mineral rubber	Bituminous petroleum product	Witco Chemical Company
Monex	Tetramethylthiuram monosulfide	Naugatuck Chemical Division, United States Rubber Company
2MT	2-Mercaptothiazoline	Rubber Chemicals Division, E. I. du Pont de Nemours & Company, Incorporated
NBC	Nickel dibutyldithiocarbamate	Ditto
Neophax A	Vulcanized vegetable oil	Stamford Rubber Supply Company
Neophax D	Ditto	Ditto
Neoprene S	Polychloroprene	E. I. du Pont de Nemours & Company, Incorporated
Neozone A	Phenyl-alpha-naphthylamine	Ditto

Material	Composition	Source
Nitrophenol	Nitrophenol	Distillation Products Industries, Division of Eastman Kodak Company
Nonyl phenol	Nonyl phenol	Battelle
ODN Plasticizer	Octadecene nitrile	Harwick Chemical Company
Paracril AJ	Copolymer of butadiene and acrylonitrile	Naugatuck Chemical Division, United States Rubber Company
Paracril B	Ditto	Ditto
Paraplex G-25	Polymeric-type plasticizer	Rohm & Haas Company
Parazone	p-Phenyl phenol	Rubber Chemicals Division, E. I. du Pont de Nemours & Company, Incorporated
PDA-10	Poly diaryl amine	Benson Process Engineering Company
Pentex	Tetrabutyl-thiuram monosulfide	Naugatuck Chemical Division, United States Rubber Company
Phenol	Phenol	J. T. Baker Chemical Company
Phenothiazine	Phenothiazine	The Neville Company
p-Phenyl Phenol	p-Phenyl phenol	Battelle
Philblack A	MAF carbon black	Phillips Chemical Company
Philblack E	SAF carbon black	Ditto
Philblack O	HAF carbon black	"
Philprene VP	Copolymer of butadiene and vinyl pyridine	"

Material	Composition	Source
Phloroglucinol	Phloroglucinol	J. T. Baker Chemical Company
Picco 100	Para coumarone-indene resin	Pennsylvania Industrial Chemical Corporation
Piccopale	Unsaturated hydrocarbon resin	Ditto
Plasticizer SC	Glycol ester of vegetable oil fatty acid	E. F. Drew & Company, Incorporated
Polyac	Poly p-dinitroso benzene	E. I. du Pont de Nemours & Company, Incorporated
Polyester HA-5-A	Resinous alkyd-type plasticizer	C. P. Hall Company
Polyrez B	Polymerized resin	Harwick Chemical Company
PPO 375	Not disclosed	Wright Air Development Center
Propyl gallate	Propyl gallate	Distillation Products Industries, Division of Eastman Kodak Company
Pyrogallol	Pyrogallol	J. T. Baker Chemical Company
Resorcinol	Resorcinol	E. I. du Pont de Nemours & Company, Incorporated
RN-34	Epoxy resin	Shell Chemical Corporation
Salol	Phenyl salicylate	Monsanto Chemical Company
Santocure	Condensation product of mercaptobenzothiazole and cyclohexylamine	Ditto

Material	Composition	Source
Santoflex AW	6-Ethoxy-1, 2-dihydro-2, 2, 4-triethyl quinoline	Monsanto Chemical Company
Santovar	Alkylated polyhydroxy phenol	Ditto
Santowhite	Alkylated phenol sulfide	Ditto
Silene EF	Hydrated calcium silicate	Columbia-Southern Chemical Corporation
Silicone SE76	Silicone gum	General Electric Company
Stabilizer A-5	Epoxy resin	Carbide & Carbon Chemical Corporation
Standard Micronex	Medium processing channel black	Binney & Smith Company
Statex B	Fine furnace black	Ditto
Stearic acid	Stearic acid	"
Sulfur	Sulfur	Stauffer Chemical Company
Super Multifex	Coated calcium carbonate	Diamond Alkali Company
Talc	Magnesium silicate	Witco Chemical Company
Telloy	Tellurium	R. T. Vanderbilt Company
Tetrone	Tetramethylthiuram tetrasulfide	Rubber Chemicals Division, E. I. du Pont de Nemours & Company, Incorporated
Tetrone A	Dipentamethylene-thiuram tetrasulfide	Ditto
Thermax	Medium thermal black	R. T. Vanderbilt Company
Titanium dioxide	Titanium dioxide	Witco Chemical Company
Tribasic lead maleate	Tribasic lead maleate	National Lead Company

Material	Composition	Source
Triethylene tetramine	Triethylene tetramine	Carbide & Carbon Chemical Corporation
Trimene base	Reaction product of ethyl chloride, formaldehyde, and ammonia	Naugatuck Chemical Division, United States Rubber Company
Triphenyl phosphite	Triphenyl phosphite	Monsanto Chemical Company
Ethyl Tuex	Tetraethylthiuram disulfide	Naugatuck Chemical Division, United States Rubber Company
Vandex	Selenium	R. T. Vanderbilt Company
Versene (regular)	Tetrasodium salt of ethylene diamine tetraacetic acid	Bersworth Chemical Company
Vixtanex B-100	Polyisobutylene	Standard Oil Company of New Jersey
Vultac No. 2	Alkyl phenol disulfide	Sharples Chemicals, Incorporated
Vultac No. 3	Alkyl phenol disulfide	Sharples Chemicals, Incorporated
Wingstay S	Not disclosed	The Goodyear Tire & Rubber Company
Wyex	Easy processing channel black	J. M. Huber Corporation
Zinc oxide	Zinc oxide	The New Jersey Zinc Company
Zinc stearate	Zinc stearate	J. T. Baker Chemical Company

APPENDIX E

LITERATURE SURVEY ON ACRYLATE POLYMERS

APPENDIX E

LITERATURE SURVEY ON ACRYLATE POLYMERS

Types

Acrylate polymers can be classed in three groups according to the monomers used in their production. These are (1) polyalkylacrylates, (2) saturated copolymers of an acrylate and a second monomer, and (3) unsaturated copolymers of an acrylate and a second monomer.

Several polyalkylacrylates have been described in the literature, but only polyethylacrylate has reached the commercial stage. This polymer, formerly sold by B. F. Goodrich Chemical Company as Hycar PA, is not now available.

Saturated copolymers of an acrylate and a second monomer are currently the most important. Much more has been written about this group, and the three commercial acrylate polymers which are available today are in this group. Most work has been done with ethyl and butyl acrylate, while the second monomer has usually been chloroethyl vinyl ether, acrylonitrile, or methacrylonitrile. The trade names of polymers in this group are shown in Table 1.

Unsaturated copolymers of ethyl acrylate with allyl maleate[12], isoprene[11], dimethylbutadiene[11], and twenty-six other unsaturated materials[11] have been described. However, as far as is known, none of these are available commercially.

Polymerization

Polyethylacrylate can be polymerized by both the emulsion and granular processes. These methods have been described in detail in the literature[9][13]. Saturated acrylate copolymers can also be prepared by emulsion polymerization. Pilot-plant production of Lactoprene EV has been described in great detail[7], and work has been recorded on the polymerization of acrylates with acrylonitrile[1][3]. Unsaturated copolymers have received less attention, but have been polymerized and reported[11][12].

Compounding

The effect of compounding ingredients, except for accelerators, has been mentioned only briefly by previous investigators. Polyethylacrylate (Hycar PA) compounding has been described by Goodrich[4]. This company

TABLE 1. DESCRIPTION AND SOURCE OF ACRYLATE POLYMERS

Type Polymer	Source: United States Department of Agriculture	Source: B. F. Goodrich Chemical Company	Source: American Monomer Corporation
Polymer of ethyl acrylate*	-	Hycar PA (Hycar PA-11)	-
Copolymer of ethyl acrylate and chloroethyl vinyl ether	Lactoprene EV	Hycar 4021** (Hycar PA-21) (Hycar PA-31)	-
Copolymer of ethyl acrylate and acrylonitrile	Lactoprene EN	-	Acrylon EA-5**
Copolymer of butyl acrylate and acrylonitrile	-	-	Acrylon BA-12**

* Identified by Goodrich as "an elastomeric polymer of an acrylic acid ester", but actually as "ethyl acrylate polymer" by Dietz and Hansen(1).

** Indicates polymers currently available. Numbers in parentheses are obsolete designations.

has also published a very complete booklet on the compounding of Hycar 4021 (ethyl acrylate-chloroethyl vinyl ether copolymer)[5]. The effect of a variety of plasticizers in Lactoprene EV (comparable to Hycar 4021) has been described[10]. Except for the three publications listed above, no literature of significance has been noted on the effect of compounding ingredients.

Vulcanization

Vulcanization of acrylate polymers has been studied and reported by approximately a dozen authors. For this discussion, the acrylate polymers will be broken down into four classes, namely, (1) polyethylacrylate, (2) copolymers of ethyl acrylate and chloroethyl vinyl ether (e.g., Hycar 4021), (3) other saturated copolymers of an acrylate, and (4) unsaturated copolymers of an acrylate.

Polyethylacrylate. Vulcanization of polyethylacrylate has been studied, but it is only recently that the theory behind this vulcanization has been explained. According to Semegen[15], two molecules of the ethyl acrylate form a Claisen type of condensation and split off an alcohol group. This reaction is accelerated by basic materials, and it will be noted that most of the recommended accelerators are basic. The earliest publication[14] recommended quinone dioxime (GMF) and benzoyl peroxide as curing agents, but reported unsatisfactory results for a Captax-sulfur-Tuads cure. Later, it was reported[6] that triethylene tetramine with stearic acid gave good cures, while Trimene base showed no inclination to cure. Goodrich[4] reported that the most heat-resistant cures were obtained with a mixture of hydrated lime and sodium metasilicate pentahydrate. A mixture of litharge and a thiazole accelerator showed less heat resistance. Only recently, a series of patents was issued to Semegen[16][17][18] listing many basic sodium compounds which vulcanize polyethylacrylate. These compounds were further described in a paper by the same author[15], and include sodium metasilicate pentahydrate, sodium metasilicate monohydrate, sodium hydroxide, sodium orthovandate, and sodium stannate. The author reports that the potassium and lithium compounds also work, but less effectively than the sodium compounds.

Copolymers of Ethyl Acrylate-Chloroethyl Vinyl Ether. The vulcanization of copolymers of this type, represented by 4021 and Lactoprene EV, has been extensively studied with encouraging results. This type copolymer has two mechanisms for vulcanization. The acrylate groups can condense, as in the case of the polyethylacrylate. Therefore, all accelerators for polyethylacrylate should also be effective with this type of polymer. In addition, Hycar 4021 has an active chlorine group which can be used in the vulcanization. Thus, compounds which vulcanize the Hycar 4021 are not necessarily effective with polyethylacrylate (Hycar PA).

Six distinct types of curing systems have been recognized which can vulcanize through the halogen group[19]. These are (1) quinone dioxime and red lead, (2) sulfur, alone or in combination with sulfur-liberating compounds, (3) peroxides, (4) dinitrobenzene and lead oxides, (5) polymerized dinitrosobenzene (Polyac), and (6) amines. Of these six types, only three are reported in the literature as giving vulcanizates with good heat resistance. Sulfur and sulfur-liberating compounds are highly recommended, with Monex[8] and Tuads[8] being most highly rated. Amines such as triethylene tetramine and Trimene base, and polymerized dinitrosobenzene (Polyac)[8] give heat-resistant vulcanizates. The quinone dioxime-red lead combination is reported to give vulcanizates with poor properties after air aging at 300 F, due to the continued accelerator action of the red lead[2][5]. No mention has been found of the effect of peroxides or dinitrobenzene on the heat aging of vulcanizates.

Sulfur and sulfur-liberating compounds act quite differently than amines in vulcanizing Hycar 4021. Amines give rapid cures which tend to revert on continued heat aging. On the other hand, sulfur compounds give much slower cures, but retain their properties better after heat aging. Generally, a mixture of an amine and a sulfur compound is used to obtain the best balance between curing time and hot-air resistance[8]. For example, Goodrich suggests that, for a compound with good heat resistance, the vulcanization system be 3.0 parts Trimene base + 0.5 part sulfur[5].

<u>Other Saturated Acrylate Copolymers</u>. Vulcanization of this class of copolymers, which includes types other than Hycar 4021 and Lactoprene EV, has also been studied. In this group are copolymers of an alkyl acrylate in which the second monomer may be acrylonitrile or another acrylate, either alkyl or aryl. The curing system most frequently mentioned as giving heat-resistant vulcanizates is a triethylene tetramine-sulfur mixture[3][14]. However, quinone dioxime (GMF) and benzoyl peroxide have also been mentioned as giving good cures[14][19]. Sulfur-Captax-Tuads vulcanizates ranged from very good to very poor, depending upon the comonomer used with the alkyl acrylate[14]. A recent patent[20] lists a variety of compounds which may be used to vulcanize the saturated copolymers, although no mention is made of the heat resistance of the vulcanizates obtained. The curing agents listed in this patent are sulfur, alone or in combination with aldehyde amines, guanidines, thiazoles, thiuram sulfides, and dithiocarbamates.

<u>Unsaturated Acrylate Copolymers</u>. Vulcanization of these copolymers has received much less attention. Combinations of quinone dioxime, quinone dioxime dibenzoate, red lead, and lead peroxide produced better vulcanizates than did sulfur[12]. However, combinations of mercaptobenzothiazole, sulfur, and Tuads also cured this type of polymer[11].

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APPENDIX F

TABLES AND ILLUSTRATIONS

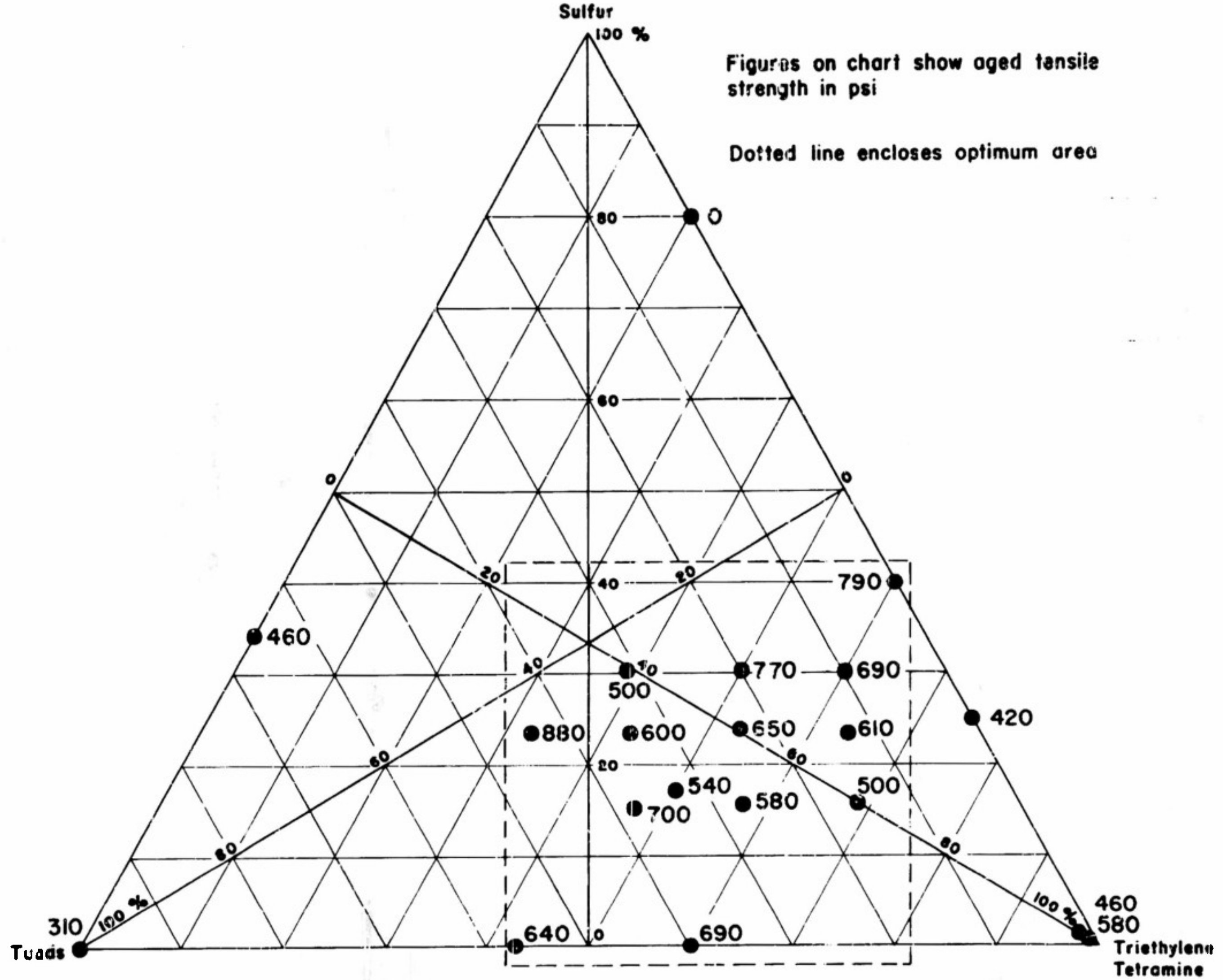

FIGURE 5. EFFECT OF TRIETHYLENE TETRAMINE - SULFUR - TUADS RATIO ON TENSILE STRENGTH AFTER AGING IN ESSO TURBO OIL-15 168 HOURS AT 350 F

0-21766

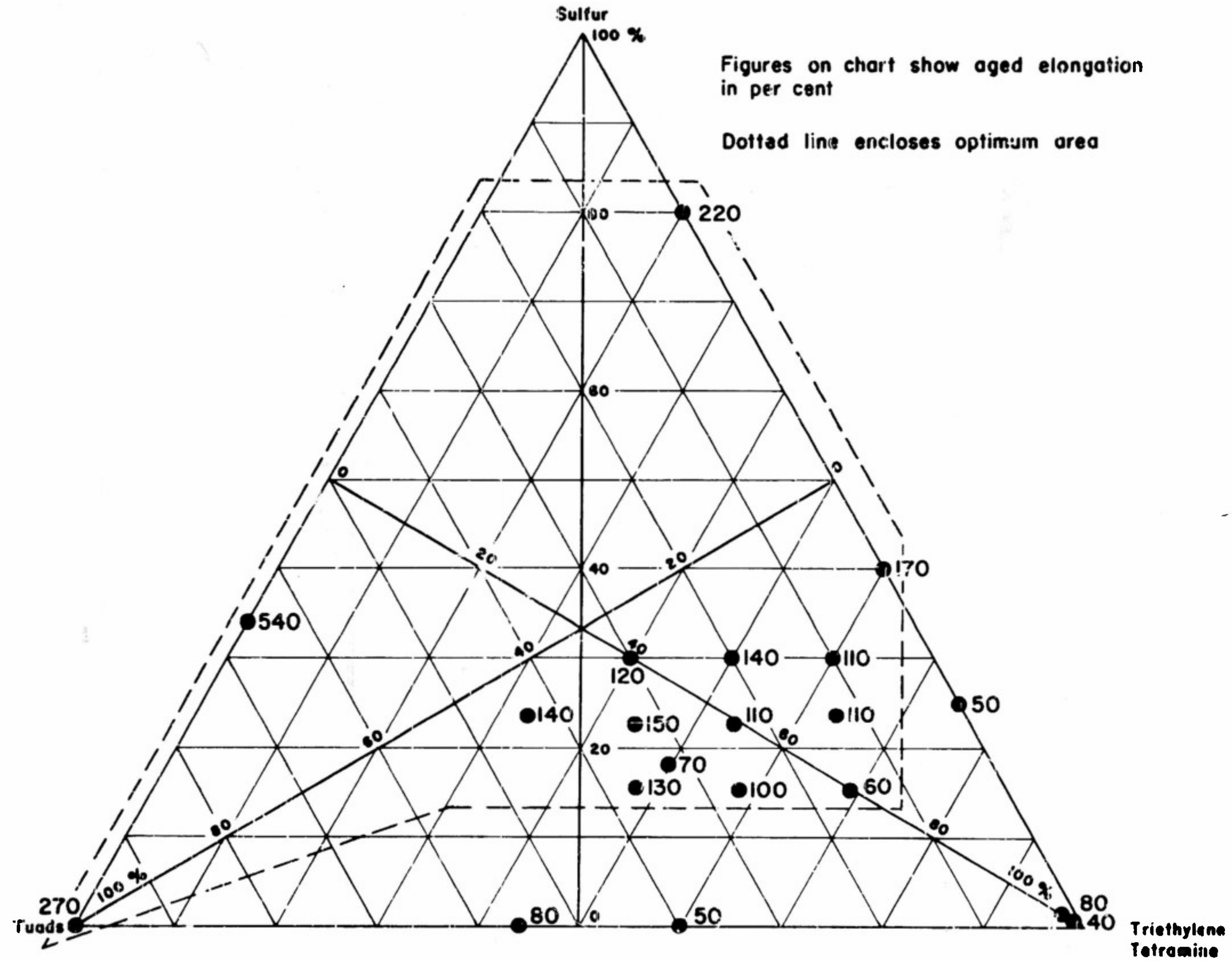

FIGURE 6. EFFECT OF TRIETHYLENE TETRAMINE - SULFUR - TUADS RATIO ON ELONGATION AFTER AGING IN ESSO TURBO OIL-15 168 HOURS AT 350 F

0-21766

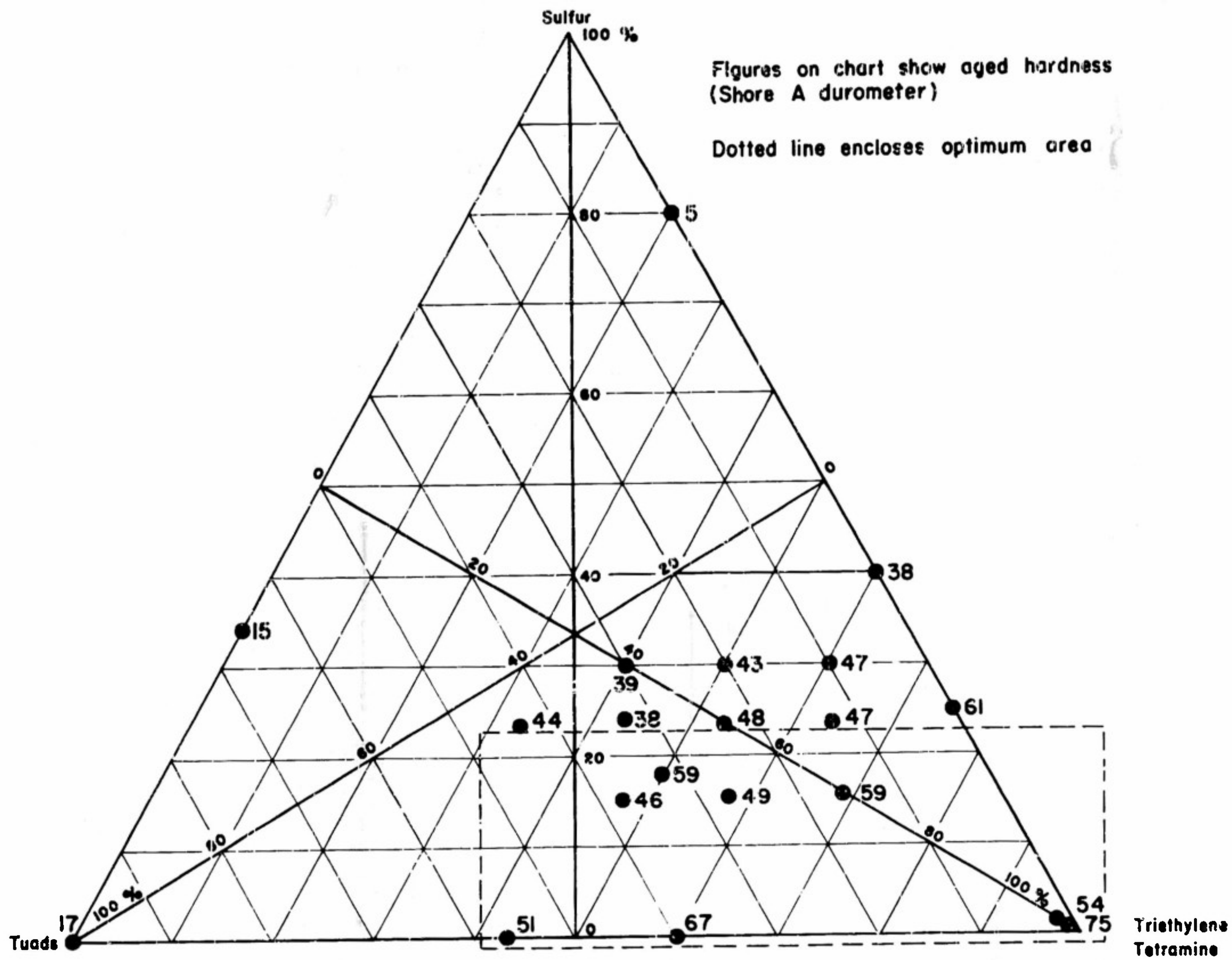

FIGURE 7. EFFECT OF TRIETHYLENE TETRAMINE-SULFUR-TUADS RATIO ON SHORE A HARDNESS AFTER AGING IN ESSO TURBO OIL-15 168 HOURS AT 350 F

0-21767

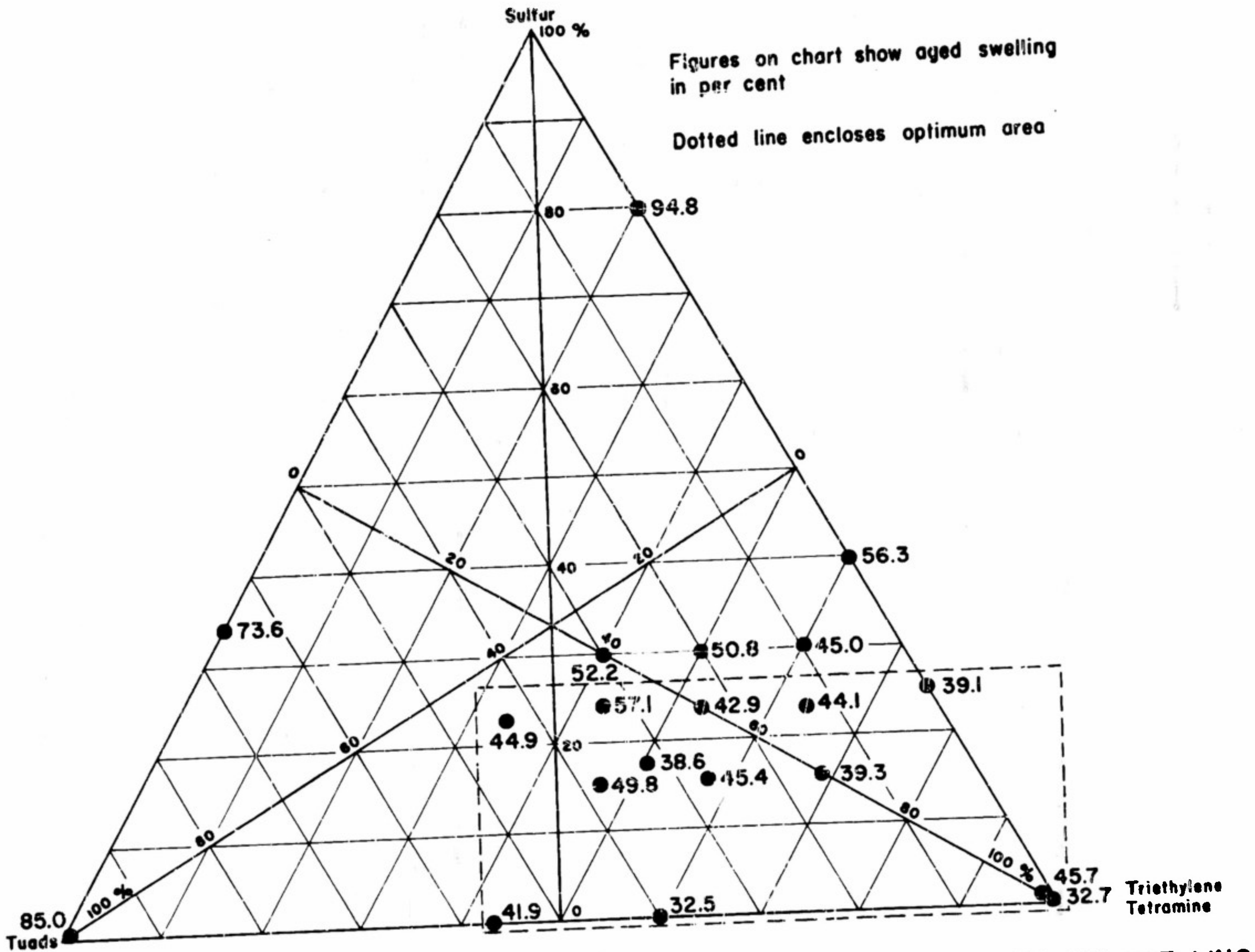

FIGURE 8. EFFECT OF TRIETHYLENE TETRAMINE - SULFUR - TUADS RATIO ON SWELLING AFTER AGING IN ESSO TURBO OIL-15 168 HOURS AT 350 F

0-21768

TABLE 2 RECIPES FOR PRELIMINARY EXPERIMENTAL COMPOUNDING OF NITRILE-TYPE RUBBER

	Recipe No (1)															
	A-1	A-2	A-3	A-4	A-5	A-6	A-7	A-8	A-9	A-10	A-11	A-12	A-13(2)	A-14(3)	B-1	B-2(4)
Hycar 1001	100	100	100	100	100	100	100	100	100	100	100	100	100	100	45	45
Paracril 26NS90	–	–	–	–	–	–	–	–	–	–	–	–	–	–	45	–
Paracril BJ	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	45
Neoprene S	–	–	–	–	–	–	–	–	–	–	–	–	–	–	15	15
Zinc oxide	5	5	5	5	5	5	5	5	5	5	5	5	5	5	4.5	4.5
Magnesium oxide	–	–	–	–	–	–	–	–	–	–	–	–	–	–	0.6	0.6
Stearic acid	1	1	1	1	1	1	1	1	1	1	1	1	1	1	0.9	0.9
Philblack O	40	40	60	80	–	–	–	–	60	60	80	80	–	–	–	–
Philblack A	–	–	–	–	–	–	–	–	–	–	–	–	60	50	–	–
MT Black	–	–	–	–	80	80	140	200	–	–	–	–	–	–	–	–
MPC Black	–	–	–	–	–	–	–	–	–	–	–	–	–	–	60	60
SRF Black	–	–	–	–	–	–	–	–	–	–	–	–	–	–	45	45
Methyl Tuads	3	3	3	3	3	3	3	3	3	3	3	3	3	0.5	–	–
Altax	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1.2	1.2
Tetrone A	–	–	–	–	–	–	–	–	–	–	–	–	–	–	1.2	1.2
Vultac 3	–	–	–	–	–	–	–	–	–	–	–	–	–	5	–	–
Dibutyl Sebacate	–	–	–	–	–	–	–	–	10	20	10	20	–	–	9	9
Plasticizer SC	–	–	–	–	–	–	–	–	–	–	–	–	–	–	9	9
PPO 375	–	–	–	–	–	–	–	–	–	–	–	–	10	10	–	–
AgeRite Powder	–	3	3	3	–	3	3	3	3	3	3	3	3	3	0.9	–
BLE	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	0.9

Note: The sources for compounding ingredients used in this research appear in Appendix D to this report.

(1) Ingredients expressed in parts by weight.

(2) Recipe 5-2 from WADC Technical Note WCRT-53-5 (February, 1953).

(3) Recipe 6-2 from WADC Technical Note WCRT-53-5 (February, 1953).

(4) Recipe No. 40513-B, a U. S. Rubber recipe submitted to WADC.

TABLE 3. PHYSICAL PROPERTIES OF PRELIMINARY EXPERIMENTAL COMPOUNDS

			Original Properties			Properties After Air Aging at 350 F				Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F (2)				Properties After Aging in Esso Turbo Oil-15 at 350 F (2)				
Recipe No. (1)	Cure Time, minutes	Cure Temperature, F	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Aging Time, hours	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Aging Time, hours	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi
A-1	30	298	65	520	4030	51-1/2	92	20	700	11.6	56	210	1030	156	10.6	63	150	830
A-1	60	298	66	490	4180	51-1/2	93	20	670	11.8	57	200	990	156	11.2	61	160	780
A-2	30	298	65	560	3900	51-1/2	93	20	600	9.9	57	220	1140	156	8.7	63	160	830
A-2	60	298	64	530	4100	51-1/2	91	20	670	9.9	57	230	1170	156	8.9	62	160	890
A-3	30	298	76	400	3830	51-1/2	95	30	640	9.2	68	180	1460	156	7.9	73	90	690
A-3	60	298	73	370	4220	51-1/2	95	20	670	9.5	66	160	1280	156	8.6	71	120	1020
A-4	30	298	82	270	3890	51-1/2	97	20	660	8.3	76	90	970	156	7.2	80	70	920
A-4	60	298	82	270	4270	51-1/2	97	20	730	8.0	75	100	990	156	7.6	78	70	830
A-5	30	298	66	630	1920	168	92	0	590	–	–	–	–	128	9.8	60	220	850
A-6	30	298	65	660	1950	168	94	0	580	–	–	–	–	128	8.3	58	230	830
A-7	30	298	78	350	1690	168	98	10	630	–	–	–	–	128	6.3	73	170	1100
A-8	30	298	90	350	1900	168	100	10	750	–	–	–	–	128	5.5	85	80	1040
A-9	30	298	63	480	3220	51-1/2	95	10	680	1.7	68	160	1190	156	0.9	73	110	840
A-9	60	298	64	470	3450	51-1/2	96	10	590	1.7	68	160	1040	156	0.6	73	120	840
A-10	30	298	54	570	2870	51-1/2	96	20	640	-4.6	68	150	940	156	-5.1	75	100	710
A-10	60	298	55	560	3300	51-1/2	96	20	650	-4.2	68	160	1320	156	-5.4	75	90	700

TABLE 3. (Continued)

Recipe No.[1]	Cure Time, minutes	Cure Temperature, F	Original Properties: Hardness, Shore A	Original Properties: Elongation, per cent	Original Properties: Tensile Strength, psi	Properties After Air Aging at 350 F: Aging Time, hours	Properties After Air Aging at 350 F: Hardness, Shore A	Properties After Air Aging at 350 F: Elongation, per cent	Properties After Air Aging at 350 F: Tensile Strength, psi	Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F[2]: Swell, per cent	Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F[2]: Hardness, Shore A	Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F[2]: Elongation, per cent	Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F[2]: Tensile Strength, psi	Properties After Aging in Esso Turbo Oil-15 at 350 F[2]: Aging Time, hours	Properties After Aging in Esso Turbo Oil-15 at 350 F[2]: Swell, per cent	Properties After Aging in Esso Turbo Oil-15 at 350 F[2]: Hardness, Shore A	Properties After Aging in Esso Turbo Oil-15 at 350 F[2]: Elongation, per cent	Properties After Aging in Esso Turbo Oil-15 at 350 F[2]: Tensile Strength, psi
A-11	30	298	71	340	3120	51-1/2	98	20	620	1.5	76	100	1030	156	0.9	81	80	880
A-11	60	298	73	310	3300	51-1/2	99	10	690	1.3	78	100	1070	156	1.1	83	70	850
A-12	30	298	66	410	2840	51-1/2	98	10	600	-4.5	81	100	940	156	-5.0	86	50	650
A-12	60	298	67	380	2970	51-1/2	100	20	760	-4.1	80	110	1170	156	-5.0	85	50	680
A-13	30	298	73	200	2650	168	98	0	600	-0.1	68	110	1200	-	-	-	-	-
A-14	30	298	66	530	2630	168	95	0	580	2.8	61	160	1180	-	-	-	-	-
B-0[3]	40	298	76	280	2410	168	99	50	1017	-	-	-	-	168	25	61	113	1250
B-1	20	298	73	220	1800	168	98	10	680	-	-	-	-	168	20.9	75	30	510
B-1	40	298	72	250	2450	163	97	10	950	-	-	-	-	168	22.6	76	30	420
B-1	60	298	74	260	2540	168	97	10	1000	-	-	-	-	168	21.0	75	40	360
B-2	40	298	76	270	2060	168	100	0	740	24.7	65	60	440	-	-	-	-	-

(1) Recipes given in Table 2.

(2) Aged in circulating-air oven.

(3) Results obtained at United States Rubber Company on their Recipe No. 40513-B.

TABLE 4. THE EFFECT OF LOW LOADINGS OF VARIOUS CARBON BLACKS ON THE AGING PROPERTIES OF HYCAR 1001

		Original Physical Properties			Physical Properties After Air Aging 72 Hours at 350 F			Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F(1)				Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F(1)			
Recipe No.	Carbon Black	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi
A-49	Standard Micronex (MPC)	59	580	2700	94	20	760	11.4	43	220	400	11.3	52	160	310
A-50	Wyex (EPC)	58	520	2250	93	20	700	12.3	48	210	375	11.7	51	140	270
A-51	Continex (SRF)	55	610	2200	88	20	670	16.8	42	250	375	11.2	51	200	310
A-52	Statex B (FF)	56	590	2450	91	20	625	14.4	45	200	280	11.4	55	190	375
A-53	Thermax (MT)	53	570	1250	90	20	700	14.2	38	270	175	12.2	46	200	130
A-54	P-33 (FT)	53	680	1670	91	20	620	13.5	38	300	200	12.3	46	250	250

(1) Aged in circulating-air oven.

Base Recipe:

Ingredients	Parts by Weight
Hycar 1001	100
Zinc oxide	5
Stearic acid	1.5
Carbon black	25
Methyl Tuads	3
Age Rite Powder	3

Cure: 30 minutes at 310 F.

TABLE 5. THE EFFECT OF VARIOUS MAGNESIA-

Recipe No.	Filler	Loading, phr[1]	Original Physical Properties: Hardness, Shore A	Original Physical Properties: Elongation, per cent	Original Physical Properties: Tensile Strength, psi	Physical Properties After Air Aging 72 Hours at 350 F: Hardness, Shore A	Physical Properties After Air Aging 72 Hours at 350 F: Elongation, per cent	Physical Properties After Air Aging 72 Hours at 350 F: Tensile Strength, psi	Physical Properties After Air Aging 72 Hours at 350 F: Crack Resistance
A-23	ELC Magnesia	100	77	410	1810	96	30	860	Cracked
A-130	ELC Magnesia Philblack O	75 25	83	330	2840	98	10	880	Cracked
A-131	ELC Magnesia Philblack O	50 50	86	180	2600	98	10	870	Cracked
A-132	ELC Magnesia Philblack O	25 75	89	130	2610	98	10	810	Cracked
A-129	Philblack O	100	91	90	2330	99	10	800	Cracked

(1) phr = parts per hundred parts of rubber.

(2) Aged in aluminum-block heater.

Base Recipe:

Ingredient	Parts by Weight
Hycar 1001	100
Zinc oxide	5
Stearic acid	1.5
Sulfur	0.5
Methyl Tuads	0.25
ELC magnesia	As indicated
Philblack O	As indicated

Cure: 60 minutes at 298 F.

PHILBLACK O LOADINGS ON HYCAR 1001

Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F(2)					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F(2)				
Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
17.8	77	250	1790	Crazed	16.2	83	130	1240	Cracked
24.6	81	130	1090	Cracked	22.7	88	70	800	Cracked
21.7	83	70	650	Cracked	20.4	88	30	490	Cracked
19.0	86	50	500	Cracked	17.3	88	20	420	Cracked
9.2	92	40	600	Cracked	8.7	95	20	480	Cracked

TABLE 6. THE EFFECT OF ZINC OXIDE AND

			Original Physical Properties			Physical Properties After Air Aging 72 Hours at 350 F			
Recipe No.	Filler (s)	Loading, phr	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
A-19	Zinc oxide	5	42	550	325	87	10	310	Cracked
A-20	Zinc oxide	100	58	580	700	92	20	450	Cracked
A-21	Magnesia (ELC)	5	45	500	340	87	10	450	Cracked
A-22	Zinc oxide Magnesia (ELC)	5 5	46	520	460	85	20	300	Cracked
A-23	Zinc oxide Magnesia (ELC)	5 100	76	360	2125	95	60	1025	Cracked
A-69	Zinc oxide Magnesia (ELC)	5 25	47	670	1160	85	10	570	Cracked
A-70	Zinc oxide Magnesia (ELC)	25 25	49	600	1325	86	10	480	Cracked
A-71	Zinc oxide Magnesia (ELC)	50 25	50	610	1480	87	10	580	Cracked
A-72	Zinc oxide Magnesia (ELC)	50 50	58	620	1580	91	10	570	Cracked
A-68	Zinc oxide Magnesia (ELC)	5 50	57	640	1670	88	40	520	Cracked
A-67	Zinc oxide Magnesia (ELC) Zinc stearate	5 75 2	62	630	2290	91	50	580	Cracked
A-96	Zinc oxide Magnesia (ELC) Zinc stearate	5 100 2	75	615	2340	95	60	600	Cracked
A-97	Zinc oxide Magnesia (ELC) Zinc stearate	5 100 5	73	630	1875	93	70	550	Cracked
A-98[2]	Zinc oxide Magnesia (ELC) Zinc stearate	5 100 2	73	600	2120	94	70	650	Cracked

(1) Compounds A-19 through A-23 were aged in circulating-air oven; compounds A-67 through A-72, and A-96 through A-98, were aged in aluminum-block heater.

(2) Methyl Tuads replaced by Ethyl Tuads in Compound A-98.

Base Recipe:

Ingredients	Parts by Weight
Hycar 1001	100
Stearic acid	1.5
Sulfur	0.5
Methyl Tuads	0.25
Zinc oxide	As indicated
Magnesia (ELC)	As indicated
Zinc stearate	As indicated

Cure: 60 minutes at 298 F.

MAGNESIA ON AGING PROPERTIES OF HYCAR 1001

Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F[1]					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F[1]				
Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
18.4	36	210	50	Cracked	15.2	43	180	30	Cracked
15.2	55	300	500	Cracked	13.9	62	160	530	Cracked
24.4	39	140	60	Cracked	22.2	42	100	20	Cracked
24.0	39	170	90	Cracked	24.4	42	140	10	Cracked
17.0	73	200	2425	Crazed	17.7	73	160	2400	Cracked
24.4	48	180	140	Crazed	23.5	55	20	160	Cracked
23.6	50	200	220	Crazed	19.7	60	80	240	Cracked
19.7	54	240	280	Crazed	20.2	59	130	240	Cracked
19.2	62	240	590	Crazed	15.9	78	30	360	Cracked
20.4	55	250	440	Crazed	20.0	65	140	330	Cracked
20.1	62	280	650	Crazed	17.1	71	150	380	Cracked
20.6	65	290	770	Crazed	17.2	79	100	400	Cracked
27.5	53	230	320	Crazed	23.2	59	150	280	Cracked
18.6	67	280	980	Crazed	16.6	73	140	640	Cracked

TABLE 7. THE EFFECT OF VARIOUS

Recipe No.	Magnesia (ELC), phr	Original Physical Properties: Hardness, Shore A	Original Physical Properties: Elongation, per cent	Original Physical Properties: Tensile Strength, psi	Physical Properties After Air Aging 72 Hours at 350 F: Hardness, Shore A	Physical Properties After Air Aging 72 Hours at 350 F: Elongation, per cent	Physical Properties After Air Aging 72 Hours at 350 F: Tensile Strength, psi	Physical Properties After Air Aging 72 Hours at 350 F: Crack Resistance
A-23	100	77	410	1810	96	30	860	Cracked
A-123	125	91	300	2530	100	30	1040	Cracked
A-124	150	94	290	2790	95	20	1250	Cracked
A-125	175	100	260	3210	100	20	1320	Cracked

(1) Aged in aluminum-block heater.

Base Recipe:

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

Cure: 60 minutes at 298 F.

LOADINGS OF ELC MAGNESIA ON HYCAR 1001

Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F (1)					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F (1)				
Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
17.8	77	250	1790	Crazed	16.2	83	130	1240	Cracked
21.1	90	130	2280	Crazed	18.8	86	80	1440	Cracked
18.0	91	100	2480	Crazed	17.9	94	50	1460	Cracked
16.1	99	120	3130	Crazed	16.7	100	50	1980	Crazed

TABLE 8. THE EFFECT OF NONBLACK FILLERS

			Original Physical Properties			Physical Properties After Air Aging 72 Hours at 350 F			
Recipe No.	Fillers	Loading, phr	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
A-88	Alumina hydrate	50	60	740	2070	93	10	610	Cracked
A-89	Alumina hydrate	100	98	600	2170	100	10	2190	Cracked
A-105	Aluminum oxide	100	65	540	370	92	10	480	Cracked
A-117	Aluminum oxide	150	73	600	390	94	10	660	Cracked
A-92	Aluminum silicate	50	51	600	475	83	10	540	Cracked
A-93	Aluminum silicate	100	60	730	750	87	30	530	Cracked
A-99	Barytes	100	55	570	330	88	10	620	Cracked
A-111	Barytes	150	56	680	310	86	10	650	Cracked
A-106	Calcene TM	100	64	720	1150	80	130	390	Cracked
A-118	Calcene TM	150	74	700	1120	88	120	630	Cracked
A-102	Dixie Clay	100	73	670	2120	88	60	930	Cracked
A-114	Dixie Clay	150	80	390	1630	93	30	910	Cracked
A-84	Hi-Sil C	50	51	740	2490	95	20	750	Cracked
A-86	Hi-Sil C	100	96	650	2450	100	10	2150	Cracked
A-101	Light precipitated chalk	100	66	580	600	84	10	580	Cracked
A-113	Light precipitated chalk	150	78	520	660	91	10	570	Cracked
A-103	Litharge	100	57	720	770	81	70	350	Cracked
A-115	Litharge	150	61	800	1120	85	90	490	Cracked
A-94	Magnesium carbonate	50	48	710	1350	90	10	540	Cracked
A-95	Magnesium carbonate	100	50	760	1160	88	20	460	Cracked
A-104	Mica	100	78	540	920	89	60	830	Cracked
A-116	Mica	150	85	290	940	93	30	1040	Cracked
A-85	Silene EF	50	60	570	1675	92	10	550	Cracked
A-87	Silene EF	100	78	585	2085	99	10	1030	Cracked
A-90	Super multifex	50	50	730	1060	89	10	440	Cracked
A-91	Super multifex	100	61	615	1645	94	30	520	Cracked
A-100	TiO_2	100	56	670	860	90	10	500	Cracked
A-112	TiO_2	150	60	730	980	92	10	570	Cracked

ON THE AGING PROPERTIES OF HYCAR 1001

Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F(1)					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F(1)				
Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
10.6	75	170	580	Cracked	9.3	78	130	480	Cracked
6.0	99	100	1650	Cracked	3.0	99	10	1100	Cracked
12.6	58	200	130	Cracked	11.7	70	10	310	Cracked
12.1	68	190	230	Cracked	10.4	73	40	400	Cracked
13.5	55	200	100	Cracked	11.9	71	10	430	Cracked
11.3	64	250	250	Cracked	10.5	77	20	330	Cracked
13.2	52	130	50	Cracked	12.0	64	10	160	Cracked
10.7	57	190	120	Cracked	9.8	66	10	320	Cracked
14.2	54	330	150	Cracked	12.8	77	10	340	Cracked
14.0	59	380	300	Cracked	13.0	67	130	260	Cracked
10.5	75	100	1040	Cracked	9.9	83	50	530	Cracked
8.6	81	70	1410	Cracked	7.9	88	40	760	Cracked
13.2	80	190	930	Cracked	12.0	85	100	590	Cracked
6.8	98	20	2160	Cracked	6.3	100	10	1730	Cracked
13.5	55	240	120	Cracked	12.3	66	10	220	Cracked
12.0	69	220	330	Cracked	11.1	75	60	430	Cracked
73.5	70	10	250	Cracked	67.3	85	10	530	Cracked
127.7	65	0	30	Cracked	117.6	75	10	240	Cracked
16.0	65	70	200	Cracked	16.5	80	20	580	Cracked
22.9	45	300	120	Cracked	22.4	54	100	120	Cracked
10.7	80	80	500	Cracked	11.1	80	70	540	Cracked
9.3	84	60	880	Cracked	8.4	89	30	870	Cracked
12.8	72	180	580	Cracked	12.8	72	130	430	Cracked
11.6	88	130	1030	Cracked	10.5	94	60	670	Cracked
17.0	52	240	130	Cracked	14.9	70	10	410	Cracked
15.9	60	310	420	Cracked	15.2	68	140	270	Cracked
14.5	54	240	170	Cracked	12.8	63	40	230	Cracked
12.6	60	380	320	Cracked	12.1	70	130	310	Cracked

TABLE 8.

Recipe No.	Fillers	Loading, phr	Original Physical Properties: Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Physical Aging in Penala: Swell, per cent	Hardness, Shore A
A-216	Aerosil	20	58	710	1680	14.9	68
A-233	Aerosil	50	83	640	3010	12.7	90
A-217	Aerosil	100	100	340	4230	10.5	100
A-234	LM-3 Coated Hi-Sil C	50	81	730	3050	12.9	89
A-235	LM-3 Coated Hi-Sil C	100	100	490	2430	8.8	100
A-236	Gilsonite	50	70	630	2160	32.9	62
A-237	Gilsonite	100	76	590	1670	45.1	55

(1) Aged in aluminum-block heater.

Base Recipe:

Ingredients	Parts by Weight
Hycar 1001	100
Zinc oxide	5
Stearic acid	1.5
Sulfur	0.5
Methyl Tuads	0.25
Filler	As indicated

Cure: 60 minutes at 298 F.

(Continued)

Properties After Turbo Oil-15 72 Hours at 350 F			Physical Properties After Aging in Penola Turbo Oil-15 168 Hours at 350 F				
Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
190	330	Cracked	13.9	77	20	370	Cracked
160	900	Crazed	11.0	94	80	630	Cracked
30	1420	Cracked	9.7	99	20	1330	Cracked
190	1200	Crazed	11.9	92	120	840	Cracked
40	1430	Cracked	7.8	100	20	1460	Cracked
290	740	Crazed	35.3	71	130	390	Cracked
290	480	Crazed	50.7	65	90	290	Cracked

TABLE 9. THE EFFECT OF THIURAM POLYSULFIDE CURING SYSTEMS ON AGING PROPERTIES OF HYCAR 1001

			Original Physical Properties			Physical Properties After Air Aging 72 Hours at 350 F (1)			Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F (1)				Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F (1)			
Recipe No.	Curing Agent(s)	Loading, phr	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi
A-27	Monex	1	57	1100	810	92	20	700	17.2	52	220	625	17.9	57	150	500
A-28	Monex	3	58	1040	660	93	20	525	11.3	50	230	825	13.7	60	160	730
A-29	Pentex	1	58	900	710	92	20	480	18.3	48	240	780	14.2	59	160	725
A-30	Pentex	3	57	960	970	90	20	550	16.4	46	260	730	12.4	58	160	710
A-31	Tetrone	1	58	720	2540	94	20	500	14.3	52	160	700	13.6	64	140	680
A-32	Tetrone	3	71	275	3125	95	20	600	12.3	60	130	910	11.6	71	100	775
A-33	Tuex	1	60	660	2400	92	20	600	14.7	48	190	650	15.3	58	140	630
A-34	Tuex	3	62	490	4350	95	10	480	6.5	52	200	1075	13.1	61	130	550
A-35	Tuex Sulfur	3 0.2	65	250	2150	94	20	625	10.0	56	140	780	12.0	61	120	700
A-15	Methyl Tuads Sulfur	0.25 0.5	63	400	2600	94	20	630	14.5	59	160	775	13.6	61	120	630
A-16	Methyl Tuads Sulfur	0.25 0.25	59	540	2250	93	20	600	14.5	56	210	830	13.4	62	130	670
A-17	Methyl Tuads Vultac No. 2	0.25 2.0	58	620	3175	94	20	625	17.1	56	180	920	16.0	55	140	670
A-18	Methyl Tuads Vultac No. 2	0.25 1.0	59	730	2250	93	20	575	15.4	54	[illegible]10	800	14.4	57	150	650

(1) Aged in circulating-air oven.

Base Recipe:

Ingredients	Parts by Weight
Hycar 1001	100
Zinc oxide	5
Stearic acid	1.5
Philblack 0	40
Curing agent(s)	Variable

Cure: Batches A-15 through A-18 – 60 minutes at 298 F.
Batches A-27 through A-35 – 30 minutes at 310 F.

TABLE 10. A STUDY OF LOW-SULFUR AND NONSULFUR

Recipe No.	Curing-System Ingredient	Loading, phr	Original Physical Properties: Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Physical Properties After Air Aging 72 Hours at 350 F: Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
A-23	Methyl Tuads Sulfur	0.25 0.5	77	630	1875	96	30	860	Cracked
A-126	Methyl Tuads(2)	4.0	80	580	3240	97	60	880	Cracked
A-127	Methyl Tuads Vandex	2.0 1.0	80	560	3130	96	50	1060	Cracked
A-128	Methyl Tuads Telloy	2.0 1.0	79	580	2610	97	60	930	Cracked
A-133	Methyl Tuads Telloy	3.0 0.5	80	710	3330	97	30	990	Cracked
A-134	Methyl Tuads Telloy	3.0 1.0	82	690	3090	98	20	940	Cracked
A-135	Methyl Tuads Vandex	3.0 0.3	83	630	3100	100	50	980	Cracked
A-136	Methyl Tuads Vandex	3.0 0.6	82	610	2800	99	40	930	Cracked
A-137	Litharge Sulfur Altax	1.5 0.5 1.0	80	530	2130	97	70	880	Cracked
A-110	Litharge	5.0	75	870	1960	96	110	750	Cracked
A-168	Altax Manganese dioxide G.M.F.	4.0 10.0 2.0	82	590	3420	96	70	930	Cracked
A-169	Methyl Tuads Santocure	3.0 2.0	81	600	1970	96	60	930	Cracked
A-186	2-MT	2.0	75	880	1430	96	90	980	Cracked
A-187	Antox Methyl Tuads	1.0 3.0	82	700	2870	99	70	850	Cracked
A-188	Ethyl Tellurac	3.0	82	660	2370	96	80	930	Cracked
A-200	Cadmium oxide	5.0	85	540	1880	98	50	900	Cracked
A-201	Calcium oxide	5.0	81	570	1890	94	70	950	Cracked
A-202	Litharge Dinitrobenzene	10.0 4.0	84	640	2450	99	30	930	Cracked

(1) Aged in aluminum-block heater.

(2) Curing systems in A-126 through A-136 recommended by R. T. Vanderbilt Company for heat-resistant stocks.

Base Recipe:

Ingredients	Parts by Weight
Hycar 1001	100
Magnesia	100
Zinc oxide (except in A-168)	5
Stearic acid	1.5
Curing agents	As shown

Cure: 60 minutes at 298 F.

CURING SYSTEMS IN A HYCAR 1001 RECIPE

Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F (1)					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F (1)				
Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
17.8	77	250	1790	Crazed	16.2	83	130	1240	Cracked
23.6	74	310	1180	Crazed	22.6	84	170	940	Cracked
25.9	79	260	1490	Cracked	23.9	83	130	1100	Cracked
29.2	77	210	1270	Cracked	24.1	84	130	830	Cracked
26.2	73	370	1480	Cracked	23.9	85	170	910	Cracked
24.0	75	300	1250	Crazed	22.6	85	160	900	Cracked
22.0	78	390	1560	Crazed	22.0	85	200	1060	Cracked
20.9	80	290	1280	Crazed	20.1	87	140	940	Cracked
24.5	75	250	1510	Crazed	23.7	83	130	1010	Cracked
30.0	61	310	770	Crazed	29.9	88	30	460	Cracked
24.7	72	390	1220	Crazed	24.4	82	170	700	Cracked
21.1	78	190	1260	Crazed	18.8	86	100	950	Cracked
26.6	75	420	1490	Crazed	25.5	89	150	930	Cracked
25.3	75	340	1210	Cracked	24.2	86	160	870	Cracked
21.5	81	260	1380	Cracked	20.8	89	140	910	Cracked
24.2	78	240	1270	Crazed	26.0	85	100	660	Cracked
24.3	76	310	1530	Crazed	26.1	82	120	900	Cracked
22.4	90	70	1330	Crazed	20.9	97	30	880	Cracked

TABLE 11. THE PHYSICAL PROPERTIES OF PEROXIDE-CURED HYCAR 1012 COMPOUNDS AFTER AGING IN ESSO TURBO OIL-15

			Original Physical Properties (1)			Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F (2)					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F (2)				
Recipe No.	Curing Agent	Loading, phr	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
520-14-23-1	Methyl Tuads	3.5	–	425	2650	41.6	49	60	130	Cracked	40.0	90	10	610	Cracked
520-14-23-2	t-Butyl HP	5	–	250	2330	35.8	76	10	210	Cracked	34.4	92	10	640	Cracked
520-14-23-7	DIP	2.5	–	510	1430	48.3	57	30	70	Cracked	44.8	89	10	420	Cracked
520-14-23-8	DIP	5	–	335	2050	41.1	68	10	230	Cracked	40.1	90	10	470	Cracked
520-14-23-10	PMH	2.5	–	525	1790	45.4	56	30	90	Cracked	46.3	89	10	430	Cracked
520-14-23-12	CHP	2.5	–	320	2200	41.1	69	20	200	Cracked	39.9	90	10	560	Cracked
520-14-25-3	t-Butyl HP	3	–	390	2270	38.3	69	10	160	Cracked	36.9	91	10	430	Cracked
520-14-25-4	t-Butyl HP	5	–	280	2460	33.7	72	20	210	Cracked	32.2	90	10	580	Cracked
520-14-25-5	PMH	5.6	–	360	2450	35.6	75	20	200	Cracked	34.5	79	10	180	Cracked
520-14-25-6	CHP	2	–	350	1970	38.2	71	10	150	Cracked	37.1	90	10	180	Cracked
520-14-25-7	CHP	3.4	–	230	1850	33.8	59	70	310	Cracked	31.8	90	10	480	Cracked
520-14-25-8	Methyl Tuads	3.5	–	450	2880	42.1	62	20	110	Cracked	41.0	89	10	570	Cracked
520-14-25-9	CHP	2.5	–	310	2350	40.7	67	20	130	Cracked	37.9	90	10	440	Cracked

(1) Tensile slabs and original physical properties supplied by the B. F. Goodrich Chemical Company.
(2) Aged in aluminum-block heater.
t-Butyl HP = tert-butyl hydroperoxide (approximately 60 per cent purity).
DIP = diisopropylbenzene monohydroperoxide (50.4 per cent purity).
PMH = paramenthane hydroperoxide (47.9 per cent purity).
CHP = cumene hydroperoxide (70.4 per cent purity).

Base Recipe:

Ingredients	Parts by Weight
Hycar 1012	100
Zinc oxide	5
Philblack A	40
Stearic acid	1
Curatives	Variable

Cure: 30 minutes at 310 F.

TABLE 12. THE EFFECT OF COMMERCIAL AND EXPERIMENTAL ANTIOXIDANTS ON THE AGING PROPERTIES OF HYCAR 1001

Recipe No.	Antioxidant	Original Physical Properties			Physical Properties After Air Aging 72 Hours at 350 F			Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F(1)				Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F(1)			
		Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi
A-34	None (Control)	62	490	4350	95	10	480	6.5	52	200	1075	13.1	61	130	650
A-36	AgeRite Alba	53	590	4290	94	10	650	12.2	49	180	665	17.1	62	130	650
A-37	AgeRite Hipar	58	590	4125	93	20	700	11.6	50	160	590	11.5	58	120	610
A-38	AgeRite Stalite	60	540	4200	95	10	610	10.2	50	170	710	10.6	59	130	670
A-39	Aminox	60	590	4400	94	20	700	12.7	49	190	650	10.7	60	130	650
A-40	Parazone(2)	57	530	4000	94	20	700	11.8	49	130	680	11.4	58	140	650
A-41	Santovar A	57	650	3275	93	20	575	10.8	55	60	310	7.1	85	20	390
A-42	Santowhite	58	560	4050	94	20	550	12.3	50	150	680	10.4	62	120	620
A-43	Wingstay S	60	560	3975	93	20	575	10.8	51	170	760	10.9	61	120	575
A-44	Flectol H(2)	58	570	4000	94	20	630	11.6	47	210	875	12.2	58	150	720
A-45	Neozone A	59	540	3875	95	20	575	11.0	55	190	840	10.8	60	120	550
A-46	PDA-10(2)	62	540	4050	94	30	625	11.2	53	210	1025	11.9	61	130	620
A-47	Santoflex AW	59	630	4000	93	20	575	11.0	53	200	990	11.6	60	130	650
A-48	2, 5-Dicyclohexyl hydroquinone(3)	63	610	3900	94	10	625	9.9	68	130	350	7.7	80	85	350

TABLE 12. (Continued)

Recipe No.	Antioxidant	Original Physical Properties: Hardness, Shore A	Original Physical Properties: Elongation, per cent	Original Physical Properties: Tensile, Strength, psi	Physical Properties After Air Aging 72 Hours at 350 F: Hardness, Shore A	Air Aging 72 Hours at 350 F: Elongation, per cent	Air Aging 72 Hours at 350 F: Tensile, Strength, psi	Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F(1): Swell, per cent	Esso Turbo Oil-15 72 Hours: Hardness, Shore A	Esso Turbo Oil-15 72 Hours: Elongation, per cent	Esso Turbo Oil-15 72 Hours: Tensile, Strength, psi	Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F(1): Swell, per cent	Esso Turbo Oil-15 168 Hours: Hardness, Shore A	Esso Turbo Oil-15 168 Hours: Elongation, per cent	Esso Turbo Oil-15 168 Hours: Tensile, Strength, psi
A-55	t-butyl catechol(3)	65	600	3625	96	20	640	14.4	49	210	625	12.5	56	140	525
A-56	Hydroquinone(2)	64	640	3575	95	20	550	14.4	58	180	350	11.0	62	150	550
A-57	Versene (Regular)(3)	67	470	3950	94	20	640	14.8	54	180	740	9.8	60	140	600
A-58	Disodium Lead Versenate(4)	67	450	3850	94	20	600	15.7	52	160	500	12.5	62	140	600
A-59	Pyrogallol(3)	66	720	2500	94	20	575	13.2	54	240	930	11.6	56	150	780
A-60	Aniline(3)	64	530	3300	95	20	650	13.9	51	180	600	11.0	60	150	550
A-61	Nitrophenol(3)	65	450	3325	96	20	580	11.2	61	120	600	9.0	68	80	575
A-62	p-Benzy amino-phenol(3)	63	550	2980	95	20	570	14.0	51	210	590	12.5	55	160	575
A-63	Resorcinol(3)	64	560	4120	95	20	570	12.8	54	160	500	11.4	62	130	575
A-64	2-Hydroxyquino-line(3)	64	490	4010	94	20	570	12.0	57	150	525	11.8	59	130	550

(1) Aged in circulating-air oven.
(2) Good crack resistance.
(3) Short-stopping agent.
(4) Sequestering agent.

Base Recipe:

Ingredients	Parts by Weight
Hycar 1001	100
Zinc oxide	5
Stearic acid	1.5
Philblack O	40
Methyl Tuads	3
"Antioxidant"	3

Cure: 30 minutes at 310 F.

TABLE 13. THE EFFECT OF PHENOLIC-TYPE MATERIALS

			Original Physical Properties			Physical Properties After Air Aging 72 Hours at 350 F			
Recipe No.	Antioxidant	Loading, phr	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
A-23	None	--	77	410	1810	96	30	860	Cracked
A-151	p-Phenyl phenol	3	79	530	2340	96	60	830	Cracked
A-152	p-Tert-amyl phenol	3	78	540	2210	97	60	670	Cracked
A-153	o-Amyl phenol	3	78	440	1960	95	60	830	Cracked
A-154	Nonyl phenol	3	78	540	2280	96	60	800	Cracked
A-155	Phenol	3	77	570	2120	96	60	830	Cracked
A-156	Triphenyl phosphite	3	80	570	2180	97	50	830	Cracked
A-183	Phloroglucinol	3	74	650	1850	96	70	830	Cracked
A-184	Catechol	3	73	690	2630	95	90	1050	Cracked
A-185	Di-tert-butyl-para-cresol	3	76	620	1670	93	80	950	Cracked
A-247	Parazone AgeRite Resin D	3 3	69	620	2350	--	--	--	--
A-248	Flectol H AgeRite Resin D	3 3	71	640	2540	--	--	--	--

(1) Aged in aluminum-block heater.

Base Recipe:

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

Cure: 60 minutes at 298 F.

ON THE AGING PROPERTIES OF HYCAR 1001

Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F[1]					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F[1]				
Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
17.8	77	250	1790	Crazed	16.2	83	130	1240	Cracked
25.7	76	230	1530	Crazed	22.6	85	140	930	Cracked
25.4	77	270	1680	Crazed	24.0	85	150	1120	Cracked
23.9	81	220	1530	Crazed	22.7	85	140	1060	Cracked
23.9	80	210	1580	Crazed	22.2	86	140	1180	Cracked
24.0	80	250	1750	Crazed	23.7	85	150	1190	Cracked
25.6	78	250	1410	Crazed	24.1	86	140	990	Cracked
26.2	72	280	1570	Crazed	25.5	81	130	880	Cracked
29.5	72	320	1400	Cracked	28.1	85	120	730	Cracked
25.4	74	260	1280	Crazed	23.1	87	90	740	Cracked
14.5	68	390	1050	Crazed	13.2	73	180	630	Cracked
14.3	66	380	1090	Crazed	13.8	72	210	770	Cracked

TABLE 14. THE EFFECT OF VINYL STABILIZERS

Recipe No.	Stabilizers	Loading, phr	Original Physical Properties: Hardness, Shore A	Original Physical Properties: Elongation, per cent	Original Physical Properties: Tensile Strength, psi	Physical Properties After Air Aging 72 Hours at 350 F[1]: Hardness, Shore A	Physical Properties After Air Aging 72 Hours at 350 F[1]: Elongation, per cent	Physical Properties After Air Aging 72 Hours at 350 F[1]: Tensile Strength, psi	Crack Resistance
A-23	None	–	81	390	1820	96	60	970	Cracked
A-144	Ferro 1820[2] Ferro 903[3]	3.5 1.5	74	700	1880	93	100	820	Cracked
A-145	Dibutyl tin maleate	5	66	660	2750	96	80	820	Cracked
A-146	Tribasic lead maleate	5	73	650	2820	92	80	870	Cracked
A-147	Stabilizer A-5[4]	5	70	550	1930	97	60	780	Cracked
A-148	Dyphos[5]	5	73	420	1990	93	60	770	Cracked
A-149	RN-34[6]	5	72	500	2270	95	50	770	Cracked
A-150	Mark XI[7] Mark XX[8]	3.5 1.5	65	780	1730	95	90	710	Cracked

(1) Aged in aluminum-block heater.

(2) Barium stabilizer – Ferro Chemical Corporation.

(3) Cadium stabilizer – Ferro Chemical Corporation.

(4) Epoxy type – Carbide and Carbon Chemicals Company.

(5) Dibasic lead phosphite – National Lead Company.

(6) Resinous epoxide – Shell Chemical Corporation.

(7) Cadmium-barium combination – Argus Chemical Company.

(8) Epoxy type – Argus Chemical Company

ON THE AGING PROPERTIES OF HYCAR 1001

Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F(1)					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F(1)				
Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
18.0	79	210	1960	Crazed	17.6	82	140	1330	Cracked
27.9	71	320	1160	Crazed	26.7	80	190	810	Cracked
20.9	64	430	1300	Crazed	24.5	72	320	1120	Cracked
29.1	73	260	1340	Cracked	27.0	83	120	950	Cracked
26.4	77	250	1630	Cracked	24.9	75	240	1640	Cracked
26.2	73	230	1570	Crazed	24.4	83	130	1120	Cracked
24.3	77	220	1800	Cracked	26.0	81	120	1200	Cracked
27.8	68	420	980	Cracked	26.2	78	210	730	Cracked

Base Recipe:

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

Cure: 60 minutes at 298 F.

TABLE 15. THE EFFECT OF LARGE AMOUNTS OF

Recipe No.	Antioxidant	Loading, phr	Original Physical Properties: Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Physical Properties After Air Aging 72 Hours at 350 F: Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
A-23	None	–	77	630	1875	96	30	860	Cracked
A-181	Pyrogallol	3	74	630	2450	94	70	860	Cracked
A-138	Pyrogallol	10	90	510	3090	100	20	1260	Cracked
A-139	Pyrogallol	15	95	470	2840	100	20	1480	Cracked
A-180	o-Cresol	3	75	700	1660	94	90	970	Cracked
A-140	o-Cresol	10	72	750	1530	95	70	820	Cracked
A-141	o-Cresol	15	68	750	1680	96	70	830	Cracked
A-182	AgeRite Resin D	3	76	670	1740	95	70	840	Cracked
A-142	AgeRite Resin D	10	77	630	1860	94	70	950	Cracked
A-143	AgeRite Resin D	15	76	720	1460	93	100	920	Cracked

(1) Aged in aluminum-block heater.

Base Recipe:

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

Cure: 60 minutes at 298 F

ANTIOXIDANT ON THE AGING PROPERTIES OF HYCAR 1001

Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F (1)					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F (1)				
Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
17.8	77	250	1790	Crazed	16.2	83	130	1240	Cracked
22.3	74	220	1710	Crazed	20.2	86	100	880	Cracked
14.8	91	110	1890	Crazed	13.1	96	60	1220	Cracked
13.9	94	100	2160	Crazed	12.6	98	40	1410	Cracked
25.4	76	310	1430	Crazed	23.2	82	150	910	Cracked
22.6	75	340	1560	Crazed	22.4	85	170	980	Cracked
18.1	76	340	1230	Crazed	19.3	84	170	830	Cracked
25.1	75	290	1560	Crazed	22.3	85	150	860	Cracked
18.4	73	320	1750	Crazed	16.9	81	190	1340	Cracked
16.5	71	400	1520	Crazed	14.9	79	210	1180	Cracked

TABLE 16. THE EFFECT OF ADDING ANTIOXIDANT TO A HYCAR 1001 COMPOUND AND TO THE AGING OIL

				Original Physical Properties			Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F(2)					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F(2)				
Recipe No.(1)	Antioxidant in Rubber, phr	Loading, phr	Antioxidant in Oil, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
A-23	None	–	0	77	410	1810	17.8	77	250	1790	Crazed	16.2	83	130	1240	Cracked
A-180	o-Cresol	3	0	75	700	1660	25.4	76	310	1430	Crazed	23.2	82	150	910	Cracked
	"	3	0.15	–	–	–	23.6	78	290	1400	Cracked	22.0	83	160	970	Cracked
	"	3	1	–	–	–	23.8	75	320	1450	Crazed	21.9	87	120	830	Cracked
A-181	Pyrogallol	3	0	74	630	2450	22.3	74	220	1710	Crazed	20.2	86	100	880	Cracked
	"	3	0.15	–	–	–	14.0	82	150	1410	Crazed	13.7	88	80	950	Cracked
	"	3	1	–	–	–	17.9	93	80	1830	Cracked	17.4	98	40	1640	Cracked
A-171	Parazone	3	0	80	540	1910	23.6	77	220	1310	Crazed	21.4	83	110	930	Cracked
	"	3	0.15	–	–	–	23.0	78	230	1380	Crazed	21.8	85	120	900	Cracked
	"	3	1	–	–	–	25.9	76	240	1370	Crazed	22.9	84	120	870	Cracked
A-172	Flectol H	3	0	80	550	1980	20.2	78	210	1510	Crazed	18.5	84	130	1020	Cracked
	"	3	0.15	–	–	–	20.2	77	250	1440	Crazed	20.8	83	130	1130	Cracked
	"	3	1	–	–	–	20.3	71	210	1150	Crazed	24.4	79	160	1190	Crazed
A-159	o-Cresol	20.1	0	66	670	2020	15.1	76	310	1010	Crazed	12.5	83	170	820	Cracked
	"	20.1	0.15	–	–	–	12.5	74	330	1140	Crazed	13.5	87	150	750	Cracked
	"	20.1	1	–	–	–	15.0	76	350	1260	Crazed	13.5	87	150	740	Cracked
A-160	Pyrogallol	20.1	0	98	350	2820	10.4	99	60	1930	Cracked	9.6	100	30	1610	Cracked
	"	20.1	0.15	–	–	–	7.8	100	40	1740	Cracked	8.1	99	30	1700	Cracked
	"	20.1	1	–	–	–	9.4	100	30	2270	Cracked	10.3	100	30	2250	Cracked
A-161	Parazone	20.1	0	69	610	2180	13.3	70	290	770	Crazed	11.8	80	130	590	Cracked
	"	20.1	0.15	–	–	–	12.0	71	310	830	Crazed	11.5	88	80	500	Cracked
	"	20.1	1	–	–	–	14.3	66	330	940	Crazed	17.0	84	80	480	Cracked
A-162	Flectol H	20.1	0	79	550	1990	13.6	73	310	1350	Crazed	12.3	79	210	1180	Cracked
	"	20.1	0.15	–	–	–	13.2	74	310	1430	Crazed	13.2	80	180	1110	Cracked
	"	20.1	1	–	–	–	11.8	71	360	1440	Crazed	16.8	76	240	1390	Crazed

(1) Base recipe same as for Table 14.
(2) Aged in aluminum-block heater.

TABLE 17. THE EFFECT OF ADDING ANTIOXIDANT ONLY TO THE AGING OIL ON THE AGING PROPERTIES OF HYCAR 1001

Recipe No.	Antioxidant in Oil		Original Physical Properties			Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F(1)				
	Antioxidant	Per Cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
A-23-e	None	–	80	460	1960	14.6	85	150	1220	Cracked
A-23-e	Phenothiazine	0.15				21.5	83	120	1130	Cracked
A-23-e		1				24.5	83	150	1200	Cracked
A-23-e	Parapoid 10C	0.15				23.7	88	110	910	Cracked
A-23-e		1				24.0	85	100	990	Cracked
A-23-e	Flectol H	0.15				22.1	89	90	840	Cracked
A-23-e		1				22.0	81	150	1350	Cracked

(1) Aged in aluminum-block heater.

Base Recipe:

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

Cure: 60 minutes at 298 F.

TABLE 18. THE EFFECT OF STEARIC ACID LOADING ON THE AGING PROPERTIES OF HYCAR 1001

		Original Physical Properties			Physical Properties After Air Aging 72 Hours at 350 F			Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F (1)				Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F (1)			
Recipe No.	Stearic Acid, phr	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi
A-24	0	45	330	375	86	20	590	17.6	41	190	150	13.9	44	170	80
A-25	0.5	42	460	350	87	10	525	17.9	38	220	80	12.3	40	180	70
A-19	1.5	42	550	325	87	10	310	18.4	36	210	50	15.2	43	180	30
A-26	5.0	42	630	450	88	20	575	18.2	33	230	60	19.6	37	110	0

(1) Aged in circulating-air oven.

Base Recipe:

Ingredients	Parts by Weight
Hycar 1001	100
Zinc oxide	5
Methyl Tuads	0.25
Sulfur	0.5
Stearic acid	Variable

Cure: 60 minutes at 298 F.

TABLE 19. THE EFFECT OF ZINC OXIDE, STEARIC ACID, AND

Recipe No.	Curing Aids	Loading, phr	Original Physical Properties: Hardness, Shore A	Original Physical Properties: Elongation, per cent	Original Physical Properties: Tensile Strength, psi	Physical Properties After Air Aging 72 Hours at 350 F: Hardness, Shore A	Physical Properties After Air Aging 72 Hours at 350 F: Elongation, per cent	Physical Properties After Air Aging 72 Hours at 350 F: Tensile Strength, psi	Physical Properties After Air Aging 72 Hours at 350 F: Crack Resistance
A-23	Zinc oxide	5.0	77	410	1810	96	30	860	Cracked
	Stearic acid	1.5							
A-204	Zinc oxide	3.0	85	520	2000	98	60	910	Cracked
	Stearic acid	1.5							
A-205	Zinc oxide	1.5	83	490	2660	96	70	1040	Cracked
	Stearic acid	1.5							
A-164	Stearic acid	1.5	82	520	2410	97	70	940	Cracked
A-206	Zinc oxide	5.0	84	480	2600	97	70	1010	Cracked
	Stearic acid	1.0							
A-207	Zinc oxide	5.0	84	450	2800	97	60	1040	Cracked
	Stearic acid	0.5							
A-163	Zinc oxide	5.0	85	400	2420	96	50	850	Cracked
A-208	Zinc oxide	2.5	85	490	2880	97	60	830	Cracked
	Stearic acid	0.75							
A-165	None	–	85	380	2450	97	40	830	Cracked
A-97	Zinc oxide	5.0	73	630	1875	93	70	550	Cracked
	Stearic acid	1.5							
	Zinc stearate	5.0							
A-167	Zinc oxide	5.0	79	570	2290	94	90	740	Cracked
	Stearic acid	1.5							
	Magnesium stearate	5.0							

(1) Aged in aluminum-block heater.

Base Recipe:

Ingredients	Parts by Weight
Hycar 1001	100
Magnesia	100
Sulfur	0.5
Methyl Tuads	0.25
Curing aids	As indicated

Cure: 60 minutes at 298 F.

ZINC STEARATE ON THE AGING PROPERTIES OF HYCAR 1001

Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F (1)					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F (1)				
Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
17.8	77	250	1790	Crazed	16.2	83	130	1240	Cracked
24.9	83	210	1660	Crazed	23.9	88	120	1070	Cracked
22.5	83	210	1760	Crazed	21.5	88	90	880	Cracked
22.0	82	220	1710	Crazed	19.1	85	130	1010	Cracked
23.6	82	210	1740	Crazed	22.6	84	120	1230	Cracked
22.0	83	190	1930	Crazed	20.6	87	100	1110	Cracked
21.3	83	160	1630	Crazed	19.4	85	90	950	Cracked
25.1	84	220	1790	Crazed	20.6	88	130	1110	Cracked
20.9	84	40	520	Crazed	19.4	86	70	780	Cracked
27.5	53	230	320	Crazed	23.2	59	150	280	Cracked
25.6	71	220	790	Crazed	25.3	77	130	530	Cracked

TABLE 20. THE EFFECT OF PROCESSING AIDS ON THE AGING PROPERTIES OF HYCAR 1001

			Original Physical Properties			Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F (1)					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F (1)				
Recipe No.	Processing Aid	Loading, phr	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
A-23	Stearic acid	1.5	77	410	1810	17.8	77	250	1790	Crazed	16.2	83	130	1240	Cracked
A-214	Acrawax CT	1.5	73	560	2900	21 2	74	290	1570	Cracked	18.8	81	180	980	Cracked
A-215	Talc	1.5	74	550	3010	23.0	77	270	1490	Cracked	21.4	82	140	820	Cracked

(1) Aged in aluminum-block heater.

Base Recipe:

Ingredients	Parts by Weight
Hycar 1001	100
Magnesia	100
Zinc oxide	5
Sulfur	0.5
Methyl Tuads	0.25
Processing aids	As indicated

Cure: 60 minutes at 298 F

TABLE 21. THE EFFECT OF NONEXTRACTIBLE PLASTICIZERS ON THE AGING PROPERTIES OF HYCAR 1001

Recipe No.	Additive	Loading, phr	Original Physical Properties			Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F(1)					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F(1)				
			Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
A-23	None	–	77	630	1875	17.8	77	250	1790	Crazed	16.2	83	130	1240	Cracked
A-243	ODN Plasticizer	3	73	570	2460	16.8	70	330	1120	Crazed	14.7	76	190	800	Cracked
A-221	ODN Plasticizer	10	59	750	2270	8.5	68	400	1060	Crazed	7.5	77	240	775	Cracked
A-222	ODN Plasticizer	5	65	690	2390	13.0	69	350	1050	Crazed	11.7	79	220	750	Cracked
A-198	Hycar 1012 × 41	10	79	630	1770	24.0	75	280	1400	Crazed	24.4	82	190	1110	Cracked
A-226	Hycar 1012 × 41	15	66	780	2020	18.5	64	420	840	Crazed	16.3	76	230	730	Cracked
A-225	Hycar 1012 × 41	20	61	850	1690	16.3	61	390	670	Crazed	15.2	73	280	550	Cracked
A-199	Hycar 1011 × 15	10	82	500	1850	25.0	78	250	1380	Crazed	23.7	84	120	850	Cracked
A-191	Paraplex G-25	10	78	660	1980	23.1	78	330	860	Crazed	26.3	91	150	630	Cracked
A-192	Glyptal Plasticizer 2557	10	76	660	2020	21.3	77	320	810	Crazed	23.1	90	160	650	Cracked

(1) Aged in aluminum-block heater.

Base Recipe:

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

Cure: 60 minutes at 298 F.

TABLE 22. THE EFFECT OF SOFTENERS AND OTHER ADDITIVES ON THE AGING PROPERTIES OF HYCAR 1001

Recipe No.(1)	Additive	Loading, phr	Original Physical Properties			Physical Properties After Aging in Essa Turbo Oil-15 72 Hours at 350 F(2)					Physical Properties After Aging in Essa Turbo Oil-15 168 Hours at 350 F(2)				
			Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
A-23	None	–	77	630	1875	17.8	77	250	1790	Crazed	16.2	83	130	1240	Cracked
A-193	Piccopale	10	87	590	2040	36.6	72	200	940	Crazed	37.2	79	110	530	Cracked
A-194	Picco 100	10	84	580	1950	38.9	76	250	1180	Crazed	29.7	81	150	850	Cracked
A-195	Vistanex B-100	10	83	480	1830	35.9	76	170	1110	Crazed	36.3	82	100	600	Cracked
A-196	Reclaim	10	84	460	2080	32.6	77	230	1220	Crazed	33.0	83	100	690	Cracked
A-197	GR-I 18	10	81	600	1910	37.6	77	170	1090	Crazed	34.8	82	120	820	Cracked
A-213	Mineral Rubber	10	84	570	2590	29.6	77	240	1280	Crazed	28.2	81	130	890	Cracked
A-220	Polyrez D	10	75	490	2610	9.9	79	150	1080	Crazed	8.8	85	90	760	Cracked
A-219	Indulin-Hycar OR-25(3)	25	50	700	1160	19.3	56	120	90	Cracked	17.7	62	20	180	Cracked
A-218	Indulin-Hycar OR-25(3)	50	52	730	1430	22.1	57	110	125	Cracked	19.4	62	70	160	Cracked
A-240	Neophax D	10	67	630	2080	25.4	63	310	730	Crazed	24.9	67	180	450	Cracked
A-241	Neophax D	20	65	570	1980	32.3	60	200	500	Crazed	31.9	65	140	350	Cracked
A-254	Neophax A	10	68	600	2450	23.4	67	270	820	Crazed	21.8	68	170	630	Cracked
A-255	Neophax A	20	66	590	2160	30.1	63	170	530	Crazed	28.3	66	110	340	Cracked
A-251	DPR Synthetic N-27	10	67	720	2100	18.3	60	400	830	Crazed	17.3	72	230	620	Cracked
A-253	GE Silicone Gum SE-76	10	68	610	1120	20.7	65	250	440	Crazed	21.9	74	130	450	Cracked
A-231	Polyester HA-5-A	5	76	650	2470	22.3	79	350	990	Crazed	19.2	86	170	730	Cracked
A-232	Polyester HA-5-A	10	67	720	1930	17.4	72	430	650	Crazed	16.0	84	220	630	Cracked

(1) Base recipe same as shown in Table 21.
(2) Aged in aluminum-block heater.
(3) Coprecipitate of 50 parts Indulin with 100 parts Hycar OR-25. Supplied by West Virginia Pulp and Paper Company.

TABLE 23. THE EFFECT OF ACRYLONITRILE CONTENT OF COPOLYMER

Recipe No.	Copolymer	Acrylonitrile Content, per cent	Original Physical Properties: Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Physical Properties After Air Aging 72 Hours at 350 F: Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
A-211	Hycar 1000 × 70	60	100	70	3500	–	–	–	–
A-173	1457-60	55	98	170	2180	100	30	1640	Cracked
A-157	Chemigum N3NS	45	86	510	1680	98	60	920	Cracked
A-23	Hycar 1001	40-45	77	630	1875	96	30	860	Cracked
A-175	Hycar 1002	30-35	81	320	1250	92	70	780	Cracked
A-177	Paracril B	26	73	380	1120	91	140	900	Cracked
A-178	Paracril AJ	18	65	390	1110	83	50	510	Cracked

(1) Aged in aluminum-block heater.

Base Recipe:

Ingredients	Parts by Weight
Base copolymer	100
Magnesia	100
Zinc oxide	5
Stearic acid	1.5
Sulfur	0.5
Methyl Tuads	0.25

Cure: 60 minutes at 298 F

TABLE 24. THE EFFECT OF ACRYLONITRILE CONTENT OF COPOLYMER

Recipe No.	Copolymer	Acrylonitrile Content, per cent	Original Physical Properties: Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Physical Properties After Air Aging 72 Hours at 350 F: Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
A-212	Hycar 1000 × 70	60	100	110	2880	–	–	–	–
A-158	Chemigun N3NS	45	68	600	4660	95	10	830	Cracked
A-174	Hycar 1002	30-35	68	400	2030	93	10	580	Cracked
A-176	Paracril B	26	58	610	2360	92	10	600	Cracked
A-179	Paracril AJ	18	62	420	2070	95	10	480	Cracked

(1) Aged in aluminum-block heater.

Base Recipe:

Ingredients	Parts by Weight
Base copolymer	100
Philblack O	40
Zinc oxide	5
Stearic acid	1.5
Methyl Tuads	3
AgeRite Powder	3

Cure: 60 minutes at 298 F.

ON AGING PROPERTIES OF A MAGNESIA-FILLED COMPOUND

Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F(1)					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F(1)				
Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
3.5	100	40	3030	Cracked	3.3	97	20	2680	Cracked
6.9	96	60	2030	Crazed	6.3	98	30	1530	Cracked
27.1	80	270	1320	Crazed	23.3	88	150	1010	Cracked
17.8	77	250	1790	Crazed	16.2	83	130	1240	Cracked
44.2	62	150	490	Cracked	40.1	73	50	300	Cracked
73.5	47	140	260	Crazed	73.3	55	70	140	Cracked
134.3	31	90	80	Crazed	125.1	28	70	25	Cracked

ON AGING PROPERTIES OF A CARBON-BLACK-FILLED COMPOUND

Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F(1)					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F(1)				
Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
3.0	100	40	2500	Cracked	3.3	100	10	1930	Cracked
11.0	63	160	410	Cracked	9.8	84	80	570	Cracked
32.4	54	100	220	Cracked	30.2	68	10	260	Cracked
75.0	26	120	40	Cracked	67.4	52	0	50	Cracked
137.9	26	120	50	Cracked	113.0	26	80	25	Cracked

TABLE 25. THE EFFECT OF CURING CONDITIONS ON THE AGING PROPERTIES OF HYCAR 1001

Recipe No.	Curing Conditions	Original Physical Properties			Physical Properties After Aging in Turbo Oil-15 72 Hours at 350 F(1)						Physical Properties After Aging in Turbo Oil-15 168 Hours at 350 F(1)					
		Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Type of Turbo Oil	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Type of Turbo Oil
A-23-f	30 min at 298 F	71	640	2480	24.6	68	230	780	Crazed	Esso	15.7	80	160	710	Cracked	Penola
A-23-f	60 min at 298 F	72	590	2470	25.5	70	250	800	Crazed	Esso	17.3	81	180	690	Cracked	Penola
A-23-f	30 min at 350 F	73	550	2500	25.2	70	260	820	Crazed	Esso	16.9	80	180	730	Cracked	Penola
A-23-f	60 min at 350 F	73	540	2550	23.2	71	270	810	Crazed	Esso	16.1	81	170	810	Cracked	Penola
A-23-f	30 min at 400 F	75	540	2450	24.5	72	270	980	Crazed	Esso	16.8	81	160	780	Cracked	Penola
A-23-f	60 min at 400 F	72	530	2360	22.3	71	250	920	Crazed	Esso	16.0	80	150	770	Cracked	Penola

(1) Aged in aluminum-block heater.

TABLE 26. PHYSICAL PROPERTIES OF COMPOUND A-23 (MAGNESIA-FILLED HYCAR 1001) AFTER AGING AT 350 F FOR VARIOUS PERIODS IN ESSO TURBO OIL-15[1]

System	Aging Time, hours	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
Tube covered with loose-fitting Petri dish	168	15.7	74	160	2175	Crazed
Tube covered with loose-fitting Petri dish	336	16.3	84	70	1150	Cracked
Tube covered with loose-fitting Petri dish	500	14.8	85	50	990	Cracked
Bottle fitted with ground-glass stopper (limited air)	500	50.4	64	110	1520	Cracked

(1) Aged in circulating-air oven.

TABLE 27. THE EFFECT OF METALS ON OIL-AGED PROPERTIES OF HYCAR 1001

Recipe No.	Cure Time, minutes	Cure Temperature, F	Metal Used	Weight of Metal Before Aging, gram	Weight of Metal After Aging, gram	Appearance of Metal After Aging	Original Physical Properties: Hardness, Shore A	Original: Elongation, per cent	Original: Tensile Strength, psi	After Aging in Esso Turbo Oil-15 at 350 F for 72 Hours: Swell, per cent	After Aging: Hardness, Shore A	After Aging: Elongation, per cent	After Aging: Tensile Strength, psi
A-13(1)	30	298	Low-carbon steel	0.1113	0.1112	Yellowed	73	200	2650	–0.5	68	110	1290
A-13	30	298	Electrolytic Cu foil(2)	0.0517	0.0512	Dark-brown stain	73	200	2650	–0.6	67	120	1440
A-13	30	298	Al foil(3)	0.0065	0.0065	No change	73	200	2650	–0.6	68	130	1480
A-13	30	298	Mg ribbon(4)	0.0251	0.0249	No change	73	200	2650	–0.5	69	130	1670
A-13	30	298	Ag sheet(5)	0.3762	0.3761	Blackened	73	200	2650	–0.4	69	120	1520
A-13	30	298	All glass	–	–	–	73	200	2650	–0.5	68	120	1360
A-13	30	298	Chromel A wire plus Al-wrapped cork	–	–	No change	73	200	2650	–0.1	68	110	1200
A-14(1)	30	298	Low-carbon steel	0.1159	0.1159	Yellowed	66	530	2630	2.5	59	150	1000
A-14	30	298	Electrolytic Cu foil	0.0505	0.0500	Lightly blackened	66	530	2630	2.7	60	170	1090
A-14	30	298	Al foil	0.0065	0.0065	No change	66	530	2630	2.5	63	160	1010
A-14	30	298	Mg ribbon	0.0261	0.0261	No change	66	530	2630	2.5	62	150	980
A-14	30	298	Ag sheet	0.3904	0.3904	Blackened	66	530	2630	2.5	62	160	1000
A-14	30	298	All glass	–	–	–	66	530	2630	2.4	63	140	980
A-14	30	298	Chromel A wire plus Al-wrapped cork	–	–	No change	66	530	2630	2.8	61	160	1180
B-2(1)	40	298	Low-carbon steel	0.1159	0.1159	Yellowed	76	270	2060	24.9	63	70	460
B-2	40	298	Electrolytic Cu foil	0.0499	0.0495	Blackened	75	270	2060	25.5	62	70	440
B-2	40	298	Al foil	0.0071	0.0071	No change	76	270	2060	25.2	63	60	340
B-2	40	298	Mg ribbon	0.0247	0.0244	No change	76	270	2060	24.5	65	70	390
B-2	40	298	Ag sheet	0.3550	0.3550	Blackened	76	270	2060	25.2	64	60	350
B-2	40	298	All glass	–	–	–	76	270	2060	24.6	64	60	370
B-2	40	298	Chromel A wire plus Al-wrapped cork	–	–	No change	76	270	2060	24.7	65	60	440

(1) Recipes are shown in Table 2.
(2) J. T. Baker Chemical Company, Lot 101747.
(3) J. T. Baker Chemical Company, Lot 111644, Impurities Fe, 0.45 per cent; S, 0.05 per cent; Cu, 0.01 per cent.
(4) Coleman & Bell, Lot NM 052550.
(5) Pure electrolytic silver.

TABLE 28. RESULTS OF AGING SPECIMENS UNDER IDENTICAL CONDITIONS TO DETERMINE REPRODUCIBILITY OF RESULTS

		Original Physical Properties			Physical Properties After Air Aging 72 Hours at 350 F				Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F				
Recipe No.	Sample No.	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
Aged in Aluminum-Block Heater																		
A-23	1	77	410	1810	96	30	860	Cracked	18.0	79	210	1960	Crazed	17.6	82	140	1330	Cracked
A-23	2	–	–	–	96	50	970	Cracked	17.1	76	250	1980	Crazed	1[illegible].5	81	120	1270	Cracked
A-23	3	–	–	–	95	40	830	Cracked	16.8	81	210	1650	Crazed	15.3	85	130	1110	Cracked
A-97	1	71	790	2670	92	130	760	Cracked	25.4	54	340	670	Crazed	27.5	68	200	510	Cracked
A-97	2	–	–	–	92	120	740	Cracked	26.7	51	340	740	Cracked	24.9	65	220	570	Cracked
A-97	3	–	—	–	91	110	750	Cracked	25.9	56	340	720	Cracked	25.7	68	180	470	Cracked

Base Recipe:

Ingredients	Parts by Weight A-23	Parts by Weight A-97
Hycar 1001	100	100
Zinc oxide	5	5
Stearic acid	1.5	1.5
Magnesia (ELC)	100	100
Sulfur	0.5	0.5
Methyl Tuads	0.25	0.25
Zinc stearate	–	5

Cure: 60 min at 298 F.

TABLE 29. THE REPRODUCIBILITY OF BATCH-TO-BATCH COMPOUNDING

Recipe No.	Original Physical Properties			Physical Properties After Air Aging 72 Hours at 350 F				Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F[1]					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F[1]				
	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
A-23-a	77	410	1810	96	30	860	Cracked	17.8	77	250	1790	Cr[illegible]	16.2	83	130	1240	Cracked
A-23-b	84	440	2140	95	40	1020	Cracked	14.7	85	240	1740	Crazed	13.8	88	100	1100	Cracked
A-23-c	80	460	1960	95	60	900	Cracked	16.2	81	260	1780	Crazed	14.6	85	150	1220	Cracked

(1) Aged in aluminum-block heater.

Base Recipe:

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

Cure: 60 minutes at 298 F.

TABLE 30. THE COMPARATIVE EFFECTS OF ESSO TURBO OIL-15 AND DI-(2-ETHYLHEXYL) SEBACATE ON THE AGING PROPERTIES OF HYCAR 1001

		Original Physical Properties			Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F(1)					Physical Properties After Aging in Di-(2-Ethylhexyl) Sebacate 72 Hours at 350 F(1)				
Recipe No.	Sample No.	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
A 23	a	77	410	1810	17.9	77	210	1630	Crazed	9.2	85	170	1540	Crazed
	b				15.6	80	230	1770	Crazed	10.1	83	190	1440	Crazed
	c				14.6	83	210	1630	No cracking	9.8	82	190	1710	No cracking
	d				14.6	79	220	1600	No cracking	10.7	80	210	1760	No cracking
	e				13.6	83	200	1760	Crazed	10.9	82	190	1680	No cracking
	f				12.6	83	170	1530	Crazed	11.7	84	180	1630	Crazed
	g				13.3	83	190	1730	Crazed	11.9	85	200	1730	Crazed
A-97	a	71	790	2670	26.5	55	310	540	No cracking	15.7	68	290	590	Crazed
	b				21.7	59	290	660	No cracking	15.1	65	330	670	Crazed
	c				21.7	60	270	600	No cracking	15.1	63	300	780	Crazed
	d				19.4	59	310	730	No cracking	16.2	63	300	800	No cracking
	e				20.2	62	300	630	Crazed	16.0	65	290	640	Crazed
	f				18.5	65	310	760	Crazed	15.8	66	280	720	Crazed
	g				19.6	62	310	710	Crazed	17.7	65	300	710	Crazed

(1) Fresh samples were aged 72 hours, removed, and tested. New samples were then aged in the same oil.

Ingredients	Recipe No. A-23	Recipe No. A-97
Hycar 1001	100	100
Magnesia	100	100
Zinc oxide	5	5
Stearic acid	1.5	1.5
Sulfur	0.5	0.5
Methyl Tuads	0.25	0.25
Zinc stearate	–	5

Cure: 60 minutes at 298 F.

TABLE 31. THE DEGRADATION OF ESSO TURBO OIL-15 AND DI-(2-ETHYLHEXYL) SEBACATE AT 350 F

	Per Cent Active Oxygen	
Aging Time, hours	Esso Turbo Oil-15	Di-(2-Ethylhexyl) Sebacate
Closed System		
0	.002	.0005
24	.001	.0005
72	.001	.0005
168	.002	.0005
Open System		
0	.002	.0005
24	.005	.006
72	.009	.004
168	.004	.003

TABLE 32. COMPARISON OF AGING RESULTS OBTAINED WITH ESSO AND PENOLA TURBO OIL-15

Recipe No.	Aging Time, hours	Type of Turbo Oil	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Crack Resistance
A-198(1)	168	Penola	15.2	89	160	940	Cracked
A-198	168	Esso	24.4	82	190	1110	Cracked
A-199(1)	168	Penola	14.4	90	140	980	Cracked
A-199	168	Esso	23.7	84	120	850	Cracked
A-200(2)	168	Penola	20.6	91	150	880	Cracked
A-200	168	Esso	26.0	85	100	660	Cracked
A-201(2)	168	Penola	22.9	88	140	980	Cracked
A-201	168	Esso	26.1	82	120	900	Cracked

(1) Recipes shown in Table 21.

(2) Recipes shown in Table 10.

TABLE 33. THE EFFECT OF TRIETHYLENE TETRAMINE, SULFUR,

Recipe No.	Vulcanizing Agent, phr TETA	Methyl Tuads	Sulfur	Cure, minutes at 310 F	Original Physical Properties Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking
PA-39	–	2.0	–	120	1390	800	51	560	330	22	85.0	None
PA-17	–	2.0	1.0	120	1350	780	44	700	480	22	72.7	None
PA-18	–	–	2.0	120	(Did not cure)		–	–	–	–	–	–
PA-37	–	–	4.0	120	(Did not cure – full of pinholes)			–	–	–	–	–
PA-38	0.5	–	2.0	60	1050	980	55	0	530	6	94.9	None
PA-2	1.5	2.0	–	30	1540	350	63	640	70	57	39.1	None
PA-19	1.5	–	–	30	1740	290	54	430	70	54	42.0	None
PA-7	1.5	2.0	1.0	30	1610	360	65	830	140	43	42.7	None
PA-20	1.5	–	1.0	30	1810	230	62	840	190	38	52.2	None
PA-21	3.0	2.0	–	30	1580	280	58	730	60	65	30.6	None
PA-22	3.0	–	–	30	1500	160	63	590	40	72	27.2	None
PA-23	3.0	2.0	1.0	30	1420	170	65	740	90	57	30.9	None
PA-24	3.0	–	1.0	30	1540	110	74	830	80	62	38.3	None
PA-48	1.6	1.2	1.2	30	1540	360	67	740	150	43	45.9	None
PA-49	2.0	0.8	1.2	30	1600	290	67	740	150	44	44.1	None
PA-50	2.4	0.4	1.2	30	1560	200	72	870	140	48	41.4	None
PA-51	1.7	1.4	0.9	30	1500	480	62	780	170	41	48.4	None
PA-52	2.1	1.0	0.9	30	1590	300	67	820	130	47	42.2	None
PA-53	2.5	0.6	0.9	30	1530	190	70	900	130	47	41.2	None
PA-54	1.9	1.5	0.6	30	1510	390	62	780	130	46	44.8	None
PA-55	2.3	1.1	0.6	30	1590	330	64	650	110	48	43.2	None
PA-56	2.7	0.7	0.6	30	1260	200	65	610	80	56	37.3	None

Note: All samples untempered.

Base Recipe:

Ingredients	Parts by Weight
Hycar 4021	100
Philblack A	40
Stearic acid	1
Vulcanizing agent	As shown

AND METHYL TUADS ON AGING PROPERTIES OF HYCAR 4021

Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F					Physical Properties After Aging in Esso Turbo Oil-15 500 Hours at 350 F				
Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking	Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking
310	270	17	82.2	None	–	–	–	–	–
400	540	15	73.6	None	–	–	–	–	–
–	–	–	–	–	–	–	–	–	–
–	–	–	–	–	–	–	–	–	–
0	220	5	94.8	None	–	–	–	–	–
640	80	51	41.9	None	530	100	46	50.2	None
460	80	54	45.7	None	–	–	–	–	–
880	140	44	44.9	None	–	–	–	–	–
790	170	38	56.3	None	–	–	–	–	–
690	50	67	32.5	None	–	–	–	–	–
580	40	75	32.7	None	–	–	–	–	–
590	70	59	38.6	None	–	–	–	–	–
420	50	61	39.1	None	–	–	–	–	–
500	120	39	52.2	None	–	–	–	–	–
770	140	43	50.8	None	–	–	–	–	–
690	110	47	45.0	None	410	140	35	56.6	None
600	150	38	57.1	None	–	–	–	–	–
650	110	48	42.9	None	520	130	44	57.4	None
610	110	47	44.1	None	470	120	43	54.5	None
700	130	46	49.8	None	–	–	–	–	–
580	100	49	45.4	None	730	150	41	66.8	None
500	60	59	39.3	None	480	90	54	50.2	None

TABLE 34. OPTIMUM RATIOS OF TRIETHYLENE

		Original Physical Properties			Physical Properties After Aging in Turbo Oil-15 72 Hours at 350 F					
Recipe No.	Cure, minutes at 310 F	Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil
PA-2	30	1540	350	63	640	70	57	39.1	None	Esso
PA-88	30	1560	490	64	510	90	56	41.1	None	Penola
PA-52	30	1590	300	67	670	130	52	43.4	None	Penola
PA-82	30	1820	230	72	870	110	55	39.7	None	Penola

Note: All samples untempered.

Recipes Used:

Ingredients	Parts by Weight PA-2, PA-88	Parts by Weight PA-52, PA-82
Hycar 4021	100	100
Philblack A	40	40
Stearic acid	1	1
Sulfur	–	0.9
Methyl Tuads	2	1
Triethylene tetramine	1.5	2.1

TETRAMINE, SULFUR, AND TUADS IN HYCAR 4021

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, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil	Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil
640	80	51	41.9	None	Esso	530	100	46	50.2	None	Esso
390	80	56	44.4	None	Penola	610	120	48	58.7	None	Penola
760	130	52	46.2	None	Penola	820	150	46	55.4	None	Penola
460	80	58	41.2	None	Penola	620	110	50	53.5	None	Penola

TABLE 35. THE EFFECT OF CURING TIME AND AMOUNT OF VULCANIZING AGENT ON AGING PROPERTIES OF HYCAR 4021

Recipe No.	Cure, minutes at 310 F	Original Physical Properties: Tensile Strength, psi	Original Physical Properties: Elongation, per cent	Original Physical Properties: Hardness, Shore A	After Aging in Penola Turbo Oil-15 72 Hours at 350 F: Tensile Strength, psi	72 Hours: Elongation, per cent	72 Hours: Hardness, Shore A	72 Hours: Swell, per cent	72 Hours: Cracking	After Aging in Penola Turbo Oil-15 168 Hours at 350 F: Tensile Strength, psi	168 Hours: Elongation, per cent	168 Hours: Hardness, Shore A	168 Hours: Swell, per cent	168 Hours: Cracking	After Aging in Penola Turbo Oil-15 500 Hours at 350 F: Tensile Strength, psi	500 Hours: Elongation, per cent	500 Hours: Hardness, Shore A	500 Hours: Swell, per cent	500 Hours: Cracking
PA-82	15	1920	260	70	750	110	52	42.1	None	400	80	56	43.1	None	550	100	47	53.2	None
	30	1820	230	72	870	110	55	39.7	None	460	80	58	41.2	None	620	110	50	53.5	None
	60	1940	200	73	860	100	55	39.1	None	560	70	59	39.8	None	600	90	53	52.2	None
	120	1920	160	74	820	100	58	36.1	None	690	70	60	40.3	None	790	110	53	46.5	None
PA-112	60	1740	160	71	500	80	54	39.0	None	640	70	57	43.6	None	–	–	–	–	–
	120	1420	140	74	520	80	56	41.0	None	660	80	58	42.0	None	–	–	–	–	–
	240	1460	120	76	460	70	57	38.6	None	610	70	59	42.3	None	–	–	–	–	–
PA-113	15	1290	200	68	900	140	49	37.4	None	810	110	53	37.9	None	590	90	57	44.5	None
	30	1430	140	70	700	130	50	33.8	None	580	80	60	35.1	None	580	70	60	36.8	None
	60	1350	120	72	860	130	52	32.4	None	510	80	61	32.7	None	520	80	57	41.6	None
	120	1070	100	74	880	110	55	32.1	None	660	90	60	33.7	None	550	70	64	34.9	None
	240	1410	110	76	1000	120	54	29.4	None	740	70	66	31.0	None	700	70	64	36.6	None

Note: All samples untempered.

Ingredients	Recipes: PA-82	PA-112	PA-113
Hycar 4021	100	100	100
Philblack A	40	40	40
Stearic acid	1	1	1
Sulfur	0.9	0.9	1.8
Methyl Tuads	1	1	2
Triethylene tetramine	2.1	2.1	4.2

TABLE 36. THE EFFECT OF VARYING TRIMENE BASE, SULFUR,

Recipe No.	Vulcanizing Agent, phr: Trimene Base	Vulcanizing Agent, phr: Methyl Tuads	Vulcanizing Agent, phr: Sulfur	Cure, minutes at 310 F	Original Physical Properties: Tensile Strength, psi	Original Physical Properties: Elongation, per cent	Original Physical Properties: Hardness, Shore A	Physical Properties After Oil 15 72 Hours: Tensile Strength, psi	Physical Properties After Oil 15 72 Hours: Elongation, per cent	Physical Properties After Oil 15 72 Hours: Hardness, Shore A
PA-3	3.0	2.0	0.5	30	1430	440	60	720	120	45
PA-40	3.0	–	0.5	30	1360	400	60	820	190	36
PA-41	3.0	2.0	–	30	1200	730	56	660	170	39
PA-42	1.5	2.0	0.5	30	960	1020	52	620	240	25
PA-43	1.5	–	0.5	30	1370	810	55	390	880	16
PA-44	1.5	2.0	1.5	30	1020	1050	53	290	220	18
PA-45	1.5	–	1.5	30	1390	730	55	210	880	7
PA-46	0.5	2.0	3	60	1120	970	54	450	500	15
PA-47	0.5	–	3	(Did not cure in 120 minutes)						
PA-76	3.0	2.0	–	30	1480	780	55	690	190	33
				60	1580	690	57	690	190	35
				120	1670	540	55	700	150	37
PA-119	6.0	4.0	–	30	1390	510	56	740	100	56
				60	1450	410	57	730	90	60
				120	1350	320	60	790	100	60
				240	1420	250	61	870	100	61
PA-115	2.1	1.0	0.9	30	1680	620	63	700	200	31
				60	1540	520	63	660	200	32
				120	1720	450	65	910	210	35
				240	1850	400	65	860	200	36
PA-116	2.6	1.0	0.9	30	1810	520	65	830	230	32
				60	1780	460	66	980	240	34
				120	1820	390	67	800	180	36
				240	1730	320	68	730	150	38
PA-117	4.2	1.0	0.9	30	1780	320	63	790	150	43
				60	1870	260	65	880	160	45
				120	1720	250	65	900	150	48
				240	1860	240	65	810	130	48

Note: All samples untempered.

Base Recipe:

Ingredients	Parts by Weight
Hycar 4021	100
Philblack A	40
Stearic acid	1
Vulcanizing agent	As shown

AND METHYL TUADS ON AGING PROPERTIES OF HYCAR 4021

Aging in Turbo at 350 F			Physical Properties After Aging in Turbo Oil-15 168 Hours at 350 F					
Swell, per cent	Cracking	Type of Turbo Oil	Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil
48.3	None	Esso	510	90	44	57.6	None	Esso
52.4	None	Esso	490	150	38	55.1	None	Esso
50.5	None	Esso	460	120	42	51.4	None	Esso
59.7	None	Esso	410	230	28	74.2	None	Esso
78.6	None	Esso	160	730	12	72.5	None	Esso
62.3	None	Esso	380	250	22	70.7	None	Esso
85.7	None	Esso	200	840	10	86.1	None	Esso
70.7	None	Esso	470	540	10	82.8	None	Esso
64.5	None	Esso	430	160	29	72.9	None	Esso
59.7	None	Esso	540	160	34	66.0	None	Esso
57.2	None	Esso	690	180	36	60.7	None	Esso
38.7	None	Penola	820	110	57	38.7	None	Penola
37.8	None	Penola	730	90	60	39.9	None	Penola
38.0	None	Penola	640	80	63	37.4	None	Penola
36.6	None	Penola	810	80	65	35.4	None	Penola
65.0	None	Penola	730	210	30	68.9	None	Penola
60.8	None	Penola	670	180	34	63.9	None	Penola
57.7	None	Penola	680	160	36	60.8	None	Penola
53.4	None	Penola	450	120	37	59.8	None	Penola
60.3	None	Penola	550	160	32	62.6	None	Penola
57.4	None	Penola	660	170	33	57.7	None	Penola
55.3	None	Penola	650	150	35	57.4	None	Penola
53.5	None	Penola	650	140	36	57.2	None	Penola
52.5	None	Penola	610	130	38	55.6	None	Penola
49.8	None	Penola	610	120	40	54.5	None	Penola
48.6	None	Penola	660	130	41	50.1	None	Penola
48.3	None	Penola	700	120	42	52.5	None	Penola

TABLE 37. COMPARISON OF VULCANIZING AGENTS ON AGING PROPERTIES OF HYCAR 4021

Recipe No.	Vulcanizing Agent, phr					Cure, minutes at 310 F	Original Physical Properties			Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F				
	Monex	Polyac(1)	Methyl Tuads	Sulfur	TETA		Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking	Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking
PA-8	1.0	–	–	2.0	–	60	670	1210	51	270	780	13	86.4	None	70	930	14	80.2	None
PA-34	1.0	–	–	2.0	–	120	1180	1020	52	220	1140	10	92.3	None	170	880	8	88.9	None
PA-35	2.0	–	–	2.0	–	120	1430	780	51	620	610	16	81.1	None	290	560	12	88.5	None
PA-36	1.0	–	–	2.0	0.5	120	1460	760	55	430	350	20	76.8	None	460	410	15	77.8	None
PA-77	1.2	–	–	1.2	1.6	30	1780	460	54	410	140	35	55.6	None	360	140	34	62.1	None
PA-78	1.0	–	–	0.9	2.1	30	1670	300	57	490	120	41	45.3	None	690	140	42	50.7	None
PA-79	0.7	–	–	0.6	2.7	30	1780	300	59	400	80	51	40.1	None	490	100	51	46.2	None
PA-85	–	4.8	–	1.2	1.6	30	1470	670	55	380	180	33	63.2	None	790	200	28	65.9	None
PA-86	–	4.0	–	0.9	2.1	30	1760	320	65	670	140	44	47.2	None	690	110	43	50.3	None
PA-87	–	2.8	–	0.6	2.7	30	1480	230	65	580	100	52	42.1	None	430	70	53	41.8	None

Note: All samples untempered.

(1) Polyac contains 25 per cent active material and 75 per cent inert filler. Thus, the amount of active material in the Polyac, Monex, and Methyl Tuads recipes was the same.

Base Recipe:

Ingredients	Parts by Weight
Hycar 4021	100
Philblack A	40
Stearic acid	1
Vulcanizing agent	As shown

TABLE 38. THE EFFECT OF HI-SIL

Recipe No.	Filler, phr by weight	Filler, phr by volume	Plasticizer, phr by weight	Treatment After Cure	Cure, minutes at 310 F	Original Physical Properties: Tensile Strength, psi	Original Physical Properties: Elongation, per cent	Original Physical Properties: Hardness, Shore A	Physical Properties After Oil-15 72 Hours: Tensile Strength, psi	Physical Properties After Oil-15 72 Hours: Elongation, per cent	Physical Properties After Oil-15 72 Hours: Hardness, Shore A	Physical Properties After Oil-15 72 Hours: Swell, per cent
PA-69	43.3	24.4	0	None	60	1590	480	76	970	130	57	44.9
PA-69	43.3	24.4	0	Tempered(1)	60	1690	180	83	890	100	59	42.7
PA-72	43.3	24.4	10	None	30	1320	620	72	620	120	53	42.9
PA-72	43.3	24.4	10	Tempered	30	1350	190	81	710	110	59	37.1
PA-73	43.3	24.4	10	None	60	1330	520	74	620	110	53	40.2
PA-73	43.3	24.4	10	Tempered	60	1390	180	83	720	100	60	35.6
PA-70	54.2	30.6	0	None	30	1400	580	83	900	120	59	41.8
PA-70	54.2	30.6	0	Tempered	30	1430	120	93	950	100	65	40.4
PA-71	65.0	36.7	0	None	30	1380	530	92	1010	110	73	40.7
PA-71	65.0	36.7	0	Tempered	30	1580	110	95	1360	100	79	34.6
PA-71	65.0	36.7	0	None	30	1380	530	92	840	120	73	42.9
PA-71	65.0	36.7	0	Tempered	30	1580	110	95	1170	100	76	34.6
PA-97	65.0	36.7	10	None	30	1380	690	84	700	140	64	45.3
PA-97	65.0	36.7	10	Tempered	30	1780	190	95	920	110	71	34.5
PA-99	65.0	36.7	20	None	30	1200	900	75	500	190	51	52.0
PA-99	65.0	36.7	20	Tempered	30	1530	270	88	850	160	62	38.4
PA-96	75.8	42.7	0	None	30	1470	600	98	1170	120	81	40.2
PA-96	75.8	42.7	0	Tempered	30	1350	100	100+	1220	100	83	33.7
PA-98	75.8	42.7	10	None	30	1350	710	95	900	130	76	42.2
PA-98	75.8	42.7	10	Tempered	30	1820	190	100	1110	120	75	32.7
PA-100	75.8	42.7	20	None	30	1150	790	87	590	170	64	46.6
PA-100	75.8	42.7	20	Tempered	30	1360	220	99	900	140	70	34.1

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

Base Recipe:

Ingredients	Parts by Weight
Hycar 4021	100
Hi-Sil	As shown
Stearic acid	1
Methyl Tuads	2
Triethylene tetramine	1.5
Plasticizer (Flexol R2H)	As shown

ON AGING PROPERTIES OF HYCAR 4021

Aging in Turbo 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, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil	Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil
None	Esso	820	120	54	53.6	None	Esso	–	–	–	–	–	–
None	Esso	960	140	55	51.7	None	Esso	–	–	–	–	–	–
None	Esso	580	140	49	46.3	None	Esso	–	–	–	–	–	–
None	Esso	690	110	56	40.9	None	Esso	–	–	–	–	–	–
None	Esso	650	130	53	42.6	None	Esso	–	–	–	–	–	–
None	Esso	730	120	59	40.4	None	Esso	–	–	–	–	–	–
None	Esso	730	100	60	50.9	None	Esso	–	–	–	–	–	–
None	Esso	830	110	62	47.2	None	Esso	–	–	–	–	–	–
None	Esso	1130	120	75	43.1	None	Esso	910	180	70	40.2	None	Esso
None	Esso	1150	100	77	38.3	None	Esso	940	140	75	44.8	None	Esso
None	Penola	1060	120	74	42.9	None	Penola	1060	190	62	50.8	None	Penola
None	Penola	930	90	76	39.7	None	Penola	1190	130	71	49.5	None	Penola
None	Penola	610	130	63	44.9	None	Penola	800	190	66	48.3	None	Penola
None	Penola	890	120	68	38.1	None	Penola	820	120	67	46.3	None	Penola
None	Penola	460	170	52	53.7	None	Penola	–	–	–	–	–	–
None	Penola	750	150	60	40.7	None	Penola	–	–	–	–	–	–
None	Penola	980	100	82	42.0	None	Penola	1050	180	81	49.8	None	Penola
None	Penola	1110	90	84	37.2	None	Penola	1220	120	91	46.8	Cracked	Penola
None	Penola	840	130	77	43.0	None	Penola	830	200	75	44.7	None	Penola
None	Penola	1010	110	77	37.1	None	Penola	940	120	80	41.3	None	Penola
None	Penola	540	160	65	43.5	None	Penola	–	–	–	–	–	–
None	Penola	770	130	71	35.9	None	Penola	–	–	–	–	–	–

TABLE 39. THE EFFECT OF FILLERS

Recipe No.	Filler, type	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, per cent	Original Physical Properties: Hardness, Shore A	Physical Properties After Oil-15 72 Hours: Tensile Strength, psi	Physical Properties After Oil-15 72 Hours: Elongation, per cent	Physical Properties After Oil-15 72 Hours: Hardness, Shore A	Physical Properties After Oil-15 72 Hours: Swell, per cent
PA-2	Philblack A	40	24.4	30	None	1540	350	63	640	70	57	39.1
PA-2	Philblack A	40	24.4	30	Tempered(1)	1710	130	74	570	70	60	41.1
PA-88	Philblack A	40	24.4	30	None	1560	490	64	510	90	55	41.1
PA-61	Philblack A	50	30.6	30	None	1360	530	68	660	120	45	47.6
PA-62	Philblack A	60	36.7	30	None	1310	390	73	710	100	53	43.1
PA-89	Philblack A	70	42.7	30	None	1350	330	88	910	90	67	39.1
PA-90	Philblack A	80	48.8	30	None	1360	240	95	1100	80	72	35.9
PA-91	Philblack A Calcene TM	40 13.9	24.4 6.2	30	None	1320	490	65	540	90	59	39.4
PA-91	Philblack A Calcene TM	40 13.9	24.4 6.2	30	Tempered	1540	140	87	400	70	65	39.4
PA-92	Philblack A Calcene TM	40 27.8	24.4 12.3	30	None	1220	470	70	640	80	64	35.1
PA-92	Philblack A Calcene TM	40 27.8	24.4 12.3	30	Tempered	1400	110	94	590	60	72	32.8
PA-93	Philblack A Calcene TM	40 55.6	24.4 24.4	30	None	970	470	78	800	100	66	34.2
PA-93	Philblack A Calcene TM	40 55.6	24.4 24.4	30	Tempered	1210	100	98	750	70	75	32.3
PA-15	Philblack O	40	24.4	30	None	2110	420	67	660	100	55	37.8
PA-65	ELC Magnesia	40	13.8	60	None	1000	540	47	230	60	51	81.3
PA-30	ELC Magnesia	71	24.4	30	None	1640	510	62	450	50	65	66.8
PA-30	ELC Magnesia	71	24.4	30	Tempered	1500	120	82	580	40	72	63.1
PA-60	Silene EF	46.6	24.4	30	None	1080	630	63	390	100	48	47.1
PA-60	Silene EF	46.6	24.4	30	Tempered	1030	140	67	570	90	57	38.1
PA-94	Silene EF	58.3	30.6	30	None	1070	490	75	550	90	64	39.7
PA-94	Silene EF	58.3	30.6	30	Tempered	1180	120	85	760	90	71	32.7
PA-95	Silene EF	70	36.7	30	None	1080	430	85	760	80	74	35.4
PA-95	Silene EF	70	36.7	30	Tempered	1260	100	93	900	80	80	28.6
PA-66	Hi-Sil C	43.3	24.4	30	None	1670	700	71	840	170	53	48.4
PA-66	Hi-Sil C	43.3	24.4	30	Tempered	1410	210	85	830	140	55	47.6
PA-103	Aerosil	46.6	24.4	30	None	1330	630	84	590	130	65	42.1
PA-103	Aerosil	46.6	24.4	30	Tempered	1280	230	94	550	90	72	43.2

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

Base Recipe:

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

ON AGING PROPERTIES OF HYCAR 4021

Aging in Turbo 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, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil	Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil
None	Esso	640	80	51	41.9	None	Esso	530	100	46	50.2	None	Esso
None	Esso	500	70	58	50.5	None	Esso	650	110	49	62.5	None	Esso
None	Penola	390	80	56	[illegible]	None	Penola	610	120	48	58.7	None	Penola
None	Esso	870	170	43	52.6	None	Esso	–	–	–	–	–	–
None	Esso	660	120	51	49.1	None	Esso	–	–	–	–	–	–
None	Penola	600	70	67	41.2	None	Penola	–	–	–	–	–	–
None	Penola	860	70	74	40.3	None	Penola	–	–	–	–	–	–
None	Penola	350	100	54	42.3	None	Penola	–	–	–	–	–	–
None	Penola	400	80	64	41.4	None	Penola	–	–	–	–	–	–
None	Penola	550	70	66	39.5	None	Penola	790	100	53	46.2	None	Penola
None	Penola	530	70	70	34.8	None	Penola	620	90	55	50.5	None	Penola
None	Penola	390	80	64	37.8	None	Penola	–	–	–	–	–	–
None	Penola	660	70	72	34.1	None	Penola	–	–	–	–	–	–
None	Esso	620	80	55	47.0	None	Esso	–	–	–	–	–	–
None	Esso	260	60	54	106.6	None	Esso	–	–	–	–	–	–
Cracked	Esso	480	60	66	86.3	Cracked	Esso	–	–	–	–	–	–
Cracked	Esso	580	40	74	101.4	Cracked	Esso	–	–	–	–	–	–
None	Esso	450	120	45	50.4	None	Esso	500	150	49	63.6	None	Esso
None	Esso	570	100	57	45.4	None	Esso	430	100	56	58.1	None	Esso
None	Penola	580	90	64	40.9	None	Penola	920	110	57	46.9	None	Penola
None	Penola	840	90	70	33.9	None	Penola	970	90	65	42.8	None	Penola
None	Penola	800	90	71	37.6	None	Penola	1000	100	64	42.7	None	Penola
None	Penola	940	80	79	30.8	None	Penola	1240	90	72	35.7	None	Penola
None	Esso	670	160	51	51.8	None	Esso	–	–	–	–	–	–
None	Esso	730	150	53	50.4	None	Esso	–	–	–	–	–	–
None	Penola	520	90	70	46.3	None	Penola	–	–	–	–	–	–
None	Penola	570	90	72	47.4	None	Penola	–	–	–	–	–	–

TABLE 40. THE EFFECT OF LUBRICANTS ON AGING PROPERTIES OF HYCAR 4021

Recipe No.	Lubricants, phr	Milling Behavior	Cure, minutes at 310 F	Original Physical Properties			Physical Properties After Aging in Esso Turbo Oil-15 72 Hours at 350 F					Physical Properties After Aging in Esso Turbo Oil-15 168 Hours at 350 F				
				Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking	Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking
PA-63	0.5 Stearic acid	Bad sticking	60	1580	490	61	710	130	44	47.4	None	700	150	41	51.8	None
PA-2	1.0 Stearic acid	Sticking	30	1540	350	63	640	70	57	39.1	None	640	80	51	41.9	None
PA-25	2.0 Stearic acid	No sticking	30	1620	520	58	700	110	45	52.4	None	310	80	39	58.5	None
PA-26	3.0 Stearic acid	No sticking	30	1550	510	56	500	100	44	44.5	None	400	100	44	57.4	None
PA-27	2.0 Acrawax CT	Slight sticking	30	1490	700	55	610	140	38	53.5	None	490	130	35	76.1	None
PA-28	2.0 Lanolin	Bad splitting and sticking	30	1570	620	52	660	140	39	52.7	None	290	120	36	61.3	None

Note: All samples untempered.

Base Recipe:

Ingredients	Parts by Weight
Hycar 4021	100
Philblack A	40
Lubricant	As shown
Methyl Tuads	2
Triethylene tetramine	1.5

TABLE 41. COMPARISON OF AGING HYCAR 4021

Recipe No.	Treatment After Cure	Cure, minutes at 310 F	Original Physical Properties: Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Physical Properties After Aging in Turbo Oil-15 72 Hours at 350 F: Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil
PA-52	None	30	1590	300	67	820	130	47	42.2	None	Esso
PA-52	None	30	1590	300	67	670	130	52	43.4	None	Penola
PA-71	None	30	1380	530	92	1010	110	73	40.7	None	Esso
PA-71	None	30	1380	530	92	940	120	73	42.9	None	Penola
PA-71	Tempered(1)	30	1580	110	95	1360	100	79	34.6	None	Esso
PA-71	Tempered(1)	30	1580	110	95	1170	100	76	34.6	None	Penola
PA-82	None	15	1920	260	70	970	130	52	41.8	None	Esso
PA-82	None	15	1920	260	70	750	110	52	42.1	None	Penola
PA-82	None	30	1820	230	72	950	120	52	40.3	None	Esso
PA-82	None	30	1820	230	72	870	110	55	39.7	None	Penola
PA-82	None	60	1940	200	73	1060	120	53	39.0	None	Esso
PA-82	None	60	1940	200	73	860	100	55	39.1	None	Penola
PA-82	None	120	1920	160	74	1140	120	55	36.1	None	Esso
PA-82	None	120	1920	160	74	920	100	58	36.1	None	Penola

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

(2) Insufficient Esso Turbo Oil-15 was available to run 168- and 500-hour aging tests on PA-82.

Ingredients	Recipes: PA-52	PA-71	PA-82
Hycar 4021	100	100	100
Philblack A	40	–	40
Hi-Sil	–	65	–
Stearic acid	1	1	1
Sulfur	0.9	–	0.9
Triethylene tetramine	2.1	1.5	2.1
Methyl Tuads	1	2	1

COMPOUNDS IN ESSO AND PENOLA TURBO OIL-15

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, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil	Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil
650	110	48	42.9	None	Esso	520	130	44	57.4	None	Esso
760	130	52	46.2	None	Penola	820	150	46	55.4	None	Penola
1130	120	75	43.1	None	Esso	910	180	70	48.2	None	Esso
1060	120	74	42.9	None	Penola	1060	190	62	50.8	None	Penola
1150	100	77	38.3	None	Esso	940	140	75	44.8	None	Esso
930	90	76	39.7	None	Penola	1190	130	71	49.5	None	Penola
(2)	-	-	-	-	-	-	-	-	-	-	-
400	80	56	43.1	None	Penola	550	100	47	53.2	None	Penola
-	-	-	-	-	-	-	-	-	-	-	-
460	80	58	41.2	None	Penola	620	110	50	53.5	None	Penola
-	-	-	-	-	-	-	-	-	-	-	-
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

TABLE 42. THE EFFECT OF ADDING HYCAR

Recipe No.	Hycar 1001, phr	Treatment After Cure	Cure minutes at 310 F	Original Physical Properties: Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Physical Properties After Aging in Turbo Oil-15 72 Hours at 350 F: Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil
PA-88	0	None	30	1560	490	64	510	90	55	41.1	None	Penola
PA-80	5	None	30	1750	500	64	690	110	53	44.3	None	Esso
PA-81	10	None	30	1590	590	63	800	120	52	44.8	None	Esso
PA-101	15	None	30	1450	510	64	590	90	60	39.7	None	Penola
PA-101	15	Tempered(1)	30	1340	130	85	880	80	75	35.1	None	Penola
PA-102	20	None	30	1380	560	64	780	110	57	40.6	None	Penola
PA-102	20	Tempered	30	1590	170	83	740	80	72	36.9	None	Penola

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

Base Recipe:

Ingredients	Parts by Weight
Hycar 4021	100
Hycar 1001	As shown
Philblack A	40
Stearic acid	1
Methyl Tuads	2
Triethylene tetramine	1.5

1001 TO A HYCAR 4021 COMPOUND

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, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil	Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil
390	80	56	44.4	None	Penola	610	120	48	58.7	None	Penola
[illegible]	100	58	48.3	None	Esso	860	160	59	53.4	None	Esso
630	100	62	52.5	None	Esso	930	170	68	57.3	None	Esso
860	90	68	41.0	None	Penola	970	80	85	54.4	None	Penola
900	70	77	37.7	None	Penola	1120	70	91	49.2	None	Penola
740	90	63	46.2	None	Penola	930	100	75	60.4	None	Penola
850	80	74	39.6	None	Penola	950	80	83	51.5	None	Penola

TABLE 43. PROPERTIES OF ACRYLON

Recipe No.	Vulconizing Agents, phr: TETA	Vulconizing Agents, phr: Methyl Tuads	Vulconizing Agents, phr: Sulfur	Filler: Type	Filler: Parts	Cure, minutes at 310 F	Treatment After Cure	Original Physical Properties: Tensile Strength, psi	Original Physical Properties: Elongation, per cent	Original Physical Properties: Hardness, Shore A	Physical Properties Oil-15 72 Hours: Tensile Strength, psi	Physical Properties Oil-15 72 Hours: Elongation, per cent	Physical Properties Oil-15 72 Hours: Hardness, Shore A	Physical Properties Oil-15 72 Hours: Swell, per cent
PA-109	0.8	0.8	2.4	Philblack O	40	60	None	1770	790	63	350	1380	35	49.9
PA-110	0.8	2.4	0.8	Philblack O	40	–	(Undercured at 120 min)				–	–	–	–
PA-57	1.5	2.0	–	Philblack O	40	–	(Undercured at 120 min)				–	–	–	–
PA-58	1.5	–	1.0	Philblack O	40	30	None	1790	670	70	1150	520	38	43.4
PA-83	1.5	–	1.0	Philblack O	50	15	None	2370	520	81	1180	430	43	48.1
						30	None	2410	470	82	1200	330	48	45.3
						60	None	2320	450	84	1120	360	50	47.1
PA-84	1.5	–	2.0	Philblack O	50	30	None	2270	490	64	1080	390	47	47.4
						60	None	2310	410	87	1260	330	53	42.4
PA-108	2.1	1.0	0.9	Philblack O	40	30	None	2430	450	71	1220	310	46	42.0
PA-122	2.1	1.0	0.9	Philblack O	50	30	None	2370	400	82	1280	250	55	39.0
						60	None	2400	310	82	1310	180	60	34.8
						120	None	2380	250	85	1300	150	69	30.2
PA-122	2.1	1.0	0.9	Philblack O	50	30	Tempered(1)	2150	260	84	1470	220	62	37.3
						60	Tempered(1)	2020	180	86	1410	170	67	37.5
						120	Tempered(1)	1850	130	90	1100	120	72	33.0
PA-123	2.1	1.0	0.9	Philblack O	40	30	None	2290	440	73	1360	230	54	37.7
						60	None	2450	360	74	1270	180	57	33.9
						120	None	2500	310	75	1130	150	60	30.5
PA-123	2.1	1.0	0.9	Philblack O	40	30	Tempered	2290	290	77	1200	190	55	39.5
						60	Tempered	2290	200	81	1100	150	60	32.3
						120	Tempered	1970	160	81	1160	140	64	33.1
PA-105	0.8	0.8	2.4	Philblack A	40	–	(Undercured at 120 min)				–	–	–	–
PA-106	0.8	2.4	0.8	Philblack A	40	–	(Undercured at 120 min)				–	–	–	–
PA-5	1.5	2.0	–	Philblack A	40	30	None	1310	820	66	750	800	32	52.1
PA-5	1.5	2.0	–	Philblack A	40	30	Tempered	1690	450	71	850	600	34	48.7
PA-107	1.8	1.8	0.4	Philblack A	40	60	None	1640	620	63	720	920	34	54.6
PA-104	2.1	1.0	0.9	Philblack A	40	30	None	1520	450	71	980	300	43	39.0
PA-120	2.1	1.0	0.9	Philblack A	40	30	None	2120	300	75	1200	150	55	32.3
						60	None	2120	230	78	1150	130	64	30.1
						120	None	2110	190	82	1300	120	69	25.8
PA-120	2.1	1.0	0.9	Philblack A	40	30	Tempered	2180	200	82	1140	140	60	35.3
				Philblack A		60	Tempered	2150	150	86	1060	120	67	31.8
				Philblack A		120	Tempered	1980	130	88	1050	90	73	28.8
PA-121	2.1	1.0	0.9	Philblack A	50	30	None	1930	250	80	1170	150	63	32.6
						60	None	1980	200	82	1220	120	72	28.9
						120	None	2180	180	84	1350	110	73	27.3
PA-121	2.1	1.0	0.9	Philblack A	50	30	Tempered	1840	170	86	1110	140	64	36.0
						60	Tempered	1970	130	86	1260	110	73	30.2
						120	Tempered	2020	130	86	1120	90	76	28.1
PA-31	1.5	2.0	–	Philblack E	40	–	(Undercured at 120 min)				–	–	–	–
PA-32	1.7	2.0	–	Philblack E	40	–	(Undercured at 120 min)				–	–	–	–
PA-33	1.7	2.0	0.5	Philblack E	40	–	(Undercured at 120 min)				–	–	–	–

All tempering was for 7 hours at 350 F.

Base Recipe:

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

EA-5 VULCANIZATES AFTER AGING

After Aging in Turbo 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, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil	Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil
None	Penola	400	1150	34	58.1	None	Penola	–	–	–	–	–	–
–	–	–	–	–	–	–	–	–	–	–	–	–	–
–	–	–	–	–	–	–	–	–	–	–	–	–	–
None	Esso	520	330	40	39.5	None	Esso	–	–	–	–	–	–
None	Esso	520	300	41	42.4	None	Esso	–	–	–	–	–	–
None	Esso	500	230	49	42.6	None	Esso	360	110	71	27.2	None	Penola
None	Esso	450	210	52	40.9	None	Esso	–	–	–	–	–	–
None	Esso	540	290	48	42.6	None	Esso	–	–	–	–	–	–
None	Esso	350	140	55	36.3	None	Esso	–	–	–	–	–	–
None	Penola	1200	300	46	44.1	None	Penola	460	100	68	24.1	Cracked	Penola
None	Penola	1320	200	60	39.0	None	Penola			Tests in progress			
None	Penola	1080	160	63	35.6	None	Penola			Tests in progress			
None	Penola	830	100	70	32.3	None	Penola			Tests in progress			
None	Penola	1120	230	60	38.5	None	Penola			Tests in progress			
None	Penola	770	110	67	37.9	None	Penola			Tests in progress			
None	Penola	660	80	72	34.4	None	Penola			Tests in progress			
None	Penola	940	170	55	38.8	None	Penola			Tests in progress			
None	Penola	820	140	60	32.5	None	Penola			Tests in progress			
None	Penola	590	110	62	30.0	None	Penola			Tests in progress			
None	Penola	950	170	55	40.8	None	Penola			Tests in progress			
None	Penola	670	120	60	34.7	None	Penola			Tests in progress			
None	Penola	620	90	67	31.7	None	Penola			Tests in progress			
–	–	–	–	–	–	–	–	–	–	–	–	–	–
–	–	–	–	–	–	–	–	–	–	–	–	–	–
None	Esso	340	450	32	43.6	None	Esso	–	–	–	–	–	–
None	Esso	410	400	34	40.4	None	Esso	–	–	–	–	–	–
None	Penola	800	890	33	54.4	None	Penola	–	–	–	–	–	–
None	Penola	1140	260	47	37.6	None	Penola	480	50	83	24.9	Cracked	Penola
None	Penola	540	90	63	31.8	None	Penola			Tests in progress			
None	Penola	710	90	67	28.9	Cracked	Penola	–	–	–	–	–	–
None	Penola	610	60	75	25.6	Cracked	Penola	–	–	–	–	–	–
None	Penola	910	110	64	35.4	None	Penola			Tests in progress			
None	Penola	540	70	68	31.3	Cracked	Penola	–	–	–	–	–	–
None	Penola	600	60	77	29.9	Cracked	Penola	–	–	–	–	–	–
None	Penola	960	110	67	34.2	None	Penola			Tests in progress			
None	Penola	720	80	71	28.3	None	Penola			Tests in progress			
None	Penola	750	80	74	26.9	None	Penola			Tests in progress			
None	Penola	930	100	69	38.9	None	Penola			Tests in progress			
None	Penola	730	70	75	30.6	Cracked	Penola	–	–	–	–	–	–
None	Penola	700	60	77	28.3	Cracked	Penola	–	–	–	–	–	–
–	–	–	–	–	–	–	–	–	–	–	–	–	–
–	–	–	–	–	–	–	–	–	–	–	–	–	–
–	–	–	–	–	–	–	–	–	–	–	–	–	–

TABLE 44. AGING PROPERTIES

Recipe No.	Polymer	Cure, minutes at 310 F	Treatment After Cure	Original Physical Properties: Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Physical Properties After Air Aging 72 Hours at 350 F: Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Cracking
PA-11	Hycar PA	120	None	1250	260	69	1460	40	97	Cracked
		120	Tempered[1]	1470	70	72	1540	40	98	Cracked
PA-13	Acrylon BA-12	30	None	1630	430	61	1480	210	74	None
		30	Tempered	1570	300	67	1430	210	76	None
VP-1	Philprene VP	30	None	1290	190	68	–	–	–	–
VP-2	Philprene VP	30	None	2790	300	73	–	–	–	–

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

Ingredients	Recipes: PA-11	PA-13	VP-1	VP-2
Hycar PA	100	–	–	–
Acrylon BA-12	–	100	–	–
Philprene VP	–	–	100	100
Philblack A	40	40	–	–
Philblack O	–	–	40	40
Stearic Acid	1	1	–	1.5
Triethylene tetramine	5	1	–	–
Litharge	10	–	10	–
Sulfur	–	1	–	1.5
Benzol chloride	–	–	10	20
Zinc oxide	–	–	–	3
Altox	–	–	–	1.5

OF MISCELLANEOUS POLYMERS

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	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil	Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil
470	50	75	31.9	None	Esso	430	50	75	34.0	None	Esso
660	50	77	31.0	Cracked	Esso	590	40	77	34.7	Cracked	Esso
130	170	22	219.5	None	Esso	20	120	17	208.3	None	Esso
200	130	26	182.7	None	Esso	10	110	15	212.5	None	Esso
80	40	61	97.9	Cracked	Penola	20	10	63	85.9	Cracked	Penola
220	40	75	58.2	Cracked	Penola	80	10	79	54.1	Cracked	Penola

TABLE 45. AGING PROPERTIES OF SILICONE RUBBERS

Sample No.	Commercial Name and Number	Original Physical Properties			Physical Properties After Aging in Esso Turbo Oil-15 at 350 F				
		Hardness, Shore A	Elongation, per cent	Tensile Strength, psi	Aging Time, hours	Swell, per cent	Hardness, Shore A	Elongation, per cent	Tensile Strength, psi
Si-1	Silicone 15060(1)	60	250	850	168	−3.6	60	60	300
Si-2	Silicone 15080(1)	80	250	1000	168	14.7	57	30	140
Si-3	Cohrlastic R-11143(2)	75–90	25	773	128	Sample decomposed			
Si-5	Silastic 50(3,4)	60	300	830	128	20.8	15	0	20
Si-6	Silastic 50(3,5)	55	320	810	128	23.7	5	50	10
Si-7	Silastic 80(3)	77	240	720	128	13.2	40	30	0
Si-8	Silastic 7181(3)	86	70	610	128	15.7	47	60	220
Si-9	Silastic 8154(3)	72	220	660	128	19.4	20	20	40
Si-10	Silastic 250(3)	40	300	560	128	31.6	2	110	0
Si-11	Cohrlastic R-10323(2)	70	70	560	128	15.2	38	120	260

(1) Sample and original data supplied by courtesy of General Electric Company.
(2) Sample and original data supplied by courtesy of Connecticut Hard Rubber Company.
(3) Sample supplied by courtesy of Monsanto Chemical Company.
(4) Colored pink.
(5) Colored white.

TABLE 46. COMPRESSION SET AFTER AGING IN ESSO TURBO OIL-15 AT 350 F

Recipe No.	Treatment After Cure	Per Cent Aged Permanent Set, Aging Time, hours: 72	168
PA-16	None	92	97
	Tempered	74	81
PA-52	None	94	96
	Tempered	64	69

Ingredients	Recipes: PA-16	PA-52
Hycar 4021	100	100
Philblack A	–	40
Hi-Sil	45.2	–
Stearic acid	1	1
Sulfur	–	0.9
Methyl Tuads	2	1
Triethylene tetramine	1.5	2.1

Cure: 30 minutes at 310 F.

TABLE 47. SUMMARY OF COMPOUNDS

Recipe No.	Cure, minutes at 310 F	Treatment After Cure	Original Physical Properties: Tensile Strength, psi	Original Physical Properties: Elongation, per cent	Original Physical Properties: Hardness, Shore A	Physical Properties After Aging in Turbo Oil-15 72 Hours at 350 F: Tensile Strength, psi	Elongation, per cent	Hardness, Shore A	Swell, per cent	Cracking	Type of Turbo Oil
PA-83	30	None	2410	470	82	1200	330	48	45.3	None	Esso
PA-95	30	None	1080	430	85	760	80	74	35.4	None	Penola
	30	Tempered (1)	1260	100	93	900	80	80	28.6	None	Penola
PA-98	30	None	1350	710	95	900	130	76	42.2	None	Penola
	30	Tempered	1820	190	100	1100	120	75	32.7	None	Penola
PA-94	30	None	1070	490	75	550	90	64	39.7	None	Penola
	30	Tempered	1180	120	85	760	80	71	32.7	None	Penola
PA-82	15	None	1920	260	70	750	110	52	42.1	None	Penola
	30	None	1820	230	72	870	110	55	39.7	None	Penola
	60	None	1940	200	73	860	100	55	39.1	None	Penola
	120	None	1920	160	74	820	100	58	36.1	None	Penola
PA-2	30	None	1540	350	63	640	70	57	39.1	None	Esso
PA-52	30	None	1590	300	67	670	130	52	43.4	None	Penola
PA-88	30	None	1560	490	64	510	90	55	41.1	None	Penola

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

Ingredients	Parts by Weight: PA-2	PA-52	PA-82	PA-83	PA-88	PA-94	PA-95	PA-98
Hycar 4021	100	100	100	–	100	100	100	100
Acrylon EA-5	–	–	–	100	–	–	–	–
Philblack A	40	40	40	–	40	–	–	–
Philblack O	–	–	–	50	–	–	–	–
Silene E F	–	–	–	–	–	58.3	70	–
Hi-Sil	–	–	–	–	–	–	–	75.8
Flexol R2H	–	–	–	–	–	–	–	10
Stearic Acid	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Methyl Tuads	2.0	1.0	1.0	–	2.0	2.0	2.0	2.0
Triethylene tetramine	1.5	2.1	2.1	1.5	1.5	1.5	1.5	1.5
Sulfur	–	0.9	0.9	1.0	–	–	–	–

HAVING BEST 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	Elongation, per cent	Hardness Shore A	Swell, per cent	Cracking	Type of Turbo Oil	Tensile Strength, psi	Elongation, per cent	Hardness Shore A	Swell per cent	Cracking	Type of Turbo Oil
500	230	49	42.6	None	Esso	360	110	71	27.2	None	Penola
800	90	71	37.6	None	Penola	1000	100	64	42.7	None	Penola
940	80	79	30.8	None	Penola	1240	90	72	35.7	None	Penola
840	130	77	43.0	None	Penola	830	200	75	44.7	None	Penola
1010	110	77	37.1	None	Penola	940	120	80	41.3	None	Penola
580	90	64	40.9	None	Penola	920	110	57	46.9	None	Penola
840	90	70	33.9	None	Penola	970	90	65	42.8	None	Penola
400	80	56	43.1	None	Penola	550	100	47	53.2	None	Penola
460	80	58	41.2	None	Penola	620	110	50	53.5	None	Penola
560	70	59	39.3	None	Penola	600	90	53	52.2	None	Penola
690	70	60	40.3	None	Penola	790	110	53	45.5	None	Penola
640	80	51	41.9	None	Esso	530	100	46	50.2	None	Esso
760	130	52	46.2	None	Penola	820	150	46	55.4	None	Penola
390	80	55	44.4	None	Penola	610	120	48	58.7	None	Penola