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A STUDY OF THE EFFECTS OF NUCLEAR RADIATIONS ON ELASTOMERIC COMPOUNDS AND COMPOUNDING MATERIALS

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FOREWORD

This report was prepared by the B.F. Goodrich Company Research Center, Brecksville, Ohio, under Supplemental Agreement S3(57-1027), Project No. 2133, Task No. 73071, Nuclear Radiation Resistant Materials. The contract was initiated under Project 1252, "ANPP Development Support Project," Task No. 73023, "Radiation Effects," on 1 January 1954. This is the fourth annual report, designated Part IV. The work was administered under the direction of the Materials Laboratory, Wright Air Development Center, Wright-Patterson Air Force Base, Ohio, with Mr. Warren R. Griffin as project engineer.

Said report covers work conducted from 1 January 1957 to 28 February 1958.

The text was compiled by Mr. John W. Born from individual summary reports of the technical men who conducted the separate phases of research. Their names appear above the sections in the text with which they were directly concerned.

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The Aircraft Laboratory at WADC has conducted tire tests under the supervision of Mr. W. M. Roberts. The Materials Laboratory at WADC has supplied fluorinated elastomers for study.

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Finally, recognition and appreciation are due to the assistants and research mechanics who have contributed so importantly to the total accomplishment.

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ABSTRACT

The research in this fourth year under the contract included the following: fundamental studies of the mechanism of radiation damage to high polymers and of the influence of molecular structure on the relative degree of radiation effects; basic attempts to protect textile filaments and cords from radiation damage; further screening of anti-rads for other conventional and newer elastomers; a search for and attempted synthesis of new and more effective anti-rads; applied studies of the separate and combined effects of heat and radiation on aircraft rubber compounds; irradiation and full-scale indoor testing of aircraft tires, with and without potential anti-rad protection; and formulation of a detailed program for the next end-item study, which will deal with "O" ring seals.

The work which is reported thus included fundamental, basic, applied, and enditem research. The results are stated very briefly in the following summary.

PUBLICATION REVIEW

This report has been reviewed and is approved.

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SUMMARY

The gamma radiation-induced cis-trans isomerization of polybutadiene has aided in understanding energy transfer in polymers. It has also indicated methods of studying the mechanism of anti-rad protection. As a bonus it has been made the basis for a promising new radiation dosimeter.

The rate of gel formation of butadiene-acrylonitrile copolymers during irradiation increases with increasing molecular weight. In the lower range of radiation exposures this rate increases with increasing acrylonitrile content. In the higher exposure range such copolymers showed increased resistance to crosslinking with increasing acrylonitrile content up to about 30 percent. Increasing the styrene content of butadienestyrene copolymers increased their resistance to radiation-induced crosslinking.

Various types of tire cord have been rated in terms of radiation resistance. Anti-rads consistently protected the cords from radiation damage. Dacron showed the best retention of stress-strain properties during irradiation, both with and without anti-rad present. Potential anti-rads have been mixed homogeneously into polymers from which filaments have been spun and drawn successfully. Their radiation resistance is now being evaluated.

New potential anti-rads were both synthesized and purchased for evaluation. This second screening study bore out the results of the first one and revealed new anti-rads. It further indicated the importance of having a good anti-oxidant present with the anti-rad. A screening study of potential anti-rads for elastomers other than natural rubber is nearing completion.

The separate and combined effects of heat and radiation on practical aircraft rubber compounds have been measured at room temperature and 158°F. so far. The effectiveness of anti-rads in such highly compounded stocks has been marginal, reaching a maximum of three-fold protection.

Three aircraft tires have received radiation exposures of 8.4 x 10⁹ ergs per gram DC, and the fourth is now being irradiated. The two irradiated tires of conventional composition still possessed some landing capabilities. The other two tires, which contain an anti-rad, have yet to undergo similar testing at the WADC Aircraft Laboratory.



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INTRODUCTION

Nuclear radiation can cause damage to rubber products in military aircraft. The essential goals of this contract research are to define and prevent that damage.

In the present sense, nuclear radiation is the radiant energy which emanates from nuclear reactions, specifically in nuclear reactors and radioisotopes. As radiation passes through a material, it transfers part of its energy to the material. The percent transfer depends upon many factors. Among them are the kind and energy of the radiation, the radiation flux, and the chemical composition and density of the material. The results of the energy transfer may also depend upon a number of factors. They include the state of the material (solid, liquid, gas), its class (molecular, atomic, ionic) and its form if solid (crystalline, amorphous), and its environment.

Elastomers, the substantial basis of rubber compounds, are both organic molecular solids and high polymers. The principal immediate effects of high energy radiation on polymers are ionization, excitation, and transmutation. All of the changes which are induced in the polymer by irradiation ensue from these effects. The critical results are changes in molecular structure which produce changes in physical properties and service performance.

This report is Part IV of a series dealing with efforts to define and understand the detrimental effects of nuclear radiation on elastomeric materials and to increase the resistance of such materials to radiation damage. Part I dealt with a broad survey of radiation effects on some 200 rubber compounds as a function of elastomer, curing system, reinforcing filler, non-reinforcing filler, antioxidant, and antiozonant (1). It provided the basis for more specific, directed research; and it resulted in the discovery of inhibitors of radiation damage, which were named anti-rads. Part II concerned an extensive screening program to obtain superior anti-rads based on a carbon black-reinforced natural rubber screening compound (2). It also involved dynamic testing of irradiated samples of ASTM rubber compounds and preliminary efforts to relate molecular structure of the polymer to radiationinduced changes in physical properties by infrared and mass spectral analysis and by measurements of continuous stress relaxation at various temperatures during irradiation. Part III dealt with the extension and completion of the studies of Part II and laid the experimental foundations for radiation testing of actual aircraft rubber end items such as tires, hose, "O" ring seals, fuel cells, and wire insulation (3). It made even more apparent the need for an expansion of emphasis toward both more basic studies and end-item testing. The work of Parts I, II, and III supported and led straightforwardly to the research discussed in this report. It

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revealed first that, while anti-rads provide significant protection against radiation damage, greater protection is needed. Achieving ultimate radiation resistance may well require the synthesis of new elastomers with inherent superior resistance. Second, it indicated the likelihood that the resistance of rubber end-items to radiation damage may be greater than expected. That is, resistance as measured in terms of the actual service performance of an irradiated end-item may exceed the apparent resistance as judged by laboratory tests of irradiated component materials of the end-item.

SECTION 2

FUNDAMENTAL RESEARCH

Radiation research prior to this year increasingly emphasized the need for fundamental studies relating to radiation effects on high polymers. Specifically, the most effective development of anti-rads and radiation-resistant polymers would require a working knowledge of the mechanisms of radiation damage and of protection against it. Greater, more permanent radiation resistance was required than compounding research alone could probably provide. Recognition of this need led to the following three studies.

2.1. Mechanisms of Radiation Damage and Protection

(This research was conducted and reported by M. A. Golub.)

2.1.1. Introduction

The study of the mechanisms of radiation-induced changes in elastomers under the subject contract began this year. It resulted from the need for greater, more permanent resistance to radiation damage than anti-rads now appear able to provide. As an initial effort it was considered worthwhile to explore possible analogies between photochemical and radiation chemical processes in high polymers. This approach was selected to obtain a better understanding of energy transfer in polymer systems during irradiation. The modes of energy dissipation are deemed very important in the overall process of radiation damage and protection. The photosensitized cis-trans isomerization of polybutadiene had been studied previously (4). Therefore, it was logical to consider the possibility of achieving a similar reaction initiated by gamma radiation in place of ultraviolet radiation. Experiments then showed that gamma radiation did indeed convert the cis double bonds into trans double bonds. Polybutadiene of high cis content in solution was exposed to Cobalt 60 gamma rays in the presence of a suitable sensitizer, such as an organic bromide, disulfide, or mercaptan. The isomerization proceeded toward an equilibrium cis/ trans ratio of 5/95. This discovery afforded a novel experimental introduction to the energy transfer problem. As a bonus it also provided the basis for a new, practical, stable radiation dosimeter for exposures up to 10^{10} ergs per gram DC



or higher (Appendix II). Further research led to an "unsensitized" gamma rayinduced isomerization in the solid state as well as in the solution. That is, the same cis-trans conversion occurred, although none of the above sensitizers was added to the polymer before irradiation. This discovery indicated the possibility of measuring radiation doses up to 10^{12} ergs per gram DC. The possibilities which were offered by a detailed consideration of this reaction led to two studies. First, the kinetics of the isomerization sensitized by organic bromides were investigated. This was followed by a similar investigation of the much slower "unsensitized" isomerization. Some work deserved to be done on the mercaptyl radical-sensitized reaction, but time did not permit any detailed treatment of this aspect. Much of the work described below was incorporated in a paper which was presented at the 132nd Meeting of the American Chemical Society. The paper is scheduled to appear in the April 1958 issue of the Journal of the American Chemical Society.

2.1.2. Experimentation

The high cis-polybutadienes which were used in this work were supplied by R. J. Minchak. They had been prepared previously by stereospecific catalysis and had viscosity average molecular weights of 300,000-500,000. They contained approximately 91% cis-1, 4 and 5% trans-1, 4 addition units. The rest of the unsaturation consisted of vinyl side groups resulting from 1, 2 addition of monomer (see repeating structural units below).

$$-CH_{2}$$
 H
 $C = C$
 H
 $-CH_{2}$
 $C = C$
 H
 $-CH_{2}$
 CH_{2}
 CH_{2}
 CH_{2}
 CH_{2}
 CH_{2}
 CH_{2}
 CH_{3}
 CH_{4}
 CH_{2}
 CH_{2}
 CH_{3}
 CH_{4}
 CH_{4}
 CH_{5}
 CH_{5}

The latter type of double bond was not involved in this study. Only the percent cis double bonds (or cis/trans ratio) in the polymer before and after irradiation was significant in this study. The initial cis/trans ratio of the polymer was thus taken to be 95/5.

Stock solutions of polybutadiene in benzene were prepared to a concentration of about 10 grams per liter for gamma irradiation. Various amounts of allyl, ethyl, or ethylene bromide were added to the solution as sensitizers. Portions of the sensitized solutions were placed in ordinary screw-cap glass bottles. They were irradiated at room temperature for various periods of time. A 360-curie Cobalt 60 gamma-ray source was used which had a flux of 1.2×10^7 ergs per gram DC per hour. Aliquots of the irradiated solutions were withdrawn after receiving the desired radiation doses and were analyzed for residual cis content.

It was found that gelation of the polymer set in during irradiation in nitrogen gas after an exposure of about 4×10^7 ergs per gram DC. This gel formation made it impossible to obtain infrared spectra after the higher doses. Therefore, irradiations were ordinarily carried out in air. Irradiation in air avoided gelation but led to very rapid degradation to quite low molecular weights. However, the rate of isomerization was independent of the initial molecular weight. Also, it was unaffected



by the accompanying chain scission during irradiation in air and by the crosslinking at least up to the point of gelation during irradiation in nitrogen. Moreover, although the molecular weight of the polymer irradiated in air rapidly became very low, the spectrum was still acceptable. Except for the differences in cis/trans ratio, the spectrum resembled that for a customary high molecular weight polymer.

Thin solid polymer films were cast from the irradiated benzene solutions onto rock salt plates. The polymer structures were determined by infrared absorption measurements of the films with a Perkin-Elmer Model 21 spectrophotometer. These analyses were made by M. J. Ferguson of the Infrared Analytical Section. Analysis was based on the characteristic absorption bands for the cis and trans double bonds at 13.6 and 10.35 microns, respectively. The cis/trans ratios were calculated from the optical densities of these two bands. Use was made of the fact that the extinction coefficient of the trans band is 1.63 times that of the cis band. Typical infrared spectra of the high cis polybutadiene before and after gamma irradiation with allyl bromide as sensitizer are shown in Figure 1. The observed decrease in the cis band corresponds quantitatively to the observed increase in the trans band, if the absorbance of the methylene band at 6.9 microns is taken as an internal standard. The polymer was isomerized from the initial cis/trans ratio of 95/5 to a structure having a cis/trans ratio of about 10/90 in the example given in the figure. At room temperature the isomerization can proceed further until an equilibrium ratio of about 5/95 is attained. In determining the optical density of the trans band the background in each spectrum was corrected for the overlapping bands at 10.1 and 11.0 microns. The latter bands are due to the cis and the vinyl configurations about the double bonds, respectively. The nature of the corrections are shown by the dotted lines in Figure 1. The spectrum of the irradiated polymer was compared with that of a high trans polybutadiene polymerized directly by stereospecific catalysis. Apart from any change in molecular weight as a result of irradiation, the chief alteration in polymer structure was that of a true cis to trans isomerization.

The change in molecular weight during irradiation was indicated by changes in dilute solution viscosity. Measurements were carried out on polymer solutions exposed to gamma rays for various periods of time with and without sensitizer, in air and in nitrogen. Standard Ostwald-Fenske viscometer measurements were made at $20.0 + 0.1^{\circ}$ C.

The polybutadiene contained the antioxidants N, N' di-2-naphthyl-p-phenylene diamine and di-tertiary-butyl-hydroquinone. Their initial content was about 0.50% and 0.25% by weight, respectively, based on polymer. This polymer underwent a small autocatalytic increase in the rate of isomerization. The further addition of about 0.50 weight percent of hydroquinone completely suppressed the increase. On the other hand, the use of N, N'-diphenyl-p-phenylene diamine or some other antioxidant in place of the hydroquinone resulted in a very pronounced autocatalytic effect. Accordingly, the optimum hydroquinone concentration of about one percent, based on polymer, was used throughout this work.

Typical kinetic plots appear in Figure 2 for the isomerization of polybutadiene with allyl bromide as sensitizer. The corresponding plot is included for the unsensitized isomerization, also in solution. These plots correspond to the expression



for a first-order reversible process

$$k' = -\frac{2.303K}{(1 + K)} \cdot \frac{d \log (1 - x/x_e)}{dR}$$
 (1)

where K is the quilibrium constant for the cis-trans interconversion, x is the percent of cis changed to trans during a radiation exposure R, and x_e is the percent cis converted at equilibrium. The quantity k' is related to the rate constant k for the sensitized isomerization of the cis double bonds through the further expressions

$$k' = k_s + k_u$$
 and (2)

$$k' = k \text{ [bromide]}^b + k_u$$
 (3)

Here k_u for the unsensitized reaction is just equal to k^t in the absence of bromide. The quantity k_s represents the rate of sensitized reaction for a given concentration of bromide. The values of k_s were graphed on log-log paper versus the concentrations bromide used in the various isomerizations. The value of the slope of the best line through the points of this graph, which is the exponent b in Equation (3), was 0.45. That value indicated that the rate of isomerization was somewhat less than half-order in organic bromide concentration. A similar 0.45 - order in bromide concentration was obtained for the rates of isomerization using ethyl bromide or ethylene bromide. Since the cis/trans ratio was initially 95/5 and became 5/95 at equilibrium, K and x_e had the values 19 and 90%, respectively. The value of ΔF^0 for the equilibrium cis-trans interconversion reaction in benzene at 25°C. was thus approximately - 1.74 kilocalories per mole.

The results of the various rate measurements are summarized in Table I. The reaction yield G is defined here as the number of cis double bonds isomerized per 100 ev of energy absorbed by the entire solution. Since the value of G falls off logarithmically with dose, a value G_0 was determined for each isomerization by extrapolating the G values to zero radiation dose. These G_0 values are given in the table. In the calculation of the reaction yields, allowance was made for the different absorption coefficients of the various components of the irradiated systems. Consider further the idealized quantity G_0^S , which may be considered the analog of the chemical quantum yield in the corresponding photochemical reaction. It is evaluated as the number of cis double bonds which are isomerized per 100 ev of absorbed energy. Said energy is that absorbed by the bromide itself through the direct action of the gamma rays. There is assumed to be no energy transfer between the solvent or polymer and the sensitizer, or vice versa. The values of G_0^S appear in the last column of Table I.

Although the yield G_0 increased with increasing concentration of the sensitizer, the value of G_0^s decreased markedly. In this respect G_0^s was a measure of the bromide sensitization. Thus, the value of G_0 increased only about seven-fold for a two hundred-fold increase in the concentration of sensitizer. At the same time, the efficiency of sensitization dropped to about one twenty-fifth of that for the solution having the smallest bromide concentration.



Figure 3 shows the change in the reduced viscosity (see Glossary) of the polybutadiene in benzene solution as a function of Cobalt 60 gamma irradiation in air versus in nitrogen and in the presence and absence of sensitizer. The molecular weight dropped rapidly from about 380,000 to 8,000 - 10,000 during irradiation in air with allyl bromide. On the other hand, the molecular weight increased during similar irradiation in nitrogen to approximately 570,000. Beyond this point extensive gelation set in. Polymer degradation also occurred on gamma irradiation in the absence of a sensitizer, being somewhat slower in nitrogen than in air.

Irradiations were also carried out at various temperatures over the range from 30° to 60°C. A set of polybutadiene solutions, all with the same concentration of allyl bromide, was studied. It was found that the kinetic plots superimposed, indicating that the radiation-induced isomerization was independent of temperature.

Investigations showed that it is possible to induce a solid state isomerization of the polymer by very prolonged gamma irradiation. Films were cast from benzene solution onto rock salt crystals for infrared analysis. Solid pieces of polymer could not be used, because they became so highly crosslinked by an exposure of 1.2×10^8 ergs per gram DC. The polymer became completely insoluble, and no suitable infrared samples could be prepared. An exposure of 5×10^9 ergs per gram DC resulted in a 2.5% decrease in the cis content of the polymer as compared with a 23% change in the unsensitized solution.

The results of the sensitized isomerization of polybutadiene can be explained by the following mechanism:

$$RBr \longrightarrow R \cdot + Br \cdot$$
 (I)

$$Br \cdot + Br \cdot \longrightarrow Br_2$$
 (V)

$$Br_2 + R$$
 \longrightarrow $RBr + Br$ (VI)

Other processes undoubtedly take place during gamma irradiation of the bromide-polymer-solvent system. Examples result in the radiolytic formation of hydrogen bromide, the crosslinking and chain scission in the polymer, and the usual radiolytic products from organic materials. These various other processes are considered to make a negligible contribution to the isomerization kinetics in the sensitized case. However, excited and ionized molecules or radicals from benzene may be very important in the case of the "unsensitized" solution.

Steps II to IV, inclusive, are readily reversible. They involve π -complexing between the bromine atoms and the cis and trans olefinic double bonds of the polymer. Corresponding radical transition states result which are capable of free



rotation and interconversion. The complexed bromine atoms are subsequently released, and the double bonds are re-established. The geometric configuration which predominates around the double bonds is that of the more stable isomeric form. In the case of polybutadiene, the trans form is evidently the more stable one.

Since the isomerization rate was independent of temperature, little or no energy of activation can be associated with Steps II or III. This is generally true for simple olefins. Therefore, it is not surprising that it is also true for double bonds in a high polymer molecule. The energy of activation for the overall isomerization is involved mainly in the formation of free bromine atoms (Step I). This energy is amply supplied by the gamma rays.

The recombination of the organic radical and the free bromine molecule, which is depicted in Step VI, constitutes in effect the reverse of the bromide radiolysis given in Step I. Reactions I, V, and VI together determine the steady state concentration of bromine atoms. This was found to be proportional to the 0.45 - power of the organic bromide concentration.

While the mechanism for the sensitized isomerization appears fairly well understood, little is known for certain about the details of the unsensitized reaction. A reasonable hypothesis is that in solution excited molecules, radicals, or ions from the solvent act in place of the sensitizer. That is, they may excite the T- electrons of the double bonds of the dissolved polymer by impact. Slow secondary electrons of 20 to 100 ev energy may also participate in the energy transfer to some extent. Impact with such slow electrons is considered to be the principal means of excitation in the solid state isomerization. The impact is considered to raise the double bonds to a higher energy level sufficient to overcome the barrier to free rotation. Interconversion between cis and trans isomers can then take place. The transitory excited species releases its excitational energy and returns to the ground state. The double bonds are again formed, but the more stable form will predominate. The possibility of sensitization by trace impurities in the otherwise unsensitized polymer system was ruled out. A sample of polymer which had been carefully purified by reprecipitation from benzene solution not only continued to show isomerization but also isomerized at the same rate as formerly.

Some recent work by Charlesby (5) is of pertinent interest. In studying the crosslinking which ionizing radiation produced in long-chain olefins, he found incidentally that molten cis and trans octadecenes isomerized to an equilibrium cis/ trans ratio of 65/35. This result was qualitatively analogous to the unsensitized radiation-induced isomerization of polybutadiene in solution. An estimate of the first-order rate constant for conversion of cis-octadecene-2 to the trans isomer was made using Charlesby's data and Equation (1) above. The rate constant obtained in this way was 1.5×10^{-11} grams of carbon per erg (DC). In comparison, a value of 4.5×10^{-11} grams of carbon per erg (DC) had been obtained for the unsensitized isomerization of polybutadiene in solution. The mechanism of the octadecene isomerization may be pictured as involving excitation of the double bonds in these molecules. Collisions of the second kind (see Glossary) with excited species from other octadecene molecules would be mainly responsible for the excitations. This process would be comparable to the assumed excitation of polymer double bonds by impact



with energetic species from benzene. Consider now that the octadecene molecule has one double bond for each 18 carbon atoms, whereas in the polymer molecule the ratio is one to four. Assume that the above mechanism is substantially correct and that energy is randomly absorbed from the gamma rays in both systems. Then, the probability that an energetic mobile entity will encounter and excite a double bond in the polymer should be roughly 4.5 times the probability in octadecene. Accordingly, the rate of isomerization of the former should be 4.5 times that of the latter. Actually, the rate constant for the polymer was 3 times that for the octadecene. The agreement is thus very close between the rate constants for two very different olefinic compounds. This close correspondence strongly suggests that the mechanisms of the two isomerizations are very similar, whatever the detailed nature of the processes may be.

2.1.3. Conclusions

Since no information is available concerning the extent of energy transfer between the polymer or solvent molecule and the sensitizer molecule, both types of G values (that is, G_0 and G_0^s) were calculated. Consider that the sensitizer molecule interacts with the surrounding solvent or polymer molecules by collision. Next, assume that the net energy transfer is negligible, that is, that the energy given up and the energy acquired by the sensitizer molecules are nearly equal. Then the G_0^s values in Table I can be regarded as a fair measure of the very high efficiency of sensitization of the polybutadiene isomerization. The efficiency increases rapidly upon dilution of the sensitizer. This result would be expected on the basis of the longer, more tortuous path that the free bromine atom can travel in its isomerizing action before it is removed by collision with an organic radical or by some other terminating reaction. Thus, at an allyl bromide concentration of 0.0144 moles per liter, the number of double bonds isomerized per 100 ev of energy absorbed directly by the bromide was nearly 10,000.

The "unsensitized" isomerization of polybutadiene in a 1% benzene solution was about ten times as rapid as in a solid polymer film. However, the G value for isomerization in solution (0.92) was about one-tenth that in the solid film (ca. 11), where the polymer alone absorbs the energy. This is demonstrated by the following calculations. Let x/y represent G for the film, where x is the number of cis double bonds isomerized and y is the amount of energy absorbed by the film in 100 ev units. Then G for the solution is $10 \times 100 \,\mathrm{y}$, or one-tenth the G value for the film. These results may be stated a second way. Absorption of 100 ev by the polymer film will result in the isomerization of 10 cis double bonds. In comparison, in a 1% polymer solution absorption of 10,000 ev by the entire solution results in the isomerization of 100 cis double bonds. Ten of the latter 100 bonds may be considered to have changed through direct absorption of 100 ev by the polymer itself. Consequently, 9900 ev of the energy acquired by the benzene molecules are responsible for the interconversion of 90 bonds, or about 110 ev per bond. As a rough approximation assume that one cis bond is converted for every 10 ev of energy transferred from the excited benzene molecules to the polymer molecule. It would follow that only about one-tenth of all such excited benzene molecules take part in this process. One would intuitively expect this numerical relationship to be dependent upon concentration, and some further experiments to investigate this point would be in order.



No doubt the problem of energy transfer is quite complex. However, further insight into the mechanism of this process would be of great value in the study of radiation damage and protection. Once the details of the benzene system are fully understood, the knowledge can be applied to a consideration of the radiation sensitivity of butadiene styrene copolymers. This work is planned for 1958.

It must be pointed out that the formation of trans structure in the solid polymer may be due partly to new vinylene unsaturation. Such unsaturation could result from very large gamma radiation doses. Until the solid state isomerization study is completed, it will not be possible to say just how many trans double bonds, if any, are formed in this manner and how many actually result from conversion of cis double bonds. This complication, which is presumably absent or negligible in the solution case, is an important factor to be considered. It is known that trans vinylene groups are produced in such molecules as polyethylene, polymethylene, and octacosane by gamma irradiation. Values of G for trans formation of about 2 have been generally reported for these materials. It is entirely possible that the overall G value of 11 for the solid state represents a combination of vinylene formation and cis-trans isomerization.

The cis-trans isomerization of polybutadiene is proving to be the basis for a radiation dosimeter of great potential usefulness. The particular merits of this system in addition to its complete stability, before and after irradiation, are several. Since the polybutadiene is an elastomeric high polymer, it is an ideal material for the dosimetry in studies of radiation effects on elastomers and elastomeric compounds. Equally important, the polybutadiene system can measure accrued radiation doses ranging as high as 5×10^9 to probably 1×10^{12} ergs per gram DC. The latter feature plus the compactness of such a dosimeter permit packaging it among the samples for the total exposure period. The procedure would make use of a standardized high cis-polybutadiene sample in approximately a 1% benzene solution. A graph depicting the rate of isomerization of this polymer in the presence of a given concentration of sensitizer would be constructed similar to Figure 2. For doses up to 5 x 10 ergs per gram DC the polymer solution can contain about 0.01 moles per liter of allyl bromide. For doses up to 5 x 10 10 ergs per gram DC the polymer solution should be employed without a sensitizer. Furthermore, for doses up to 10^{12} ergs per gram DC a thin polymer film which has been cast on a rock salt crystal should be used. After exposure of the polymer to some unknown radiation dose, the infrared spectrum of the polymer would be graphed. The residual cis content would be calculated, and the dose corresponding to this amount of cis double bonds would be read from the appropriate graph. Both film and solution dosimeters are being used in the current aircraft tire irradiation at the MTR Gamma Facility (see Section 4.1 below) as part of a general evaluation of the new dosimetry technique.

The emphasis in future research will gradually be shifted to other promising topics of the fundamental investigation of the mechanisms of radiation damage and protection in elastomers. These topics include new detailed consideration of energy transfer processes; the role of aromatic substituents or additives in inhibiting radiation effects; kinetic treatment of the competing processes of crosslinking and chain scission; and the effect of type and arrangement of bound monomer units in the polymer chain on susceptibility to radiation damage, for example in graft, block, and



ordinary copolymers of the same overall composition. Much of this work will be conducted with butadiene-styrene copolymers. A basic search will also proceed for materials having non-destructive gamma ray absorptivity analogous to ultraviolet absorbers. Such absorbers would be tested as rubber additives to inhibit radiation damage.

2.2. Radiation Resistance Versus Molecular Structure

(This research was conducted and reported by J. A. Glantz and R. G. Bauman.)

2.2.1. Introduction

The research prior to this year created a growing interest in basic studies of the relationships between molecular structure of polymers and radiation-induced changes in physical properties. In Section I reference was made to the early work of this type involving infrared and mass spectral analysis and stress relaxation. The results were not very enlightening in this respect, largely because of the complexity of composition of the rubber samples. It was recognized that the further studies should involve samples either of pure polymer or of polymer plus a small amount of some particular additive of interest. Also, evaluation tests should avoid interference by oxidation with interpretation of the results. It was intended that such studies should lay the foundation for the development of radiation-resistant polymers.

2.2.2. Experimentation

The polymers which were chosen for study were natural rubber (hevea), Neoprene GN, butyl rubber, and two series of copolymers. One series consisted of copolymers of butadiene and styrene and the other, of butadiene and acrylonitrile. In both series the charge weight percents of butadiene ranged from 50 to 90. The copolymers were prepared according to standard recipes. The purpose of this study was to make a preliminary determination of how variations in polymer composition influence resistance to radiation damage. Samples were prepared from molded sheets of both pure polymer and gum compound 0.025 of an inch thick. The pure polymers were all molded into sheets at 212°F. The recipes and the time and temperature of molding and vulcanizing (curing) of the gum polymer compounds appear in Table II. Sample strips which measured 2.75" x 0.25" were then cut from the molded sheets and were extracted for 16 hours to remove soap, catalyst, or other impurities. The natural rubber samples were refluxed in acetone and the samples of the other polymers, in methanol in Soxhlet extractors. After extraction the strips were vacuum-dried and stored in nitrogen gas until used.

The samples thus processed were wrapped in aluminum foil and exposed to 8.4×10^8 , 1.7×10^9 , 2.5×10^9 , 4.2×10^9 , 5.9×10^9 , and 8.4×10^9 ergs per gram DC from Cobalt 60 gamma radiation. Following irradiation, volume swell and percent gel were measured as a function of radiation exposure. The sample lengths were determined both before and after swelling for 24 hours in a suitable swelling agent at room temperature. Each swelling agent contained one weight percent phenyl beta-naphthyl amine to protect the polymers from oxidation. Table III identifies the swelling agent and presents the experimental data for each polymeric



material. The ratio ∞ , which is proportional to volume swell, is defined as the ratio of the final, swelled length L to the initial, unswelled length L₀. Figures 4 and 5 show the effect of the various styrene and acrylonitrile comonomer contents in the two polymer series upon volume swell. These results in turn reveal the influence upon the net degree of radiation-induced crosslinking. That is, the greater the extent of net crosslinking becomes, the less swelling occurs.

The procedure for measuring the sol and gel contents of the irradiated materials employed the following techniques. Approximately 0.2 gram of the irradiated sample in 100 ml. of an appropriate solvent was placed in a constant temperature bath at 50°C. The solvent was drained off and fresh solvent was added twice a day for one week. At the end of this period the samples were removed and dried in a vacuum oven for 24 hours at approximately 50°C. Each sample now consisted solely of insoluble gel. All sol had been extracted. The final weight divided by the initial weight, multiplied by 100, gave the percent gel. The percent sol was calculated simply by subtracting the percent gel from 100%. Table IV presents the sol-gel data. Figures 6, 7, and 8 express the percent gel as a function of the radiation exposures for these stocks.

2.2.3. <u>Discussion of Results</u>

If the effect of molecular structure upon radiation-induced crosslinking in polymers were known, it would theoretically be possible to synthesize a radiation-resistant polymer. The data from the studies of natural rubber, Neoprene GN, and butyl rubber have not been completely evaluated yet. It is expected that the results will appear in the next quarterly report. However, the evaluations for the two series of copolymers with various monomer ratios have been completed and represent interesting results.

The swelling measurement is the more revealing result in that it details the molecular weight between effective crosslinks. The sol-gel measurements determine the amount of polymer that has been crosslinked past the limit of solubility. Figure 4 shows that the styrene units in the polymer have a beneficial effect. At each radiation exposure the crosslinking efficiency decreased with increasing styrene content. In the case of the butadiene-acrylonitrile copolymer series the evaluation of results is somewhat more complex. Increasing the acrylonitrile content has a beneficial effect up to an exposure of 2.5 x 109 ergs per gram DC and an acrylonitrile content of 30 percent by weight. Above this exposure the crosslinking efficiency increases with increasing acrylonitrile content and with exposure, as shown in Figure 5. The percent gel data in Figures 6, 7, and 8 show a general sharpincrease in gel content with irradiation, even at the lowest exposure. All the gelation curves approach an asymptotic percent gel value which is considered to indicate the equilibrium ratio of crosslinking to chain scission induced by irradiation. In order of decreasing ratio of chain scission to crosslinking, the elastomers are rated as butyl rubber, natural rubber, Neoprene GN, SBR, and Hycar. Unlike in the other elastomers, chain scission predominates in butyl rubber. In contrast, the order of decreasing rate of crosslinking is Hycar, SBR, Neoprene GN, and natural rubber. The initial percent gel increases with acrylonitrile content as shown in Figure 8. The curves for the two copolymer series are more informative



when a correction has been applied for the initial percent gel. The percent of gel which is induced by irradiation may be calculated with the following equation:

$$F = (F_0 - a)(100\%)/(100\% - a)$$

where F is the percent gel resulting from irradiation,

Fo is the measured percent gel, and

a is the initial percent gel.

The corrected forms of the curves in Figures 6, 7, and 8 appear in Figures 9, 10, and 11, respectively.

2.2.4. Conclusions

Increasing the styrene content in butadiene-styrene copolymers increased resistance to radiation induced crosslinking. Butadiene-acrylonitrile copolymers showed increased resistance to such crosslinking with increasing acrylonitrile content up to 30 weight percent of the latter. The elastomers rated in order of decreasing ratios of chain scission to crosslinking during irradiation are butyl rubber, natural rubber, Neoprene GN, SBR, and Hycar (butadiene-acrylonitrile). In order of increasing rates of radiation-induced crosslinking the elastomers were natural rubber, Neoprene GN, SBR, and Hycar. The initial percent gel also increased with increasing acrylonitrile content in the butadiene-acrylonitrile series.

2.3. Development of Radiation-Resistant Polymers

(This research was conducted and reported by E. Witt and T. R. Paxton.)

2.3.1. Introduction

The ultimate objective of this contract is the development of elastomeric components, particularly end-items, which will be immune or at least resistant to deterioration by ionizing radiation. A logical attack upon this problem is to learn how polymer structure affects susceptibility to radiation-induced deterioration. Such fundamental knowledge is essential to the preparation of an elastomer which is inherently radiation-resistant.

The effects of ionizing radiations on polymeric materials comprise a broad subject, which many persons have studied. It is generally accepted that the primary immediate effect of irradiation is bond cleavage within the polymer to produce free radicals. The radiation may interact to remove substituent groups or atoms or to sever main chains. Subsequent reactions depend upon the nature of the free radicals. Disproportionation, recombination, propagation through residual double bonds, or radical transfer with neighboring structural groups may occur. The principal net final change in structure is crosslinking or chain scission or a combination of both. Attempts to correlate the observed changes with particular initial polymer structures have been partially successful (6, 7). Charlesby and fellow workers have made fundamental studies of radiation-induced changes in the structure of polymers such as polystyrene, polyisobutylene, and polyethylene (8). These studies showed that the number of crosslinks is proportional to the radiation dose in polymers which



undergo predominant crosslinking. They also showed the important influence of molecular weight distribution upon the rate of gel formation with irradiation. However, few systematic studies have been made of a series of copolymers of selectively varied monomer ratio which would show the effect of molecular structure on susceptibility to radiation change (9).

Irradiation studies of simpler molecules in solution give evidence that one molecular component can protect the others in the solution. It is therefore reasonable to scan the range of monomer ratios for a copolymer system in search of similar protective effects and of a monomer ratio which leads to minimum radiation damage. This type of investigation was encouraged by the results of the research which is described above in Section 2.2. Much of the previous work by other organizations has dealt with changes in commercially available rubber compounds in the cured state. In order to avoid the attendant complicating effects of the added compounding ingredients, the present investigation has been limited to raw polymers. The chemical reactions between chain segments which result from the high-energy activation by nuclear radiation can be assumed to be the same whether the polymer is initially crosslinked or not. Any differences would be due to the vulcanizing agents and were to be avoided.

The copolymer system selected for this study was butadiene-acrylonitrile (NBR), largely because of the broad experience of the B. F. Goodrich Company with this system. Polymerization and evaluation techniques were thus already available. Much valuable time could be saved by eliminating preliminary ground work. The butadiene-styrene (SBR) copolymer also has not yet been studied extensively from the present viewpoint. It is worthy of investigation, since it is known that the arcmatic ring in the styrene unit provides radiation protection.

2.3.2. Experimentation

Polymers were prepared using standard techniques. They were evaluated by measuring percent gel (insoluble polymer), swelling index of the gel, and viscosity of the sol (soluble portion of the polymer) after various gamma radiation exposures. The basic polymerization recipe is as follows:

Ingredient	Percent by Weight
Butadiene Acrylonitrile Water S. F. Flakes Ammonium Persulfate Sulfole B-8	Variable Variable 200 5.0 0.5 0.5

Polymerization Temperature: 40°C.

Variations were made in monomer ratios as indicated in the discussion of results below (Section 2.3.3). For one series the Sulfole B-8 chain transfer agent modifier was varied to provide a variety of molecular weights. In one of the preliminary



series of polymer preparations (81GH1834), the persulfate catalyst was supplemented by azoisobutyronitrile (Porofor N), using 0.3 parts per hundred parts of monomers (phm) of each. Polymerizations were carried out using standard quart bottle emulsion techniques, charging a total of 100 grams of monomer. At the desired percent conversion of monomers into polymer the reaction was short-stopped, and the charge was promptly vented. The unreacted monomer was removed from the polymer system in a Rinco Rotating Evaporator at a reduced pressure of 4-6 cm. Hg. After thus being stripped, the latex was creamed with sodium chloride and coagulated with ethyl alcohol. The crumbs of polymer were washed with hot water until free from chloride ion and were dried partially in a circulating air oven and finally in a vacuum oven at 60°C. Samples of dried polymer to be irradiated were placed in glass ampoules, vacuum-flushed with nitrogen, and evacuated to about 0.5 microns Hg pressure. The ampoules were then sealed by fused-closure while still evacuated. The only exceptions to this general procedure are noted in the discussion of results. _The ampoules were exposed to Cobalt 60 gamma radiation at a flux of 1.10 x 10 ergs (that is, 1.10 joules) per gram of carbon per hour. The total exposures ranged from 0.21 to 67.12 joules per gram of carbon. The doses are reported in terms of energy absorbed per gram of dry carbon (Appendix II). Other samples were submitted to the Analytical Section for determination of nitrogen content, heat loss, and ash. Heat loss and ash were usually negligible, and nitrogen analysis was used to calculate the actual monomer ratio which was obtained.

Standard sol-gel screens were used in the determination of percent gel and swelling index. Chlorobenzene was chosen as the solvent because it has a solubility parameter appropriate for this polymer system and is not highly volatile. In order to guard the polymer against air-curing 0.2 percent Santovar A, an antioxidant, was dissolved in the chlorobenzene. After static swelling and dissolution for 20 hours, most of the solution was decanted from the sol-gel cup and filtered through a medium-grade glass frit. It was found that in order to obtain reproducible viscometer flow times, the filtration must be repeated until the solution passes through the filter easily. The swelling index was determined from the swollen weight of gel on the screen after 30 minutes' draining. The total solid content of the filtered solution was measured by evaporation of the solvent to determine the percent soluble. The percent gel was calculated by difference. Hence, it included both the gel left on the screen and that removed by filtration. Small amounts of very highly swollen gel were sometimes not retained on the screen, and in these cases the swelling index was indeterminable.

Viscosity measurements were made with Ostwald-Cannon-Fenske capillary pipettes having flow times of about 40 to 60 seconds at 25.0°C. Flow times were measured at four concentrations, if the percent gel was high enough to make such successive dilutions significant. The intrinsic viscosity was obtained as usual by extrapolation of $\gamma_{\rm sp}/c$ to c=0. Corrections for kinetic energy were not applied, since the relative comparison of values was of principal interest.

Since the predominant overall result of irradiation proved to be crosslinking to form gel, it was desirable to start with polymers having low gel content before irradiation. To accomplish this it was necessary to halt polymerization before it



was complete. Furthermore, it was also necessary to give the polymer some slight protection against air during coagulation and drying. Presumably a short-stop must provide such protection. This requirement introduced a difficulty, because many of the effective short-stops are very similar to compounds which inhibit radiation damage, that is, anti-rads (3). Therefore, a series of similar copolymers were prepared, using the same charge ratio of 67/33 for butadiene/acrylonitrile. Each polymerization was stopped at about 80% conversion, as shown in Table V. A portion of each copolymer was aged for 72 hours in air at 60°C. Such treatment was similar to, although more severe than, that which is customary in preparing and drying samples. Other portions of the same copolymers received various gamma ray exposures. Analysis for percent gel in these samples showed that hydroquinone and sodium dimethyl dithiocarbamate (SDD) protected adequately against air aging without noticeably changing the effect of radiation. For this reason 0.25 parts per hundred parts of rubber (phr) of hydroquinone was selected as the short-stop for all subsequent preparations.

2.3.3. Discussion of Results

After several preliminary preparations a series of copolymers of various composition was obtained which had low gel content and similar molecular weight. Table VI presents the data. Comparison of the monomer charge ratios with the resulting acrylonitrile contents of the polymers shows that it is increasingly difficult to prepare copolymers containing much more than 35% VCN. This was the reason that the high-VCN charge, 81GH1854F, was stopped at low conversion and consequently has such a low molecular weight. It is an inherent disadvantage of the butadiene-acrylonitrile system that the complete range of copolymer composition is not available for study.

Figure 12 presents a typical set of results for the irradiation of a 67/33 butadiene-acrylonitrile copolymer. As the radiation exposure increased, the gel content grew larger and approached a limiting value above 90 percent. This indicates
that crosslinking is the predominant reaction. Concurrently, with increasing exposure and hence crosslinking, the swelling index of the gel decreased rapidly to a
fairly low value. The higher molecular weight polymer present requires fewer
crosslinks for gelation and so is the more rapidly converted to gel by random crosslinking. Therefore, the intrinsic viscosity of the remaining soluble portion decreased with increasing radiation exposure. Whether the viscosity would have
reached a constant limit or would have increased eventually with continued irradiation cannot be decided from these data. Furthermore, as the gel content becomes
very high, the polymer concentration in the soluble fraction becomes very low. As
a result the measurement of intrinsic viscosity is not sufficiently precise to have
meaning. For this reason viscosity measurements were sometimes omitted entirely.

The changes in gel content and swelling index are shown in detail in Figures 13, 14, and 15. Figure 16 shows the change in gel content and swelling index as a function of the acrylonitrile content of the copolymer and of the radiation exposure. Gel formation is slightly more rapid for the copolymers with higher acrylonitrile content. The swelling index at any given exposure shows a more pronounced variation with composition. The direction of the variation is consistent with the gel



data. That is, crosslinking is more rapid the higher the percent acrylonitrile. However, part of the variation in swelling index must be due to the fact that the polymer-solvent interaction coefficient changes with polymer composition. This change proceeds in such a way as to give lower swelling with high VCN content in the copolymer.

The gel-formation curves (Figures 13, 14, and 15) exhibit a peculiar feature! namely, the curve rises rapidly to a plateau and then later rises again, apparently approaching a second plateau. All five polymers show this same behavior. Previous experiments had shown that the curious interruption in the initial gel formation was not due to shortstop. A comparison of radiation effects in air versus in vacuum was made for polymer 81GH1844F. The data for both percent gel and swelling index fall on the same curves in the two cases, and both curves have plateaus. Careful consideration led to a reasonable explanation for the plateau interruption in terms of molecular weight distributions. Suppose that the distribution contained two distinct peaks, one normal and one very high molecular weight fraction. The latter fraction could be formed by some slight crosslinking during polymerization. It would certainly be converted to gel by relatively very little radiation-induced crosslinking.

If this explanation is accepted for the observed plateau, it becomes necessary to explain why a plateau is obtained for sample 81GH1854F, whose polymerization was stopped at low conversion. It had a low intrinsic viscosity (see Table VI) and therefore would not be expected to contain crosslinked polymer. Also, the swelling indexes for this polymer seem exceptional (Figure 16). The original polymer was prepared from a monomer charge ratio of 30/70 butadiene/acrylonitrile. In this range butadiene tends to enter the polymer in greater proportion than is indicated by the charge ratio. The ratio of unreacted monomer concentrations would thus become less than 30/70. The polymer which is formed just before shortstopping could well be exceptionally rich in VCN content as compared with the more normal initial composition. The curve for percent gel versus radiation exposure has an inflection which suggests the presence of two kinds of molecules. If the exceptional species of polymer molecules present had high VCN content, they would form gel more rapidly. Furthermore, the gel would have a lower swelling index than expected.

In order to test the effect of molecular weight on the rate of gelation by gamma irradiation, two series of polymers were prepared, using the 67/33 BN/VCN ratio. The percent conversion was varied in the first series, as shown in Table VII, and the gel curves of Figure 17 were obtained. Only the sample with high conversion shows a distinct inflection suggesting a plateau, and in the case of the two lower conversions the formation of gel is delayed. These results reflect the fact that crosslinking during polymerization begins between 60 and 70% conversion for this mercaptan concentration (0.5 phm).

In the other series the amount of mercaptan modifier was varied, as described in Table VIII, from 0.5 to 10.0 phm. The resulting variations in percent gel and intrinsic viscosity with radiation exposure for this series are shown in Figures 18 and 19. Some of the data for the polymers with higher mercaptan contents have

Contrails

been omitted from the figures for the sake of clarity. The omitted data are fully consistent with the data shown. So long as no appreciable gel is formed, the radiation-induced crosslinking results in an increase in molecular weight and intrinsic viscosity. However, as the proportion of polymer in the form of gel rises, there is a corresponding drop in the molecular weight of the soluble fraction. Again, it is seen that when conditions do not lead to crosslinking during polymerization, the gel curve has no plateau or inflection. For all samples but the first, namely 81GH1859A, an initial radiation exposure is required before gelation commences. This initial exposure increases as the intrinsic viscosity of the original polymer decreases.

Flory (10) and also Charlesby (11) have made statistical calculations of the relationship between the fraction of gel in a polymer and its degree of crosslinking. Baskett (12) has applied this theory to the determination of molecular weight distributions of polyethylenes from percent gel versus radiation exposure data. Charlesby (13) has tested his equations with data for polystyrene. In his notation the relationship is

$$W_{s} = [1 + \sqrt{(1 - W_{s})}]^{-2}$$
 (1)

in which W_s is the weight fraction of soluble polymer and γ , the number of crosslinks per number-average molecule. The crosslinking function depends upon the radiation dose:

$$\gamma = \langle R/(P_0 + \beta R) \rangle$$
 (2)

in which R is the radiation dose, $\not \in$ is a constant of proportionality between dose and the number of crosslinks formed, $\not \in$ is a similar constant for scission, and P_0 is an inverse measure of the initial molecular weight. The constants $\not \in$ and $\not \in$ are characteristics of the particular polymer involved. A concise way of reporting the results of the present research would be to list values of these two parameters for the various copolymers studied. Unfortunately, the present data are not quite sufficient yet to permit this.

A log-log plot of soluble fraction against radiation exposure is given in Figure 20 for samples 81GH1859 A, B, and C. Also shown are plots of Equation (1) for $\beta/\alpha=0$ (no chain scission) and for $\beta/\alpha=0.1$. The latter curve matches the experimental slopes fairly well. However, this gives only a crude estimate of the ratio of scission to crosslinking. Results at much higher exposures will give a better determination, since the effect of chain scission becomes more prominent as the system approaches an equilibrium concentration of soluble polymer. When the data for the series of copolymer of various compositions (from Figures 13, 14, and 15) are replotted in the manner of Figure 20, the plateaus which represent the crosslinking stage prior to gelation are accentuated. Attempts to correct for this initial crosslinking by adding a constant, R_0 , to the actual radiation exposure have not eliminated the plateau. Furthermore, they cancel the possibility of a unique fit to a theoretical slope. Additional data on polymers which are free from crosslinking are needed to clarify these remaining relationships.



2.3.4. Conclusions

At the beginning of this first attempt to relate molecular structure to resistance of polymers to radiation damage, there were few guideposts. Yet the success of the attempt to develop radiation-resistant polymers depends fundamentally upon knowing such relationships. This year's research has provided the techniques for building the first guideposts.

In all the copolymers of butadiene and acrylonitrile which were studied, the predominant radiation effect is the production of crosslinks. A dynamic equilibrium between crosslinking and chain scission appears to result when the gel content reaches approximately 95 percent. On the basis of this study it appears that the rate of crosslinking increases with an increase in the proportion of acrylonitrile in the polymer. The change in the rate of scission cannot be obtained from the present experimental results.

The molecular weight distribution in these polymers has a profound effect upon their rate of gelation. The higher the initial molecular weight, the more rapid is the gel formation. Furthermore, the shape of the curve of percent gel versus radiation exposure is quite sensitive to the initial molecular weight distribution as well as to the kinds of molecules which are initially present. Research personnel who are interested in the characterization of polymers are strongly urged to become acquainted with the possible usefulness of radiation-induced crosslinking in estimating molecular weight distribution.

The methods which were developed in this research are satisfactory for the study of the effect of polymer composition on radiation stability. With some refinements it is recommended that these techniques be applied to studies of other copolymer systems. Monomer pairs which merit examination next include butadiene and styrene, particularly, and butadiene and methacrylonitrile.

In short, the goal is clear: either the percent gel limit or the rate of gelation, or both, must be reduced markedly in the majority of polymers. The techniques have been developed now for evaluating relative radiation resistance. Now a patient analysis of structure versus radiation stability of polymers has in fact begun.

SECTION 3

APPLIED RESEARCH

3.1. Introduction

Certainly the evaluation of radiation resistance of rubber end-items such as tires, hose, and seals for aircraft is vitally necessary. However, such effort is not sufficient to insure that the end-items which eventually would be used in aircraft



would possess the same required resistance to radiation damage. It is a fact that the extent of damage varies with recipe for any given rubber compound. Therefore, data for one specific end-item cannot be attributed to a similar end-item made of different rubber and plastic compounds. Until final design specifications have been established for aircraft rubber end-items for radiation applications, it is necessary to acquire extensive information on a variety of rubber polymers and compounds. From such a fund of data an approximate but useful prediction can be made of the resistance of a given end-item to radiation damage. It is a fair approximation that a rubber product is little more resistant than its least-resistant component. It is equally important to recognize that the combination of component materials may increase the resistance of one or more of the components. The following applied research has been conducted against this general background of understanding.

3.2. Protection of Present Tire Cords from Radiation Damage

(This research was conducted and reported by H. R. Voelkl.)

3.2.1. Introduction

On the basis of separate tests of individual components it is an experimental fact that at present rubber compounds will outlast textile materials with which they are associated in a nuclear radiation field. Examples of products in which such an association exists are tires, hose, and fuel cells. Therefore, if such end-items are to be made more radiation-resistant, methods are required for protecting organic textile cords from radiation damage.

Efforts to provide such protection proceeded along two lines. First, cords were treated with crosslinking agents prior to irradiation. Among other effects, irradiation causes chain scission and produces free radicals. The purpose of cross-linking was to counterbalance chain scission which occurs during irradiation. If excess crosslinks were formed beforehand, possible irradiation would regenerate an equivalent of the original polymer structure. Second, it was reasoned that chemical additives similar to the anti-rads for natural rubber might inhibit radiation damage to textile cords. Since certain aromatic chemical compounds have proved quite effective for rubber, several such agents were chosen for pioneering study. It is significant to note that this study concerns the protective treatment of commercial cords, as opposed to the case in Section 3.3 below.

3.2.2. Experimentation

Experimental polyamide (EPA), nylon, and perlon (nylon 6) tire cords were crosslinked by heating to 120°C. for 30 minutes in 10% chlorobenzene solutions of 3,3'-bitolylene-4,4'-diisocyanate (N200), diphenylmethane-4,4'-diisocyanate (N300), and hexamethylene diisocyanate (HDI). Following this treatment the cords were washed in toluene and air-dried.

In addition, tire cords of Dacron, nylon, perlon, perlon with age-resister, and rayon were treated with the following organic compounds: 2-naphthol, phenothiazine, N-phenyl-2-naphthyl amine, pyrogallol, quinhydrone, and quinone. The



treatments were accomplished by placing loops of tire cord in an Erlenmeyer flask containing 1% solutions by weight of the potential anti-rad. All anti-rads were in alcohol solution except phenothiazine, which was in benzene. A partial vacuum was applied to the system in order to fill all the voids in the cords with solution. The cords were then dried in air prior to irradiation and physical testing.

Dacron, experimental polyamide (EPA), nylon, perlon, perlon with age resister, and rayon tire cords were also treated with the following crosslinking agents in a third study: 37% aqueous formaldehyde, 25% aqueous glutaraldehyde, 2% Mondur TM (4,4',4"-triisocyanato triphenyl methane), and 2% Hylene DP (2,4,4'-triisocyanato diphenyl ether). The formaldehyde and glutaraldehyde treatments were brought about by dipping cords into hot solutions (100°C.) of the aldehydes and then curing at 150°C. for ten minutes. The triisocyanate treatments were effected by a dipping process followed by a five minute cure at 150°C.

All gamma irradiations of treated tire cords were conducted at Brookhaven National Laboratory. The radiation exposures which were used in this study were 8.39×10^7 , 4.19×10^8 , 8.39×10^8 , and 1.68×10^9 ergs per gram DC. These values correspond to 1, 5, 10, and 20 MR, respectively.

A Scott Testers IP-4 tension testing machine was used to measure the breaking strength and ultimate elongation of all the tire cords. The IP-4 Tensile Tester applies the full machine load in 18 seconds. It is an inclined plane, constant rate of load machine of the general type described in Standard Specifications for Textile Testing Machines (15).

Flexing tests of treated and irradiated tire cords were performed on the rotary flexing machine which is represented in Figure 21. The apparatus, which was developed at the Research Center, consists of rigid base (A) and supports (B), with a motor driven rod (H) extending its full length. To this rod are fastened circular metal disks. Each pair of disks is connected by two polished fixed rods (C). There is provision for six cords (E) to be fastened to a removable bar (G) by means of clamps (D). The cord samples were clamped to the bar and were passed over the polished rods. Their other ends were attached to metal containers (F) with clamps (D). The containers were loaded to various weights with lead shot to subject the samples to selected stresses. Rotation of the motor driven rod then caused the cords to flex cyclically under stress. All cords were flexed continuously for 30 minutes at 5 percent of the breaking load for the original, untreated cord in each series. The entire apparatus had been designed to be placed in an oven for flex testing at elevated temperatures. In this study the flexing took place at room temperature.

3.2.3. Discussion of Results

Table IX presents the stress-strain data for the four variations in crosslinking treatment of the three types of textile cords as a function of Cobalt 60 gamma irradiation. The results are tabulated as breaking load in pounds, tenacity in grams per denier (see Glossary), and ultimate elongation in percent. These properties are customarily measured to characterize textile cord. Values are included for



the unirradiated cords as control samples for reference. Measurements were made both on unflexed cords and on otherwise identical cords which had been flexed in the manner described above after irradiation. Textile cords which are destined for dynamic service involving cyclic or repeated deflection or distortion of the rubber end-item require good flex fatigue resistance. Table X expresses the data of Table IX in terms of percent retention of the initial property values.

From the latter two tables several conclusions can be drawn. First, on the basis of both absolute values and percent retention the untreated cords rank perlon, EPA, and nylon in order of decreasing resistance to a radiation exposure of 1.68 x 10⁹ ergs per gram DC. This order applies to both the unflexed and the flexed cords. The flexing reduced the tenacity of unirradiated, untreated cord by approximately 20 percent for EPA, 8 percent for nylon, and 4 percent for perlon. After the 1.68 x 10⁹ ergs per gram DC exposure, the tenacity of untreated cord was reduced by about 40 percent for nylon and 25 percent for perlon as a result of flexing. In general, therefore, perlon cords are the best of these three both before and after irradiation, at least as high as the latter radiation exposure, in terms of tenacity.

Second, on the basis of retention of tenacity and ultimate elongation the treated nylon and perlon cords are quite similar after an exposure of 1.68 x 10⁹ ergs per gram DC. In terms of absolute values of the two properties, the treated perlon cords are superior to the nylon cords by roughly 20 percent before flexing and 30 percent afterwards. The unflexed treated EPA cords are generally from 10 to 41 percent superior to the corresponding cords of perlon and nylon in retention of tenacity after the latter exposure. The corresponding unflexed treated cords of the three polymers all retained approximately the same percent of the initial ultimate elongation after the latter irradiation. In terms of absolute tenacity values the unflexed treated cords decreased in the order of EPA, perlon, and nylon. In the case of absolute values of ultimate elongation there was little practical difference between the latter perlon and EPA cords. Both cords were superior to the corresponding nylon cords in this respect. After flexing, the irradiated treated perlon cords were superior in absolute stress-strain values to the like EPA and nylon cords in that order.

The crosslinking agents were consistent in the majority of cases in their effect upon the retention of initial properties after irradiation. In order of decreasing effectiveness they were HDI, N200, and N300. The cords containing HDI underwent little or no change in ultimate elongation as a result of the maximum exposure.

The final practical question is which of these cords is most promising for use in end-items for radiation service. The answer is that perlon cord which has been treated with HDI retains its initial properties best under the various test conditions. After flexing and an irradiation of 1.68 x 10⁹ ergs per gram DC said cord has 48 percent of the tenacity and 110 percent of the ultimate elongation of unflexed unirradiated nylon cord. The corresponding figures for the flexed irradiated nylon cord are 30 percent and 68 percent, respectively. In spite of the improvement which the perlon-HDI cord represents, even that cord may have inadequate radiation resistance for some applications.



The results of the study of anti-rad protection of cords of various polymers appear in Tables XI and XII. These two tables show the amount of inhibition which the six additives provided for the five cord polymers. In all but one of the 30 cases the additives gave significant protection. The cords were generally well protected up to 8.4 x 10⁸ ergs per gram DC, as the data show. They were fairly well protected at 1.7 x 10⁹ ergs per gram DC. These tables permit both an absolute and a relative comparison of unprotected and protected cords. Table XI gives tenacity in grams per denier (see Glossary) and ultimate elongation in percent. Table XII gives the initial values before irradiation in the same units and subsequent values after irradiation in percent of initial value.

The additives which were chosen had proved to be effective in inhibiting radiation damage to a natural rubber compound. Addition of the potential anti-rad to the cords had little effect in most cases on the tenacity and ultimate elongation before irradiation. With only one possible exception every additive protected every cord polymer. The degree of protection varied but in the majority of cases was marked.

All subsequent remarks concerning the protection afforded the cords will refer to the properties after a radiation exposure of 1.68 x 109 ergs per gram DC and without flexing. Flexing tests are scheduled but have not been run yet. Of the untreated cords Dacron possessed the best inherent resistance. It retained 80 percent of its initial tenacity and 94 percent of its ultimate elongation. In contrast, nylon underwent a 50 percent decrease in tenacity and a 15 percent increase in ultimate elongation. The tenacity of rayon decreased by 72 percent, and the elongation at break decreased by 44 percent. Currently, nylon and rayon cords are used most commonly in rubber products. Like nylon, untreated perlon experienced a 47 percent decrease in tenacity and a 36 percent increase in ultimate elongation. The cords of perlon plus age-resister (Perlon-AR) showed a 73 percent decrease in tenacity and a 51 percent decrease in ultimate elongation. Thus, the age-resister had a detrimental effect on radiation resistance.

No single anti-rad was most effective for all five cords alike. Table XI shows that both quinone and pyrogallol give a fourfold extension in radiation service "life" of nylon cords, based on absolute values. The same order of protection is given to each of the other four cords by the most effective anti-rads. In the case of Dacron, the addition of phenothiazine results in nearly the same tenacity and ultimate elongation after the irradiation as the untreated cord has without irradiation. Dacron thus exhibits the best retention of stress-strain properties after irradiation, both with and without an anti-rad present. In absolute stress-strain properties after the 1.68×10^9 ergs per gram DC exposure, the best cords are Dacron with phenothiazine, perlon with quinhydrone, and perlon plus age resister with phenothiazine. All three irradiated cords closely approximate unirradiated nylon cord in tenacity. Furthermore, irradiated Dacron cord with phenothiazine present nearly matches unirradiated nylon cord in both tenacity and ultimate elongation.

Two observations are of interest in a comparison of perlon with perlon plus age-resister. First, in the absence of an anti-rad the age-resister has an adverse effect on resistance to radiation damage. Second, phenothiazine is much more



effective in perlon-AR than in perlon, whereas pyrogallol is about equally less effective. It may be that synergism exists between these anti-rads and the ageresister.

The results in Tables XI and XII are presented graphically in Figures 22 through 27. Figure 22 shows that both Dacron and experimental polyamide cords underwent linear decrease in tenacity with increasing radiation exposure. The other four cord polymers deteriorated in an exponential manner. All but five of the 30 cord polymers containing an anti-rad also decreased linearly in tenacity as the cumulative exposure increased. Furthermore, the rate of deterioration was approximately the same for all 25 of those protected polymers and was similar to that for untreated Dacron. This raises the interesting speculation that it is the presence of aromatic molecular structures in the polymer system and not how they are introduced that is important. To put it differently, the aromatic nucleus appears to be approximately as effective an inhibitor of radiation damage when it is a part of the polymer molecule as when it is present in a separate additive. The behavior of the Dacron cord containing quinhydrone (Figure 25) is noteworthy. Apparently the rate of deterioration approached zero while the tenacity still exceeded 4 grams per denier and the ultimate elongation approximated 16 percent.

Time did not permit irradiation of the samples of the six cord polymers which had been treated with the four crosslinking agents. However, successful treatment was accomplished, as Table XIII attests. In general, addition of a crosslinking agent increased the ultimate elongation of the unflexed unirradiated cord and had little effect on the breaking load, just as before (see Table IX). Formaldehyde particularly benefitted nylon cord, leading actually to a higher tenacity and only slightly lower ultimate elongation after flexing. In a number of other cases treated cords had practically as large tenacities after flexing as the corresponding untreated cord had without flexing. Examples are Dacron plus formaldehyde, Dacron plus glutaraldehyde, nylon plus Hylene DP, perlon plus glutaraldehyde, perlon with ageresister plus glutaraldehyde, and rayon plus glutaraldehyde. These results suggest that treatment of a cord first with an effective crosslinking agent and then with an anti-rad would give optimum radiation stability.

3.2.4. Summary and Conclusions

Again, in this discussion all irradiated cords received an exposure of 1.68×10^9 ergs per gram DC.

In terms of absolute values of tenacity and ultimate elongation, the perlon cords which were treated with crosslinking agents were superior to the like-treated nylon cords by roughly 20 percent before flexing and 30 percent thereafter. Perlon cord which had been treated with HDI retained its initial properties the best of the specially-crosslinked polymers under the various test conditions. After irradiation and flexing, this treated cord had 48 percent of the tenacity and 110 percent of the ultimate elongation of unflexed unirradiated nylon cord. The corresponding figures for the flexed irradiated nylon cord were 30 percent and 68 percent, respectively. However, even the perlon-HDI cord may have inadequate radiation resistance for some applications.



In the second study every potential anti-rad protected every cord polymer except one from radiation damage. The degree of protection varied but was marked in most cases. Of the untreated cords Dacron possessed the best inherent resistance, retaining 80 percent of its initial tenacity and 94 percent of its ultimate elongation after irradiation. Dacron cord with a phenothiazine treatment had nearly the same tenacity and ultimate elongation after irradiation as the untreated cord had without irradiation. Dacron thus exhibited the best retention of stress-strain properties after irradiation, both with and without an anti-rad present. Irradiated Dacron cord with phenothiazine present nearly matched unirradiated untreated nylon in both tenacity and ultimate elongation.

The tenacities of untreated Dacron and EPA cords decreased linearly with increasing radiation exposure. The same behavior was noted for nearly all cord polymers containing anti-rads. In contrast, the tenacities of the other four untreated polymer cords decreased exponentially. These results suggest that deterioration tends to be linear for polymer systems containing aromatic nuclei and exponential for those which do not have any aromatic character. Moreover, an aromatic nucleus appears to be approximately as effective an inhibitor of radiation damage when it is present in a separate additive as when it is part of the polymer molecule.

These experimental observations indicate that Dacron which has been treated with phenothiazine or quinhydrone is the best selection for use in radiation-resistant rubber end-items. However, this conclusion may be subject to later qualification when the flexing test data have been evaluated. It is reasonable to assume that either the degree or duration of protection, or both, could be improved in general by increasing the concentration of anti-rad in the polymer system.

The initial results of the second study of crosslinking agents are sufficiently interesting in terms of flex fatigue resistance that irradiation and subsequent testing should be carried out.

3.3. Development of Radiation-Resistant Textile Cord Filaments

(This research was conducted and reported by S. E. Smith.)

3.3.1. Introduction

An alternate approach to the above means of protecting textile cords against radiation damage is the homogeneous dispersion of the protective agent in the cord polymer. The most feasible method of dispersion is to mix the polymer and the additive intimately prior to extruding the filaments from which the cord is prepared. Solid protective agents are the logical choice in order to minimize loss by volatilization at elevated temperature or by migration. Since physical mixing of the solid additive with the polymer replaces the previous liquid immersion techniques, there are fewer practical limitations such as solubility on what additives may be tried. Another advantage of this approach is a more definite control of the concentration of the potential protective agent in the polymer. Probably the greatest advantage is that such homogeneous mixing offers the best chance of inhibiting the effects of



radiation uniformly within the polymer. However, these and other advantages are accompanied by the difficulties which are peculiar to the preparation of monofilaments from new polymer mixtures. The greatest difficulty lies in obtaining reproducibility from one extrusion run to another for a given mixture of polymer and additive.

3.3.2. Experimentation

The first steps in this study were the selection of the textile polymers for extrusion into filaments and the choice of the potential protective agents. Two textile polymers of high tenacity were chosen. One, Polyamide A, was an experimental polyamide of a proprietary nature which had been developed in a different research project. The other, Polyamide B, was nylon 6.

The selection of protective additives depended upon an understanding of the fundamental changes which nuclear radiation induces in such polymers. It also depended in the same sense upon the relationships between polymer structure and macromolecular configuration on the one hand and stress-strain properties on the other. The physical properties of critical interest were tenacity, ultimate elongation, and resistance to flex fatigue. The tenacity is related to the degree of orientation or crystallinity and to the molecular weight or degree of crosslinking of the polymer. Thus, there appeared to be three practicable ways of offsetting a radiationinduced decrease in tenacity. One was by mechanically drawing the filament to increase the degree of crystallinity and hence the initial tenacity. The second involved additional crosslinking of the polymer in order to distribute the breaking load more efficiently throughout the polymer network. The third was to prevent chain scission which would lead to regions of local weakness where stress failure could occur. Extensibility depends upon restrictions of freedom of movement of the polymer chains under an applied force. Such restrictions may result from radiation-induced crosslinking. Conversely, chain scission would remove restrictions and increase ultimate elongation. Furthermore, a polymer is less extensible in the oriented state, because of the restricting crystallite phases. When the crystallinity is removed by heating, the polymer will shrink. The ultimate elongation consequently will increase, providing there are no counter processes occurring. Finally, the relationships between molecular structure or configuration and flex fatigue resistance are less well understood. Possibly the same factors are involved in flex fatigue resistance as in retention of tenacity and ultimate elongation but act repeatedly and locally in the same region of the filament.

The ideal textile filament or cord would maintain its initial static and dynamic physical properties in spite of high nuclear radiation exposures. Such a polymer would undergo no net change in degree of crosslinking versus chain scission (neither qualitatively nor quantitatively) and would show no change in degree of crystallinity. Additives which would tend to provide the necessary protection include metals, which can readily provide electrons to neutralize organic molecular ions, "energy sink" molecules which can absorb radiation energy and dissipate it without affecting the polymer, and free radical stabilizers or captors which can prevent the polymer from undergoing crosslinking or chain scission via free radicals. The additives which were selected are identified in Table XIV.



All of the monofilament extrusions were made with a microextruder, a machine which had been developed at this laboratory. A charge of polymer or of polymer thoroughly mixed with additive was poured into the cylinder, which had been preheated to 250°C. The plunger was driven into the cylinder mechanically by hand until a 150-pound pressure was indicated in order to compact the material. The plunger was removed, the cylinder was refilled, and the charge was again compacted as before. The plunger was then driven mechanically by a motor, and timing began. Temperature and pressure data were taken frequently throughout the extrusion process.

A single continuous filament was extruded through a 0.0136-inch diameter die. The plunger was set to move at the maximum speed of approximately 0.11 of an inch per minute. The filament take-up rate was about 25 feet per minute, and each extrusion required from 30 to 35 minutes. At these speeds a filament of between 300 and 400 denier was spun. (The denier is the weight in grams of 9000 meters of the filament.) The spun filaments were all first drawn over a block heated to 90°C. Each filament was subsequently further drawn to achieve the best tensile properties obtainable at 90°C. Because each additive affected the optimum obtainable tensile strength differently, each filament was drawn at a different tension.

A sample length of approximately 240 cm. was employed for each radiation exposure. Each 240-cm. filament sample was wound on a separate rectangular stainless steel plate for irradiation. The plates were placed in frames in the manner of ladder rungs, and each assembly was placed in a cylindrical aluminum canister. The canisters were closed at both ends with aluminum disc caps and steel expansion rings, which fitted into grooves in the inner canister wall on each side of the discs. The filaments were then irradiated in air at room temperature in the Gamma Facility at Brookhaven National Laboratory. Cobalt 60 gamma radiation exposures of 8.4×10^7 , 4.2×10^8 , 8.4×10^8 , and 1.68×10^9 ergs per gram of carbon were conducted.

Following irradiation each 240-cm. sample was cut into five 45-cm. lengths, and each length was weighed. The length of median weight was tested on the Scott Testers IP-4 tension testing machine, and breaking load and elongation at break were recorded. The tenacity was then calculated from the breaking load and the filament denier. Two lengths, one on each side of the median, were kept for auxiliary testing. The remaining two lengths were kept for reference.

Auxiliary tests were considered for possible future use in order to obtain a better basic understanding of radiation damage to textile cords. Specifically, it was recognized that major changes in molecular weight and in resistance to flex fatigue as a result of irradiation affect the service performance of filaments and cords. Intrinsic viscosity determinations are the customary means of measuring molecular weight quantitatively. However, both the amount of sample and the time required for viscosity measurements make their use impracticable in the present case. The samples to be studied are too numerous. Dilute solution viscosity measurement, an alternate procedure, also requires more sample than is available. However, the measurement of the crystalline melting points of the filaments permits a qualitative determination of changes in molecular weight as a result of radiation—



induced crosslinking and chain scission. An optical microscope with crossed Nicol prisms and a Koffler hot stage would be used to detect and measure the melting points.

The torsional modulus of elasticity, G, is a fundamental property of any material. The magnitude of G depends upon both the molecular structure of the polymer and the physical structure of filament. The G value of a filament may be affected during irradiation by crosslinking, chain scission, molecular rearrangement, oxidation, a decrease in the degree of crystallinity, alteration of the cross-sectional area and shape, and formation of cracks in the filament surface for example. The change in torsional modulus which is measured will be the resultant of many such radiation effects. Since the measurement of flex fatigue of a single filament involves unavoidable complications, torsional modulus measurements would be substituted. The value of G would be calculated from the following equation.

$$\frac{\Gamma}{\Theta} = \frac{\pi r^4 G}{21}, \text{ or } G = \frac{2 \Gamma 1}{\pi r^4 \Theta}$$

by measuring the filament radius r, the length l, and the rotational deflection Θ produced by the torque Γ . The techniques which are required for these measurements have been worked out, and the apparatus for measuring Θ and Γ has been designed and is ready to be made.

3.3.3. <u>Discussion of Results</u>

Samples of all the filaments were tested prior to irradiation to provide control data. The results appear in Table XV for the two series of polymers. Whenever a poor filament resulted, it was very poor. Examples were filaments of Polyamide A having the additive contents 1.0M, 5.0P, 1.0R, 1.0R plus 2.0X, 1.0T, 0.5V, 1.0V, and 5.0V (see Table XIV). In the Polyamide A series nine of the polymer mixtures gave filaments which were superior and three produced filaments practically equivalent to those of the pure polymer. The tenacity of the pure polymer, 4.81 grams per denier, is approximately 20 percent lower than expected because of extrusion difficulties. The same difficulty affected most of the polymer mixtures in the Polyamide A series, although in different degrees. The measured values of tenacity and ultimate elongation for each filament are considered accurate within plus or minus 10 percent of their stated magnitudes.

In the Polyamide B series seven polymer mixtures were superior, six were practically equivalent, and seven were somewhat inferior to the pure polymer in tenacity. The filament properties of the pure Polyamide B were equivalent to those which are customarily reported. The polymer containing barium nitrate had the best filament properties. The polymer mixtures containing 1,5-dihydroxynaphthalene, N, N'-di-beta-naphthyl-p-phenylenediamine, and p-methoxyphenol all appeared superior to the pure polymer in tenacity. This statement should be qualified by recognition of the difficulty in obtaining reproducibility of properties in two successive overall filament preparations from the same polymer mixture. Accurate reproducibility is difficult to achieve in such experimental preparations because the final properties depend so critically upon such factors as the draw ratio, for example.



Radiation test results are expected to show whether additives such as the latter three are effective in protecting the present two polymers from radiation damage.

In certain respects the preparation of filaments, especially from experimental polymers and polymer mixtures, is an art. Delays which arose in processing these mixtures delayed irradiation and testing. However, all samples have now been irradiated, and the physical testing is approximately half finished. It is expected that testing and evaluation will be completed by the end of March, 1958.

Tests so far have given the following results. Irradiation initially has caused a general increase in filament tenacity. In many of the cases the filament containing an additive has resisted radiation-induced change better than the control filament of pure polymer. All filaments have undergone a 5 to 10 percent shrinkage in length. However, no change in denier has been detected. The shrinkage was probably due to the disappearance of crystallite regions in the polymer and to the resulting retraction of the chains during irradiation.

The lack of an increase in denier to correspond to the decrease in length is surprising from the standpoint of conservation of filament volume. Presumably internal changes within the polymer compensate for the decrease in length. If so, there should be approximately a 5 to 10 percent decrease in specific gravity of the polymer in the filament. An understanding of this phenomenon might help to clarify why the flex life of a textile cord may be less resistant to radiation-induced deterioration than are its tenacity and ultimate elongation.

3.3.4. Summary and Conclusions

Since only about half of the samples, namely those which received the lower radiation exposures, have been tested, only preliminary conclusions can be drawn. The initial, low exposures have caused a 5 to 10 percent shrinkage in the lengths of all filaments. Presumably, radiation-induced disorientation of the polymer was responsible for the shrinkage. These first exposures in the series also increased the tenacities in general, probably as a result of crosslinking. In many cases so far the additive has improved the resistance of the polymer to radiation-caused change. A detailed report of the results will be made when the testing and evaluatio have been completed.

Complementary measurements of melting point and torsional modulus of elasticity have been proposed and described in detail. Such additional future tests could provide a better basic understanding of radiation effects on textile filaments.



3, 4, Development of New Anti-Rads

3.4.1 Synthesis of Potential New Anti-Rads

(This research was conducted and reported by J. S. Eden and J. C. Westfahl.)

In 1955 and 1956 a search was made for more effective anti-rads for a standard natural rubber tread stock (3). The potential protective agents were selected from available existing chemicals on the basis of their structures and probably modes of dissipation of radiation energy. A considerable number of quite effective inhibitors of radiation damage were discovered in this screening study. The best such anti-rad, N-phenyl-N'-cyclohexyl-p-phenylenediamine, led to a retention of 99 percent of the initial tensile strength and 88 percent of the initial ultimate elongation by the natural rubber compound in spite of an 8.4 x 10^9 erg per gram DC gamma radiation exposure.

The latter results suggested that it might be possible to synthesize chemical compounds specifically for anti-rad effectiveness by considering the structures of the most effective anti-rads in the screening study. If new anti-rads furnishing significantly greater protection could be found, the service life of polymeric materials could be increased. The weight of radiation shielding material required to protect the rubber components might then be reducible.

The data from the foregoing screening study (3) were used in an attempt to correlate the structures of the organic compounds which proved to be anti-rads with their observed activities. These compounds were assigned quality ratings by multiplying the percent retained tensile strength by the percent retained ultimate elongation and dividing by 100. The rating was from best to worst in order of decreasing magnitude of the index values. As a result of this evaluation, two classes of organic compounds were selected as being capable of yielding anti-rads of increased activity.

The first class included molecular complexes of phenols with quinones. The interest in these complexes stemmed from the following consideration, where the numbers are the index ratings. Here the complex of two chemical compounds

having good anti-rad properties was significantly superior to either and in fact was among the best studied. Molecular complexes of p-quinone with pyrogallol

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(rated 65.6), beta-naphthol (60.3), hydroquinone monobenzyl ether (42.6), and p-cresol (22.9, but rated highly on the basis of retention of modulus) were selected for synthesis as potential anti-rads. All except the complex with pyrogallol were prepared in sufficient quantity for anti-rad testing. Difficulty was encountered in the preparation of complexes of p-quinone with readily oxidized phenols, such as pyrogallol. When these two components were mixed, a portion of the quinone was reduced to hydroquinone with a simultaneous oxidation of the pyrogallol. The remaining quinone combined with the hydroquinone to form quinhydrone. The crystalline product isolated from the reaction was, therefore, quinhydrone rather than the expected complex.

The synthesis of several other complexes of this general type was attempted. Naphthoquinone was successfully complexed with hydroquinone. An effort to prepare a complex of the quinhydrone type from naphthoquinone and p-aminophenol produced reddish-brown needles of melting point 261°C. Quantitative chemical analysis for naphthoquinone showed that this product was not the desired complex. It was assigned the following structure on the basis of its elementary composition, infrared absorption spectrum, and chemical behavior.

Reduction with aqueous sodium hydrosulfite gave a colorless compound which was reoxidized by atmospheric oxygen to the original colored compound. Reductive acetylation with zinc dust and acetic anhydride gave a colorless solid of melting point 193°-194.5°C. Based on elemental analysis, its structure was judged to be that of a triacetoxy N-phenyl-beta-naphthylamine.

(II). The N-(beta-naphthoquinonyl) -p-aminophenol (I) and N-(4-acetoxyphenyl)-1, 4-diacetoxy-2-naphthylamine (II) were considered promising, and large samples were prepared for anti-rad evaluation. Furthermore, a quantity of N-cyclohexylaniline was prepared for testing by the reaction of cyclohexyl bromide and aniline.

Work was then begun on the synthesis of chemical compounds of the second class, namely unsymmetrically disubstituted N, N'-diphenyl-p-phenylene diamine derivatives of type (III).

$$R_1 \longrightarrow NH \longrightarrow NH \longrightarrow R_2$$
(III)

In particular, three compounds were desired: one where R_1 was CH_3 and R_2 was H, one where R_1 is NO_2 and R_2 is H, and one where R_1 is CH_3 and R_2 is NO_2 . Since no general method was available for the synthesis of these compounds, several routes were investigated.

The first method which was tried involved the reaction p-aminodiphenyl amine with p-iodotoluene and p-iodonitrobenzene, employing the conditions described in German Patent 185, 663 (16). When p-aminodiphenyl amine, p-iodotoluene, potassium carbonate, and a small amount of cuprous iodide were heated for 18 hours at 200°C., a blue high-melting solid was formed. This material did not have the properties predicted for the desired product. The reaction was repeated using an inert atmosphere to give a liquid product. None of the desired compound was isolated from the liquid product.

The unsubstituted compound, N, N'-diphenyl-para-phenylene diamine, can be prepared by heating p-hydroxydiphenyl amine with aniline, using hydrogen chloride and ammonium bromide as catalysts. When this reaction was carried out using p-nitroaniline in place of aniline, a black resinous solid was formed. Apparently the p-hydroxydiphenyl amine was oxidized by the nitro group of p-aniline, and the preparation was unsuccessful. When the same reaction scheme was followed using p-toluidine ($CH_3C_6H_4NH_2$) instead of aniline, a crystalline solid of melting point $186^{\circ}-188^{\circ}C$. was formed. This product was identified as the dimethyl compound ($R_1=R_2=CH_3$) containing traces of the monomethyl compound by comparisons of infrared spectra and melting points of this substance and the authentic dimethyl compound (melting point $187^{\circ}C$.) prepared by Mr. D. Y. Miller. The authentic monomethyl compound ($R_1=CH_3$, $R_2=H$) was prepared by the Chapman synthesis, which will be described below. The inability to prepare the monomethyl compound from p-hydroxydiphenyl amine and p-toluidine was due to an interchange reactions such

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Attempts to repeat the work of Goldberg (17) on the reaction of aryl halides with acetylated aromatic amines were unsuccessful. Reaction of p-N-acetamidodiphenyl amine with p-iodotoluene did not give the desired monomethyl compound. Therefore, a method developed by Chapman of preparing substituted diphenylamines of unequivocal structure (18) was tried. The Chapman synthesis as extended to substituted diphenyl-p-phenylene diamines consists of the following steps.

$$C_6H_5CONH - R_1 + PCl_5 \longrightarrow C_6H_5C = N R_1$$
 (I)

$$C_6 H_5 \stackrel{C1}{C} = N \stackrel{C}{\bigcirc} R_1 + R_2 \stackrel{C}{\bigcirc} NH \stackrel{C}{\bigcirc} \stackrel{NH}{\bigcirc} \stackrel{R_2}{\longrightarrow} C_6 H_5 \stackrel{C}{C} = N \stackrel{NH}{\bigcirc} \stackrel{R_1}{\bigcirc} (II)$$

Authentic monomethyl compound was prepared by this method (R_1 - CH_3 , R_2 - H). Its melting point was 150.5° 151.5°C. Time did not permit the preparation of a larger amount for anti-rad testing. A study of the literature indicated that Step III of the Chapman method will not proceed if R_1 or R_2 are nitro groups. It was therefore planned to synthesize the corresponding chloro compounds, in which R_1 - Cl, R_2 -H and R_1 -Cl, R_2 -H and R_1 -H and H and H and H and H are H are H are H and H are H are H and H are H are H are H are H are H and H are H and H are H are

The specially-synthesized potential new anti-rads were given to Mr. H. J. Kehe for incorporation into the screening master rubber compound and evaluation by irradiation and physical testing. This work will be described in the next section of this report.



3.4.2. Selection and Evaluation of Potential New Anti-Rads

(This research was conducted and reported by H. J. Kehe.)

3.4.2.1. Introduction

The growing importance of nuclear propulsion places an ever greater emphasis upon the resistance of component products to radiation damage. Wherever propulsion is concerned, rubber products are traditionally vitally involved. Often, the very success of nuclear propulsion depends importantly upon the ability of end-items containing rubber and plastic compounds to continue to function satisfactorily in spite of irradiation. This phase of research concerns the continuing search for and development of additives to increase the resistance of rubber compounds to radiation damage.

Research in previous years (1,2) had led to the discovery of radiation damage inhibitors which were named "anti-rads." Subsequently, an extensive screening study resulted in an efficiency rating of over eighty such inhibitors, of which many imparted significant protection to a standard natural rubber compound (2, 3). The present research was an attempt to develop new, superior anti-rads. The potential inhibitors were selected on the basis of the screening study. Among others, they included specially-prepared chemical compounds which are discussed in Section 3, 4, 1 above.

3.4.2.2. Experimentation

Rubber compounds consist of elastomers into which numerous other materials have been mechanically mixed. Some of the additives are soluble, whereas others are insoluble but intimately mixed in the form of very fine particles. Air, particularly oxygen, becomes one of the dissolved constituents as a result of the way in which the rubber is processed. In this state the oxygen is easily activated and may become extremely reactive toward the elastomer. The energy which is imparted to the rubber system during irradiation activates the molecules and atoms in general, thereby promoting numerous reactions including oxidation.

Rubber must serve under a wide variety of conditions. In regular applications rubber is surrounded by oxygen in an environment which may include heat, cold, wide fluctuations in humidity, fumes, fluids, and several kinds of radiation. Simultaneously, the mechanical forces of vibration, stretching, compression, torsion, and abrasion may act on the material. To these environmental factors must today be added nuclear radiation.

Satisfactory use of rubber requires a knowledge of how it will perform under a given set of conditions. The following tests were employed to evaluate the performance of rubber samples which had received a graduated series of high exposures to gamma radiation. ASTM methods and procedures were used in all cases. The static testing included measurements of tensile strength, ultimate elongation, and modulus of die C dumbbells with the Scott machine (ASTM Test D412) and of hardness with the Shore A durometer (Test D676). These tests were chosen as being of the



broadest interest to give information about the strength, stretchability, resistance to stretching, and the load-bearing and deflection characteristics of a rubber compound. Further evaluation tests were made of rubber performance under dynamic conditions. It is in this area that rubber serves most outstandingly and encounters the severest demands. The Goodrich Flexometer (Test D623) provided information on the extent of heating caused by continously repeated flexure. It also demonstrated whether the sample would permanently assume a different shape under the combined effects of heat and cyclic compression. For a material that becomes deformed in use, at least an approximate return to the original shape is a persistent vital requirement. The Yerzley Oscillograph (Test D945) revealed how radiation and the other environmental conditions affected resilience and resistance to dynamic deformation.

Identical groups of samples were exposed to gamma radiation on the one hand versus accelerated oxidation on the other. The purpose was to compare the effects of oxidation in the absence of nuclear radiation with the sum of the effects induced by irradiation. The irradiations took place at the Materials Testing Reactor (MTR) Gamma Facility of the National Reactor Testing Station (NRTS), according to the customary procedure. Spent reactor fuel elements served as the source of radiation. The fluxes ranged mainly from 1.3 x 10^8 to 2.0 x 10^8 , averaging approximately 1.5 x 108, ergs per gram of carbon. A change in the container for sample irradiation effected large savings in time and cost. Inexpensive conventional "tin" cans were substituted for the aluminum canisters which had been customarily used. The economies stemmed from the roughly 50 percent greater inner volume of the can with no greater outer volume. Since irradiation charges are based upon the number of containers as well as the exposure for each container, the ability to pack many more samples into a container resulted in lower overall cost. Furthermore, the cost of the "tin" cans and can sealer was nominal compared with that of an equal number of the thick-walled aluminum canisters. The irradiations all took place in air at room temperature.

The accelerated oxidation (Test D572) involved exposure of samples to pure oxygen in a bomb at 70°C. and a pressure of 300 psi for 48 hours in place of nuclear irradiation. The object of oxidation testing was to learn whether any correlation exists between antioxidant and anti-rad effectiveness. If a correlation were established, there would then be available a simple, quick, and well-known procedure for predicting anti-rad capability.

The rubber compounding and sample preparation involved the masterbatch technique. A standard ASTM natural rubber tread stock recipe, namely D15-55T, 5a, was selected as in the first screening study to be the master rubber compound. The screening recipe thus was as follows.

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Ingredient	Parts by Weight
Natural Rubber Easy Processing Channel Black Zinc Oxide Stearic Acid Sulfur Benzothiazyl Disulfide	100.0 50.0 5.0 3.0 3.0 1.0
Potential Anti-Rad	<u>0 - 4*</u> 162. 0 - 166. 0*

*The additive concentration varied. Both identity and concentration are given in Table XVI.

The rubber, carbon black, zinc oxide, and stearic acid were mixed in a Banbury mill of 1000-ml. capacity. The specifications were 77 rpm speed for the rotors, 10 psi steam pressure in the shell, and cold water in the rotors. The standard operating procedure was as follows.

Time (Minutes)	<u>Operation</u>
0	Placed rubber in Banbury mixer.
1	Added first 1/3 of carbon black and all of zinc oxide.
3	Added second 1/3 of carbon black and all of stearic acid.
5	Added final 1/3 of carbon black.
6	Cleaned ram and added cleanings to charge.
7	Discharged compound; maximum and final temperatures were recorded.

The several Banbury charges were subsequently blended together on a 10-inch roll mill at 158 \pm 9°F. Small aliquots from the resulting large batch were finally mixed with the curatives and the experimental anti-rad. The mixing was done on a 6-inch roll mill. The aliquot was first warmed on the mill, which was set at 0.075 inches' clearance and 158 \pm 9°F. The sulfur was then added, followed by benzothiazyl disulfide and finally the potential anti-rad. Three alternate diagonal cuts, three-quarters of the way across the band of rubber on the roll, hastened proper mixing. Thereafter, the stock was stripped from the mill, and the roll separation was set at 0.030 of an inch. The rolled sheet was passed endwise through the mill six times to insure complete mixing. The batch was sheeted off at a thickness of 0.080-0.090 of an inch.



In the early part of the study tensile sheets were vulcanized for 10, 20, 40, and 80 minutes at 284°F. Later, curing took place for 10, 20, 30, and 40 minutes at 293°F. This increase in temperature speeded the production rate for tensile sheets. Simultaneously, the Mooney cure rate (D1077) was determined to corroborate the curing rate given by the tensile test (D412). Inspection of the tensile strength, modulus, and elongation values and the Mooney cure rate led to the selection of the optimum cure for each individual stock. Enough tensile sheets were prepared at the optimum cure to furnish all the dumbbells required for subsequent tests. At the same time samples for the Goodrich Flexometer and Yerzley Oscillograph tests were cured in the pellet mold (D623).

Various ASTM methods describe the sample preparations and test procedures in complete detail; hence they will not be repeated. However, the packaging of samples for irradiation is not covered and so will be described here.

Each radiation exposure for each rubber compound required 5 ACS dumbbells. The 5 samples were carefully stacked into a 5-ply bundle. An identifying number on aluminum foil (3 mil) was adhered to Scotch tape (1/2 inch), and the tape was wound around one wide end of the stack of dumbbells. Plain Scotch tape was wrapped around the other wide end. Each such stack of 5 identical samples was then separately wrapped in 3-mil aluminum foil (at least 2 turns). The foil ends were crimped and folded over in a regular package fold. Care was exercised that the narrow portion of the dumbbell was not bent, twisted, compressed, or deformed in any other way. The wrapping bore the same identification number that was on the wide end of the dumbbells. Goodrich Flexometer pellets were wrapped with about two turns of Scotch tape carrying an identifying number on a small piece of 3-mil aluminum foil. Each pellet was then separately wrapped in a piece of aluminum foil and was properly identified.

After being wrapped, the dumbbells and Flexometer pellets were placed in either an aluminum canister (Figure 93) or a size 300 x 509 "tin" can for gamma irradiation. The canister was capable of holding 8 packets of 5 dumbbells each in addition to 8 pellets. When the pellets were omitted, the canister could hold 9 packets without deforming them. Toward the close of 1957, trials showed that a 300 x 509 conventional "tin" can could satisfactorily replace the heavy-walled aluminum canister. The can had a capacity of 13 packets alone or 12 packets plus 12 pellets. In all cases, lightly packing the unfilled spaces with Pyrex brand glass wool protected the rubber samples from mechanical injury during shipment and handling.

3.4.2.3. Discussion of Results

Table XVII presents the physical property values of the various rubber compositions which were obtained by the static and dynamic testing. The values before and after irradiation are given in absolute terms. The quality of compounding and curing of the rubber stocks was generally both consistent within any series and good. In approximately 90 percent of the cases the stress-strain values deviated by no more than 5 percent from the average for each set of 5 dumbbells. In almost all cases the deviation was less than 10 percent.



Addition of an anti-rad caused no change in the stress-strain properties of the unirradiated compounds in each series within the limits of error, regardless of concentration. No significant change occurred in dynamic modulus. However, the temperature rise during flexing generally decreased with increasing anti-rad concentration. In the majority of cases the hardness underwent little or no change. When a real change occurred, increasing the amount of anti-rad increased the hardness.

Because of the variations from series to series in initial tensile strength, for example, comparisons of radiation effects and anti-rad protection are more meaningful on the basis of the relative change. Therefore, the results of irradiation are shown in the form of the percent of the initial property value which was retained by selected groups of stocks in Table XVIII.

In order to evaluate reproducibility of test results, consider Compound 81GHC 6, 41, 58, and 82 in Table XVII. There are four separate sets of results for the same compound, namely, the unprotected master Compound 81GHC6, measured at different times and with separate handling. The tensile strengths before irradiation were 3640, 3740, 3620, and 3600 psi, respectively, averaging 3650 \pm 70 psi (a \pm 2% deviation). The corresponding ultimate elongations were 560, 540, 520, and 570 percent, averaging 550 \pm 20 percent (a \pm 4% deviation). Thus, reproducibility appeared good.

Before comparing anti-rad protection, examine the results for the 81GHC2-6 and 81GHC41-45 series, at radiation exposures up to 8.7×10^9 in distinction to 21.8 x 108 ergs per gram DC, in Table XVIII. Both series contain PBNA (phenylbeta-naphthyl amine) as the protective agent in the same graduated amounts. However, the former series was slightly undercured on purpose, and the latter series received optimum cure. Two observations are important. First, undercured Compound 81GHC6 without PBNA equals Compound 81GHC45, which has 4 phr of PBNA and an optimum cure, in radiation resistance through 8.7 x 109 ergs per gram DC. Second, in these two series and, in fact, throughout the study the percent retention of tensile strength and elongation after a 21.8 x 109 erg per gram DC exposure was roughly the same for a large majority of the compounds. This suggests that, with but several exceptions, the protective effect of an additive is lost before this highest exposure is reached. Since varying degrees of protection are afforded through an exposure of 8.7×10^9 ergs per gram DC, all further comparisons will be made principally after the latter exposure. Also, the benefit to be gained in radiation resistance by a slight undercure, which has been pointed out in earlier reports, is re-emphasized here.

Figures 28 and 29 permit a graphic comparison of radiation effects on tensile strength for series 81GHC2-6 and 81GHC41-45, respectively. Deterioration of the former group of undercured compounds is delayed by the completion of cure during the initial radiation. It is interesting that after this completion of cure the rate of decrease in tensile strength is faster for the former series. Finally, after the 21.8×10^9 erg per gram DC exposure, both series had lost practically the same percent of the initial tensile strengths.



The same remarks which were made in the last paragraph about tensile strength can be made for the radiation-induced changes in ultimate elongation. Figures 30 and 31 make the corresponding comparison. Similarly, the rate of increase in 100 percent modulus was greater for the undercured series, as shown in Figures 32 and 33.

Table XVIII permits a comparison of the relative protective effects of PBNA, Antiox 4010, and 32CH60G. Antiox 4010 (N-cyclohexyl-N'-phenyl-p-phenylene diamine) had proved to be the best anti-rad in the first screening study (3). The 32CH60G additive had exhibited very good antioxidant properties in earlier independent research.

Although Compound 81GHC82 should have received an optimum cure, its modulus indicated that it was slightly undercured (Table XVI). Therefore, the optimum-cured Compound 81GHC41 will be used as the control for general comparisons.

At a concentration of 1.0 phr the retentions of tensile strength and elongation after an exposure of 8.7×10^9 ergs per gram DC were 89 and 44 percent for Antiox 4010; 77 and 46 percent for 32CH60G; and 62 and 40 percent for PBNA, respectively. At a concentration of 4.0 phr both 32CH60G and PBNA gave maximum protection. The comparison values were 81 and 51 percent for the former and 82 and 55 for the latter. Thus, Antiox 4010 was the best in the entire series in terms of retention of tensile strength. Furthermore, the value of 89 percent agrees fairly well with the 99 percent figure of the first screening study. It was among the best in retention of ultimate elongation. However, its value of 44 percent compares poorly with the 88 percent figure of the previous study. Indeed, the retentions of elongation were generally surprisingly low in most cases in this second study. The figures for 32CH60G should perhaps be tempered somewhat by the possibility that the compounds containing it may have been slightly undercured as judged by the moduli. The problem of obtaining exactly equivalent, optimum cures for each compound in a series of this size is a difficult one, which is aggravated by time considerations. When allowances are made for differences in moduli and thus probably in states of cure, it appears that PBNA and 32CH60G gave approximately equal protection.

The data in Table XIX show the percent of each initial stress-strain property which was retained by a large number of stocks after irradiation. The results were evaluated in a less detailed manner than in the case of Table XVIII. The substances which were examined in Table XIX included such additives as commercial and experimental antioxidants, ultraviolet radiation absorbers, quinhydrones, quinones, fluorescent brightening agents, dyes, color pigments, and some miscellaneous compounds.

A careful study of Table XIX showed that, for each stress-strain property measured, the additives could be grouped according to the following scheme:

Superior - a small number of substances;

Average - a large number of substances; and

Inferior - a small group of substances.

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Figures 34, 35, and 36 present these results graphically. The plots show at least the best three for each property, the blank or control, some of those giving average results, and the poorest. Table XIX reveals the following:

Superior in tensile retention: Compounds 81GHC60, 84, 91, 96, 97, 102, 122, 124, and 126 (having 75 percent retention or more);

Superior in elongation retention: Compounds 81GHC59, 60, 61, 84, 91, 96, 97, 99, 100, 102, and 126 (having 45 percent retention or more); and

Superior in modulus retention: Compounds 81GHC60, 97, and 99.

Of these, only Compounds 81GHC60 and 97 appear in all three categories. They are C2.00-VIII-132 [2,2,4-trimethyl-6-(\$\infty\$, \$\infty\$-dimethylbenzyl)-1,2-dihydroquinoline] (19) and "Santowhite," respectively. Compounds 81GHC84 (Antiox 4010), 91 (cyclohexyl aniline), 96 ("Tinopal PCR + PCRP"), 102 (2-tertiarybutyl-4-methoxy phenol), and 126 (2-(\$\infty\$-naphthylthio)-quinone) were superior in the first two of the three categories only. Compound 81GHC99 ("Vultac 3") was superior in the last two categories alone. Descriptions of the exact structures of the trade-marked materials whose names are in quotation marks are not available.

A review of the data in Table XVII reveals that seven of the eight additives listed above are antioxidants as well as anti-rads. The eighth, namely 81GHC102, has no appreciable antioxidant properties. There are other such additives in the table which are relatively good anti-rads but have little antioxidant effectiveness. See the following compounds, for example: 81GHC106-109, 111, 113-115, 118-120, 123, and 125.

In this study an antioxidant other than the potential anti-rad was purposely excluded from each rubber compound. The object was to have each protective agent stand alone in inhibiting radiation damage. In the previous screening study each prospective anti-rad was incorporated into a master rubber compound containing one phr of PBNA as an antioxidant. The presence of the PBNA complicated the evaluation of anti-rad efficiency. The questions of possible incompatibility versus synergism between the PBNA and the anti-rad arose. This complication was avoided by omitting the antioxidant per se. The evaluations of results of this study should be made with this omission clearly in mind.

The retention of tensile strength after an exposure of 8.7 x 10⁹ ergs per gram DC varied from 36 to 89 percent. The retention of elongation ranged from 20 to 55 percent. An anti-rad was rated superior in tensile strength if the retention was 70 percent or greater, average if from 55 through 69, and inferior if below 55 percent. Similarly, it was classified as superior if the elongation retention was 45 percent or above, average if from 38 through 44 percent, and inferior if below 38 percent.

The best anti-rad on the basis of tensile strength alone was Antiox 4010 (Compound 81GHC84). On the basis of tensile strength and ultimate elongation, $2-(\propto -$



naphthylthio)-quinone (Compound 81GHC126), 2-tertiary butyl-4-methoxyphenol (Compound 81GHC102), and C2.00-VIII-132 (Compound 81GHC60) were the best antirads. Considering all three stress-strain properties in Table XIX, C2.00-VIII-132 (Compound 81GHC60) was the best all-round anti-rad. The names of the suppliers of the latter superior commercial anti-rads appear in Appendix VIII.

In the previous screening study (3), the Antiox 4010 compound retained 99 percent of its initial tensile strength and 88 percent of its ultimate elongation after the same exposure (8.7 x 109 ergs per gram DC) involving the same master recipe plus one phr of PBNA as an antioxidant. In this study the exactly corresponding values were 89 and 44 percent, respectively. Therefore, the rubber compound containing Antiox 4010 had 10 percent lower retention of tensile strength and 44 percent less retention of ultimate elongation than the equivalent compound containing Antiox 4010 plus PBNA. The most obvious conclusion is that there was synergism between these two additives which resulted in greater protection by their combination than was obtained with either alone. Table XVII demonstrates that in most cases a good antioxidant was much more effective in inhibiting decreases in ultimate elongation than in tensile strength during accelerated oxidation. It is possible that when the two additives were combined during irradiation, one was freer to act as an antioxidant and the other as an anti-rad. This implies a specificity of action to a certain extent, which would seem to be a reasonable characteristic. The uncertainty as to the exact reasons for the better protection by the combination emphasizes anew the importance of the fundamental studies of the mechanism of radiation damage and of its inhibition.

The potential anti-rads in Compounds 81GHC76, 77, 78, 79, 92, and 93 were the chemical substances which were especially synthesized as described in Section 3.4.1. The additives in the series 81GHC76-79, which were complexes of quinone or naphthoquinone with various aromatic hydroxy compounds, were average to inferior as anti-rads. However, the additives in Compounds 81GHC92 and 93 were both superior in inhibiting loss of tensile strength and closely approached superiority in the retention of ultimate elongation.

A cursory review of the results of the Goodrich Flexometer and Yerzley Oscillograph tests revealed no significant differences between the protected and the unprotected stocks as far as radiation resistance was concerned. An exhaustive evaluation of the data may reveal differences, but time did not permit such a study.

3.4.2.4. Summary and Conclusions

From the sample processing standpoint, conventional "tin" cans, size 300 x 509, were satisfactorily used in place of the customary aluminum canisters for MTR gamma irradiations. The volume savings were approximately 50 percent and resulted in marked savings in time and money. However, the cans are unsafe if a large gas pressure develops during irradiation.

Addition of the anti-rad caused no significant change in stress-strain properties of the unirradiated and unaged samples.



In general, the loss of tensile strength was roughly the same during oxygenbomb aging as during exposure to 8.7×10^9 ergs per gram DC of gamma radiation. However, the decrease in ultimate elongation of compounds containing anti-rads was generally much less during the accelerated oxidation than during the irradiation.

Radiation resistance of a rubber compound can be increased appreciably by a slight undercure without seriously affecting its physical properties. The onset of radiation damage is delayed by the radiation-induced completion of cure.

Antiox 4010 was the best in the entire series of 61 potential anti-rads in terms of retention of tensile strength after irradiation. It was among the best in retention of ultimate elongation. In terms of both tensile strength and ultimate elongation, 2-(α -naphthylthio)-quinone was best. Considering all three stress-strain properties (the latter two plus 100 percent modulus), 2, 2, 4-trimethyl-6-(α , α -dimethylbenzyl)-1, 2-dihydroquinoline imparted the best radiation resistance.

This study corroborated the conclusion from the previous screening research that a good anti-rad is not necessarily a good antioxidant, and vice versa.

There appears to be a great advantage in combining a good antioxidant, such as PBNA, with a good anti-rad, such as Antiox 4010. The antioxidant enhances the ability of the anti-rad to inhibit radiation damage. The improvement is principally in the retention of ultimate elongation during irradiation. It would almost seem that the anti-rad mainly inhibits the loss of tensile strength, while the antioxidant inhibits chiefly the loss of ultimate elongation. Although this is obviously an oversimplification, it is a tempting conclusion.

Two of the specially-synthesized anti-rads were superior in retention of tensile strength by their rubber compounds and approached superiority in the retention of ultimate elongation.

The combination of various antioxidants with the substances which have shown superior anti-rad properties when used alone should give outstanding results. It is recommended that the next stage in anti-rad development should include a study of such combinations.

These results emphasize the importance of fundamental studies of the mechanism of protection against radiation damage.

3.5. Determination of Anti-Rads for Other Elastomers

(This research was conducted and reported by J. A. Glantz.)

3.5.1. Introduction

An obvious question arose from the results of the study of anti-rad protection of natural rubber compounds. The question concerned the degree of protection which the best anti-rads might afford to practical compounds of elastomers other than natural rubber.



As the first approach to obtaining the answer, a study was made of the radiation resistance of 12 production-type recipes to which three of the best anti-rads were separately added, giving 36 different compounds containing anti-rads. The various elastomers which were used have long been in conventional use. The recipes were in commercial use by the B. F. Goodrich Company.

The results of the latter research led to a screening study of the effectiveness of anti-rads in recipes containing base polymers other than natural rubber. Commercial recipes were not used.

Recently, thermally stable new elastomers have become available commercially. The possible use of these elastomers in aircraft applications has made it necessary to determine their radiation resistance and possible later to protect them partially from radiation damage by the application of anti-rads.

One of the most promising of the latter elastomers is Viton A, which is an alternating copolymer of vinylidene fluoride and hexafluoropropylene. Therefore, work has begun to determine and also to try to improve its radiation resistance by the addition of anti-rads.

3.5.2. Experimentation

The selection of elastomers and recipes, incorporation of anti-rads, determination of optimum cure, sample preparation, irradiation, and evaluation of the results have been completed for the production-type recipes.

The latter steps constituted a preliminary study of the effect of radiation on well-compounded factory stocks and of their protection by anti-rads from damage in a radiation environment. The final selection of the twelve factory stocks used in this study was made from twenty-eight production type recipes. The twelve recipes were selected as the most representative for use in given end-items. The polymers used were natural rubber, neoprene, SBR, and NBR rubbers, and the end-items ranged from tires through gasketing materials. Table XX summarizes the polymer and end-item use of each recipe. The recipes discussed above are of actual industrial items, and as such they are proprietary in nature.

The anti-rads selected for use in each of the twelve recipes were N-cyclo-hexyl-N'-phenyl-para-phenylene diamine (A), 65 percent phenyl-alpha-naphthyl-amine plus 35 percent diphenyl-para-phenylene diamine (B), and N-phenyl-N'-ortho-tolylethylene diamine (C). The commercial names and suppliers of these anti-rads appear in Appendix VII. The anti-rads were then mixed singly into each of the selected recipes on the basis of five parts anti-rad per hundred parts of elastomer in the above order. This procedure gave one control stock and three anti-rad stocks for each of the twelve recipes. The above additives were selected, because they were rated 1, 2, and 5 in the aforementioned anti-rad screening study. The optimum cures for both the control stocks and the anti-rad stocks were then determined obtained from families of cures. Tensile strength, ultimate elongation, and modulus were used as criteria. One of the major difficulties which arose was that the control stock at optimum cure exhibited different stress-strain



values from the anti-rad stocks at their optimum cures. It was felt that rather complicated compounding research would be needed to produce equivalent stress-strain values. Therefore, a compromise was reached. The optimum cure for the control stock was obtained, and then that cure for the anti-rad stock was selected which gave the closest correlation in tensile strength, ultimate elongation, and modulus to the control stock. In some cases this gave either a slightly undercured or overcured anti-rad stock. In all subsequent studies all stocks should be at their optimum cure regardless of stress-strain values. Microdumbbells were then cut from the selected optimum-cured sheets, packaged, and irradiated at the MTR Gamma Facility. The exposures used were 8.71×10^7 , 8.71×10^8 , 2.61×10^9 , 8.71×10^9 , and 7.96×10^{10} ergs per gram DC.

The test program consisted of stress-strain measurements on both the unirardiated and the irradiated samples. Emphasis was placed on tensile strength, ultimate elongation, and 100 percent modulus (taken from the stress-strain graph), since these are of most interest and use. The modulus at 100 percent elongation was selected because the elongation decreases very quickly under irradiation. Most of the samples retained at least 100 percent ultimate elongation through most of the exposures. The results of the stress-strain testing are shown in Table XXI. These data are shown graphically in Figures 37 through 48 and represent the percent retention of the original ultimate elongation at the specified radiation exposures.

The selection, compounding, determination of optimum cure, sample preparation, and irradiation of the stocks for the extended anti-rad screening study have been completed. Testing and evaluation of the data will be completed as soon as possible.

The results of the study on the effect of radiation on the protected factory stocks indicated a need for an extended anti-rad screening study in which the stocks contained base polymers other than natural rubber. The study has closely paralleled the pattern established for the anti-rad screening study for natural rubber. Since a number of different polymers were used, it was necessary to use six structurally different anti-rads to represent the total list of anti-rads that had previously been used (3) in order to complete this study in a reasonable length of time. The original premise is still valid for this study. That is, since each anti-rad used would be incorporated in the same amount into the same master recipe for each polymer, any increased resistance to radiation damage would result from anti-rad action.

The polymers selected for use in this study were SBR 1500/1501, Hycar 1002, Butyl 218, Neoprene GN, Hypalon "20", Hycar PA-21, Thiokol ST, and Thiokol FA. These polymers were chosen as being representative of their respective series of elastomers. The recipes selected for use in this study are both simple and either ASTM recipes or manufacturer-recommended recipes. In all cases, the recipes are carbon black-reinforced. All of the recipes used in this study are presented in Table XXII. The anti-rads were selected first as being the best on the basis of their rating by chemical structure and second according to their commercial availability. The six chemical structures represented are (1) amine-diamine, (2) quinone-hydroquinone, (3) amine, (4) phenol, (5) diamine, and (6) quinone. The anti-rads which were selected to represent the above structures in the extended anti-rad



screening study were (1) 65 percent phenyl-alpha-naphthyl amine plus 35 percent N, N'-diphenyl-para-phenylene diamine (2) quinhydrone, (3) alpha-naphthylamine, (4) beta-naphthol, (5) N-phenyl-N'-ortho-tolylethylene diamine and (6) anthraquinone. The commercial names and suppliers of these anti-rads are presented in Appendix VII. This order of addition was used in compounding the anti-rad stocks, as shown in Table XXII.

All of the anti-rads were successfully mixed into the eight basic recipes using 5 parts of anti-rad per hundred parts of rubber. One of the important factors in determining the benefit derived from an anti-rad, as was shown in the study of the effects of radiation on factory stocks, is the state of cure. Each compound used in this study, therefore, was vulcanized to its own optimum cure, according to the earlier recommendation in this report. In order to determine both the optimum and the equivalent state of cure for each compound, three test methods were employed. These methods were (1) the Mooney Scorch Test, (2) families of cures, and (3) reasonably close duplication of the recognized physical properties for the elastomers of the control stocks and reasonably close duplication of the physical properties of the control stocks for the anti-rad stocks. All of the stocks were successfully given optimum cures except the Thiokol ST with quinhydrone present and the Thiokol FA with quinhydrone, alpha-naphthylamine, and N-phenyl-N'-orthotolyl-ethylene diamine, which did not cure even with variations in the recipes and curing conditions. The curing times and temperatures which were required to produce optimum cures for all of the stocks are summarized in Table XXII.

ASTM dumbbells were then cut from optimum-cured sheets, wrapped in aluminum foil to prevent cross contamination, packaged, and sent to the MTR Gamma Facility to be irradiated to exposures of 4.36×10^9 , 8.71×10^9 , and 1.31×10^{10} ergs per gram DC. The lowest exposure was selected to produce a significant change in the initial physical properties. The middle exposure was chosen to include a major loss of the original physical properties. The purpose of the highest exposure was to show the extent of any protection from radiation damage afforded by the anti-rad.

Testing of the unirradiated and irradiated samples consists of the measurement of stress-strain properties. As stated previously, the properties of most interest and use are tensile strength, ultimate elongation, and 100 percent modulus taken from the stress-strain graph. Testing of the samples and evaluation of the data will take place as soon as the samplesare returned from the radiation facility.

All of the newer elastomers that were to be studied this year have been obtained, and compounding and curing have proceeded as far as time has permitted. The elastomers are of the thermally stable group. Those which were selected are Kel-F, 1F4, 2F4, a methyl phenyl silicone, a dimethyl silicone, and Viton A. All of these elastomers are of interest from a military point of view. With the exception of Viton A, which will be reported later in this section, the goal for all the other newer elastomers was to produce samples of these elastomers with physical properties comparable to those reported by the manufacturer, irradiate them, and determine their radiation resistance. The recipes and curing conditions used for these elastomers, all of which are those recommended by the manufacturer, are



summarized in Table XXIII. At the end of the period covered by this report, the samples for irradiation of the methyl phenyl silicone and the dimethyl silicone have been prepared. Although the physical properties reported by the Materials Laboratory for Kel-F, 1F4, and 2F4 compounds have been duplicated, the values which the manufacturer reports have not been matched. Therefore, no samples of these compounds have yet been irradiated.

The exception to the above procedure for the newer elastomers is Viton A, in which considerable interest has been shown. The decision was made not only to determine the radiation resistance of a well-compounded Viton A stock but also to determine if the radiation resistance of the base compound could be improved through the use of anti-rads.

Sufficient amounts of Viton A were obtained from the manufacturer to complete this study. A recipe then obtained from Wright Air Development Center which will be found in Table XXIII. The anti-rads which were selected for incorporation into the base recipe are the same as for the previous extended anti-rad screening study: namely, (1) 65 percent phenyl-alpha-naphthylamine plus 35 percent N, N'-diphenyl-para-phenylene diamine, (2) quinhydrone, (3) alpha naphthylamine, (4) beta-naphthol, (5) N-phenyl-N'-ortho-tolylethylene diamine and (6) anthraquinone. Initially, an experimental batch was mixed and cured to familiarize ourselves with the handling of Viton A and also to find out what physical properties to expect from this recipe. Then both the master stock and the anti-rad stocks were compounded and cured. All of the stocks were oven cured for twenty-four hours after completion of the press cure. A summary of the physical property values of the master and anti-rad stocks appears in Table XXV. At this point Mr. E. E. Mooney took up and continued the Viton A study, conducting the evaluations of radiation resistance.

3.5.3. Discussion of Results

A major difficulty in the evaluation of the radiation resistance of factory stocks was the problem of producing samples with and without anti-rads present having equivalent states of cure. As has been discussed before, the state of cure of a rubber sample has a significant influence on its apparent resistance to radiation damage. If it is undercured, the initial radiation effect is completion of cure. The onset of damage is accordingly delayed. If the sample is overcured prior to irradiation, the ultimate radiation-induced failure is hastened. Therefore, when the evaluation of the data was made this factor was taken into account in reaching the conclusions. Table XXVI presents the results in one of the most useful forms: namely, the extended service life in a nuclear radiation environment due to addition of anti-rads. These exposures are the ones required to cause a 50 percent decrease in ultimate elongation from the initial value. In the case of the anti-rad stocks this figure represents the maximum protection obtained as determined from Figures 37 through 48. The letters (A, B, or C) following the compound numbers indicate the anti-rads which were used. They were (A) N, N'-cyclohexyl phenyl-para-phenylene, (B) 65 percent phenyl-alpha-naphthylamine plus 35 percent diphenyl-para-phenylene diamine, and (C) N-phenyl-N'-ortho-tolylethylene diamine. The Hycar (NBR) stocks were protected the most, the Neoprene stocks were well protected, the SBR stocks were somewhat protected and the natural rubber stocks were not protected at all. The



discrepancies in results between this study and the anti-rad screening study for natural rubber are probably due in a large measure to differences in the complexity of the recipes. In the original natural rubber screening study, an ASTM recipe with a minimum number of ingredients was used. In a well-compounded factory recipe, there are bound to be a number of other ingredients present whose influence upon the radiation resistance of the compound is unknown and therefore could be quite substantial. The results showed that (1) the state of cure, (2) the polymer system, and (3) possibly some other agents which are present, influence the amount of improved resistance derived by the addition of an anti-rad.

The final results of the extended anti-rad screening study have not been obtained yet, due to an unexpected and unavoidable delay in having the samples irradiated at the MTR Gamma Facility. However, all of the anti-rads were successfully mixed into all of the master compounds. The anti-rads exercised a plasticizing effect upon the master stocks. This was shown by decreased tensile strength and modulus and increased ultimate elongation of the anti-rad stocks at optimum cure as compared with the control stocks at optimum cure. Several compounds did not cure satisfactorily. The probable reason is an interaction of unknown mechanism between the anti-rad and the curing ingredients of the compounds.

Difficulties were encountered with the compounding and curing of most of the newer, thermally stable elastomers which were due to a lack of available compounding and curing information. This lack made it necessary to do some compounding research for these elastomers.

All of the master and anti-rad stocks of Viton A were successfully mixed and press cured. All of the Viton A variations except the one containing quinhydrone were successfully oven cured with comparable equivalent cures, as shown in Table XXV. The quinhydrone variation appeared to be undercured and even with as much time as one week still produced an undercured stock. The amount of time necessary to produce an optimum-cured stock is probably too long for this particular stock to be of any practical use.

3.5.4. Summary and Conclusions

Twelve factory stocks, both with and without anti-rads present, which were subjected to irradiation showed both the effects of radiation upon a well-compounded factory stock and the added protection against radiation damage provided by anti-rads.

An extended anti-rad screening study has been partially completed for polymers other then natural rubber. Six anti-rads which were representative of the chemical structures of the anti-rad screening study for natural rubber were mixed into each of the eight master stocks. Optimum cures for all of the master stocks and all of the variations have been selected, and all of the stocks have been irradiated. The physical testing is completed, and evaluation of the results is now in progress.

Compounding research has been completed for some of the newer thermally stable elastomers. An extended anti-rad screening study is being completed for



Viton A. Well-cured compounds of these newer elastomers have been obtained as judged by the reported physical properties for stocks with the same recipes.

Increased radiation resistance can be obtained for factory stocks by the incorporation of anti-rads. The amount of increase in resistance which is imparted by anti-rads depends upon three principal factors: namely, (1) the elastomer system which is involved, (2) the state of cure of the stock, and (3) any other ingredients already present which may provide protection against radiation damage to the stock.

There seemed to be no single anti-rad which was universally most effective in the radiation protection of all of the physical properties of any given factory stock. When all 36 compounds are considered grossly, the anti-rads rank Akroflex.C first, Antiox 4010 second, and FLX third. However, the rating has little practical value. In many cases there was little real difference among the three in effectiveness. Also, lack of optimum cure affected the resistance to radiation damage in some cases.

Among the factory-type compounds which are listed in Table XXVI, the three natural rubber stocks have the best radiation resistance of those without anti-rads by a considerable measure. Of the stocks containing an anti-rad, natural rubber Armorite Packing with N-phenyl-N'-ortho-tolylethylene diamine shows the best retention of ultimate elongation. This compound and the protected Hycar (NBR) compounds gained the greatest relative protection by the addition of an anti-rad. The neoprene stocks acquired the least protection. The marked difference in radiation resistance between Compounds 614FGA4 and 5 on the one hand and 614FGA13 on the other emphasizes that changes in the compound recipe for any given elastomer can change the radiation resistance appreciably.

Anti-rads can be successfully mixed into all of the polymer systems which have been studied to date, and satisfactory cures can be obtained. To get equivalent states of cure for the control and the anti-rad stocks, it is necessary to readjust the curing times for the anti-rad compounds.

Physical property values have been obtained for compounds of the newer thermally stable elastomers which duplicate results reported by other laboratories. However, the values given by the manufacturer have not yet been achieved.

The reported physical property values for a selected Viton A compound have been duplicated. Anti-rads have been successfully mixed into Viton A stock, and most of the resulting recipe variations have been cured to states equivalent to those of the control compound. These curing conditions have been employed in the preparation of samples for irradiation and testing, as described in the next section.



3.6. Radiation Testing of Practical Rubber Compounds

(This research was conducted and reported by E. E. Mooney and S. T. Semegen.)

3.6.1. Introduction

In general terminology, practical rubber compounds are individual rubber stocks which are used in the construction of a commercial product or end-item. Each such end-item has a specific service function. Examples of rubber products which are important in aircraft design include tires, hose, seals, fuel cells, and wire insulation. The majority of the rubber applications fall into these principal categories. Prior research (4) had made it both feasible and desirable to go directly to end-item testing of a selected current aircraft tire. Section 4 below will discuss the latter. However, in the case of the remaining four categories exploratory research involving practical compounds was a necessary prerequisite to effective radiation-testing of end-items.

End-items may and often do contain several different rubber compounds. The composition may be further complicated by the incorporation of textile cord for additional strength and shape retention. Each component material has its own resistance to radiation damage. Since specifications for rubber end-items are highly subject to change as experimental aircraft undergo development, it is necessary to know the individual radiation resistances of the various types of rubber compounds which may be combined to produce a future end-item.

The resistance of a rubber material to damage by nuclear radiation may be influenced by other environmental factors. These include heat and various fluids in contact with it such as oxygen, fuels, lubricants, and hydraulic fluids. Of these, the two which are most universally certain to be associated in nuclear-propelled vehicles are heat and ionizing radiation.

A two-phase program has been agreed on for the development of aircraft compounds and rubber components to meet the challenge of high temperature and nuclear irradiation. The first phase includes short-range studies to provide engineering data on conventional elastomers in practical compounds up to 300°F. The second phase comprises long-range investigations of new elastomers for service above 300°F. Both phases have been initiated this year.

The first phase was designed to provide three types of information: namely, (1) the extent of damage done to control aircraft compounds by heat and radiation combined, (2) evaluation of the relationship between the sum of the separate effects of heat and radiation and their combined effect, and (3) the extent of radiation resistance which is imparted to aircraft rubber compounds by the addition of potential anti-rads.

The long-range program of the second phase involves Viton A primarily, at the request of WADC. Combined heat and radiation studies similar to the above are now underway using six different potential anti-rads. These protective agents were



selected from six different classes of chemical compounds evaluated in a prior screening study (3) involving only natural rubber as the elastomer.

The present program was designed as the beginning of an exhaustive study of the damage to practical compounds which is caused by heat and radiation under various service environmental conditions. Methods have been established for logical extension of the present work to higher temperature regions, different potential antirads, new polymers, and environmental testing. Such studies will logically be extended to rubber end-items as soon as it is practicable to do so.

3.6.2. Experimentation

At the outset the question arose of which few recipes should be selected to represent the many practical compounds in aircraft rubber parts. With the help of B. F. Goodrich technical personnel in production, two representative compounds were chosen for each of the four major classes of aircraft products: namely, hose, seals, fuel cells, and wire insulation. In selecting the compounds an effort was made to include (1) typical production compounds, (2) compounds doing the primary job in end-items of complex construction, and (3) a variety of elastomers.

Careful consideration and discussion indicated the necessity of determining both the separate and combined effects of heat and radiation. The ultimate goal is to be able to predict the resistance of any conventional type of rubber compound to any reasonable combination of heat aging and radiation exposure. From an engineering viewpoint a knowledge of the interaction of these two environmental factors is important to achieve the goal of predicting service performance of rubber compounds in future aircraft applications. Comprehensive heat aging studies were necessary for these particular compounds in order to select proper sampling intervals for radiation exposures at elevated temperatures.

Stress-strain testing of the selected rubber compounds was done in accordance with ASTM Method D412-51T on standard die C dumbbells. The data were compiled on the basis of the median value of five dumbbells of each compound for tensile strength, ultimate elongation, and modulus. Exposure to heat and radiation was made in closed containers using test specimens wrapped in aluminum foil. Dumbbells were wrapped compactly in sets of five. Each dumbbell in a set was separated by foil from the others. This procedure accomplished three goals: namely, (1) maximum use of the limited space in the containers for irradiation at BNL and MTR, (2) prevention of cross-contamination of samples, and (3) simulation of the environment of many aircraft rubber compounds by limiting the supply of oxygen.

The entire testing of experimental dumbbells was done by the same operator on the same Scott Tester machine. Dumbbells which were used for equivalent cure determinations (discussed below) were tested by a different operator on a different Scott Tester. The modulus values from the latter were consistently lower than those from the former. This is shown by a comparison of the values in Figures 50-57 with the data in Tables XXIX, XXX, and XXXI. Shore Durometer A hardness was measured at room temperature in accordance with ASTM Method D-676. Figure 49 shows the equipment used in packaging the dumbbells for their exposure to heat and radiation.



The potential anti-rads which were selected for evaluation this year in the practical compounds were Akroflex C (65% phenyl-alpha-naphthylamine plus 35% N, N'-diphenyl-p-phenylene diamine) and quinhydrone. The standard additive concentration was 5 phr.

The following compounds were selected to represent the above-mentioned classes of practical aircraft compounds. The increasing numbers in each series correspond to the master compound without potential anti-rad, with Akroflex C, and with quinhydrone, respectively. The recipes for the 24 control and experimental com-

- (1) 81GH311, 317, 318: neoprene wire insulation
- (2) 81GH313, 327, 328: SBR wire insulation
- (3) 81GH357, 359, 360: neoprene packing compound
- (4) 81GH358, 365, 366: Hycar packing compound
- (5) 81GH386, 391, 393: Hycar-Vinylite bladder stock
- (6) 81GH387, 395, 397: Hycar self-sealing liner stock
- (7) 81GH420, 423, 425: Hycar-SBR hose
- (8) 81GH421, 427, 429: neoprene hose

pounds are given in Table XXVII.

Equivalent cure is defined here as that state of vulcanization which (1) most nearly reproduces the best physical properties of the control stock, especially the modulus, and (2) is intermediate between the bare attainment of adequate cure and the beginning of "overcure" or aging induced by either heat or radiation or both. Equivalent cure was chosen in the present study as the basis for comparison of potential anti-rad stocks versus the control stocks. The procedure in equivalent cure determinations includes Mooney Cure Method ASTM D1077, plots of stress-strain properties versus cure time, and reasonable duplication of physical properties of the control stock by those of the experimental stocks in each series.

The present research is concerned with aging, that is, the deterioration of physical properties with time. Since equivalent cure establishes the approximate point of zero aging time, the validity of results of radiation aging studies depends on the care with which this point is determined. Apparent protection of a particular rubber compound must be interpreted in terms of the accuracy of the determination of equivalent cure. An undercured stock, with or without anti-rad, will use the initial radiation energy to complete the cure before any apparent aging begins. Therefore, this stock would show falsely high radiation resistance. Conversely, an overcured stock will show aged properties before irradiation begins and increased aging with irradiation. Such a stock would show falsely low radiation resistance. Figures 50-57 (graphs of modulus and ultimate elongation as a function of curing



time) illustrate how equivalent curing time was selected for each of the 24 stocks in the present study. The calculated Mooney cure time and selected equivalent cure time for each stock are given in Table XXVIII. Mooney cure time is given as M7 and M_{10} , which are defined as follows:

$$M_7 = T_5 + 7 \triangle 30$$
 and $M_{10} = T_5 + 10 \triangle 30$,

where T_5 is the time required for a 5-point rise in Mooney plasticity, T_{35} is the time required for a 35-point rise, and Δ 30 equals T_{35} minus T_5 . The measurements were all made on a large rotor Mooney machine at 293°F., abbreviated as ML293. The latter temperature is the one which was selected for the cures.

The Mooney cure times M_7 and M_{10} generally bracket that region of the curing rate curve of conventional polymers where the slope changes rapidly, that is, where the vulcanization reaction nears its completion. This region is normally selected as "optimum cure." Since aircraft rubber compounds must meet certain property specifications, equivalent cure for the stocks containing potential anti-rads was not always synonymous with the Mooney optimum cure.

Equivalent cures were determined from dumbbells tested at room temperature. Theoretically, a separate determination of equivalent cure should have been made for dumbbells pulled at elevated temperatures. This latter equivalent cure was assumed to be the same as the former. However, this point should be considered in evaluating the elevated temperature data.

Different combinations of heat and radiation exposure and testing conditions were imposed on the dumbbell samples. The purpose was to test thoroughly how much radiation protection the potential anti-rads afford to the aircraft rubber compounds. In addition, the combinations of exposure and testing simulate many actual service conditions, thus providing useful engineering data.

Six methods of such exposure and testing were employed this year for the 24 compounds, as follows.

Method I: Irradiation at room temperature and testing at room temperature

Method II: Irradiation at 158°F. and testing at room temperature

Method III: Irradiation at 158°F. and testing at 158°F.

Method IV: Irradiation at room temperature and testing at 158°F.

Method V: Heat aging at 158°F. and testing at room temperature

Method VI: Heat aging at 158°F. and testing at 158°F.

Irradiations at room temperature were conducted to cumulative exposure doses of 2.1×10^9 , 4.2×10^9 , 8.4×10^9 , and 12.6×10^9 ergs per gram of carbon. The irradiations at 158° F. were carried out at 8.4×10^8 , 16.8×10^8 , 33.6×10^8 , and



 58.7×10^8 ergs per gram DC. Heat aging at $158^{\rm o}F$. was conducted for periods of time which were suitable for each stock. Tables XXIX, XXX, and XXXI list data obtained by Test Methods I, II, and III. Data from Methods IV, V, and VI will be evaluated in a later report. Figures 58-65, 66-75, and 76-83, corresponding to Test Methods I, II, and III, respectively, illustrate graphically the effect of radiation alone and of combined heat and radiation on the percent retention of initial elongation for all compounds.

The curves for samples irradiated at room temperature (Figures 58-65) are not too well defined in the region of $0-4.2 \times 10^9$ ergs per gram DC ($0-5 \times 10^7$ roentgens). Samples are now being irradiated to provide additional points for the curves in said region.

Tables XXXII, XXXIII, and XXXIV, corresponding to Test Methods I, II, and III, respectively, are tabulations of the radiation exposures required to reduce the ultimate elongation of each of the rubber compounds to 50 percent of the initial value before irradiation. These tables also present reductions of percent of initial ultimate elongation that result from exposures of either 12.6 x 10^9 ergs per gram DC at room temperature or 5.87 x 10^9 ergs per gram DC at $158^{\rm o}$ F. Also, the dose rates are given for the gamma irradiation of each set of samples at Brookhaven National Laboratory.

Radiation protection ratios are listed in Table XXXV. The protection ratio is the ratio of the exposure required to cause a reduction of 50 percent of the initial ultimate elongation for a compound containing a potential anti-rad to the corresponding exposure for the control compound.

3.6.3. Discussion of Results

Neoprene, SBR, Hycar (NBR), and a Hycar-Vinylite blend were the types of elastomers used in this study. The compounds that were tested included aircraft wire insulation, packing, fuel cells, and hose. The predominant apparent radiation effect was crosslinking of the elastomer. That is, modulus increased and ultimate elongation decreased with increasing radiation exposure. These two physical properties provide reliable measurements of radiation-induced aging. Tables XXIX, XXX, and XXXI give the changes in these properties for the above compounds with and without the potential anti-rads Akroflex C and quinhydrone present.

The tensile strengths of most of the stocks except the low durometer neoprene and Hycar packing compounds (81GH357, 365, 366, and 81GH358, 365, 366, respectively) remained quite constant throughout the range from 0-58.7 x 10⁸ ergs per gram DC. The anti-rads generally improved the retention of initial ultimate elongation slightly and moderately inhibited increases in modulus. The rapid decrease in both tensile strength and ultimate elongation of the packing compounds in the range of 0-2.1 x 10⁹ ergs per gram DC accounts for the small increase in modulus. All compounds exhibited increases in hardness with increasing radiation exposure. Apparently the lower the initial hardness the greater is the percent increase in hardness as a function of irradiation.



As discussed in Section 3.6.2., the validity of the comparison of stocks containing potential anti-rads with control stocks depends importantly on the accuracy with which equivalent cures are determined. The equivalent cures of the eight air craft compounds and their potential anti-rad variations are illustrated in Figures 50-57. A tabulation of Mooney curing times M₇ and M₁₀ and the selected equivalent cure times appears in Table XXVIII for all compounds. The selections of equivalent cure were based primarily on a matching of modulus at room temperature of stocks containing potential anti-rads and control stocks. Mooney cure time, tensile strength, and ultimate elongations were used to confirm the equivalent cure determinations based on modulus. Table XXVIII shows that (1) quinhydrone greatly retards the equivalent cure of Hycar, accelerates neoprene slightly, and has little effect on SBR, and (2) Akroflex C slightly accelerates the equivalent cure of both neoprene and Hycar.

Figures 58-83 show graphically the changes in percent retention of ultimate elongation of all 24 compounds as a function of the heat and radiation exposure and test conditions of Methods I, II, and III given above. Radiation exposures of 83.9 x 10^8 ergs per gram DC at room temperature and 33.6 x 10^8 ergs per gram DC at 158^{0} F. caused a major change in the physical properties of all the compounds.

In the better-defined curves of percent retained elongation versus radiation exposure, each curve may be considered in two portions. The first or "reaction" portion, which always fell within the latter two exposure ranges depending upon the irradiation temperature, was generally characterized by an S-shape indicating relatively rapid radiation aging. The second or "reaction tail" portion was characterized by relatively slow radiation aging. Reference to both sections of the radiation aging curves is important for a complete description of radiation effects on rubber.

Tables XXXII, XXXIII, and XXXIV summarize the comparative radiation resistances of the aircraft compounds in the "reaction" and "reaction tail" portions of each curve. The results for Methods I, II, and III are given in terms of (1) the radiation exposure required to reduce ultimate elongation to 50 percent of the initial value and (2) the reduction in percent retained elongation at two high exposures. These three tables present the totals and averages of each of these two values for the 24 rubber compounds. These resulting figures show that (1) Methods II and III are more drastic than Method I in the rate of reducing the percent retained elongation to 50 percent but (2) all three test methods result in approximately the same reduction in percent retained elongation after the 58.7 x 10⁸ ergs per gram DC exposure.

Several further observations can be made from the latter three tables. First, SBR wire insulation compounds (81GH313, 327, 328) and Hycar 1043-SBR 1001 tube stocks (81GH420, 423, 425), the only two compounds containing SBR elastomer, show significantly more radiation resistance than the other compounds. This perhaps is not too surprising, since SBR is known to possess inherent good resistance to high energy radiation. Hycar 1043 is also used in the bladder stocks 81GH386, 391, and 393, whose radiation resistance is apparently inferior. It is interesting to speculate that perhaps as little as 13.67 phr of SBR can so enhance the resistance of the Hycar. Second, the Hycar self-sealing fuel cell liner stock (81GH387, 395, 397) received the most overall benefit from the potential anti-rads Akroflex C and quinhydrone. Its

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maximum improvement, as shown in Tables XXXII-XXXV, can be expressed as 2.8/2.1/1.6 for Method I/Method II/Method III, respectively.

Generally, these two potential anti-rads provided at best only marginal radiation protection, ranging up to 3.2-fold improvement, for the compounds involved. The protection ratios which appear in Table XXXV indicate this clearly. In most cases where protection occurred, the potential anti-rads provided less protection in Test Methods II and III than in Method I.

In order to obtain some measure of the duplicability and limits of experimental error of a test method, a duplicate set of dumbbells for the neoprene and Hycar packing compounds (81GH357, 359, 360 and 81GH358, 365, 366, respectively) was tested using Method II. Figures 69 and 71 show the duplicate results. A comparison of the latter two graphs with the graphs of their duplicate sets in Figures 68 and 70 reveals that the two sets of data agree fairly closely. Table XXXIII, which makes a further comparison of the two sets, indicates an 8 percent maximum variation in the former set and a 14 percent maximum variation in the latter set. In view of the normal experimental variations, this degree of duplicability is considered satisfactory.

In Section 3.5 above, data are given for the room temperature irradiation of eight compounds which have been repeated in the present study. The data from the two independent studies have been plotted together in Figures 62 through 65. The agreement between these sets of data ranges from very good to mediocre. The difference in the radiation exposure which effected a 50 percent reduction of retained ultimate elongation varied from zero to 8.4 x 10^8 ergs per gram DC (i.e., a maximum deviation of 40 percent in the exposure). It is believed that the reproducibility should be roughly $\frac{1}{2}$ 10 percent. The greater variation here is attributed to nonequivalent cure in certain cases in the earlier study reported in Section 3.5, which took place over a year ago. The later study, which has been described in this section, benefitted importantly from the knowledge gained from the previous similar research.

Section 3.6.4. Summary and Conclusions

Eight rubber compounds were selected to represent each of four major classes of aircraft rubber products: namely, hose, packing compounds, fuel cells, and wire insulation. Each compound was then modified by the separate addition of two potential anti-rads, Akroflex C and quinhydrone, at a concentration of 5 phr.

The separate and combined effects of heat and radiation were studied using stress-strain, hardness, and compression set measurements for evaluation. Portions of the work are still being evaluated and will be summarized in a later report.

To test the effectiveness of these two potential anti-rads, Akroflex C and quinhydrone, two approaches were taken. All compounds were carefully given equivalent cures; and equivalent samples of the compounds underwent identical combinations of exposure to radiation and heat and testing, according to Test Methods I through VI, inclusive. The results of testing by Methods I, II, and III appear in this



report. Methods II and III are more drastic than I in aging the compounds in this study.

The elastomers that were employed in these compounds were neoprene, SBR, Hycar (NBR), and a Hycar-Vinylite blend. Gamma radiation causes predominantly crosslinking of said polymers. Their compounds increase in modulus and hardness with increasing radiation exposure. The heat and radiation aging processes were most satisfactorily tracked by determining the change in percent retained ultimate elongation. The greater change for these compounds consistently came in the region of $0-83.9 \times 10^8$ ergs per gram DC at room temperature (Method I) and $0-33.6 \times 10^8$ ergs per gram DC at 158° F. (Methods II and III).

Comparisons of radiation resistance have been made with graphs and tables. The comparisons were on the basis of the gamma radiation exposure required for a 50% reduction in ultimate elongation and of the reduction in the percent retained elongation after both 58.7×10^8 and 126×10^8 ergs per gram DC at room temperature and after 58.7×10^8 ergs per gram DC at 158° F.

SBR wire insulation and Hycar-SBR hose stock (85/14) showed the most inherent radiation resistance. Hycar self-sealing liner stocks apparently received the most overall benefit from the potential anti-rads Akroflex C and quinhydrone. The order of improvement (expressed as the protection ratio of quinhydrone-containing stock to control stock) according to Methods I, II, and III was 2.8/2.1/1.6, respectively. This rating was based on the radiation exposure required to reduce the ultimate elongation to 50 percent of the initial value.

Protection ratios were calculated for all compounds. Akroflex C and quinhydrone offered at best only marginal radiation protection to these highly pigmented, complex practical rubber compounds. These results support the growing conviction that many of the factory rubber stocks have coincidentally been protected against radiation damage in the course of being protected against more conventional types of aging. The best protection ratios are obtained when exposure and testing occur at room temperature. When irradiation takes place at 158°F., the anti-rad provides less apparent protection. It is conceivable that the difference may be due to heat damage, against which the potential anti-rad perhaps affords less or no protection, rather than to greater radiation damage per se.

A measurement of the duplicability of the test methods in the present study plus a comparison of independent results for duplicate compounds indicates approximate limits of variation of 4-10 percent.

The importance of accurate equivalent cures and careful specification and administration of exposure and testing was demonstrated again in this study.

Further compilation of stress-strain data for heat and radiation aging of the aircraft rubber compounds is still in progress. In addition, compression set studies for the same stocks are soon to be finished. Evaluation of the protection of Viton A against radiation-induced changes in stress-strain and compression set values by potential anti-rads is also in progress.

Contrails

Thus, the investigation of the relationship between heat aging and radiation aging and of their interaction has begun. In addition to the continuation of this research to permit the prediction of radiation resistance under any heat aging conditions, further work should be done to explore the protection offered to rubber by compounding ingredients. Also, work is recommended to extend the temperature range of investigation and to commence component and end-item testing under simulated service environmental conditions.

SECTION 4

RADIATION-RESISTANT RUBBER AIRCRAFT END-ITEMS

4.1. Aircraft Tire Irradiation Study

(This research was conducted and reported by J. W. Born.)

4.1.1. Introduction

The radiation research under this contract has two final goals. Chronologically, the first is to provide, insofar as is presently feasible, aircraft rubber end-items to exceed minimum engineering requirements for nuclear radiation service. Second only in probable time of achievement is to develop elastomeric materials with many times the best present radiation resistance in anticipation of the much more severe requirements of the future.

The above sections constitute a progress report on all phases except end-items. This section deals with the current status of end-item development.

The concern with actual end-items in this program at the Research Center dates from late in 1956. There had been a strong interest in the subject before that, but obvious technical difficulties of irradiating and testing any but the smallest rubber products were strong deterrents. However, recognition of the growing need for such research led to our decision to at least begin with the small items.

In the summary technical report for 1956 (3) it was concluded that sufficient knowledge had been accumulated to warrant a study of tires. It was stated, "It appears likely that such an airplane tire (as was described) would survive at least one landing after receiving a radiation exposure of 100 megareps at about 25°C., particularly if the rubber compounds and tire cord were protected from radiation damage by the addition of anti-rads." A tentative program of tire evaluation was then out lined in that report. This is a status report on said program.

The Type VII, $26'' \times 6.6''$, 14-ply rated, tubeless natural rubber tire with nylon cord was chosen. It was a tire in current use on military aircraft. In addition to four conventional tires, four more were made which were identical except for the

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addition of 5 phr of Akroflex C (35% N, N'-diphenyl-p-phenylene diamine plus 65% phenyl-alpha-naphthyl amine) to each rubber compound used in the tire. It was reasoned that the nylon cords might acquire protection secondarily by becoming impregnated with excess Akroflex C as it migrated out of the rubber. Akroflex C had proved to be one of the best anti-rads for natural rubber (3).

The four tires of conventional composition served as controls. The two types of tires were matched in pairs, one control with one experimental tire, for testing. Four test conditions were employed: namely, without anti-rad and without irradiation, with anti-rad but without irradiation, without anti-rad but with irradiation, and with anti-rad and with irradiation. There were thus three possible cross-comparisons in one overall study.

Gamma irradiation was chosen in preference to combined neutron and gamma irradiation. The selection was based on the conviction that equal doses of high energy radiations cause the same degree of damage to any given elastomeric material, regardless of whether neutrons or gamma rays are concerned. The reason for our preference was to avoid the radioactivation which neutron irradiation induces and which complicates subsequent testing. This decision did not rule out the possibility of later studies using reactor radiations. Indeed, at the completion of the present study such an extension is planned and will be conducted, if funds become available.

Only one available gamma radiation facility had adequate capacity for irradiating a tire of even these modest dimensions. This was the Materials Testing Reactor (MTR) Gamma Facility of the National Reactor Testing Station (NRTS) at Idaho Falls, Idaho (Appendix I). Accordingly, with the very capable cooperation of the personnel at that facility, an irradiation program was formulated. It called for gamma irradiation in air at room temperature yet under water in the unclassified "swimming pool" facility, using spent reactor fuel elements. A total integrated exposure dose of 83.9 x 10⁸ ergs per gram of carbon was selected. It was to be administered in two equal increments, so that each side of the tire would receive equal doses of the latter value.

Thus, a special container was required which would (1) remain watertight during irradiation under 14 feet of water, (2) be blow-out proof, (3) permit inflation of the tire at the bottom of the pool, continuous maintenance of pressure during the exposure period, and deflation before removal from the pool, and (4) allow easy inversion of the tire-wheel assembly to obtain equal exposures of both sides.

Finally, arrangements were made with the personnel of the Aircraft Laboratory and the Materials Laboratory for full-scale indoor testing of the tires at Wright Air Development Center. Only after these advance preparations involving four technical organizations had been completed could experimentation begin.

4.1.2. Experimentation

The experimentation involved the following phases: (1) obtaining approval for tire manufacture, for use of the MTR Gamma Facility, and for tire testing at the Aircraft Laboratory at WADC, (2) construction of eight Type VII, $26'' \times 6.6''$ aircraft



tires, (3) design and construction of a container for the tire during irradiation, (4) preparation of small laboratory rubber samples of the tire compounds for irradiation with the tires, (5) dosimetry and calculation of the exposure periods, (6) irradiation of the tires and small samples, (7) tire and sample testing, and (8) evaluation of the results.

Certain criteria defined the scope and conditions of this first study of tire resistance to radiation damage. Previous research, particularly measurements of dynamic physical properties of rubber compounds after irradiation (3), led to the selection of a radiation exposure of 83.9 \times 10^8 ergs per gram of carbon for each aircraft tire. Gamma irradiation was chosen in preference to combined neutron and gamma irradiation to avoid neutron radioactivation of test materials and equipment. This choice was based on the conviction that equal doses of highly penetrating radiation energy cause equivalent degrees of radiation damage to rubber, regardless of whether neutrons or gamma rays are involved. The tires were to be inflated with nitrogen gas to minimize accelerated oxidation during irradiation. Room temperature was chosen for irradiation to simplify this initial evaluation. Personnel safety was made one of the principal criteria, since certain potential hazards were involved. Specifically, the precautions involved design of the irradiation container and the inflation pressure of the tire at various stages. The competing needs to test enough tires to get reliable results and yet to complete the study within a reasonable time dictated the use of eight aircraft tires. The size of the tire irradiation assembly necessitated the use of the MTR Gamma Facility. The convenience and close technical supervision of testing which was available at the WADC Aircraft Laboratory made it the preferred site for full-scale simulated service testing.

The Tire Testing Department of the Tire Manufacturing Division of the B.F. Goodrich Tire Company constructed the twelve aircraft tires for radiation testing. Mr. M. A. Reinhart and Mr. L. J. Vandenberg supervised the compounding, and Mr. W. N. Dickerson directed the construction of the tires. Six of the tires were of completely conventional composition. The other six differed in composition only in the incorporation of 5 phr of the anti-rad Akroflex C into each rubber compound in the tire. The Type VII, 26" x 6.6", 14 ply rating tubeless tire with nylon cord was chosen. The elastomer content was predominantly natural rubber. Akroflex C had proved to be among the most effective anti-rads for natural rubber (3). Each tire contained six compounds corresponding to the tread, the overhead ply, the carcass, the bead and plylock filler, the bead insulation, and the air-retaining liner. Except for the changes noted, this tire was in current use on military aircraft at the start of this study.

The twelve rubber formulations which were used in the eight tires plus two quality control tires of each type were compounded from the same fresh supplies of ingredients by the same standard procedures. Sufficiently large batches were prepared and cross-blended for uniformity to make all six tires of each type. The twelve tires were molded and cured in a standard manner, and nine were delivered to the Research Center for the testing program.

Since it is very difficult to make the conventional laboratory physical measurements on the compounds in a tire after its construction and cure, special samples



were made of each compound for physical testing. The tests involved standard ASTM methods for the measurement of tensile strength, ultimate elongation, and modulus (D412-51T), hardness (D314-52T), static modulus, dynamic modulus, Yerzley resilience, and deformation load (D945-55), and temperature rise and permanent set during dynamic compression (D623-52T, Method A). The samples were selected for irradiation with two of the four tires. Specifically, the samples without Akroflex C were scheduled to be irradiated with the second tire containing the same compounds. Likewise, the samples containing Akroflex C were scheduled to be irradiated with the first of the two tires containing these same compounds.

The MTR Gamma Facility appeared to be the only available gamma facility which had both a large enough space to accommodate a tire-wheel assembly and rotating apparatus and a high enough and uniform enough radiation flux throughout the minimum space to make a tire irradiation feasible. This facility had been used previously by B. F. Goodrich personnel in the first vulcanization of an uncured automobile tire by nuclear irradiation (20). Twenty spent fuel elements from the Materials Testing Reactor had been used in a planar rectangular array (Figure 84) as the gamma radiation source. The elements were located on a steel table at the bottom of the pool under approximately 16 feet of water. The same arrangement was chosen for use in this study. The tire-wheel assembly would therefore be placed under water as close to the fuel elements as practicable. Consequently, a special container was required to isolate the tire from the water of the pool during irradiation.

A schematic diagram of the canister assembly which was designed appears in Figure 85. The canister proper measured 27-1/4 inches in diameter and 8-1/2 inches in depth as interior dimensions. It consisted of two main half-shells with numerous attachments, as shown in the latter figure. The parts are as follows, using the same numbering as in the figure: (1) one half of the canister, (2) the other half of the canister, (3) the lower extension of the drive shaft, (4) the upper extension of the drive shaft, (5) the long tie-bolt, which holds the two halves securely together and whose head has a hexagonal socket for the end of the drive-shaft, (6) the 32 rimbolts for clamping the two halves together, (7) the metal hose for inflating and deflating the tire under water, (8) a 4"-diameter safety exhaust hose, (9) a needle valve in the nitrogen gas line to the tire which is open during irradiation, (10) an identical needle valve which is closed during irradiation, (11) a copper tube which connects the external needle valve to the tire valve, (12) a copper tube which connects the other external needle valve to the tire valve, and (13) the tire valve Tconnection to the two copper tubes. Gasket A is an SRB rubber seal, as are Gaskets B and C. Gasket D is a grooved aluminum gasket.

The resulting canister assembly was necessarily designed to be completely invertible. This feature was essential because of geometric characteristics of the radiation field and absorption of the radiation by the assembly. The only way to obtain equal exposure of both sides of the tire to radiation was to invert the canister, containing the tire-wheel assembly, midway through the exposure. Therefore, Parts 3 and 4 had to be readily reversible; and interchangeable connections of Part 7, the gas line, to Part 13, the tire valve T-connection, were necessary. The design in Figure 85 acheived these requirements. It was developed with the able assistance of Mr. W. R. Bidwell of the Engineering Design Group, who subsequently supervised its construction.



The basic canister was made by Frank J. Stolitzka and Son of Akron, Ohio. It was made of 3/8-inch thick aluminum sheet and had the shape of a cylindrical pillbox. The modifications and accessory parts, which were described above, were made in the machine shop at the Research Center. In addition to the parts already named, aluminum sample straps were made and installed inside the canister halves. They were arranged radially with ten in each half, equally spaced apart in mirror-image orientation, as shown in Figures 86 and 87. Figure 86 is a schematic drawing, and Figure 87 is a photograph of the completed canister. Including the gasket compressed nominally to 1/16-inch thickness the interior dimensions were 8-1/2 inches in depth and 27-1/4 inches in diameter.

After construction the canister, its accessory parts, and the first tire-wheel assembly were sent to the MTR Gamma Facility, along with all the necessary tools and materials for assembly. Then, the canister assembly was tested underwater in the pool in a simulated run. Mr. A. M. Holman and Mr. M. Johnson of the staff at the facility provided a great deal of able assistance. A number of adjustments and modifications became necessary. The valve fittings had to be tightened to prevent leakage. The tire valve T-connection had to be modified to give trouble-free service. The canister containing the tire-wheel assembly had to be weighted with lead bricks (ca. 75 pounds) to overcome its buoyancy and cause it to sink. Rubber gaskets of SBR elastomer had to be substituted for the initial soft lead gaskets to prevent the last traces of leakage. The lead gaskets had been chosen because of concern about radiation-induced failure of rubber, but the rubber gaskets served very well during the long exposures.

Finally, thanks largely to the efforts of the facility staff including Mr. D. L. Hunke, project engineer, the necessary in-service improvements in the equipment were made. The design proved entirely sound. The equipment thereafter functioned very well, requiring no further modification. Figure 88 illustrates the complete assembled apparatus in operation in the pool facility. As an additional safety feature Mr. D. L. Hunke devised an aspirator system with tubes running inside the exhaust hose to the bottom of the canister. Its purpose was to detect and offset any leakage which might conceivably have developed during irradiation.

The successful performance of the canister left only two steps before the irradiation of the tire could begin. These were initial dosimetry inside the canister and calculation of the irradiation period required for an exposure of 83.9 \times 10⁸ ergs per gram of carbon.

At the outset little information was available to indicate what the initial flux inside the canister or what its rate of decrease with time would be. The only information on hand included the fluxes at a given distance from each individual spent fuel element at some earlier date and an empirical equation which predicts the rate of radioactive decay of a typical element (21).

Ceric sulfate dosimetry such as was described in <u>Dosimetry of a Kilocurie Cobalt-60 Source</u> (22) was used to measure the flux values inside the canister. Bakelite screw-capped glass vials containing aqueous ceric sulfate solution were attached to the sample straps in the positions indicated in Figure 89. A total of 40



dosimeter bottles were placed on straps A-E, inclusive, for each dosimetry run.

The dosimeters were Scotch-taped in these positions, which corresponded to locations on the surface of the tire when it was inside the canister. The canister was then rotated at 0.97 rpm by the drive-shaft of an electric motor. The purpose of the rotation was to correct for inhomogeneities in the radiation field. The dosimeter solutions were analyzed in the MTR laboratories. The analytical values were reported as fluxes in megaroentgens per hour. A typical set of values, namely those obtained at the start of the first tire irradiation, are shown in Table XXXVI. This table also shows how the results were evaluated in every case. The dose rates were averaged for each two corresponding positions above and below, such as Al and A7, A2 and A6, A3 and A5, and so forth. Average values of the initial fluxes were thus obtained for the bead, sidewall, shoulder, and tread locations on the tire.

Since no information was available as to flux decay rate inside the canister, it was necessary to improvise in order to predict the proper exposure period. Reference (21) gives the following equation for the flux at 6 inches from the center of the face of a typical spent fuel element in water from 20 to 200 days after discharge from the reactor:

flux,
$$f(r/hr) = 4.2 \times 10^7 t^{-1.21}$$
,

where t is the time after discharge in days. Since these conditions approximated those in this study, it was reasoned that an equation of this general form could be used.

However, the flux at any point inside the canister would obviously be the synthesis of the contribution from each of the 20 elements in the array. Also, the observed rate of decay would be modified by absorption of radiation by the equipment. Therefore, the above equation had to be modified. The modification was made on the basis of the dosimeter values.

The general equation for the flux was taken as $f(r/hr) = Ct^{-1} \cdot 21$, where t is expressed in days. Ceric sulfate dosimetry gave average flux values inside the canister as a function of average age of the 20 spent fuel elements which were used. Therefore, f and t were known, and C could be calculated. Since the flux decreased with time, it was necessary to integrate the flux over a certain period of time to obtain a given radiation exposure. That is, the radiation exposure D equals the product of flux and exposure time (D = ft). By using the above flux equation and converting to units of r/day, the following expression was derived for exposure in roentgens-equivalent-physical:

D (reps) =
$$\int_{t_1}^{t_2} f dt = -114C \left[t^{-0.21} \right]_{t_1}^{t_2}$$

The t_1 and t_2 values are the average ages of the 20 spent fuel elements at the beginning and end of the period of time required for the given exposure. Since t_1 , the start of the period, is known, t_2 can be calculated. When dosimetry measurements



had been made at the end of the exposure, two sets of values for f and t were available. Consequently, the value of the exponent could be determined more accurately from the relationship $f_1/f_2 = t_1^{-x}/t_2^{-x}$. Once x was known, C could also be determined accurately. Because of the pioneer nature of this work, these calculations were made anew for each tire irradiation. The results will be discussed in the next sub-section.

Now that all of the equipment, techniques, and operations had been worked out satisfactorily, the actual tire and sample irradiations could begin.

A standard procedure was developed for each tire irradiation. New rubber and aluminum gaskets were used each time. Dosimetry measurements were made before each tire irradiation. Then, the tire, already mounted on its aircraft wheel, was put on the axle inside the canister. The gas lines were attached to the tire valve T-connector (Figure 85), and the rubber gaskets (A, B, and C) were put in place. The tie-bolt was screwed in but not tightened. Then, the rim and flange bolts were tightened evenly in turn to compress the gaskets from 25 to 50 percent. Next, the tie-bolt was tightened. The function of the tie-bolt was to resist expansion and rupture of the circular faces of the canister in case of tire blow-out during irradiation. Dr. C. H. Stockman and Mr. H. F. Neff calculated that a force in excess of 175,000 psi could become concentrated at the center of the faces as a result of bulging. As an added precaution against bulging and rupture, the exhaust tube was provided to relieve any excess pressure inside the canister. Following this, the external needlevalve on the under side was closed. The nitrogen gas hose was connected to the needle-valve on the upper side, which was then opened. The tire was inflated to 50 psi with oil-pumped nitrogen gas, and the entire canister assembly was lowered into place in the pool facility by chain hoist. The motor drive-shaft was connected to the upper drive-shaft extension rod, and the rotation of the canister assembly was begun. The inflation pressure of the tire was increased to 210 psig and was maintained at 200-210 psig throughout the irradiation. At the end of the exposure, the pressure was reduced to 50 psig before removing the canister assembly from the pool. This was safety precaution.

The lower drive-shaft extension rod rested on a ball-bearing beneath the table which supported the fuel element array. Figure 90 shows the regular arrangement of elements and the opening in the center of the table. The drive-shaft assembly was rotated at 0.97 rpm by an electric motor, thereby rotating the canister and tire to obtain uniform irradiation. Figure 88 shows the equipment assembled and in operation.

When the tire had received an average radiation exposure of 420 x 10⁷ ergs per gram DC, the canister was removed from the pool. The two drive-shaft extensions were interchanged, the gas line was connected to the other external needle-valve, and the canister was inverted and put back in place for irradiation from the other side of the tire. The second half of the total radiation exposure was applied, and then the irradiation was stopped. The concluding dosimetry measurements were made of the average flux at the conclusion of the exposure. The tire was then shipped to WADC for testing in the Aircraft Laboratory.



Standard ASTM rubber samples were wrapped individually in aluminum foil and attached to the sample straps with Scotch-brand electrical tape. The samples included four Yerzley resilience pellets, two Goodrich Flexometer buttons, and one Pico abrasion button per compound on one sample strap and 24 Die D Scott dumbbells wrapped in groups of three in foil per compound on a second sample strap. Such a set of test samples without anti-rad present was irradiated with the second tire containing no anti-rad. A similar set of samples containing the anti-rad (Akroflex C) was irradiated with the first tire having anti-rad present, which was the third tire irradiated. The purpose of irradiating and testing samples of the individual compounds was to attempt to relate changes in the properties of the compounds to changes in the test performance of the tire in question. Samples were selected to measure critical physical properties such as abrasion resistance, hysteresis, dynamic modulus, and resilience at pertinent tire locations such as tread, shoulder, sidewall, and bead. Subsequently, the small samples were returned to the Research Center for testing.

The tire testing was conducted on full-scale dynamometer equipment in the Aircraft Laboratory at WADC. The test apparatus is shown in Figure 91 at the completion of testing the first irradiated tire. Two series of high and low speed landings were made for each tire at half-hour intervals. This allowed the temperature to equalize and approximate room temperature between landings. It is general practice to conduct three (3) low speed break-in landings of a tire prior to the high speed landing tests. However, the irradiated tires were treated as exceptions because of their radiation-damaged condition. Only one low speed landing was used as a break-in. No other exceptions to the usual test conditions were made. The unirradiated control tires underwent the same landing test procedures as the irradiated experimental tires.

Appendix IX describes the specifications for the two series of tests in detail. A high speed landing requires deceleration at a controlled rate from a peripheral speed of 200 mph to less than 100 mph, until a roll distance of 7000 ± 250 feet has been covered. The series consists of 25 cycles. The low speed landing requires deceleration from 90 to 0 mph, during which the tire absorbs the 887,000 footpounds of kinetic energy. The test data comprise the number of landings, total number of flywheel revolutions, miles of roll, and temperature of the tire tread before and after landing.

For the small rubber samples, standard ASTM test methods were used as follows (22): D15-55T for compounding, mixing, molding, and curing; D412-51T for stress-strain testing; D945-55 for Yerzley measurements of static and dynamic modulus, resilience, and deformation load; D623-52T, Method A, for temperature rise and permanent set during dynamic compression. Abrasion was measured on a testing machine developed at this laboratory and named the Pico abrader. The machine is described in U. S. Patent 2,799,155 (23).

4.1.3. Discussion of Results

Three of the four tires have been irradiated. The irradiation of the fourth tire is now in progress and will end on April 14, 1958. Two of the irradiated tires have



been tested, namely the two tires without anti-rad present. All four unirradiated control tires have been tested. The irradiation equipment has functioned very well, and the radiation test procedure has been highly satisfactory.

After the early minor modifications, chiefly the replacement of lead gaskets with SBR rubber ones, the aluminum canister and accessory equipment functioned perfectly. The tire inflation pressures were maintained throughout each entire exposure period between 200 psi and the nominal operating pressure of 210 psi, with a total nitrogen gas consumption of only 280 psig over an irradiation period of 62 days, as a typical example. The irradiation equipment is therefore considered to be completely satisfactory.

The test data for dosimetry, integrated exposure, and general radiation history appear in Tables XXXVII-XXXIX for the first three irradiated tires. These included the two tires of conventional composition and one of the two containing the anti-rad Akroflex C. The dynamometer landing test data are summarized for all three tires in Table XL. For convenience the tires, which are fully identified in the former three tables, will be referred to hereafter as Test Tires 1, 2, and 3. The corresponding unirradiated tires will be designated Control Tires 1, 2, 3, and 4, similarly.

Military specifications require that a conventional aircraft tire of this type must withstand 25 high speed landings plus at least about 150 low speed "fatigue" landings on the dynamometer equipment. The following evaluations can be made of the data in the latter table.

Control Tire 1 failed during the 76th low speed landing with a ply separation in the shoulder. This tire therefore did not meet minimum specifications and so was an unsatisfactory control specimen. Its unexpected early failure illustrates that in spite of careful construction and cure, occasionally a tire will prove to be substandard. However, Control Tire 2 was satisfactory, Control Tire 3 was above average, and Control Tire 4 was considerably superior to any other tire of this type which has ever been tested in this manner before.

The testing of Control Tire 2 was stopped after 147 low speed landings because of a slight blister on the shoulder and severe bead chafing. Control Tire 3, containing anti-rad, failed after 204 low speed landings because of severe ply separation evidenced by two blisters. The 204 figure represented very good performance. Control Tire 4, also containing anti-rad, failed on the 324th fatigue landing with a ply separation in the shoulder and some air leakage in the bead region. The 324 figure was exceptionally high and represented superlative performance. Further studies are now in progress to explore this discovery and potential break-through. All four control tires showed practically the same rise of 115°F. in tread temperature during landing tests.

Test Tire 1 failed at the completion of the 18th high speed landing with a bead-to-bead blow-out and a small second break in the sidewall (see Figure 91). The main break was parallel to one cord direction and therefore across the other cord direction, apparently following a path of least cord resistance. This type of failure was consistent with our expectation that failure of the cord would determine the test



"life" of an irradiated tire. This tire failed after 26 miles of "travel," roughly one-tenth the total distance expected of a conventional unirradiated tire. Test Tire 2 withstood all 25 high speed landings and failed at the start of the first low speed landing, also by a nearly bead-to-bead blow-out. Again, the line of break cleanly paralleled one cord direction. The latter irradiated tire "traveled" roughly 36 miles before failing. The detailed report on this tire has not arrived yet.

The pressure and temperature rises during testing did not vary significantly from one to another for any of these six tires. The increase in tire pressure during the landings is proportional to the increase in temperature of the contained air. The normal increase is 25 psi for high speed landings and somewhat more for the low speed tests.

The simulated high speed landing is an attempt to impose the same dynamic forces on the tire which it would receive in some idealized landing at high speed. In continuation, the low speed test represents the deceleration of an aircraft to a stop under load, during which 887,000 foot pounds of kinetic energy are absorbed by the tire.

Test Tires 1 and 2 failed at opportune numbers of landings. That is, they showed significant damage and yet not a complete loss of landing capabilities. This raises an important point. Even after this 83.9×10^8 erg per gram DC exposure, which is large in a practical sense, it appears highly probable that a number of safe landings could be made. It was interesting that no apparent "growth" of the tire took place during irradiation.

The testing of all the small samples of the compounds of conventional composition without the anti-rad has been completed. However, not all the results have been tabulated yet. The stress-strain values are tabulated in Table XLI and evaluated in Table XLII. The results for each property and each sample location are the median of three values. The control value is the median of seven in each case. The deviation in perhaps 80 percent or more of the cases was \pm 15 percent of the average value for tensile strength. The values for ultimate elongation and 100 percent modulus showed much wider variations, as indicated in Table XLI. Variations were to be expected, however, because of the different sample locations and the geometry of the radiation field. It is more meaningful to compare the samples in locations 1 versus 7, 2 versus 6, and 3 versus 5, and to consider 4 in a comparative sense.

If both sides of the tire (Control Tire 2) actually received equal radiation doses, then each such pair of sample trios should have the same physical properties, allowing for variations from other causes. In the majority of cases the agreement is very good for tensile strength and fairly good overall for ultimate elongation and modulus. Taken altogether, the data show good agreement, considering the many possible causes of variation, and indicate that in general the two sides of the tire received the same radiation dose. Furthermore, on the basis of past results it appears fairly certain that these laboratory samples received radiation exposures of at least very nearly, if not exactly, 83.9 x 10⁸ ergs per gram DC. That is, their stress-strain data are practically the same as those of other similar natural rubber samples after a known exposure of this exact amount.

This leads us logically to the question of how close to 83.9 x 10^8 ergs per gram DC each integrated tire radiation exposure came. As stated in Tables XXXVII-XXXIX, the three integrated average exposure doses were 78.9×10^8 , 78.9×10^8 , and 91.4×10^8 ergs per gram DC for the first three irradiated tires, respectively. These correspond to 94, 94, and 109 percent of the desired exposure. Figure 92 is an empirical plot of the flux inside the canister versus the average age of the 20 spent fuel elements. The points unambiguously determine a single curve, which should be very useful in any future such tire irradiations. Similar calculations to those above indicated that in all three cases the two halves of each total exposure were equal in magnitude to well within 5 percent maximum deviation.

4.1.4. Summary and Conclusions

Eight aircraft tires of current military type were constructed. Four were of conventional composition. The other four were of identical design and had the same composition, except that each rubber compound contained 5 phr of the anti-rad Akroflex C. Two tires of each type served as unirradiated controls and have been tested with the dynamometer equipment of the Aircraft Laboratory at WADC. Of the remaining four tires, the two without anti-rad have received radiation exposures of 78.9×10^{9} and 78.9×10^{8} ergs per gram DC at the MTR Gamma Facility. These two tires have been tested and failed after 18 and 25 high speed landings and 26 and 36 miles of roll, respectively. The first of the two anti-rad tires has received an exposure of 91.4×10^{8} ergs per gram DC and is awaiting testing at WADC. The second of the latter two, which is the last tire in this opening study, is now being irradiated. Its exposure will end on April 14, 1958.

The canister in which the tires have been irradiated and its accessory equipment have functioned very well. It is capable of serving equally well in other radiation facilities for exposure to neutrons as well as gamma rays. The mathematical method which was devised for predicting the correct tire irradiation period proved accurate to within \pm 10 percent. An empirical plot was obtained which gives the average flux inside the canister as a function of the age of the spent fuel elements.

The first set of small test samples of the tire compounds without anti-rad present has been tested. The stress-strain data indicate in general that the two sides of the second tire received the same radiation dose. It appears further that these samples received exposures which were very near or equal to 83.9×10^8 ergs per gram DC.

It is concluded that the exposure period necessary for a given radiation dose can be predicted with less than 10 percent error from the initial dosimetry. The two conventional tires which received exposures of 78.9×10^8 and 78.9×10^8 ergs per gram DC, respectively, failed at opportune numbers of landings. That is, they showed both significant radiation damage and yet not a complete loss of landing. capabilities.

Thus, even after this ca. 83.9×10^8 erg per gram DC exposure, which is large in a practical sense, it appears probable that a number of safe landings could be made. It was interesting to observe that no apparent "growth" of the tire took place during irradiation.



No irradiated anti-rad tire has been tested yet. Therefore, no conclusion can be drawn for this report about protection against radiation damage to the tire.

The initial indications are that both irradiated tires failed because of cord weakness. This supports the early opinion that the nylon cord would be the least resistant part of the tire to radiation damage. Other tire cords are now known to have greater radiation resistance (see Section 3.2). Future studies involving tires with the most resistant cords appear worthwhile.

It is possible that other recommendations for the construction of more radiation-resistant tires can be made when the test results become available for the irradiated anti-rad tires.

4.2. The Proposed "O" Ring Irradiation Study

4.2.1. Introduction

The Materials Laboratory of WADC has indicated that, after tires, "O" ring seals are of most interest from the standpoint of resistance to radiation damage. Therefore, the following tentative program for radiation research has been formulated. It resulted from a discussion among E. R. Bartholomew and W. R. Griffin of the Rubber Products Section of WADC, H. G. Gillette of the Precision Rubber Products Corporation, and J. W. Born of the B. F. Goodrich Company Research Center. The program, which was mutually agreed upon by these personnel, has been approved by their respective organizations. It is scheduled to be put into effect in 1958.

4.2.2. Proposed Program

The proposed program calls for fabrication and adjustment of the resulting physical properties of "O" rings, composed of both conventional and experimental polymers with and without anti-rads present, by the Precision Rubber Products Corporation. The anti-rads will be selected and supplied by the B. F. Goodrich Company. The former company will measure the original properties of the "O" ring compounds as a quality control step as well as for experimental control purposes. The latter company will undertake nuclear irradiation of the samples of "O" ring compounds and end-items. Subsequently, the Precision Rubber Products Corporation will perform post-irradiation measurements of the same physical properties. Then, all experimental test data will be reported to the B. F. Goodrich Company, which will evaluate the test results in terms of radiation resistance and report to WADC. The following details have been approved for the program.

4.2.2.1. Sample Preparation

Only "O" ring formulations are to be considered from the general class of rubber seals. A minimum number of compounds has been chosen by the Precision Rubber Products Corporation to represent the three major applications; namely, in hydraulic oil, engine oil, and fuel. In the initial study these will be the six approved rubber compounds as stated by Mr. Gillette:



Specification	Usage	Compound
MIL-P-25732	Hydraulic, -65° to 275°F.	737-70
MIL-P-5315	Fuel, -65° to 250°F.	VL-123X5
MIL-R-7362B (Proposed)	7808 Oil, -65° to 300°F.	146-70
MIL-R-6855, Cl. I and II	Fuel and Oil, -65° to 300° F.	718-60
(Neoprene)	Silicate Esters, -65° to 400°F.	227-70
(Viton A)	Silicate Esters, -65° to 550°F.	1700-70

In addition to, and possible prior to, testing of "O"ring end-items, measurements of compression set, hardness, modulus, and volume swell between 0 and 15 percent are considered most important. Hardness measurements will be made on the compression set buttons. Modulus measurements will be made on standard Scott dumbbells (die C) along with tensile strength and ultimate elongation determinations. Volume swell measurements can be made on 1" x 2" x 0.075" samples taken from the tensile sheets. Standard ASTM samples will be used wherever possible.

Three organic chemicals which have proved to be effective anti-rads will be supplied by the B.F. Goodrich Company to the Precision Rubber Products Corporation. Each in turn will be mixed into each of the five "O" ring compounds, excepting the Viton A compound, in the various amounts of 0.5, 1.0, 2.0, and 4.0 parts per hundred parts of rubber polymer. Three other anti-rads will be supplied especially for the Viton A compound on the basis of results from the present screening study. The approved "O" ring compounds will thus serve as master compounds for the preparation of the anti-rad protected compounds. They will also serve, when molded and cured, as control sample stocks for radiation testing.

Since the addition of an anti-rad is likely to alter the time required for optimum cure at a given temperature, it may be necessary to re-establish the optimum cure conditions. In some cases the anti-rad will have a plasticizing effect, reducing the modulus at optimum cure. Compounding research will be necessary in such cases to restore the modulus to that of the control compound. It appears from our studies that increasing the carbon black content may suffice. Fortunately, addition of the anti-rads does not seem to change the hardness significantly where the concentration is 5 pphr or less.

The AN6227-19 "O" ring, which measures $1^n \times 1-1/4^n \times 1/8^n$ nominally, will be used throughout this study. This is the size desired by the Precision Rubber Products Corporation and WADC. It is also a highly convenient size for irradiation.

4.2.2.2. Sample Irradiation

All samples will be submitted to the B.F. Goodrich Company Research Center for irradiation. Tentative plans call for radiation exposures of 10, 40, 70, 100, and possibly 130 megaroentgens at the MTR Gamma Facility. A megaroentgen is 83.9×10^6 ergs per gram of carbon. Initial irradiations will be conducted at room temperature. Subsequent irradiations will be at elevated temperatures, guided by the results of the room temperature study. The compression set samples will be



compressed throughout the irradiation.

4.2.2.3. Sample Testing

The Precision Rubber Products Corporation will test samples of each type and compound prior to irradiation for two main purposes. First, it is necessary to insure that the samples meet design specifications. Second, initial control test data are necessary.

In addition to these initial control test samples, final control test samples, both irradiated and unirradiated, will be submitted with the irradiated experimental samples to the latter company for testing.

All test results will be reported to the B.F. Goodrich Company along with any necessary evaluations of the overall results. Standard ASTM or Military Specification Tests will be used wherever possible.

4.2.2.4. Size of Research Program

In order to implement this program it was necessary to consider how many samples will be involved. There will be four types of samples: namely, compression set pellets, Scott dumbbells, volume swell strips, and "O" rings. The following numbers of samples are required per compound per radiation exposure (including the "zero exposure"):

compression set pellets: 2
Scott dumbbells: 5
volume swell strips: 2
"O" rings 18

Therefore, 27 samples are required for each compound at each radiation exposure. Since there are 24 compounds and 6 exposures, a total of nearly 4,000 individual samples will be involved.



GLOSSARY

- acrylonitrile vinyl cyanide (VCN), CH2*CHCN.
- age resister an additive for rubber or plastic compounds which inhibits deterioration in physical properties caused by environmental factors such as heat, oxygen, and ozone, for example.
- anti-rad a material which inhibits radiation damage, particularly when incorporated into organic materials.
- butadiene 1,3-butadiene (vinyl ethylene), CH_2 -CH-CH- CH_2 .
- cis-trans isomerization the transformation of a cis configuration about an ethylenic double bond in a molecule to a trans configuration about the same ethylenic double bond.
- collisions of the second kind molecular interactions in which excitational energy is transferred from an excited atom or molecule to an unexcited atom or molecule.
- copolymer a very large molecule which is formed when two or more different monomers (small molecules) are combined by polymerization.
- Dacron du Pont's synthetic polyester; a condensation polymer obtained from ethylene glycol and terephthalic acid, that is, polyethylene terephthalate.
- degradation reduction in molecular weight by scission of chemical bonds.
- denier the weight in grams of 9,000 meters of a textile filament or yarn.
- dosimeter a chemical system or an instrument for the measurement of radiation energy, particularly a radiation dose.
- dumbbell a stress-strain test sample which is cut from a relatively thin sheet of rubber or plastic with a metal die.
- end-item a manufactured product, or distinct integral part thereof, which has a distinct service function.
- EPA- the abbreviation for experimental polyamide, which is the general designation for the family of polymers to which the various nylons belong.
- G the reaction yield, which is defined here as the number of cis double bonds isomerized to trans double bonds per 100 ev of energy absorbed by the whole solution. More generally G is defined as the yield of any product per 100 ev of energy absorbed by the system under study.



- Go the "initial" reaction yield, which is determined by extrapolating the curve for G versus radiation dose back to zero dose.
- Go the reaction yield in terms of the number of cis double bonds isomerized to trans double bonds per 100 ev of energy absorbed by the sensitizer, extrapolated to zero radiation dose.
- gel that portion of a polymer, usually produced by crosslinking, which is insoluble (as opposed to the soluble sol portion); that apparently solid, often jelly-like material which is formed from a colloidal polymer solution on standing.
- intrinsic viscosity the specific viscosity divided by the volume concentration of a solution or suspension (the volume concentration, c, is defined as the volume of the dispersed phase per 100 cm. ³ of the suspension or solution).

joule - 10⁷ ergs.

- monomer the simple unpolymerized form of a compound, having a relatively low molecular weight, which may be used either alone or with a different monomer or monomers to form various types and lengths of molecular chains called polymers or copolymers; simplest repeating structural unit of a polymer.
- NBR the American Chemical Society designation for an elastomeric copolymer of acrylonitrile and butadiene.

nylon 6 - polycaprolactam.

- nylon 66 a copolymer of hexamethylene-diamine and adipic acid; polyhexamethylene adipamides.
- TT complexing-the attachment, usually very brief, of an atom or free radical to a carbon-carbon double bond of an organic molecule through a coupling of the unpaired electron of the atom or free radical to one of the Telectrons of the double bond, giving rise to a complex which is again a radical (due to the second Telectron, now unpaired) in which structure the original double bond has become a single bond. Of the 4 electrons constituting a double bond, the two which are associated with the particular reactivity of the associated double bond are called Telectrons.

percent gel - the percent of a polymer mass which is insoluble.

perlon - du Pont's synthetic polymer, polycaprolactam (nylon 6).

polyamide - a polymer in which the imido group - C - NH - recurs with each repeating structural unit.

polymer - a high molecular weight material containing a large number of repeating structural units.

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- radiation damage any particular detrimental effect which is induced in a material by irradiation.
- radiation dose the actual amount of radiation energy absorbed by the material in question when it is placed in a radiation field.
- radiation exposure the amount of radiation energy which is absorbed by a material in a radiation field as computed by multiplying the irradiation time by the flux determined by dosimetry (also termed "exposure dose").
- radiolysis the dissociation of a molecule induced by ionizing radiation, analysis to photolysis for a photochemically-induced dissociation.

reduced viscosity - is give by
$$\frac{\mathcal{N}_{sp}}{c} = \frac{\mathcal{N}_{r}-1}{c}$$

- SBR the American Chemical Society designation for an elastomeric copolymer of styrene and butadiene.
- short-stop: (noun) a chemical ingredient which is added to a polymerization system to terminate the chain reactions; (verb) to terminate a polymerization by adding a chemical ingredient to the system.
- sol a general term for colloidal dispersions, as distinguished from true solutions.
- stereospecific having a specific, preferred structural configuration; as, for example, the arrangement of monomer units in all cis-1, 4 or all trans-1, 4 polybutadiene, in contrast to the random mixture of cis and trans units in an ordinary or non-stereospecific polybutadiene.
- styrene the monomer from which polystyrene is prepared; one of the comonomers in SBR (vinylbenzene).
- swelling index the ratio of the weight of a swollen gel to the weight of the dried gel.
- synergism the cooperative action of discrete agents or substances whose combined effect is greater than the sum of their separate effects.
- tenacity tensile strength, particularly of textile filaments and cords; the breaking strength of a material when subjected to a tensile (stretching) force.
- VCN the common abbreviation for acrylonitrile (vinyl cyanide).
- viscometer viscosimeter, a device for measuring the viscosity or resistance to flow.

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APPENDIX I

DESCRIPTION OF GAMMA IRRADIATION FACILITIES

Brookhaven National Laboratory

Radiation Sources: Stainless steel-clad Cobalt 60 elements in either lead shielding containers or a water tank.

Flux Values: 1.26 x 10⁷ to 5.45 x 10⁷ ergs per gram DC per (hour).

Dosimetry: Aqueous ferrous sulfate dosimeters and ion chambers are used.

B. F. Goodrich Company Research Center

(The use of this facility is restricted to the B. F. Goodrich Company and WADC under the subject contract.)

Radiation Source: A cylindrical Cobalt 60 element in a lead shielding container.

Flux Values: 1.2 x 10⁷ to 1.1 x 10⁷ ergs per gram DC per (hour).

Dosimetry: Aqueous ferrous sulfate dosimeters are used.

Materials Testing Reactor (MTR)

Radiation Source: MTR spent fuel elements in a water-filled pool.

Flux Values: The maximum flux attainable is about 4.2×10^8 ergs per gram DC per hour.

Dosimetry: Aqueous ceric sulfate dosimeters and ion chambers are used.

General Reference: Martens, J.H. and Minuth, F.G., Selected List of Neutron and Gamma Irradiation Facilities, Information Division, Argonne National Laboratory, April 1957.



APPENDIX II

"CARBON DOSE" CONVERSION FACTORS

Irradiation Facility	Type of Dosimeter	Conversion Factor Ergs per Gram/Roentgen
BNL	Ferrous Sulfate	83.9
BFG ²	Ferrous Sulfate	83.9
MTR ³	Ceric Sulfate Ionization Chamber	83.9 87.1

Brookhaven National Laboratory Gamma Facility.
 B. F. Goodrich Company Gamma Facility.
 Materials Testing Reactor Gamma Facility.



APPENDIX III

TABLES OF EXPERIMENTAL DATA

All of the tables of data which were obtained from the research which was reported above are included in this appendix. This collection was made for the convenience of the reader and because of frequent reference to many of the tables.

Technical discussions of the tables are to be found in the text of the report, and figures relating to many of the tables appear in Appendix IV. The tables are listed in the table of contents.



TABLE I RADIATION-INDUCED ISOMERIZATION OF POLYBUTADIENE*

[Bromide] mole/l.	10 ⁹ k + gram DC/erg	10 ⁹ k (gram DC/erg)(mole/l.)	$-0.45 \frac{\text{Reaction}}{\text{G}_{\text{o}} \pm}$	on Yield GoS
	<u>A</u>	llyl Bromide Sensitizer		
2.888	9.62	5.97	117	367
0.694	5.90	6.95	90	1090
0.312	3.86	6.52	67	1790
0.143	2.78	6.64	50	2870
0.0584	1.95	7.03	37	4910
0.0144	0.89	6.01	17.3	9660
		Av. 6.52 ± 0.45		
0.656 0.131 0.0437	2. 28 1. 18 0. 70	2.75 2.94 2.87 Av. 2.86± 0.10	39.4 28.7 14.0	563 2020 2940
	Eth	ylene Bromide Sensitizer		
0.923	11.97	12.41	175	1140
0.462	8.22	11,86	145	1806
0.115	4.03	10.66	81	3950
0.0575	3.18	11.49	61	5920
21 02 13		Av. 11.61 ± 0.86		:

^{*} Concentration of polymer in benzene was 11.5 g./l. $+k_u$ was 0.045 x 10^{-9} gram DC/erg. k_s values were obtained by equation (2).

 $[\]pm G_0$ for the unsensitized reaction was 0.92. The values given in this column were corrected for the unsensitized reaction.



TABLE II

THE RECIPES AND CURING INFORMATION FOR THE GUM STOCKS

Pigment	81GGB1	81GGB7	81GGB3	81RGB7	81RGB8
Natural Rubber	100				
Neoprene GN		100			
Butyl Rubber			100		
SBR (90/10)				100	
SBR (80/20)					100
Stearic Acid	0.5		1	1	1
Zinc Oxide	5	5	5	5	5
Magnesium Oxide		4			
Benzothiazyl Disulfide	1			1	1
Phenyl-Beta-Naphthylamine	0.5				
Tetramethylthiuram Disulfide			1		
Sulfur	2		2	2	2
Specific Gravity	0.98	1.31	0.97	1.00	1.00
Curing Time (Min.)	45	40	45	40	40
Curing Temperature, ⁰ F.	284	284	302	302	302

Pigment	81RG9	81RG10	81RGB12	81RGB13	81RGB14	81RGB15
SBR (70/30)	100					
SBR (50/50)		100				
Hycar (90/10)			100			
Hycar (80/20)				100		
Hycar (70/30)					100	
Hycar (50/50)						100
Stearic Acid	1	1	1	1	I	1
Zinc Oxide	5	5	5	5	5	5
Benzothiazyl Disulfide	1	1	1	1	1	1
Sulfur	2	2	2	2	2	2
Specific Gravity	1.00	1.00	1.05	1.05	1.05 1	.05
Curing Time (Min.)	40	40	40	40	40	40
Curing Temperature, oF	302	302	302	302	302	302

2.20

2.46

2.56

2.64

2.70

3.10

dissolved

in Benzene

Polymer

Ŋ

sample

×

dissolved

sample

પુ મ **8**

in Cyclohexane

Polymer

Rubber

Butyl

9

TABLE III

2.50 83.9 2.74 2.60 1.96 1.60 1.86 2.10 EFFECT OF RADIATION UPON THE SWELLING OF POLYMERS AND CURED GUM STOCKS 1.76 1.70 1.70 2.68 1.58 2.72 1,30 2.10 58.7 Exposure x 10-8 Ergs per Gram DC 1.68 2.80 42.0 2.86 2.94 1.78 2.09 1.41 1.90 2.08 3.04 1.46 25.2 1.92 3.18 1.66 3.68 2.59 1.76 2.86 1.63 1.42 1.56 2.70 1.73 16.8 3.24 2.06 2.20 1.52 1.54 2.70 1.75 2.10 4.54 8.39 3.28 2.06 3.30 3.54 1.82 4.04 1.98 1.96 1.72 2.04 1.81 dissolved sample 1.66 3.60 3.66 2.06 Values 3.06 1.84 2.10 Initial 고 ~ ¬\$ → 8 તુન 8 戈ㅋ 8 in Cyclohexane Neoprene GN Neoprene GN Butyl Rubber Cured Gum Cured Gum Cured Gum in Benzene in Benzene in Benzene Polymer Natural Natural Rubber Rubber Compound 81GGB

80

3

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Contrails

TABLE III, (Continued)

ഥ	EFFECT OF RADIATION UPON THE	rion t		SWELLING OF POLYMERS AND CURED GUM STOCKS	F POLYN	ERS ANI	CUREL	GUM S	FOCKS
Compound			Initial	Exp	Exposure x 1	10-8 Ergs	per Gram DC	m DC	
81RGB			Values	8.39	16.8	25.2	42.0	58.7	83.9
2	SBR (90/10)	ำ	1.94	1.98		1.92	1.96	1.94	1.94
	Cured Gum	'n	2.98	3.02	2.92	2.78	2.76	2.64	2.56
	in Benzene	У		1.53		1.45	1.41	1.36	1.32
œ	SBR (80/20)	ų	1.97	1.94	190	1.84	1.94	1.92	1.92
	Cured Gum	้ำ	3.22	3.06	2.94	2.76	2.80	2.70	2.60
	in Benzene	X	1.63	1.58	1.55	1.50	1.44	1.41	1.35
6	SBR (70/30)	ų	1.98	1.92	1.91	1.90	1.87	1.84	1.92
	Cured Gum	[^] 니	3.42	3.14	3.04	2.94	2.84	2.68	2.66
	in Benzene	Y	1.73	1.64	1.59	1.55	1.52	145	1.38
10	SBR (50/50)	'n,	1.90	1.90	1.87	1.88	1.88		1.90
	Cured Gum	, H	3.56	3,34	3.22	3,08	290	2.90	2.70
	in Benzene	У		1.76	1.72	1.64	1.54		1.42
12	Hycar (90/10)	ų	1.82	2.04	180	1.86			1.86
	Cured Gum	'n	2.90	3,00	5.66	2.66	2.58	2.58	2.38
	in Cyclohexanone	8	1.59	1.47	1.48	1.43			1.28
13	Hycar (80/20)	J,	2.00	1.86	1.90	1.86	1.98		2.00
	Cured Gum	, '1	3.12	2.84	2.85	2.70	2,74	2.52	2.54
	in Cyclobexanone	Y	1.56	1.53	1.48	1.45	1.38		1.27

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TABLE III, (Continued)

ਜੁਸ਼	EFFECT OF RADIATION UPON THE SWELLING OF POLYMERS AND CURED GUM STOCKS	u NOI	PON THE SWI	STTING C	F POLYM	ERS AND	CURED	GUM ST	OCKS
Compound 81RGB			Initial Values	8.39	Exposure x	10 ⁻⁸ Ergs per 25.2 42.0		Gram DC 58.7	83.9
14	Hycar (70/30) Cured Gum in Cyclohexanone	J°7 K	1.92 3.02 1.57	1.94 2.96 1.53	1.88 2.86 1.52	1.98 2.84 1.43	1.96 2.76 1.41	1.94 2.62 1.35	1.82 2.36 1.29
15	Hycar (50/50) Cured Gum in Cyclohexanone	ŋ ⊓ Å	1.86 2.80 1.51	1.92 2.82 1.48	1.98 2.92 1.47	1.82 2.54 1.40	2.02 2.80 1.38	1.88 2.48 1.32	1.76 2.18 1.24
16	SBR (90/10) Polymer in Benzene	3 u X	1.90 3.54 1.86	1.94 3.26 1.68	1.98 3.02 1.53	1.98 2.96 1.49	1.84 2.56 1.39	1.94 2.52 1.30	1.78 2.16 1.21
17	SBR (80/20) Polymer in Benzene	7,7 Å	2, 12 4, 58 2, 16	1.98 3.45 1.74	2.00 3.20 1.60	2.04 3.16 1.55	2.04 2.90 1.42	2.04 2.80 1.37	1.76 2.22 1.26
18	SBR (70/30) Polymer in Benzene	킹ㅋጰ	1.90 4.10 2.16	1.91 3.48 1.82	1.80 2.98 1.66	1.90 2.98 1.57	1.94 2.84 1.46	1.88 2.64 1.40	1.88 2.48 1.32
19	SBR (50/50) Polymer in Benzene	7,7 <i>X</i>	sample dissolved	1.96 3.62 1.85	1.98 3.46 1.75	1.98 3.20 1.62	2.08 3.10 1.49	2.00 2.90 1.45	1.70 2.40 1.41

WADC TR 55-58 Pt IV

Contrails

TABLE III, (Continued)

EFFECT OF RADIATION UPON THE SWELLING OF POLYMERS AND CURED GUM STOCKS

WADC TR 55-58 Pt IV

Compound			Initial						
SIRGB			Values	8.39	16.8	25.2	42.0	58.7	83.9
21	Hycar (90/10)	ı,	1.98	1.84	1.86	1.82	1.80	1.76	1.76
	Polymer	J	3.56	2.98	2.76	2.55	2.32	2.18	2.03
	in Cyclohexanone	У	1.80	1.62	1.48	1.40	1.29	1.24	1.15
22	Hycar (80/20)	L ₀	2.30	1.78	1.80	1.82	1.78	1.64	1.76
	Polymer	ı	4.90	2.96	2.72	2.64	2.36	2.04	1.76
	in Cyclohexanone	K	2.13	1.66	1.51	1.45	1.33	1.24	1.00
23	Hycar (70/30)	่า	2.00	1.88	1.86	1.92		1.74	1.64
	Polymer	'n	4.24	3.18	2.82	2.70	2.47	2.12	1.64
	in Cyclohexanone	Ķ	2.12	1.69	1.52	1.41	1.30	1.22	1.00
24	Hycar (50/50)		1.80	1.72	1.78	1.88	1.62	2.05	1.62
	Polymer		3.70	2.82	2.66	2.68	2.06	2.24	1.62
	in Cyclohexanone		2.06	1.64	1.49	1.43	1.26	1.11	1.00

83

^{*} The sample liquified during irradiation.

The values for L_0 and L are given in units of inches.



TABLE IV

EFFECT OF RADIATION UPON THE SOL-GEL CONTENT OF THE PURE POLYMERS

Compound			odx =	Exposure x 10-8	-8 Ergs per	r Gram DC	ي	
		0	8.39	16.8		42.0	58.7	83.9
81GGB4	% Gel	0.0	39.46	59.15	69.77	73.90	78.49	81.68
	% Sol	100.0	60.54	40.85	30.23	26.10	21.51	18.32
81GGB5	% Gel	19,67	67.02	73.93	85.65	90.90	93.03	92.52
	% Sol	80.33	32.98	26.07	14.35	9.10	6.97	7.48
81GGB6	% Gel	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	% Sol	1 00.00	100.00	100.00	100.00	100.00	100.00	100.00
81RGB16	% Gel	76.76	91.03	92.25	94.44	98.04	99.38	99.24
	% Sol	23.24	8.97	7.75	5.56	1.96	0.62	0.76
81RGB17	% Ge1	54.29	91.21	95.33	96.23	98.49	99.29	98.97
	% So1	45.71	8.79	4.67	3.77	1.51	0.71	1.03
81RGB18	% Gel	53,72	81.33	93.82	96.36	99.12	99.72	99.22
	% Sol	46.28	18.67	6.18	3.64	0.88	0.28	0.78
81RGB19	% Gel	58.35	96.18	97.84	98.81	99.21	99.08	100.00
	% So1	41.65	3.82	2.16	1.19	0.79	0.92	0.0

WADC TR 55-58 Pt IV

TABLE IV, (Continued)

ত্র দুর্ঘ ত	CT OF RADI	EFFECT OF RADIATION UPON THE SOL-GEL CONTENT OF THE PURE POLYMERS	THE SOL-	GEL CON	TENT OF	THE PUR	E POLYM	ERS
			Exp	sure x 10	Exposure x 10-8 Ergs per Gram DC	er Gram I	Ŋ	
Compound		0	8,39	16.8	25.2	42.0	58.7	83.9
81RGB21	% Gel	41.54	89.17	97.30	98.22	98.81	98.56	97.30
	% Sol	58.46	10.83	2.70	1.78	1.19	1.44	2.70
81RGB22	% Gel	48.83	94.74	98.59	97.76	76.86	100.00	100.00
	% Sol	51.17	5.26	1.41	2.24	1.03	0.0	0.0
81RGB23	%Gel	61.64	100.00	100.00	100.00	97.75	93,35	100.00
	% So1	38.36	0.0	0.0	0.0	2.25	6,65	0.0
81RGB24	% Gel	98.49	100.00	100.00	100.00	100.00	100.00	100.00
	% Sol	1.51	0.0	0.0	0.0	0.0	0.0	0.0



TABLE V

EFFECT OF SHORT-STOP CONCENTRATION ON GELATION INDUCED BY AIR-AGING VERSUS IRRADIATION

			Percent Gel	·
Sample 81GH -	Short-Stop (phm)	Original Sample	Aged 72 Hrs. @60°C. in Air	Irradiated 16.8 Joules/Gm.
1844C	0.5 Hydroxylamine 0.2 Stalite	7.8	17.3	73
1844D	0.5 Hydroxylamine	10.3	20.5	79
1844E	0.25 Hydroquinone	2.9	3. 1	71
1844F	0.25 SDD (Good-rite 3955)	10.5	8.0	77

TABLE VI

INITIAL PROPERTIES OF A SERIES OF NBR COPOLYMERS

Sample 81GH-	Monomer BN	Charge VCN	% Conv.	% VCN (Anal.)	% Gel	<u>[n]</u>
1854E	90	10	72.4	11, 1	15.1	1.50
185 4D	80	20	73.5	21.7	16.3	1.48
1844E	67	33	87.9	32.4	4.3	1.76
1854C	55	45	71.4	38,9	17.5	1.42
1854F	30	70	37.7	45.3	14.6	0.61



TABLE VII

IDENTIFICATION OF SAMPLES AT VARIOUS CONVERSIONS

Sample 81GH-	Acrylonitrile Content (Percent)	Conversion (Percent)	$[\gamma]$
1857 A	35.6	39.5	1.38
1857 B	34.5	58.6	1.50
1857 C	33.2	86.8	1.84

TABLE VIII

IDENTIFICATION OF SAMPLES OF VARYING MODIFIER CONTENT

Sample 81GH-	Acrylonitrile (Percent)	Conversion (Percent)	Sulfole-B8 (phm)	<u>[n]</u>
1859 A	34.8	65.2	0.5	1.44
1859 B	33.4	68.1	1.0	0.72
1859 C	32.6	75.0	2.0	0.42
1859 D	32.7	79.3	4.0	0.22
1859 E	31.3	80.9	6.0	0.19
1859 F	30.6	81.5	8.0	0.12
1859 G	30.0	81.1	10.0	0.10

TABLE IX STRESS-S TRAIN DATA OF CROSSLINKED TIRE CORDS

다. 전 (%)	32.0 28.0 29.0 44.0	11. 4 (brokeduring flexing) 18. 1 7. 39 1. 55 9. 74 19. 5 5. 35 1. 12 11. 3 17. 5 7. 46 1. 57 16 7	21.5 18.4 24.8 27.2	12. 2 5.97 1.46 11.9 19. 1 5.67 1.38 9.57 22.8(broke during flexing) 13. 5 6. 54 1. 59 12. 2
1. 68 x 10 ⁹ B.L. Tenacity U.E. (1b.) (g./den.) (%)				flesting
8 x l enac	2.69 3.34 2.29 3.47	uring 1 1.55 1.12 1.57	2. 49 2. 07 2. 12 2. 90	1.46 1.38 hring 1.59
	28.3 12.8 244.6 15.9 32.6 16.5	rroked 7,39 5,35	A) 10 ~ C	5.97 5.67 groke o
로 스	3.3 1.6 1.6 1.6	1.4(b 3.17 3.55 7.57	20.010.3 30.08.5 32.68.7 21.011.9	2.25 9.15 2.8(b
8.39 x 10 ⁸ B.L. Tenacity U.E. (1b.) (g./den.) (%)	32 4 4 5		23.3	12 22 22 13
k 10 ⁸	99 12 30 19	22 61 91 . 59	2.79 3.44 4.46 2.51	2.40 3.21 4.16 1.91
Ter (g.	6 3. 2 3.	55.2. 68 1. 10 1. 58 1		
8 1. 41 1. 41	19. (19. (19. (19. (19. (19. (19. (19. (5 10. 7. 9 9.	11.3 14.1 18.3 10.3	9.85 9 13, 15 17, 03 3 7, 84
이 다.	21.7 19.0 3.9 40.3 19.6 4.1 46.3 15.7 3.3 41.0 15.2 3.1	14. 5 10.55 2. 22 23. 0 7.68 1. 61 23. 0 9. 10 1. 91 22. 0 7. 58 1. 59	20.8 31.0 34.0 26.7	15.5 21.9 24.5 18.3
Ergs/Gram DC 4.19 x 10 ⁸ B.L. Tenacity U.E. (1b.) (g./den) (%)		74 35 32 14	69 50 62 61	
/Greena Pena (g. //	4. 17 4. 06 4. 25 4. 11	2222	ણ 4 . 4. 6.	3, 48 3, 48 4, 83 2, 96
14. 4. [b.)	19.8 19.3 20.2 19.5	15. 0 13. 03 25. 8 11. 15 28. 7 11. 02 26. 1 10. 17	25. 3 15. 1 33. 9 18. 4 40. 5 18. 9 39. 9 14. 8	4.4.6.5 8863
6 H (S)	29.0 1 49.7 1 59.3 2 46.6 1			17.0 14.2 21.4 14.2 25.2 19.7 23.8 12.1
ogsur ty U.	29 49 59 46	15 25 28 28 26	25 33 40 39	17 21 25 23
8.39 x 10' B.L. Tenacity U.E. (1b.) (g./den) (%)	4.57 4.82 4.57 4.21	3, 65 3, 26 3, 33 2, 88	5.54 5.15 5.23 5.25	5.04 5.08 5.52 4.81
8.39 (g	7 T C O	¥ # 8 5	C 1 4 C	3888
	21.7 23.1 21.7 20.0	17. 15. 15. 13.	22. 21. 21. 21.	20. 20. 22. 19.
U.E.	16.4 44.7 48.6 40.5	16.7 31.4 34.3 30.9	18. 0 32. 4 31. 2 27. 9	18.8 27.4 29.0 25.2
city den)	2 ~ 0 9			
Tenacity U.E. (g. /den) (%)	4.82 4.07 4.00 3.66	3.73 3.32 3.21 2.96	5.23 5.03 4.61 4.23	4.84 4.79 4.75 4.42
B.L. 7		17.75 15.72 15.25 14.10	4 9 6 %	19.82 19.60 19.42 18.10
# E	22.9 19.3 19.0 17.4	17. 15. 15.	21.4 20.6 18.9 17.3	
nt int	200 300 DI	200 300 DI	Unflexed Nylon Nylon+N200 Nylon+N300 Nylon+HDI	Flexed Nylon Nylon+N200 Nylon+N300 Nylon+HDI
Cord and Treatment	Unilexed EPA EPA+N200 EPA+N300	Flexed EPA EPA+N200 EPA+N300 EPA+HDI	Unflexed Nylon Nylon+N20 Nylon+N300 Nylon+HDI	Flexed Nylon Nylon+N20 Nylon+N30 Nylon+HDI
Col	EPA EPA EPA EPA	Flex EPA EPA- EPA- EPA-	Unfley Nylon Nylon Nylon	N N N N N N N N N N N N N N N N N N N

TABLE IX, (Continued) STRESS-STRAIN DATA OF CROSSLINKED TIRE CORDS

U.E.	32.0 22.6 26.3 35.1	18.8 11.3 39
B.L. Tenacity U.E. B.L. Tenacity	24.713.3 3.27 3 41.510.2 2.51 2 40.3 8.7 2.14 2 27.614.3 3.52 3	14.2 10.05 2.47 18.8 26.7 6.75 1.66 11.3 27.4 (broke duing flexing) 16.8 10.19 2.50 19.7
8.39 x 10 ⁸ B.L. Tenacity (lb.) (g./den.	14.2 3.49 19.6 4.82 20.0 4.92 12.3 3.02	4. 09 16.5 10.61 2. 61 4. 74 26.5 18.16 4. 47 5. 12 27. 6 19.30 4. 74 2. 80 20.1 7.98 1. 96
am DC 10 ⁸ icity U.E. len.) (%)	4. 75 24.7 5. 48 45.3 4. 99 40.0 3. 86 32.3	09 16.5 74 26.5 12 27.6 80 20.1
Exposure, Ergs/Gram DC 4.19 x 10 ⁸ acity UE B.L. Tenacity Ullow, (g./den.) (%) (1b.) (g./den.) (%)	25.7 19.3 4.36.9 22.3 5.44.9 20.3 4.43.8 15.7 3.	5. 20 19. 7 16. 63 4. 5. 98 29. 4 19. 30 4. 5. 63 29. 2 20. 81 5. 5. 00 27. 1 11. 38 2.
y U.E.	25.7 36.9 44.9 43.8	19.7 29.4 29.2 27.1
Exp 9 x 10' Fenacit g./den	5.45 5.29 6.07 5.57	5.20 5.98 5.63 5.00
B.L. Tenacity U.E. B.L. Tenacity	6.00 20.022.2 5.67 40.221.5 5.38 36.9 24.7 5.42 37.2 22.7	5.78 19621.15 5 5.39 31.224.36 5 5.21 34.322.92 5 5.11 30.520.36 5
0 Tenacity g./den.)	6.00 5.67 5.38 5.42	5.78 5.39 5.21 5.11
B.L. (1b.) (24. 4 0 23. 1 0 21. 9 22. 0	23. 52 0 21. 95 0 21. 20 20. 74
Cord and Treatment	Unflexed Perlon Perlon+ N200 23. 1 Perlon+ N300 21. 9 Perlon+ HDI 22. 0	Flexed 23.52 Perlon N200 21.95 Perlon+N300 21.20 Perlon+HDI 20.74

B.L. = Breaking Load U.E. = Ultimate Elongation N200 = 3, 3'-Bitolylene 4, 4'-Diisocyanate N300 - Diphenylmethane 4, 4'-Diisocyanate HDI - 1, 6 - Hexamethylene Diisocyanate

TABLE X

RELATIVE STRESS-STRAIN DATA OF CROSSLINKED TIRE CORDS

	× 109	U.E.	Value	((195.0	62.6	9.69	108.8	Jacks of mine fleeing		31.0	33.0	54.0		117.4	56.8	79.5	97.5	7 63	# · · · · ·		-	48.0
	1.68×10^{9}	Tenacity Percent	Initial	1	55.8	82.0	57.2	94.8	-for expense		47.0	35.1	53.0	t	4(.0	41,2	46.0	9.89	,	20.6	29.0	broke during	36.2
	× 10 ⁸	U.E. at of	Value	!	172.5	9.66	83.5	80.5	703	2.00	57.6	56.9	56.6	,	111.1	95.6	104.3	75.3	4	04.7	69.7	78.6	53.5
	8.39×10^{8}	Tenacity l Percent of	Initial		82.7	101.2	82.5	87.1	u U	0.40	48.8	59.6	53.8	(53.3	68.4	8.96	59.4	•	47.0	67.1	87.7	43.4
į	10 ⁸	U.E. it of	Value	•	132.2	90.1	95.5	101.2	0 70	00.00	73.3	67.1	71.2	1	115.5	96.7	108.9	95.7	i d	84.5	80.0	84.5	72.6
	4.19 x 10 ⁸	Tenacity Percent of	Initial		9.98	7.66	106.2	102.3	υ ((2.2	70.9	72.3	72.1		70.5	89.5	100.3	85.4		(1.9	72.6	101.7	6.99
	Exposure, 8.39×10^7	t of	Value		176.8	111.1	121.9	115.1	ć	۷۰,۸۵	82.1	83.7	84.5		140.5	104.7	129.9	143.0	;	90.5	78.1	86.9	94.5
	8.39	Tenacity Percent	Initial Value		94.9	118.3	114.3	115.0	t	7.7	98.4	104.0	97.1		106.0	102.3	113.3	124.1	•	104.0	106.1	116.1	108.8
	0	Fenacity U.E. (Initial Values)	(%)		16.4	44.7	48.6	40.5		16.7	31.4	34.3	30.9		18.0	32.4	31.2	27.9				29.0	25.2
}		Tenacity (Initial V	(g. /den.)		4.82	4.07	4.00	3.66	,	3.73	3.32	3.21	2.96		5.23	5.03	4.61	4.23		4.84	4.79	4.75	4.45
		Cord and Treatment		Unflexed	EPA	EPA+ N200	EPA+ N300	EPA+ HDI	Flexed	EPA	EPA+ N200	EPA+ N300	EPA+ HDI	Unflexed	Nylon	Nvlon+N200	Nylon+N300	Nylon+HDI	Flexed	Nylon	Nv10n+N200	Nylon+N300	Nylon+HDI

TABLE X, (Continued)

RELATIVE STRESS-STRAIN DATA OF CROSSLINKED TIRE CORDS

1.68 x 109 enacity U.E. Percent of uitial Value		159.9	5622	71.3	94.4		95 9	36.2	harke charing flexing	64.6
1.68 x 109 Tenacity U. Percent of Initial Value		54.5	44.2	39.8	64.9		42, 8	30.8	broke dr	49.1
8.39 x 10 ⁸ nacity U.E. Percent of itial Value		123.4	103.2	109.2	74.2		72.4	85.1	79.9	55.1
8.39 × 10 ⁸ Tenacity U Percent of Initial Valu		58.2	85.1	91.6	55.7		45.2	82.7	91.0	38.5
Ergs/Gram DC 4.19 x 10 Tenacity U.E. Percent of Initial Value		123.4	112.6	108.3	86.9		84.1	85.0	80,5	62.9
Ergs/Gram 4.19 x 10 Tenacity Percent Initial Val		79.1	9.96	95.9	71.2		70.9	88.0	98.2	54.9
Exposure, 8.39 x 10 ⁷ enacity U.E. Percent of Initial Value		128.3	91.6	121.7	117.8		100.4	94.2	85.1	88.9
Renac Pero		8.06	93.3	113.0	102.9		90.0	111.0	108.0	98.2
0 Tenacity U.E. (Initial Values) g./den.) (%)		20.0	40.2	36.9	37.2		19.6	31.2	34.3	30.5
Tenacity (Initial Va (g./den.)		9.00	5.67	5.38	5.42		5.78	5.39	5.21	5.11
Cord and Treatment	Unflexed	Perlon	Perlon+N200	Perlon+N300	Perlon+HDI	Flexed	Perlon	Perlon+N200	Perlon+N300	Perlon+HDI

U.E. * Ultimate elongation N200 = 3, 3'-Bitolylene 4, 4'-Diisocyanate N300 = Diphenylmethane 4, 4'-Diisocyanate HDI * 1, 6 - Hexamethylene Diisocyanate

TABLE XI

STRESS-STRAIN DATA OF ANTI-RAD TREATED TIRE CORDS

				읪	4	rgs/G			,	60.	
0			4.	4.19×10^{8}		œ			1.6	$.68 \times 10^{\circ}$	- 1
B. L. Tenacity	1.2	ity U.E.	B. L.	Tenacity	, U.E.	B. L.	Tenacity 1	U. E.	ij.	Tenaicity	ы П
(lb.) (g./den		n.) (%)	(1b.)	(g./den)	(%)	(lb.)	(g./den.)((%)	(lb.) (g.	g. /den.)	8
17.14 4.3		34 10.5	16.45	4.16	6.6	15.43	91		13.64	3.46	
4,		_	5	4.45	15.2	18.57	4.71 15	5.4	14.30	3.63	14.6
4	_	1		4.78	14.4	18.00	57 1	5.4		3.99	Ď.
		7	4	4.21	15.0	18,42	4.67 14	‡ . 4	16.90	4.28	16.0
4	4	ı	18.27	4.64	19.6	17,70	4.49 14	۰	15.57	3.94	15.2
Quinhydrone 19.80 5.02	N	14.3	17.05	4.37	16.6	17.43	4.42 13	3.8		4.27	9
	\mathcal{S}	16.3	19.52	4.95	19.4	19.20	4.87 20	3, 5	16.46	4.18	16.0
	0	18.7	15.15	3.70	8.02	11.34	2,77 20		10.20	2.49	Ξ:
21.67 5.29	6	20.8	•	4.45	27.6	15.66	3.82 26	•	10.12		
5.	∞	20.8	20.85	5.08	27.4	15.96	90	•	13.17	3.19	တ်
Phenothiazine21.25 5.19	6	20.0	18.77	4.58	25.4	15.00	3,66 20	0,4	12.05	2.94	18.0
ī,	6	18.9	20.85	5.08	27.8	17.76	33	∞.			તું
Quinhydrone 20.36 4.97	_	18.7	20.47	5.00	30.0	17.34	24		۰	3,55	;
20.55 5.	-	21.2	19.87	4.85	56.6	18.41	4.49 27.	7.2	15.21	3.71	
25.17 6.18	∞		19.32	•	23.6	14, 21	49	۵	13.30	3.27	32.0
22.80 5.61	$\overline{}$	25.7	24.11	ŗ,	41.0	3	51	5.2	14.60		28.4
21.15 5.20	0	23.0	2,	5.	30.7	7	77	•	15.40		
Phenothiazine25.57 6.28	œ	22.0	21,33	'n.	28.5	20.92	5.15 30	0.5	15.20		
Pyro Gallol 20.19 4.92	Ņ	26.1	22,95	5.64	32.0	23.22	5.71 40	•	•	4.43	29.8
6.1	ഹ	2	21.67		30.2	23.13	8 3	3.6	•	9	
	φ,	3 26.2	23.05		32.5	21.12	20 3	5.6	17.90	4	

TABLE XI, (Continued)

STRESS-STRAIN DATA OF ANTI-RAD TREATED TIRE CORDS

		v U. E.	(%)	12.7	30.5	33.6	33.0	32.2	31.6	33.0	7.0	8.0	9.0	8.3	8.6	9.5	11.0
	3×10^9	Tenacit	g./den.	2.18	4.01	3.73	4.47	4.10	4.09	4.19	0.84	1.16	1,38	1.26	1.41	1.52	1.41
	1.6	B.L.	(lb.) (g./den.) (%)	9.17	16.87	15.70	18.81	17.25	17.20	17.62	6.50	9.00	10.76	9,83	10.98	11.84	12.48
		y U.E.	.) (%)	14.9	43.2	36.2	34.2	40.0	34.7	39,8						11.6	
n DC	9 x 108	Tenacit	g./den	3, 10	4.98	4.84	5.03	5.24	5.07	4.89	1.29	1.59	1.63	1.45	1.80	4.78	1.80
Ergs/Gram DC	8.39×10^{8}	B.L.	(lb.)	13.13	20.94	20.34	21.15	22.08	21.33	20.58	10.	12.	12.	11.	13	13.83	12
e, Er		, U.E.	(%)	26.1	37.0	37.2	32.2	36.2	41.8	37.2	10.9	13.4	15.6	14.6	11.6	14.8	16.4
Exposure, Erg	9×10^8	Tenacity	(lb.) (g./den.) (%)	4.40	5.42	5.46	5.24	5.51	5.76	5.32	1.68	2.09	2.14	1.95	1.84	2.18	1.84
	4.19	B. L.	(lb.) (18.52	22.80	22.98	22.05	23.19	24. 26	22.38	12.71	16.23	16.62	15.18	14.31	16.98	16.38
		U.E	(%)	25.8	30.9	29.5	27.6	29.9	30.0	28.9	12.5	11.0	15.5	11.9	13.0	14.4	12.5
	0	Tenacity	g./den.) (%)	96.9	5.87	6.11	5.76	5.81	90.9	5.78	2.18	1.88	2.37	1.99	5.09	2.27	5.09
		B.L. I	(1b.) (g	25.09	24.70	25.70	te 24. 20	24.45	25.50	24.30	16.95	14,60	18.45	te 15.50	16.25	17.70	15.81
			Treatment	Perlon-AR None	Amine	2-Naphthol	Phenothiazine 24.20	Pyrogallol 24.45	Quinhydrone 25.50	Quinone	None	Amine	2-Naphthol	Phenothiazine 15.50	Pyrogallol 16.25	Quinhydrone 17.70	Quinone
			Cord	Perlon-							Rayon						

Amine = N-Phenyl - 2 - Naphthyl Amine.

TABLE XII

RELATIVE RETENTION OF STRESS-STRAIN PROPERTIES OF ANTI-RAD TREATED CORDS

Cord	Treatment	B. L. (1b.)	U. E. (%)	Expos 4.19 B. L. (% of	Exposure, Ergs/Gram DC 4.19 x 10 8 8.39 x 10 B.L. U.E. B.L. U. (% of Initial) (% of Initial)	gs/Gran 8.39 B.L. (% of	/Gram DC 8.39 x 10 8.1. U.E. (% of Initial)	1.68 x 10 ⁹ B. L. U. E (% of Initial)	10 ⁹ U.E.
Dacron	None Amine	17.14	10.5 14.9		1111.1		107.0	49.7 51.6	
	2-Naphthol Phenothiazine Pyrogallol Quinhydrone Quinone	18. 49 18. 55 19. 09 19. 80 20. 25	13.0 13.4 14.0 14.3	94.5 88.2 100.0 100.8	131.9 127.0 147.1 160.5 125.7	72.4 70.5 85.0 85.2 89.6	113.4 102.0 131.2 139.0 128.2	59.6 56.6 73.0 71.4 74.1	89.5 90.0 118.6 113.3
	None Amine 2-Naphthol Phenothiazine Pyrogallol Quinhydrone	20.51 21.67 22.05 21.25 20.85 20.36 20.36	18.7 20.8 20.8 20.0 18.9 18.7 21.2	75.0 111.2 90.1 97.9 88.1 95.9	87.2 121.8 100.7 122.8 89.2 102.8	59.3 84.9 58.6 72.8 86.0 78.2	60.0 94.6 71.0 85.7 110.8 80.5	38.4 61.7 58.3 63.4 67.6 67.0	56.0 72.8 58.1 69.8 75.5 63.9
Perlon	None Amine 2-Naphthol Phenothiazine Pyrogallol Quinhydrone	25.17 22.80 21.15 25.57 20.19 25.00 24.35	23.6 25.7 23.0 22.0 26.0 26.0	76.9 105.8 104.7 83.4 1113.8 86.8	104.7 159.7 133.5 128.1 122.7 116.1	56.5 80.5 100.2 81.8 115.1 92.5	104.7 140.9 178.1 137.2 153.2 129.2	52.9 64.0 72.9 59.5 89.4 75.4	135.6 110.3 118.2 111.9 114.2 131.5

TABLE XII, (Continued)

RELATIVE RETENTION OF STRESS-STRAIN PROPERTIES OF ANTI-RAD TREATED CORDS

			•	Ехров	Exposure, Ergs/Gram DC	s/Gran	B DC		
Cord	Treatment	B. L.	O.E.	$\frac{4.19 \text{ x}}{\text{B. L.}}$	$\frac{4.19 \times 10^8}{0.E}$	8.39 B. L.	8.39×10^{8} B. L. U. E.	$\frac{1.68 \times 10^9}{1.8}$	x 109
		(Ib.)	(%)	Jo %)	(% of Initial)	(% of	[2]	(% of Initial)	nitial)
Perlon-AR	None	25.09	25.8	73.9	101.2	52.4	57.8	36.6	49 2
	Amine	24.70	30.9	92.4	119.8	84.6	139.9	68.3	98.7
	2-Naphthol	25.70	29.5	89.4	126.1	79.1	122.9	61.1	113.9
	Phenothiazine	24.20	27.6	91.1	116.8	87.4	123.9	77.8	119.6
	Pyrogallol	24.45	29.9	94.8	121.1	90.4	133.9	70.5	107.8
	Quinhydrone	25.50	30.0	95.2	139.2	83.6	115.8	67.5	105.2
	Quinone	24.30	58.9	92.0	128.8	84.6	137.8	72.5	114.1
Rayon	None	16.95	12.5	95.9	94.4	90.1	90.5	79.6	93. 1
	Amine	14.60	11.0	94.4	102.0	99.7	103.3	76.9	98.0
	2-Naphthol	18.45		102.0	110.8	97.4	118.5	85.1	115.2
	Fuenothiazine Dunganiai	15.50	11.9	88.6	112.0	99.4	107.5	91.1	119.6
	ryroganol Oninhuduon	16.25	13.0	95.7	140.0	92.7	103.7	81.5	108.6
	Quinono	17.70	14.4	86.1	116.1	88.1	114.8	85.1	96.5
	X minolie	15.81	12.5	96.4	119.0	94.8	125.9	81.2	97.1



TABLE XIII

STRESS-STRAIN DATA OF UNIRRADIATED TIRE CORDS

			Unflexed			Flexed	
Cord	Treatment	B.L.	Tenacity	U., E.	B.L.	Tenacity	U.E.
Cora	* * Catinon	(lb.)	(g/den)	(%)	(lb.)	(g/den)	(%)
					_		
Dacron	Formaldehyde	21.37	5.41	17.4	18.30	4.64	12.0
2401011	Glutaraldehyde	18.42	4.67	16.8	17.73	4.50	15.1
	Hylene DP	19.07	4.84	23.8	15.18	3.85	20.7
	Mondur TM	17.80	4.51	30.8	11.75	2.98	37.7
						2 07	20.2
EPA	Formaldehyde	21.48	4.52	32.3	18.43	3.87	30.2
	Glutaraldehyde	22,76	4.78	29.8	17.10	3.59	26.5
	Hylene DP	22.57	4.75	23.5	16.16	3.40	20.0
	Mondur TM	16.97	3.56	18.6	13.86	2.91	15.9
** 1	Dld.hada	22,27	5,44	26.1	22,50	5.49	24.4
Nylon	Formaldehyde		5.13	19.0	20.07	4.90	19.8
	Glutaraldehyde	20.97	5.50	24.9	21.82	5,33	21.7
	Hylene DP	22,50		19.5	13.81	3.37	17.7
	Mondur TM	21.96	5.37	19.5	15.01	3, 31	11,7
Perlon	Formaldehyde	25.77	6.33	28.1	18.83	4.63	26.6
2 011011	Glutaraldehyde	25.10	6.17	22.9	24.22	5.96	26.3
	Hylene DP	24.52	6.03	23.4	21.90	5.39	20.8
	Mondur TM	24.30	5.98	11.4	16.20	3.98	18.9
		25 22	/ 00	24 5	19.31	4.59	27.9
Perlon-AR	Formaldehyde	25.23	6.00	34.5		5.90	29.7
	Glutaraldehyde	24.87		27.4	24,82		22.2
	Hylene DP	23,20		24.7	21.75	5.17	
	Mondur TM	24.36	5.78	25.3	14.28	3.39	20.8
Rayon	Formaldehyde	18.60	2.39	27.2	15.51	2.00	23.4
Rayon	Glutaraldehyde	18.97		19.1	17.28	2.22	19.7
	Hylene DP	17.75		11,3	10.09	1.30	9.8
	Mondur TM	17.12		14.1	11.00	1.41	9.5
	MIGHT I M	11,12	2,20				



TABLE XIV

IDENTIFICATION OF THE FILAMENT ADDITIVES

Code	Polyamide Additive	Remarks
М	pulverized activated carbon	free radical captor
N	quinhydrone	one of the best anti-rads for rubber
Р	N, N'-di-beta-naphthyl-p-phenylene diamine	antioxidant and anti-rad for rubber
Q	N, N'-diphenyl-p-phenylene diamine	antioxidant and anti-rad for rubber
R	lead nitrate	heavy metal salt
S	silver nitrate	metal salt
Т	cadmium nitrate	good neutron captor
U	barium nitrate	metal salt
v	1,5-dihydroxynaphthalene	antioxidant, anti-rad
w	p-methoxyphenol	antioxidant
x	pyrogallol	antioxidant, anti-rad
Y	Congo red	highly aromatic



A. POLYAMIDE A (EXPERIMENTAL NYLON) FILAMENT CHARACTERISTICS

Filament Composition	Tenacity	Elongation %	Polymer Characteristics	Draw Ratio
1.0 M	तक स्थाप स्थाप		Not extrudable	
1.0 N	6.79	21.8		6.80
0.5 P	6.56	20.0		5.03
1.0 P	5.01	17.2		6.84
5,0 P			Deteriorated	*===
1.0 Q	7.10	17.6		6.06
1.0 R		A = + -	Too fluxed	
1.0 R - 2.0 X	1.19	4.6	Not irradiated	5.28
1,0 S	5.27	17.0		5,82
1.0 S - 2.0 X	4.65	28.6		4.58
1.0 T	1.17	10.8		4.95
1.0 U	6.79	18.4		5,35
0.5 V			Not run	
1.0 V	1.55	18.6		4.97
5.0 V			Not run	
0.5 W	6.91	19.6		~ -
1.0 W	7.10	19.0		4.89
5.0 W	7.21	20.8		4.98
1.0 X	6.64	18.4		4.97
1.0 Y	4.56	22.6	Poor dispersion	4.69
Pure	4.81	17.6		6.30

Note: The numbers in the first column are the amounts of additive per hundred parts of polymer by weight. The letters identify the additives.



B. POLYAMIDE B (NYLON 6) FILAMENT CHARACTERISTICS

Filament Composition	Tenacity g/denier	Elongation %	Polymer Characteristics	Draw Ratio
1.0 M	6.08	19.2		3.91
1.0 N	5.97	22.8		3.26
0.5 P	6.94	19.4		3.67
1.0 P	6.88	18.6		4.05
5,0 P	6.76	22.8		2.25
0.5 Q	5.75	22.2		3.25
1.0 Q	5.51	19.0		3.68
1.0 R	6.01	21.8		4.16
1.0 S	6.19	24.4		4.29
20.0 S	4.88	18.0		
1.0 T	5.6 7	21.0		4.12
1.0 U	7.40	19.2		4.17
0.5 V	7,20	19.6		4.71
1.0 V	6.98	19.4		4.22
5.0 V	5.69	16.4		4.16
0.5 W				
1.0 W	6., 35	19.4		3.84
5.0 W	5. 4 9	19.0		4.07
1.0 X	6.32	18.4		3.66
1.0 Y	6.55	17.8	Good dispersion	4.59
Pure	6,10	20.0		3,25

Note: The numbers in the first column are the amounts of additive per hundred parts of polymer by weight. The letters identify the additives.



TABLE XVI

RUBBER COMPOUNDS USED IN SCREENING POTENTIAL ANTI-RADS

All of the rubber compounds listed below consist of the master recipe plus the indicated additive in parts per 100 of natural rubber.

81GHC	PHR	Additive (Potential Anti-Rad)
6	-	None (blank stock)
2	0.5	N-Phenyl-beta-naphthylamine (PBNA)
3	1.0	PBNA
4	2.0	PBNA
5	4.0	PBNA
41		None
42	0.5	PBNA
43	1.0	PBNA
44	2.0	PBNA
45	4.0	PBNA
5 8		None
59	3.0	PBNA
60	3.0	C2.00-VIII-132
61	3.0	Diphenylamine-acetone product (resin)
62	3,0	4, 4'-Isopropylidenedianiline
63	3.0	4, 4'-Isopropylidene-bis-diphenylamine
64	3,0	N, N'-Diphenylbenzoquinone diimide
65	$\int 1.5$	N, N'-Diphenylbenzoquinone diimide
	(1.5	N, N'-Diphenyl p-phenylene diamine (DPPD)
66	3.0	DPPD
67	3.0	Flectol L.S.
68	3.0	Flectol White
69	3.0	9, 9-Dimethylacridane
70	3.0	2, 4-Dihydroxyacetophenone
71	3.0	Benzil
72	3.0	2'-Hydroxy-4-methoxybenzophenone
73	3.0	2, 2'-Dihydroxy-4, 4'-dimethoxybenzophenone
74	3.0	Mixture of Isomers of Dihydroxydimethoxybenzophenone
75	3.0	2, 4-Dihydroxybenzophenone
76	3.0	Complex of Quinone plus 4 moles of 2-Naphthol
77	3.0	Complex of Quinone plus 2 moles of p-Cresol



RUBBER COMPOUNDS USED IN SCREENING POTENTIAL ANTI-RADS

81 GHC	PHR	Additive (Potential Anti-Rad)
78	3.0	Complex of Quinone plus 2 moles of p-benzyloxyphenol
79	3.0	Complex of 1, 4-Naphthoquinone plus Hydroquinone
80	3.0	p-Triphenylmethyl diphenylamine
81	3.0	N-p-Hydroxyphenyl morpholine
82	0	Blank
83	0.5	N-Cyclohexyl-N-phenyl-p-phenylene damine ("Antioxidant 4010")*
84	1.0	N-Cyclohexyl-N-phenyl-p-phenylene diamine
85	2.0	N-Cyclohexyl-N'-phenyl-p-phenylene diamine
86	4.0	N-Cyclohexyl-N'-phenyl-p-phenylene diamine
87	0.5	32CH60G
88	1.0	32 C H60 G
89	2.0	32CH60G
90	4.0	32CH60G
91	3.0	Cyclohexyl aniline
92	3.0	2-(p-Hydroxylanilino)-1, 4-naphthoquinone
93	3.0	2-(p-Acetoxyaniline)-1, 4-naphthohydroquinone diacetate
94	3.0	l-Phenyl-3-pyrazolidone
95	3.0	"Tinopal AN Supra"
96	3,0	"Tinopal PCR + PCRP"
97	3.0	"Santowhite"
98	3.0	"Santowhite A"
99	3.0	"Vultac No. 3"
100	3.0	"Peptone-22"
101	3.0	Benzyl disulfide
102	3.0	2-tert Butyl-4-methoxy phenol
103	3.0	l, l-Methylene di-(2-naphthol)
104	3.0	Metal-free phthalocyanine blue
105	3.0	"Toluidine Toner RT-1"
106	3.0	"Benzidine Yellow YB-14"
107	3.0	"Benzidine Yellow YB-5702"
108	3.0	"Fast Benzidine Yellow YB-5708"
109	3.0	"Pyrazolone Red R-6258"
110	3.0	"S.P. Pyranthrone Orange Toner 5XS1414"



RUBBER COMPOUNDS USED IN SCREENING POTENTIAL ANTI-RADS

81GHC	PHR	Additive (Potential Anti-Rad)
111	3.0	"Indo Brilliant Orange OV5964" *
112	3.0	"SP Dibenzanthrone Green Toner 5XS1408"
113	3.0	"SP Indo Orange GR Toner 5XS1411"
114	3.0	"SP Perylene Scarlet Toner 5XS1413"
115	3.0	"Indo Yellow Powder 5XS1427"
116	3.0	"SP Brown Toner 5XS1412"
117	3.0	"SP Indo Blue Toner B-79"
118	3.0	"SP Phoebe Red Toner MV6639 5XS1409"
119	3.0	"SP Acridone Red Toner 5XS1410"
120	3.0	"Permanent Violet Toner B-4016"
121	3.0	"Thio Fast Pink MV6632"
122	3.0	"Bright Tone Indo Maroon MV6623"
123	3.0	"Thio Fast Red MV6613"
123	3.0	"Thiosa Fast Red MV6604"
125	3.0	"Oil Soluble Red"
126	3.0	2-(& -Naphthylthio)-quinone
* - 0	•••	· • • · · · · · · · · · · · · · · · · ·

^{*} Names in quotation marks are trade-marked names. In some cases, these materials are commercially available under these names; in other cases they are available only in limited experimental quantities.

Contrails

TABLE XVII

PHYSICAL PROPERTIES OF RUBBER STOCKS: ORIGINAL vs. AGED (1) vs. IRRADIATED (2)

•									-			20		ogn	-	-											m)
ويعاقب	Irr	7			30					30				59			24	21	23	5 6	59	30	20	34	19	18	108 reps
Flexometer rise,	⋖	Notest	65	94	84	86	56	83	84	135	115	99	95	77	96	29	86	89	98	88	29	53	33	HZ	Z	Ľ	1.0 x 1
F.	o	49	44	40	39	39	40	39	61	40	36	56	43	74	41	31	33	24	97	24	30	42	40	36	34	30	or
Dyn. Mod. psi	Irr	62	65	58	1770	59	2130	2230	2410	2080	1850	2110	1850	1670	1940	2940	1860	2190	2260	2390	2420	2420	1810	2590	1700	1910	rgs/g C. or
		820	880	905	900	840	1000	0	1060	1050	096	900	930	860	885	1220	890	1150	1140	O	066	1070	930	1015	815	865	109 er
Hardness	Irr			22	74	73				75		75		7.1			92	78	80	80	42	80	74	80	74	15	8.7 ×
Haı	၁	54	28	9	55	9				63				57			9	69	29	65	61	62	58	64	29	56	se:
	Irr	300	300	310	380	380	200	230	230	300	330	210	270	300	300	100	240	150	160	140	170	180	230	150	170	180	(2) Dose:
Elongation,	4	~	3	r)	580	_	190	510	530	530	260	300	520	500	450	210	475	460	470	510	520	450	400	200	200	790	ត
Elon	0	260	580	570	610	615	540	550	580	290	605	520	260	580	009	490	575	510	520	510	570	560	580	530	540	260	ressui
is.	Irrad	3000	3020	3100	3440	3420	2210	2540	2560	3180	3400	2340	3020	2980	2900	1460	2580	1960	2000	1800	2120	2260	2720	1820	1920	2060	300 psi oxygen pressure
ensile, psi	Aged	200	2720	2860	3260	3400	420	2580	3010	3150	3010	950	2980	2420	3020	2080	2640	3540	3520	3400	3620	2240	1620	400	360	160	00 psi c
Ter	Orig	3640	3900	4100	4260	4160	3740	3920	4120	4220	4150	3620	4200	3900	4020	4080	4000	4220	4260	4060	4280	3980	4020	3820	3640	3760	and
100%	Irrad	720	200	720	640	260	840	860	096	820	160	920	840	800	800	! ! !	800	1120	1120	1180	1140	1140	096	1200	980	940	B at 70°C
Modulus,	Aged	100	100	100	100	100	290	260	280	280	290	270	300	220	280	840	280	420	440	440	380	280	240	260	220	280	48 hours BB
Mo	Orig	200	200	200	200	180	300	280	280	300	280	280	300	300	760	460	280	400	380	380	360	360	300	360	260	260	
81 GHC		9	2	3	4	5	41	42	43	44	45	28	29	09	61	79	63	64	65	99	29	89	69	70	71	72	(1) Aged:

Contrails

TABLE XVII, (Continued)

PHYSICAL PROPERTIES OF RUBBER STOCKS: ORIGINAL vs. AGED (1) vs. IRRADIATED (2)

| | ι | | | | | | | | Ī | | | _

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|-----------------|--|--|---|--|---|---|--|--|---|--|--
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r temi	Irr	

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 | | |
 | (3) | |
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| comete
rise. | A | NT | Ľ | L | 100 | 28 | 101 | 107 | 85 | 68 | 52 | 06

 | 104
 | 101 | 98 | 126

 | 122 | 113 | 109
 | (3) | |
 | | |
| Flex | 0 | 43 | 48 | 37 | 90 | 28 | 30 | 49 | 43 | 36 | 47 | 36

 | 34
 | 33 | 36 | 37

 | 39 | 34 | 34
 | (3) | |
 | | |
| Mod pei | Irr | 2190 | 2040 | 2020 | 1950 | 1820 | 2050 | 1890 | 1990 | 2410 | 2130 | 2080

 | 2085
 | 2070 | 2085 | 2100

 | 2010 | 1905 | 1815
 | (3) | |
 | | |
| Dyn. | | 915 | 830 | 885 | 915 | 870 | 955 | 1005 | 910 | 1110 | 840 | 930

 | 970
 | 940 | 980 | 935

 | 975 | 1075 | 875
 | (3) | |
 | | |
| dness | Irr | 22 | 92 | 78 | 77 | 74 | 92 | 7.7 | 75 | 48 | 72 | 92

 | 74
 | 74 | 11 | 77

 | | |
 | 73 | 11 | 75
 | 4 | 83 |
| Har | 0 | 09 | 09 | 63 | 64 | 09 | 63 | 64 | 28 | 63 | 59 | 9

 | 62
 | 9 | 63 | 56

 | 09 | 99 | 09
 | 62 | 63 | 63
 | 29 | 29 |
| % | Irr | 150 | 200 | 170 | 240 | 200 | 200 | 230 | 200 | 140 | 250 | 220

 | 260
 | 250 | 190 | 240

 | 260 | 300 | 310
 | 280 | 240 | 240
 | 220 | 120 |
| gation, | 4 | 340 | 290 | 210 | 200 | 410 | 410 | 540 | 450 | 410 | 330 | 460

 | 540
 | 530 | 550 | 540

 | 540 | 999 | 540
 | 510 | 470 | 430
 | 330 | 380 |
| Elon | 0 | 530 | 540 | 540 | 290 | 530 | 540 | 590 | 520 | 520 | 570 | 570

 | 590
 | 999 | 580 | 580

 | 570 | 009 | 909
 | 570 | 260 | 550
 | 290 | 490 |
| Bî | Irrad | 1780 | 2180 | 1980 | 2560 | 2440 | 2440 | 2440 | 2380 | 1940 | 2840 | 2760

 | 3760
 | 3040 | 2280 | 2760

 | 3060 | 3240 | 3260
 | 3200 | 2800 | 2760
 | 2440 | 1540 |
| ဌ | | 1260 | 620 | 420 | 2500 | 2300 | 2260 | 3100 | 2340 | 2620 | 980 | 2520

 | 3800
 | 3720 | 3680 | 2920

 | 3140 | 3320 | 3160
 | 2980 | 2600 | 2280
 | 1260 | 2180 |
| Ter | Orig | 3480 | 3300 | 3740 | 4040 | 4110 | 4220 | 4140 | 3700 | 4100 | 3600 | 4060

 | 4240
 | 4200 | 4140 | 3960

 | 4120 | 4040 | 4040
 | 4020 | 3880 | 3760
 | 3920 | 3800 |
| 100% | Irrad | 1020 | 940 | 1060 | 920 | 1000 | 1040 | 920 | 1040 | 1340 | 780 | 860

 | 820
 | 840 | 920 | 820

 | 860 | 720 | 100
 | 840 | 940 | 096
 | 096 | 1240 |
| lulus, | Aged | 300 | 220 | 240 | 300 | 300 | 300 | 360 | 280 | 420 | 2.80 | 300

 | 440
 | 420 | 440 | 300

 | 320 | 300 | 280
 | 320 | 340 | 380
 | 280 | 200 |
| Moč | Orig | 260 | 240 | 300 | 300 | 320 | 300 | 340 | 300 | 360 | 2,60 | 300

 | 300
 | 340 | 300 | 280

 | 260 | 260 | 240
 | 320 | 340 | 340
 | 260 | 480 |
| 81 GHC | | 73 | 74 | 75 | 92 | 7.7 | 78 | 462 | 80 | 81 | 28 | 83

 | 84
 | 85 | 98 | 87

 | 88 | 89 | 06
 | 91 | 95 | 93
 | 94 | 95 |
| | Modulus, 100% Tensile, psi Elongation, % Hardness Dyn. Mod psi | Modulus, 100% Tensile, psi Elongation, % Hardness
Orig Aged Irrad Orig Aged Irrad O A Irr O Irr | Modulus, 100% Tensile, psi Elongation, % Hardness Dyn. Mod psi Orig Aged Irrad Orig Aged Irrad O A Irr O Irr O Irr 260 300 1020 3480 1260 1780 530 340 150 60 77 915 2190 | Modulus, 100% Tensile, psi Elongation, % Hardness Dyn. Mod psi Orig Aged Irrad Orig Aged Irrad O A Irr O Irr O Irr 260 300 1020 3480 1260 1780 530 340 150 60 77 915 2190 240 220 940 3300 620 2180 540 290 200 60 76 830 2040 | Modulus, 100% Tensile, psi Elongation, % Hardness Dyn. Mod psi 260 300 1020 3480 1260 1780 530 340 150 60 77 915 2190 240 220 940 3300 620 2180 540 290 200 60 76 830 2040 300 240 3740 420 1980 540 210 170 63 78 885 2020 | Modulus, 100% Tensile, psi Elongation, % Hardness Dyn. Mod psi 260 300 1020 3480 1260 1780 530 340 150 60 77 915 2190 240 220 940 3300 620 2180 540 290 200 60 76 830 2040 300 240 3740 420 1980 540 210 170 63 78 885 2020 300 300 920 4040 2500 2560 590 540 64 77 915 1950 | Modulus, 100% Tensile, psi Elongation, % Mardness Dyn. 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TABLE XVII, (Continued)

PHYSICAL PROPERTIES OF RUBBER STOCKS: ORIGINAL vs. AGED (1) vs. IRRADIATED (2)

υp.	1																							
er ter	TI	(2)	(c)																					
Flexometer temp.	A A	(3)	(c)																					
Flex	0	(3)												٠										
Dvn. Mod. nai	Irr	(3)	2																					
Dyn	0	(3)																						
Hardness	Irr	74	7.6	7.5	73	2.2	76	72	08	7.8	80	76	7.7	. 44	7 0 2	7.8	7.7	. ~	77	77	7.6	ο α	77	- 0
Ha						09	59	56	70	67	29	20	2 %	4	, 7,	65	99	69	, 'c	99	7 2	3 79	9 00	0 4
%		280	270	250	310	270	250	290	150	200	170	230	220	230	210	220	220	140	220	210	200	180	180	220
Elongation,	A S	510	500	400	590	520	380	330	400	250	450	280	270	290	180	320	200	170	250	280	300	290	220	310
Elon	0	590	580	570	610	009	580	580	520	570	570	580	570	580	560	570	550	540	560	999	500	570	510	7 7 0
si	Irrad	2840	3000	2780	3120	2720	2560	3160	1920	2280	2060	2560	2520	2560	2460	2360	2500	1700	2620	2560	2260	2040	2040	2600
Tensile, psi	Aged	2660	2780	1920	3640	2520	1840	1240	1900	096	2440	620	620	009	099	1200	460	520	720	720	1080	096	800	1320
Ter	Orig	3800	3940	3900	4420	3800	3760	3840	3980	3900	4220	3820	3700	3820	3960	3800	3680	3880	3700	3760	3140	3540	3340	3540
100%	Irrad	820	880	860	840	800	780	800	1160	1000	1140	940	006	880	1000	880	096	1260	1000	1000	1000	980	1000	940
Modulus, 100%	Aged	320	320	280	400	3.00	280	260	340	320	380	240	240	220	420	300	320	400	280	290	300	320	400	360
Moc	Orig	280	320	300	400	300	300	260	440	340	380	300	280	760	300	280	320	380	300	280	300	220	260	240
81 GHC		96	26	86	66	100	101	102	103	104	105	106	107	108	109	110	111	11.2	113	114	115	11-6	117	118

TABLE XVII, (Continued)

PHYSICAL PROPERTIES OF RUBBER STOCKS: ORIGINAL vs. AGED (1) vs. IRRADIATED (2)

	temp. Irr	ć	(3)								
	Flexometer temp.		(3) (3)								
	Flex		(3)								
	Dyn. Mod. psi O Irr		(3) (3)								
	Oyn.		(3)								
	Hardness O Irr				11		,	28	92	2.2	92
(7)	Hare		99	99	29	29		99	29	29	29
IATEI	% Irr		220	210	190	230		210	220	230	330
RAD	Elongation, % O A Irr		320	290	300	320		330	310	470	260
vs. I	Elong O		260	550	550	540		260	530	260	909
ORIGINAL vs. AGED (1) vs. IRRADIATED (2)	i Irrad		2520	2560	2360	2680		2460	2600	2380	3220
L vs. A	Tensile, psi Orig Aged Irrad	D	1140	1000	1100	1080		1040	1040	2260	3220
RIGINA	Ten Orig	O	3640	3660	3640	3360		3440	3380	3880	4040
0	Modulus, 100% Orig Aged Irrad		940	1000	1020	980		1000	980	880	780
	ulus, 1 Aged	i 0 1	340	340	320	300		300	300	300	7 60
	Mod Orig	9	260	280	280	260		260	240	240	200
	81 GHC		119	120	121	122		123	124	125	126

(3) These tests not made in the group 81GHC91-126 inclusive.

TABLE XVIII

PERCENT OF INITIAL PROPERTY RETAINED AFTER INDICATED EXPOSURE × 10⁹ ERGS/GRAM OF CARBON

Comments on cure	2 2 2 2 3	1371111	Ξ	=	Ξ	Ontimum		Ξ	=	Ξ	Ξ	=	7	5	Ξ	=	: :	: =	· <u>=</u>
Additive Co	<i>G</i>	PRNA 0.5		PBNA 2.0		Č	PRNA 0.5	BNA 1		4.		A.O. 40100.5	A.O. 4010 1.0	A.O. 4010 2.0	A.O.4010 4.0	30 JU711 Ju		32CH60G 2.0	
21.8	16	17	. œ	20	23	17	20	2 00	19	26	14			20		7			
7.	45	52	55	62	29		42				44		44			40			
Elongation	72	29	70	72	75	37	26			55	28			99				2 6	
3.0	84	80	90	87	83	7.1	75	62	80	83	62	17		98	78	ά,			
0.87	91	95	86	95	94	91	95	26	93	98	95	90	66	36	93	ς C) O		
21.8	28	33	59	39	41	30	39	39	37	51	38	37	38	42	32	36) (C	37	42
12	83	80	92	81	82	59	9	29	75	82	62	89	68	73	55	20			
Tensile	89	26	91	26	93	55	82	ć	82	82	90	84					000		
3.0	96	100	101	66	103	88	26	26	26	66	106	66		101	93		102		
0.87	94	96			101	66	104	105	102	102		104	104 1	103 1	100	107 1			
21.8	ı	650	009	650	999	1	443	ı		450	1		ı	488	1	1			658
lus 8.7	360		360		311	280	07	342	73	7.1	300	987	273	47	306	292	350	277	92
Modu 6.1	280	300	300		233		239			214	254	254	240	200	246	250			
100% Modulus 37 3.0 6.1 8.7	180	160	200	210	178		193			161	169				160	178			175
0.87	140	200	80	80	167	113	114	121	120	114	131	133	120	106	120	114			117
Cmpd. 81GHC	9	2	3	4	ις	41	42	43	44	45	82	83	84	85	86	87	88	68	06



TABLE XIX

PERCENT OF INITIAL PROPERTY RETAINED AFTER INDICATED EXPOSURE (ERGS PER GRAM OF CARBON x 10⁻⁹)

Cmpd.	100% Modulus 4.4 8.7 21.8			Tens	ile Str	ength	Ultimate Elongation 4 4 8 7 21.8			
81GHC	$\frac{1}{4.4}$	8.7	21.8	$\overline{4.4}$	8.7	21.8	4.4	8.7	21.8	
010110										
58	236	328	_	91	6 5	39	64	40	15	
59	206	280	520	87	72	40	66	46	19	
60	187	266	446	100	76	42	77	52	19	
61	215	307	_	95	72	37	75	50	17	
62	252	_	_	38	36	32	27	20	10	
02				_						
63	214	286	-	91	65	34	68	42	16	
64	200	280	-	79	47	29	59	29	16	
65	195	294	_	78	47	30	58	31	15	
66	195	310	-	80	44	26	59	27	12	
67	178	306	_	81	50	32	63	30	12	
68	183	306	-	89	57	39	66	32	14	
69	180	320	_	100	68	35	74	40	14	
70	183	333	_	78	47	36	59	28	13	
71	223	376	_	90	53	31	65	31	11	
72	223	362	_	86	55	38	66	32	14	
73	223	393	_	92	51	36	64	3 4	23	
7 4	216	392	-	104	66	43	74	37	15	
75	206	353	_	85	53	40	61	32	17	
76	194	307	_	96	64	39	71	41	17	
77	187	312	_	92	60	35	65	37	17	
• •	-0.									
78	220	348	_	86	58	34	61	37	15	
79	171	270	-	85	59	36	66	39	17	
80	200	347	-	97	64	39	69	38	15	
81	206	372	-	80	47	37	58	27	14	
91	187	262	-	96	80	39	74	49	18	
92	188	276	-	93	72	40	68	43	16	
93	188	272	_	95	74	38	69	44	15	
94	246	361	-	90	62	37	65	37	15	
95	196	258	-	64	41	33	45	25	12	
96	214	292	-	96.4	75	41	71	48	17	
97	188	275	457	100	76	42	74	47	19	
98	193	286	-	101	71	41	74	44	18	
99	145	210	395	87	71	38	74	51	18	
100	210	267	-	88	72	37	63	45	15	



TABLE XIX, (Continued)

PERCENT OF INITIAL PROPERTY RETAINED AFTER INDICATED EXPOSURE (ERGS PER GRAM OF CARBON \times 10 $^{-9}$)

Cmpd.	100%	100% Modulus 4.4 8.7 21.8			sile Str	ength	Ultimate Elongation 4.4 8.7 21.8			
81GHC	$\frac{4.4}{}$	8.7	21.8	4.4	8.7	21.8	4.4	8.7	21.8	
101	207	260	_	97	68	3მ	69	43	16	
102	238	308	_	98	82	39	67	50	17	
103	182	264	_	81	48	30	62	30	14	
104	188	294	_	89	59	35	80	35	14	
105	195	300	-	79	49	36	58	30	14	
106	213	313	_	95	67	40	69	40	17	
107	222	322	_	97	69	38	68	39	16	
108	231	338	-	99	69	39	67	40	16	
109	234	333	_	83	62	36	61	38	16	
110	228	314	-	88	62	37	65	39	16	
111	212	300	_	91	68	38	65	40	16	
112	210	332	-	78	44	31	56	30	11	
113	220	333	_	94	71	33	66	39	18	
114	236	357	_	94	68	37	70	38	18	
115	220	333	-	106	72	38	70	40	14	
116	300	445	_	89	58	29	62	32	12	
117	262	385	-	97	61	37	67	35	14	
118	275	392	-	97	73	41	66	40	16	
119	239	362	_	9 8	69	39	66	39	14	
120	250	357	-	96	70	40	67	38	16	
121	222	364	_	99	65	39	71	35	15	
122	246	377	-	108	80	40	74	43	15	
123	231	384	_	101	72	40	68	37	14	
124	266	408	-	103	77	40	68	42	15	
125	258	366	-	89	61	31	68	41	16	
126	280	390	730	95	80	44	76	55	20	
84	-	273	-	-	89	38	-	44	15	
41	-	280	-	-	59	30	-	37	17	



TABLE XX POLYMERS AND USES OF THE FACTORY RUBBER STOCKS

Compound	Polymer	Use
614FGA4	Natural Rubber	New Carcass
614FGA5	Natural Rubber	Tread
614FGA6	Hycar 1001	Self Sealing Cell Liner
614FGA7	Hycar 1043	Bladder
614FGA9	Hycar 1043 (85 pts) GR-S 1001 (13.7 pts)	Synthetic Hose Tube
614FGA10	Hycar 1043	Synthetic Hose Tube
614FGA11	Neoprene GN-A	Synthetic Hose Tube
614FGA12	Neoprene GN-A Hycar 1042	Synthetic Hose Cover
61 4FGA 13	Natural Rubber	Armorite Packing
614FGA15	GR-S 1500/1501 GR-S 1705	Armorite Gasket
614FGA16	Neoprene GN-A Neoprene S	Neoprene Gasket
614FGA17	Hycar 1001	Heat-Resistant Packing



	Physical	Initial	Exp	osure, .	Joules p	er Gra	m DC	
614FGA-	Property	Values	8.71	87.1	261	871	2610	7960
	T.S.	3375 PSI	110.3	104.0	73.6	64.5	29.3	31.1
4	U.E.	510%	104.5	103.9	78.4	55.5	14.7	4.9
	100% Mod.	380 PSI	51.8	90.3	129.7		305.3	Ϋ́
	T.S.	3100 PSI	93.8	96.1	87.5	48.5	25.6	25.5
4A	U.E.	525%	93.7	90.5	85.7	39.6	8.0	4.8
	100% Mod.	35 7P SI	54.1	86.0	128.8		Y	Y
	T.S.	3580 PSI	85.0	76.4	78.0	39.6	20.6	16.5
4B	U.E.	5 7 5%	91.0	85.6	78.3	36.2	8.7	4.3
	100% Mod.	330 PSI	63.6	75.8	136.4	180.9	Y	Y
	T.S.	3733 PSI	98.8	88.9	82.7	49.2	20.4	23.5
4C	U.E.	575%	92.7	88.3	78.3	47.8		4.3
	100% Mod.	373 PSI	72.4	94.6	135.9	175.1	297.6	Y
	T.S.	3340 PSI	103.6	110.3	108.2	83.4	39.8	22.8
5	U.E.	512%	98.2	100.2	87 . 9	53. 7	16.2	4.9
	100% Mod.	220 PSI	87.0	153.2	153.2	324.1	618.2	Y
	T.S.	3733 PSI	104.2	98.0	84.1	54.1	32.1	27.4
5 A	U.E.	493%	102.0	91.3	74.4	33.9	11.8	5,1
	100% Mod.	397 PSI	88.9	146.1	136.8	277.8	Y	Y
	T.S.	3990 PSI	97.7	93.5	83.0	49.0	19.6	23.4
5B	U.E.	525%	89.0	90.5	76.2	36.6	6.3	4.8
	100% Mod.	410 PSI	94.4	133.4	101.7	208.0	Y	Y
	T.S.	3927 PSI	101.7	98.0	81.2	51.6	28.6	20.0
5C	U.E.	483%	105.2	100.0	81.2	4 5.1	10.4	5.2
	100% Mod.	377 P SI	108.0	151.2	123.9	238.7	Y	Y
	T.S.	2133 PSI	102.3	100.5	105.6	98.2	87.2	x
6	U.E.	392%	89.3	82.9	54.1	19.1	8.4	X
	100% Mod.	267 PSI	110.5	172.3	300.7	Ÿ	Y	Y

 $[\]textbf{A:} \quad N, \, N^{\, \text{\tiny 1}}\text{-cyclohexylphenyl-p-phenylene diamine}$

B: 65% phenyl-alpha-naphthylamine + 35% N, N'-diphenyl-p-phenylene diamine

C: N- phenyl-N'-ortho-tolylethylene diamine



Compound	Physical	Initial	Ex	posure,	Joules			
614FGA-	Property	Values	3.71	87.1	261	871	2610	7960_
								
	T. S.	1860 PSI	103.9	102.2	113.1	113.8	91.6	122.0
6A	U.E.	508%	106.9	99.4	80.3	36.0	8.3	4.9
	100% Mod.	140 PSI	166.4	137.8	271.4	792.8	Y	Y
	T. S.	1950 PSI	122.0	96.2	113.5	102.0	92.2	120.9
6B	U.E.	492%	101.6	85.4	76.2	28.9	10.2	5.1
	100% Mod.	153 PSI	169.9	135.3	272.5	836.6	Y	Y
	T.S.	1847 PSI	128.3	106.7	113.7	111.5	109.2	134.5
6C	U. E.	508%	113.2	88.6	79.3	26.2		
00	100% Mod.	93 PSI	268.8	233.3		1304.3		Y
	T. C	1700 PSI	118.6	110.6	122. 5	137.8	160.2	118.8
-	T.S. U.E.	400%	104.2	73.0	48.8	8.2		
7		233 PSI	153.2	231.8	451.9	Y	Y	Y
	100% Mod.	233 P31	195.2	231.0	* 31. /	•	•	•
	т. S.	2000 PSI	112.5	109.2	107.5	115.6	129.0	176.8
7 A	U.E.	350%	103.7	114.3	76.3	29.4	7.1	7.1
	100% Mod.	333 PSI	136.0	125.2	203.3	663.7	Y	Y
	T.S.	2150 PSI	107.3	115.5	106.4	103.7	121.9	97.2
7B	U. E.	438%	98.6	79.9	61.0	21.0		
1.0	100% Mod.	267 PSI	169.7	157.3	273.4	859.6	Y	Y
	T.S.	2070 P S I	113.5	97.9	98.2	125.7	125.6	25.8
7C	U. E.	500%	85.0	73.4	45.0	16.6		
10	100% Mod.	223 PSI	227.4	144.8		1215.2		
	100 % Mod.	223101		1 1 1 1 0	202.0			
	T.S.	2043 PSI	101.3	107.3	105.2	120.6		
9	U.E.	167%	94.6	89.8	59.9	15.0		
	100% Mod.	1043 PSI	113.8	142.2	206.1	Z	Z	Z
	T. S.	2133 PSI	102.1	104.4	84.5	125.6	97.4	154.2
9A	U.E.	150%	100.0	88.7	72.0	44.7	16.7	16.7
•	100% Mod.	1033 PSI	106.2	120.3	148.8	Z	Z	Z

A: N, N' - cyclohexylphenyl-p-phenylene diamine

B: 65% phenyl-alpha-naphthylamine +35% N, N'-diphenyl-p-phenylene diamine

C: N-phenyl-N'-ortho-tolylethylene diamine



Compound	Physical	Initial	$\mathbf{E}_{\mathbf{X}}$	posure,	Joules	per Gra	am_DC	
614FGA-	Property	Values	8.71	87.1	261	871	2610	7960
	T.S.	2107 PSI	109.8	115.0	108.1	129.9	111.9	
9B	U.E.	150%	100.0	88.7		33.3	16.7	16.7
	100% Mod.	1247 PSI	101.8	101.4	128.3	Z	Z	Z
	T.S.	1630 P S I	135.6	111.2	131.3	108.7	90.8	150.9
9C	U.E.	125%	113.6	89.6	92.0	26.4	20.0	20.0
	100% Mod.	940 PSI	152.4	167.0	190.7	Z	. Z	Z
	T. S.	2385 PSI	102.3	99.7	115.3	103.8	111.1	x
10	U.E.	375%	93.3	90.9		13.3	6.7	X
	100% Mod.	365 P S I	89.6	185.5		Y	Y	Y
	T.S.	1650 PSI	57.0	87.7	86.4	100.0	62.2	166.1
10A	U. E.	225%	77.8	74.2	59.1	25.8	11.1	11.1
	100% Mod.	380 PSI	79.7	144.7	245.5	Y	Y	Y
	T. S.	2113 PSI	86.7	97.6	87.7	92.8	104.7	143.4
10B	U. E.	283%	100.0	76.0	55.8	20.5	8.8	8.8
102	100% Mod.	420 PSI	73.8	154.0	257.8	Y	Y	Y
	T.S.	2300 PSI	92.9	86.1	90.4	71.7	90.7	156.1
10C	U. E.	283%	101.4	75.3		8.8	8.8	8.8
100	100% Mod.	347 PSI	67.1	190.2		Y	у У	Y
	- A	0040 DGT	102 /	100.0	02.5	124.0	117 6	73.4
	T.S.	2043 PSI	102.6	72.7	92.5 54.6	124.0 13.7	117.5 13.7	
11	U.E.	183% 1103 P S I	77.6 136.3	135.7		13. <i>t</i>	Ϋ́ 13. (13. 1 Y
	100% Mod.	1103 PS1	130.3	155. /	171.4	1	1	1
	T.S.	1493 P S I	113.9	112.3		138.8		
11A	U.E.	215%	93.0	81.4		11.6	11.6	
	100% Mod.	750 PS I	142.7	151.6	150.3	Y	Y	Y
	T.S.	1633 P S I	103.7	103.3	98.0	141.4		
11B	U.E.	210%	107.1	87.1		11.9	11.9	
	100% Mod.	823 PSI	129.6	138.5	143.0	Y	Y	Y

A: N, N' - cyclohexylphenyl-p-phenylene diamine

B: 65% phenyl-alpha-naphthylamine + 35% N, N'-diphenyl-p-phenylene diamine

C: N - phenyl-N-ortho-tolylethylene diamine



Compound	Physical	Initial	Exp	osure, J	oules p	er Gra	m DC	
614FGA-	Property	<u>Values</u>	8.71	87.1	26.1	871	2510	7960
	T.S.	12/2 DCI	112 4	114 0	110 2	100 5		212 =
11C	1.5. U.E.	1367 PSI 200%	112.4	114.8	110.2	138.5		210.7
110	100% Mod.	657 PS I	100.0 145.0	87.5	75.0	12.5		12.5
	100% Mod.	057 P31	145.0	149.2	180.7	Y	Y	Y
	T.S.	1223 PSI	100.8	165.4	149.0	104.7	74.4	164.3
12	U.E.	292%	91.4	88.4	54.1	8.6	8.6	8.6
	100% Mod.	257 PSI	105.0	201.2	346.3	Y	Y	Y
	T.S.	1423 PSI	85.5	111.9	137.7	100.3	54.1	150.4
12A	U.E.	383%	102.3		77.0	23.4		6.5
	100% Mod.	167 PSI	77.8	189.8	305.4	790.4		Υ Υ
		_						
	T.S.	1317 PSI ·	-	126.3	161.7	117.2		149.2
12B	U.E.	400%	93.8	91.8	83,2	27.0	6.2	6.2
	100% Mod.	127 PSI	118.1	249.6	370.1	1084.2	Y	Y
	T.S.	1337 PS I	104.7	147.7	135.4	101.9	66.6	167.5
12C	U.E.	417%	95.4	101.9	67.9	10.1	6.0	6.0
	100% Mod.	110 PS I	118.2	315.4	381.8	Y	Y	Y
	T.S.	2780 PS I	97.1	119.8	116.7	60.9	47.8	26.9
13	U. E.	383%	89.6		91.4	37.1	6.5	6.5
15	100% Mod.	270 PSI	140.7	155.6	222.2	410.0	о. э Ү	о. э Ү
	100 /0 1010 4.	270151	140.1	155.0	<i>LLL. L</i>	410.0	1	1
	T.S.	2530 PS I	98.9	91.8	107.4	88.9	49.1	32.1
13A	U.E.	400%	85.5	79.2	75.0	37.5	14.5	6.2
	100% Mod.	433 PS I	99.3	123.1	174.8	322.6	Y	Y
	T.S.	3225 PSI	95.7	82.5	106.7	73.0	31.3	14.0
13B	U.E.	438 %	97.5	85.6	88.4	55.2	15.3	5.7
	100% Mod.	237 PSI	122.4	156.1	148.9	346.0	Y	Y
	T.S.	2130 PSI	120 4	160 (140 4		5 0 5	24.5
13C	u. E.	338%	120.6	160.6	148.4	111.4	53.5	34.3
	100% Mod.	338% 340 PSI	120.7	125.7	110.9	64.2	7.4	7.4
	100% Moa.	340 L91	134.4	138.2	183.2	338.2	Y	Y

A: N, N'-cyclohexylphenyl-p-phenylene diamine

B: 65% phenyl-alpha-naphthylamine +35% N, N'-diphenyl-p-phenylene diamine

C: N-phenyl-N'-ortho-tolylethylene diamine



TABLE XXI, (Continued)

Compounded	Physical	Initial	Exp	osure,	Joules p	er Gra	m DC	
614FGA	Property	Values	8.71	87.1	261	8 71	2610	7960
	T.S.	3385 PSI	96.6	94.4	96.0	77.7	49.4	54. 5
15	U.E.	500%	95.0	83.4	70.0	31.6	10.0	5.0
	100% Mod.	310 PSI	108.7	151.6	202.2	411.9	Y	Y
	T.S.	3373 PSI	93.4	90.0	85.1	67.3	50.8	55.0
15A	U.E.	595 %	91.3	84.0	56.0	3 4. l	13.9	4.2
	100% Mod.	190 PSI	138.4	182.6	215.8	435.3	1063.2	Y
	т. S.	3320 PSI	91.9	100.2	70.9	71.6	29.8	21.9
15B	U.E.	550%	81.8	86.4	51.4	36.4	7.6	4.5
	100% Mod.	230 PS I	149. 1	156.5	172.6	420.4	Y	Y
	T.S.	3387 PSI	98.5	91.4	91.3	56.9	47.9	38.6
15C	U.E.	553%	99.4	81.4	72.9	31.6	12.1	4.5
	100% Mod.	160 PSI	208.1	206.2	30.0	50.0	Y	Y
	T.S.	1687 P S I	80.6	96.0	76.3	79.0	59.6	92.5
16	U.E.	203%	114.8	86.2	65.5	24.6	12.3	12.3
	100% Mod.	810 PSI	59.9	123.1	101.6	Y	Y	Y
	T. S.	1543 PSI	95.3	97.4	84.0	84.4	67.5	75.8
16A	U.E.	287%	84.3	84.3	78.4	23.3	8.7	8.7
	100% Mod.	590 PS I	83.0	120.8	85.2	Y	Y	Y
	T.S.	1567 PSI	90.4	97.6	83.2	63.8		186.3
16B	U.E.	450%	97.1	106.2	85.1	27.8		5.6
	100% Mod.	467 PSI	65.7	83.5	59.3	207.1	Y	Y
	T.S.	1710 PS I	87.7	105.3	93.0	101.0		149.7
16C	U.E.	425%	94.1	96.9	68.7	15.8		5.9
	100% Mod.	503 PSI	77.5	114.7	102.8	Y	Y	Y
	T. S.	1527 PSI	100.8	104.1	106.5	92,1		
17	U.E.	300%	104.0	69.3	58.3	19.3		
	100% Mod.	600 P S I	111.2	165.5	213.8	Y	Y	Y

A: N, N'-cyclohexylphenyl-p-phenylene diamine

B: 65% phenyl-alpha-naphthylamine+35% N, N'-diphenyl-p-phenylene diamine

C: N-phenyl-N'-ortho-tolylethylene diamine



Compound	•	Initial	Exposure , Joules per Gram DC								
614FGA-	Property	Values	8.71	87. 1	261	871	2610	7960			
	T.S.	1287 PSI	101.0	102.0	102.6	119.4	130.0	Х			
17A	U.E.	275%	124.4	139.3	100.0	45.4	9.1	X			
	100% Mod.	660 P S I	73.2	98.0	93.9	221.2	Y	Y			
	T.S.	1433 PSI	93.5	100.7	101.2	111.0	122.1	X			
17B	U.E.	300%	106.7	102.7	83.3	34.3	8.3	Х			
	100% Mod.	647 PS I	78. 4	108.2	105.6	238.5	Y	Y			
	T.S.	1213 PSI	103.0	104.4	108.8	97.3	148.4	х			
17C	U.E.	308%	69.2	97.4	48.7	26.9	8.1	X			
	100% Mod.	593 PSI	87.2	113.5	134.9	217.5	Y	Y			

A: N, N'-cyclohexylphenyl-p-phenylene diamine

B: 65% phenyl-alpha-naphthylamine +35% N, N'-diphenyl-p-phenylene diamine

C: N-phenyl-N'-ortho-tolylethylene diamine

X: the samples were too brittle to test.

Y: no 100% modulus values were obtained because the ultimate elongation was less than 100%.

TABLE XXII

81GHB

EXTENDED ANTI-RAD SCREENING STUDY: RECIPES AND CURING INFORMATION

Pigment	2	6	41	20	9	7	∞	01	=	12	13	14	15	16
SBR 1500/1501	100	100	100	100	100	100	100							
Hycar 1002								100	100	100	100	100	100	100
Zinc Oxide	ß	ນ	5	ß	5	ß	ហ	Ŋ	2	Ŋ	Ŋ	Ŋ	5	5
EPC Black	40	40	40	40	40	40	40							
SRF Black								20	20	20	5 0	50	20	90
Stearic Acid	1.5	1.5	1.5	1.5	1.5	1.5	1.5							
Benzothiazyl Disulfide	n	æ	3	33	33	33	33	-	П	7	-	-	-	7
Sulfur	2	7	7	2	2	7	2	1.5	1.5	1.5	1.5	1.5	1.5	1.5
A		ß							S,					
В			2							ĸ				
U				ďΩ							ហ			
D					5							Ŋ		
ഥ						ıZ							Ŋ	
Ĺų							S.							S
Specific Gravity	1.13	1.13	1.13	1.13	1.13	1.13	1.13	1.19	1.19	1.19	1.19	1.19	1.19	1.19
Curing: Time (Min.)	9	45	101	40	118	37	09	18	2.2	75	17	15	18	19
Temp. OF.	293													1

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117

S

16

293

Curing:

100

31

TABLE XXII, (Continued)

EXTENDED ANTI-RAD SCREENING STUDY: RECIPES AND CURING INFORMATION

1.38 1.38 30 14 S 1.38 59 90 100 ß .38 28 100 20 ťΩ 1.38 1.38 27 17 100 100 17 56 1.13 1.40 25 100 17 81GHB 24 19 100 1.13 23 0.5 100 38 1.13 0.5 22 100 21 1.13 100 0.5 21 ٤O 17 0.5 0.5 2.0 1.13 1.13 20 100 100 84 23 S 1.13 2.0 100 17 18 50 Benzothiazyl Disulfide Tetramethyl Thiuram Time (Min.) Temp. ^oF. Magnesium Oxide Specific Gravity Neoprene GN Pigment Stearic Acid Disulfide Zinc Oxide EPC Black Butyl 218

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Sulfur

4 E C C B A

Contrails

TABLE XXII, (Continued)

EXTENDED ANTI-RAD SCREENING STUDY: RECIPES AND CURING INFORMATION

81GHB

Pigment	33	34	35	36	37	38	39	41	42	43	44	45	46	47
	100	100	100	100	100	100	100	100	100	100	100	100	100	100
	20	20	20	20	70	20	20	40	40	40	40	40	40	40
	2.5	2.5	2.5	2.5	2.5	2.5	2.5	-	-4	-	-		-	-
	-	7	-	-	-	~	-	ъ	B	3	m	m	3	8
Magnesium Oxide	30	30	30	30	30	30	30	0.5		0.5	0.5	0.5	0.5	0.5
	÷	5							ഹ	ι				
			Ŋ	ı						ഹ	u			
				C)	1						n	u		
					v	ı						n	u	
							70						0	'n
Specific Gravity	1.34	1.33	1.33	1.33	1.33	1.33	1.33 1.23	1.23	1.2	1.2	1.2	1.22	1.22	1.2
: Time (Min.)	32	22	21	19	22	20	30	14	18	150	16	30	13	12
H	293							:						•

Contrails

TABLE XXII, (Continued)

EXTENDED ANTI-RAD SCREENING STUDY: RECIPES AND CURING INFORMATION

60 61	100 100 40 40		r. r.	1.50 1.50 X 56
59	100	0.5 10 0.1 0.3	ĸ	1.50
58	100 40	0.5		1.50 X
57	10់ 40	0.5 10 0.1 0.3		1.50 X
56	100	0.5 10 0.1 0.3		1.50
55	100			1,52
54	100	0.4. ພາບ.	5	1.40
53	100		S	1.40
52	100		ഹ	1.40
51	100			1.40
50	100	2.0 2.5 3.5 5.5 5.5		1.45 X
49	100	6.04 6.5 7		1.40
48	100	0.5 2.5 3.5		1.42
Pigment	Thiokol ST Thiokol FA SRF Black	Stearic Acid Zinc Oxide GMF DPG Benzothiazyl Disulfide A C	ሀ터ቮ	Specific Gravity Curing: Time (Min.)

X - Did not cure



Note: All of the anti-rads were added to the master stock on the basis of 5 parts anti-rad per hundred part rubber. Following in the list of symbols used in designating each anti-rad in this table.

Anti-Rad	Symbol
Phenyl-alpha-naphthylamine plus N, N', diphenyl-p-phenylene diamine	А
Quinhydrone	В
Alpha-naphthylamine	С
Beta-naphthol	D
N-phenyl-N'-ortho -tolylethylene diamine	E
Anthraquinone	F



TABLE XXIII
RECIPES AND CURING INGREDIENTS FOR THE NEWER ELASTOMERS

Pigment	\$1GHB97	81GHB98	81GHB99	81GHB107	81GHB108
Kel-F 3700 SE - 550 (Compounded stock from General Electric SE - 74	100 Co.)	100	100		
(Compounded stock frogeneral Electric Co.) 1F4				100	
2F4 SRF Black				40	100 4 0
Zinc Oxide	10				
Dyphos	10				
Sulfur				1	1
Benzoyl Perioxide Triethylene tetramine	3			1.25	1.25
Specific Gravity	2.04	1.15	1.14	1.66	1. 6 6
Press Curing:	15	10	10	30	30
Time (Min.) Temperature, ^o F.		260	260	320	320
Oven Curing: Time (Hrs.) Temperature, OF.	16 . 300	24 480	24 480	24 300	24 300

TABLE XXIV

RECIPES OF VITON A COMPOUNDS FOR ANTI-RAD SCREENING

Pigment	81GHB100	81GHB101	81GHB102 181GHB103	81GHB103	81GHB104	81GHB105	81GHB106
Viton A	100	100	100	100	100	100	100
Magnesium Oxide	20	20	20	20	20	20	20
(Heavy)							
Mountain Black	25	25	25	25	25	25	25
Hexamethylene	1.3	1.3	1.3	1.3	1.3	1.3	1,3
diamine Carbamate							
N N' Di-beta	0.2	0.2	0.2	0.2	0.2	0.2	0.2
naphthyl-para							
phenylene - diamine							
Phenyl-alpha		ιΛ					
Naphthylamine plus							
N, N' - diphenyl para							
phenylene diamine							
Quinhydrone			5				
Alpha-naphthylamine				Z.			
Beta - naphthol					5		
N-Phenyl - N- ortho-						5	
tolylethylene diamine							
Anthraquinone							un.
Specific Gravity	1.91	1.85	1.85	1.85	1.85	1.85	1.85
Press Curing:							1
Time (Min.)	30	30	30	30	30	30	30
Temperature, F.	320	320	320	320	320	320	320
Oven Curing:							
Time (Hrs.)	54	24	24	24	24	24	24
Temperature, F.	400	400	400	460	400	400	400

(1) Did not cure

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TABLE XXV

PHYSICAL PROPERTIES OF VITON A VARIATIONS

Cmpd. 81GHB	Anti-Rad	Tensile Strength (psi)	Ultimate Elongation (percent)	100% Modulus (psi)
	None	1685	175	569
101	Phenyl-alpha-naphthylamine plus N, N'-diphenyl-p-phenyl- ene diamine	1790	180	909
102	Quinhydrone	345	585	240
103	Alpha-naphthylamine	1805	155	666
104	Beta - Naphthol	1825	205	773
105	N-Phenyl-N'-ortho tolylethylene diamine	1830	140	1190
901	Anthraquinone	1710	155	840

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TABLE XXVI

EXTENSION OF SERVICE LIFE OF RUBBER STOCKS BY ANTI-RADS

("Service Life" is that radiation exposure which results in a loss of 50 percent of the initial ultimate elongation")

Compound 614 FGA-	Base Elastomer	Without Anti-Rad (Joules/Gram DC)	With Anti-Rad (Joules/Gram DC)
4	Natural Rubber	750	650
5	Natural Rubber	750	700
6	Hycar 1001	300	600
7	Hycar 1043	300	700
9	Hycar 1043 SBR 1001	400	650
10	Hycar 1043	200	300
11	Neoprene GN-A	300	450
12	Neoprene GN-A Hycar 1042	250	600
13	Natural Rubber	650	1150
15	SBR 1500/1501 SBR 1705	450	550
16	Neoprene S Neoprene GN-A	400	500
17	Hycar 1001	350	850



TABLE XXVII

RECIPES OF PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS

Neoprene Wire Insulation 81GH311		81GH317		81GH318	
Neoprene GN	100	81GH311	100 8	1GH311	100
Mineral Rubber	10	Akroflex C	_2 0	uinhydrone	2
Light Magnesium Oxide	4		102		102
Fine Clay	35				
White Talc	35				
Ultra-Fine Whiting	35				
MPC Black	10				
Paraffin (Refined) Wax	6				
Light Amber Petrolatum	3				
PBNA	2				
Zinc Oxide	10				
	250				
Equiv. Cure 28' x 293°F. Sp. Gr. 1.67		Equiv. Cure :	24' x 293 ⁰ F 1.67	. Equiv. Cu Sp. Gr.	



RECIPES OF PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS

SBR Wire Insulation 81GH313		81GH327	81GH3	28
SBR-1007	100	81GH313	100 81GH3	13 100
Altax	3	Akroflex C	1.25 Quin	hydrone 1.25
Tuads	1.5		101.25	101.25
Litharge	1.5			
EPC Black	20.			
Ultra-Fine Whiting	78 . 5			
Fine Clay	120.			
Stearic Acid	1.			
Mineral Rubber	60.			
Paraffin (Refined) Wax	6.			
Zinc Oxide	4.			
PBNA	2.			
Sulfur	2.5			
	400.0			
Equiv. Cure 12' x 293°F. Sp. Gr. 1.51				quiv. Cure 10'x 293°F. b. Gr. 1.51



RECIPES OF PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS

Neoprene Packing Comp 81GH357	pound	81GH359	81GH360	
Neoprene W	60	81GH357	100 81GH357	100
Neoprene WHV	40	Akroflex C	2.99 Quinhydr	on <u>e 2.99</u>
Magnesium Oxide	2		102.99	102.99
P BII A	2		. •	·
MT Black	35			
Zinc Oxide	5			
2-Mercaptoimidazoline	1			•
MBTS	1			•
F.A.Ester	20			
Sun Resistant Wax	1			÷
	167			
Equiv. Cure 25' x 293°	'F.	-	20' x 293° F. Equ	iv. Cure 15'x 293°F.

Sp. Gr. 1.32 Sp. Gr. 1.32

Sp. Gr. 1.32



RECIPES OF PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS

Hycar Packing Compound 81GH358		81GH365	81GH366
Hycar 1001	80	81 GH358	100 81GH358 100
Hycar 1002	20	Akro Flex	2.725 Quinhydrone 2.725
EPC Black	40		102.725 102.725
Lauric Acid	0.75		
Zinc Oxide	5		
F. A. Ester	10		
Dioctyl Phthalate	10		
Tributoxy Et. Phosphate	15		
TMTD	0.10		
Sulfur	1.25		
MBTS	1.25	ı	
	183.35		
Equiv. Cure 30 x 293°F. SP. GR. = 1.14		Equiv.Care 2 SP. GR. = 1	5 x 293°F. Equiv. Cure 135 x 293°F. 14 SP. GR. = 1.14



RECIPES OF PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS

Hycar/Vinylite Bladder Sto 81GH386	ock_	81GH391		81GH393	_
Hycar 1043	100	81GH386 1	.00	81GH386	100
SRF Black	80	Akro Flex C	2.07	Quinhydron	ie 2.07
81GH386A	52.6	1	102.07		102.07
Stearic Acid	1				
Vultrol	0.5				
TMTM	0.3				
MBTS	1.				
Zinc Oxide	5.				
Sulfur	1.				
	241.4				
Equiv. Cure 18 x 293°F. SP. GR. = 1.17		Equiv. Cure 17x 2 SP.GR. = 1.	293 ⁰ F. 17	Equiv. Cure 12 SP. GR. =	
81GH386A					
VYNV2	38.02				
2 PbCO ₃ ·Pb(OH) ₂	1.14				
Hycar 1312	22.81				
Dibutyl Sebacate	38.03				
	100.0				
SP. GR. =	1.09				



RECIPES OF PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS

Hycar Self-Sealing Lin 81GH367	ner Stock	81GH395		81GH397	_
Hycar 1011	100	61 GH387	100	81GH387	100
SRF Black	80	AkroFlex C	2.29	Quinhydro	ne 2.29
Zinc Oxide	5		102.29		102.29
Dibutyl Phthalate	30				
Stearic Acid	1.				
TMTM	0.3				
MBTS	1.				
Sulfur	1				
	218.3				

Equiv. Cure 12 x 293°F. Equiv. Cure 10x293°F. Equiv. Cure 225x293°F. SP. GR. = 1.24 SP. GR. = 1.24 SP. GR. = 1.24



RECIPES OF PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS

Hycar/SBR Tube Stock 81GH420		81GH423		81GH425	
Hycar 1043	85	81GH420	100	81GH420 10	0
SBR 1001	13.67	AkroFlex C	1.88	Quinhydrone	1.88
Agerite Powder	2.0		101.88	10	1.88
SRF Black	130.				
Zinc Oxide	5.				
F.A. Ester	15.				
Tributoxy Ethyl Phosphate	10.				
MB TS	2.				
Sulfur M.B. (60%)	3.33				
	266.				
Equiv. Cure 15 x 293°F. SP. GR. = 1.27		Equiv. Cure		Equiv.Cure 23x 7 SP. GR. = 1.	



RECIPES OF PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS

Neoprene Tube Stock 81GH421		81GH427		81GH429		
Neoprene GN	100	81GH421	100	81GH421	100	
Agerite Resin D	1.50	Akroflex C	2.385	Quinhydro	ne 2.385	
MT Black	60.		102,385		102.385	
SRF Black	30.					
Lauric Acid	2.					
Processing Oil	5.					
Zinc Oxide	5.					
MBTS	2.		•			
Magnesium Oxide	4.					
	209.50					
Equiv. Cure 13' x 2	93°F.	Equiv. Cur	e 10' x 293	^o F. Equiv.	Cure 7' x 293 ⁰ F	٦.



TABLE XXVIII

CURE TIME DATA FOR PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS

Compound	d	Mooney	Cure Time	Equivalent
81GH	<u>Material</u>	M ₇ _	M	Cure Time
	-	'_	$(Min. \times 29)$	
				·
311	Neoprene Wire Insulation-Control	28	39	28
317	Neoprene Wire Insulation-Akroflex C	24	32	24
318	Neoprene Wire Insulation-Quinhydrone	9	12	9
313	SBR Wire Insulation-Control	12	15	13
327	SBR Wire Insulation-Akroflex C	12	14	12
328	SBR Wire Insulation-Quinhydrone	12	16	13
320	bate wire madation-Quinnydrone	12	10	10
357	Neoprene Packing CmpdControl	17	22	25
359	Neoprene Packing Cmpd Akroflex C	16	21	20
360	Neoprene Packing Cmpd Quinhydrone	12	16	15
358	Hycar Packing CmpdControl	23	30	30
365	Hycar Packing CmpdAkroflex C	17	22	25
366	Hycar Packing Cmpd Quinhydrone	97	136	135
386	Hirona / Winglita Bladdan Control	1.5	10	
391	Hycar/Vinylite Bladder-Control	15	18	18
393	Hycar/Vinylite Bladder-Akroflex C	14	17	17
273	Hycar/Vinylite Bladder-Quinhydrone	91	125	125
387	Hycar Self-Sealing Cell Liner-Control	10	12	12
395	Hycar Self-Sealing Cell Liner-Akroflex C		9	10
397	Hycar Self-Sealing Cell Liner-Quinhydron		51	225
420	Hycar-SBR Tube Stock-Control	12	15	15
423	Hycar-SBR Tube Stock-Akroflex C	11	14	12
425	Hycar-SBR Tube Stock-Quinhydrone	23	31	23
421	Neoprene Tube Stock-Control	13	17	13
427	Neoprene Tube Stock-Akroflex C	10	14	10
429	Neoprene Tube Stock-Quinhydrone	5	7	7
	The state of the s	٠	,	ſ



TABLE XXIX

THE EFFECT OF ROOM TEMPERATURE GAMMA RADIATION ON THE ROOM TEMPERATURE PHYSICAL PROPERTIES OF PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS

(1 MR Equals 83.9 \times 10⁶ Ergs per Gram of Carbon)

---- Samples broke before test data could be taken.

^{*}Four dumbbells were tested instead of five.

^{**} Three dumbbells were tested instead of five.



		Co	balt 60	Radiation I	ose (MR)	
Compound	Property	0	25	50	100	150
0.0						
81GH311	T	1430	1270	1180	1490	
	E	550	420	180	70	2420
	M_1	680	820	1060		
•	M_2	870	960			
	M_3	1030	1070			
	H	76	83	87	94	
81GH317	T	1480		1360	1420	1590
	E	670		310	140	70
	Ml	560	e = = =	920	1280	
	M_2^1	820		1130		
	M ₃	950		1330		
	H	72	~ 	85	90	93
				•	, ,	,,
81GH318	\mathbf{T}	1330		1120	1270	1690
	E	580		200	150	50
	M_1	740		980	1140	es 📾 🖦 w
	M_2	930		1100*		
	M_3^{-}	1050		***		
	Н	80		88	90	95
81GH313	Т	1060	9 7 0	920	920	
	E	290	250	200	120	
	$\overline{\mathtt{M}}_{1}$	530	680	7 50*	870	
	M ₂	800	950	920		
	M ₃		m (4 m es	, _ ·		
	H	78	84	85	89	
		10	01	03	0,7	
81GH327	T	1020		930	910	960
	E	. 300		220	180	160
	M_1	490		680	750	900
	M_2	770		920		
	M_3^{5}	1040				
	н	77		85	87	88



THE EFFECT OF ROOM TEMPERATURE GAMMA RADIATION ON THE ROOM TEMPERATURE PHYSICAL PROPERTIES OF PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS

			Cobalt 60		on Dose (M	<u>(R)</u>
Compound	Property	0	_25_	50	100	150
81GH328	${f T}$	9 7 0		870	870	940
	E	360		200	200	90
	M_1	430		660	720	
	M_2	680		850	880*	
	M_3^-	860				
	H	78		85	86	90
81GE357	${f T}$	2130	740	300	350	
	E	760	270	100	40	
	M_1	80	160	300		
	M_2	200	420			~ -
	M_3	430				
	M_4	830				
	Η	45	53	62	75	
81GH359	T	2210		330	190	240
•	E	850		170	60	70
	M_1	70		150		
	M_2	160				
	M_3^2	320				
	M_4	610				
	Η	42		58	69	72
81GH360	Т	2250		530	330	280
	E	810		290	130	60
	M_1	120		140	250	
	M_2	230		330		
	м3	420				
	M_4	650				
	H	47		53	5 7	67
81GH358	Т	2040	1660	1340	1130	
	E	500	260	140	80	
	M_1	110	220	680		
	M_2	300	1030			
	м3	820				
	н	48	5 7	68	77	

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			Cobalt 60	Radiatio	n Dose (MF	₹)
Compound	Property	0	25	50	100	150
010770/5						
81GH365	<u>T</u>	2100	₩ 🙃 🖘	1730	1200	1260
	E	550		260	140	140
	\mathbf{M}_{1}	90		260	820	780
	M ₂	270		1190		
	M_3	680				
	Н	46		57	64	65
81GH366	T	2130		1850	1800	1530
	E	500		260	170	120
	\mathbf{M}_1	100		250	560	1210
	$M_2^{\frac{1}{2}}$	350		1140		
	M_3	790				
	н	50		60	65	72
						-
81GH386	T	2270	2330	2350	1750	
	E	420	170	140	40	
	M_1	400	1210	1600		
	M_2	1170				
	M_3^-	1800				
	н	65	75	80	90	
81GH391	T	2150		2220	2320	2520
	E	470		190	90	70
	M_1	380		1000		***
	M_2	1030				
	M_3	1620				
	H	65		7 5	85	90
81GH393	т	2140		3440	25.30	2400
01011373	E	2140 410	~-~-	2440	2570	2480
		370		220	140	100
	$\mathbf{M_1}$	8 7 0		890	1850	2460*
	M ₂	1610		2200		
	м ₃ Н	65		72		
	n	05		72	80	85



			Cobalt 60	Radiatio	n Dose (MR)	
Compound	Property	0	25	_50	100	150
81GH387	Т	2200				
01011301	E	2280	2480	2420	2550	
		460	220	170	80	
	M_1	190	730	1050		≈
	M ₂	840	2270			
	М ₃ Н	1630	9===			
	п	57	7 0	74	82	
81GH395	T	2120		2230	22 7 0	2150
	E	500		280	160	120
	M_1	160		530	1220	1820
	M_2	800		1760		
	M_3^-	1400			@ D == =	
	Н	55		65	74	7 5
81GH397	T	2310		2390	2390	2260
	E	490		300	180	160
	\mathbf{M}_1	190		430	970	1470
	M_2	7 30		1670	===	1110
	M_3^-	1480				
	Н	55		65	69	77
81GH420	Т	2240	2460	2610	2620	
	E	200	150	110	80	
	\mathbf{M}_{1}	890	15 7 0	2380		
	M_2^{-}		0 = = 5	pee		
	M_3^2	~				
	H	71	80	84	85	
81GH423	Т	2270	2500	2460	2310	2380
	E	210	150	110	90	90
	M_1	800	1450	2040	70	90
	M ₂	2270				
	M_3^2			===		~~==
	Н	70	. 77	83	85	82



		C	Cobalt 60	Radiation	Dose (MR)	
Compound	Property		25	50	100	150
81GH425	Т	2300	2640	2670	2770	2810
01011123	Ē	200	150	120	90	90
	M	850	1640	2350		
	M ₂	2300*				
	M ₃					
•	н	71	80	83	86	88
81GH421	т	1830	1820	1860	2150	
	E	390	250	180	100	
	\mathbf{M}_1	590	800	1180	2150*	
	M ₂	1340	1680	e -		
	M ₃	1780	c	2-0-		
	Н	73	78	80	85	
81GH427	Т	1570	1630	1670	1940	2010
4 - 4-1 -1	E	420	290	180	100	60
	M_1	480	660	1080	1940*	
	M_2	1100	1480			- 44 47 43
	M_3^2	1480	~= ~ →	e = = #		
n	н	70	7 5	80	90	95
81GH429	Т	1700	1660	1720	1980	2070
0101112,	E	370	200	110	60	40
	M_1	660	1020	1620		
	M_2	1350	1660*		p	
	M_3^2	1630	~ ~ ~ ~		~ 	
	H	76	83	88	94	95



TABLE XXX

THE EFFECT OF 158°F. GAMMA RADIATION ON THE ROOM TEMPERATURE PHYSICAL PROPERTIES OF PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS

(1 MR Equals 83.9 x 10^6 Ergs per Gram of Carbon)

⁻⁻⁻⁻ Samples broke before test data could be taken.

^{*} Four dumbbells were tested.

^{**} Three dumbbells were tested.



			Cobalt 60	Radiatio	n Dose (MR)	
Compound	Property	0	10	20	_40	70
81GH311	T	1430	1280	1180	1080*	1150
	E	550	570	330	280	220
	M_1	680	610	870	820	920
	M_2^1	870	7 90	1040	960	1080*
	M3	1030	870	1160		
	H	76	77	83	85	88
81GH317	T	1480	1260	1260	1200	1250
	E	670	600*	380	300	200
	M_1	560	550	800	840	1000
	M_2	820	720	1010	1010	1250*
	M_3^2	950	830	1130	1200*	
	н	72	7 3	80	83	86
81GH318	T	1330	1130	1150	1160	1210
	\mathbf{E}	580	410	140	160	80
	M_1	74 0	7 50	1030	1070	4 - 2 -
	$\mathbf{M}_{2}^{\mathbf{T}}$	930	910			
	M_3^2	1050	980			
	H	80	83	88	84	90
81GH313	Т	1060	960	970	940	980
	E	290	300	180	200	150
	\mathbf{M}_{1}	530	530	760	600	890
	M_2	800	780	4 B C G	900*	
	M_3^2					
	н	78	80	85	83	87
81GH327	Т	1020	930	930	950	1000
	${f E}$	300	300	180	250	170
	M_1	490	460	750	630	820
	M_2	770	740		880	
	M_3^2	1040	940*			
•	н	77	80	84	82	86



	_		Cobalt 60	Radiati	on Dose (M	R)
Compound	Property	0	10	_20	40	
81GH328	T	970	870	880	950	0.00
	E	360	310	210	850	900
	$\overline{\mathtt{M}}_{1}$	430	470		200	170
	M ₂	680	710	660	620	810
	M_3	860	870*	880	850	
	H	78	81		00	
	••	10	01	84	83	87
81GH357	T	2130	1470	600	350	310
	$\mathbf E$	760	470	290	130	80
	M_1	80	110	100	230	
	M_2	200	270	300		
	M_3^2	430	580			
	н	45	45	55	60	62
81GH359	Т	2210	1440	0.40	250	
,	Ē	850	600	940 460	350	240
	M ₁	70	90		170	100
	M ₁	160	180	90	150	240
	M_3	320	430	170		D 00 ## 6
	H	42		400		~==
	11	42	45	50	55	60
81GH360	T	2250	1670	1510	5 7 0	460
	E	810	690	660	250	150
	\mathbf{M}_{1}	120	90	100	180	280
	M_2	230	230	240	430	Mose
	M_3^2	420	440	450		
	H	47	45	45	52	56
81GH357	Т	2130	1440	760	240	220
	Ē	760	460	290	340	320
	M_1	80	110	100	130	80
	M_2	200	290	300	250	
	M_3	430	640	300	a =	
	H	45	50	55	*	
	••	13	50	22	58	61



			Cobalt 60	Radiation	n Dose (MR	
Compound	Property	0	10	20	40	70
Odinpound	<u> </u>					- (-
81GH359	T	2210	1460	9 7 0	340	260
0 0	E	850	600	440	170	100*
	M_1	70	90	110	140	260
	M_2^1	160	180	200		
	M_3^2	320	430	470	- -	
	н	42	46	50	55	58
81GH360	${f T}$	2250	1880	1480	620	360
01011900	Ē	810	710	670	280	110
	 М ₁	120	110	120	170	330
	M_2	230	220	250	430	
	M_3	420	420	420		
	H	47	45	47	53	59
81GH358	T	2040	1860	1680	1470	1260
01011550	Ē	500	320	270	180	130
	M	110	150	180	430	790
	M_2	300	600	840		
	M_3	820	1460			
	н	48	53	55	65	70
81GH365	Т	2100	1860	1500	1570	1390
81011303	Ē	550	370	290	210	170
	$\overline{\mathbf{M}}_{1}$	90	110	190	360	520
	M_2	270	460	790	1460	
	M ₃	680	1160	m a 57		0 = 0 0
	H	46	49	55	60	63
81GH366	Т	2130	2130	2230	1850	1870
81GH300	Ē	500	360	340	230	180
	\mathbf{M}_1	100	160	200	360	540
	M ₂	350	610	77 0	1600	C = 00
	M ₃	790	1630	1770		
	H	50	52	55	60	64



			Cobalt 60	Radiatio	n Dose (Mi	₹)
Compound	Property	0	10	20	40	
						
81GH358	${f T}$	2040	1 97 0	1450	1310	1370
	E	500	350	300	190	130
	\mathbf{M}_{1}	110	160	160	380	870
	M_2	300	630	74 0		
	M_3^-	820	1580	1470	# C = G	
	H	48	53	56	60	69
81GH365	T	2100	1800	1640	1600	1700
	E	550	380	320	220	150
	\mathbf{M}_1	90	110	160	320	630
	M_2	270	400	700	1530	
	M_3^2	680	1010	1560		~==6
	н	46	50	55	60	65
81GH366	Т	2130	2230	2190	1930	1850
	E	500	390	350	120	160
	Mi	100	140	170	370	580
	M_2	350	560	74 0	1690	
	M_3^2	790	1400	1690	a =	# E & &
	н	50		54	60	63
81GH386	Т	2270	2310	2290	2510	2880
	E	420	300	280	150	90
	M_1	400	580	550	1400	
	M_2	1170	1490	1690		
	M_3^2	1800		e அ சரை		
	Н	65	66	7 0	7 5	81
81GH391	T	2150	2060	2250	2400	2660
	E	470	400	280	190	110
	\mathbf{M}_1	380	360	520	1030	2410
	M ₂	1030	1200	1630		
	M_3^2	1620	1770	c1 = c3 =		G = 44 G
	н	6 5	65	62	70	7 9



			Cobalt 60	Radiatio	n Dose (Mi	₹)
Compound	Property	0_	10	20	40	
			 			
81GH393	T	2140	2360	2420	2580	2590
	E	410	390	300	200	140
	M_1	370	420	630	1130	1800
	M_2	870	1330	1760		******
	M_3	1610	2040	2360*		
	H	65	65	7 0	7 0	75
	_				_	
81GH387	<u>T</u>	2280	2310	2390	2480	2750
	E	460	320	300	190	150
	M ₁	190	360	460	940	2110
	M_2	840	1370	1520		
	M_3^-	1630	2160		~ ~ ~ ~	
	H	57	60	65	70	7 5
016***		2122	21/2		2.400	2//2
81GH395	<u>T</u>	2120	2160	2220	2400	2660
	E	500	410	350	260	190
	M ₁	160	210	280	550	1010
	M ₂	800	920	1360	1940	
	M ₃	1400	1660	1980		
	Н	55	59	60	64	68
81GH397	Т	2310	2360	2400	2430	2550
,	E	490	400	380	290	210
	M_1	190	200	340	5 4 0	880
	M_2^1	730	940	.1240	1870	2390*
	M_3^2	1480	1780	2060		
	н	55	54	60	64	65
81GH420	T	2240	2510	2580	2150	2320
	E	200	150	140	100	90
	M_1	890	1480	1810	2040*	₩ ₩ 15 ±
	M2					en
	M_3	20.0		c 		
	H	71	75	78	80	85



			Cobalt 60	Radiat	ion Dose (MR)
Compound	Property	0	_10_	_20	40	70
81GH423	Т	2270	2510	2460	2490	2630
	E	210	170	150	$\frac{2470}{140}$	
	M_1	800	1280	1580	1730	100
	M_2	2270		7500	1730	2630*
	M ₃			9000		
	H	70	74	75	75	80
81GH425	Т	2300	2420	25.10		
01011425	E		2430	2510	2470	26 7 0
		200	170	160	150	100
	$^{\mathrm{M}}_{\mathrm{1}}$	850	1280	1580	1580	2670*
	M ₂	2300*		= m +, =		** <> = m
	M_3					50 50 E
	H	71	7 5	76	75	83
81GH421	T	1830	1860	1740	1910	2050
	E	390	300	260	180	110
	\mathbf{M}_1	590	7 80	780	1030	1950
	M_2	1340	1630	1620		
	M_3^2	1780	1860*			
	н	7 3	7 5	7 5	75	85
81GH427	Т	1570	1.640	1630	1690	2010
	E	420	370	320	250	150
	M_1	480	600	630	74 0	1500
	M_2	1100	1310	1350	1540	1500
	M_3^2	1480	1640	1630		
	H	70	70	74	7 5	85
81GH429	Т	1700	1660	1720	1810	1050
	E	370	270	220	200	1950
	M ₁	660	830	920		100
	M_1	1350	1560		1020	1950*
	M ₂ M ₃	1630		1720	1810	
	H	76	75	80	80	85
	**		13	00	00	00



TABLE XXXI

THE EFFECT OF 158°F. GAMMA RADIATION ON THE 158°F. PHYSICAL PROPERTIES OF PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS

(1 MR Equals 83.9 x 10⁶ Ergs per Gram of Carbon)

⁻⁻⁻⁻ Samples broke before test data could be taken.

^{*} Four dumbbells were tested.

^{**} Three dumbbells were tested.



			Cobalt 60	Radiati	on Dose (M	R)
Compound	Property	0	10	20	40	70
81GH311	T	860	720	620	600	740
	${f E}$	650	500	310	230	100
	M ₁	410	420	470	510	740
	M ₂	490	470	550*	580	# 45 46 73
	M_3^-	540	530	2 m # 5		*****
	H	76	7 9	81	85	90
81GH317	Т	900	780	680	650	720
	E	700	580	380	250	180
	M	360	3 7 0	440	510	620
	M_2	440	480	520	620	
	M_3^-	500	500	600		
	H	72	7 5	7 9	83	85
81GH318	T	820	630	630	690	690
	E	600	380	90	130	100
	M_1	420	470	***	666	690
	M_2	490	550			
	M_3^2	530	600			
	Н	80	83	86	90	90
81GH313	T	770	680	660	670	610
	E	300	280	180	200*	140
	\mathbf{M}_1	360	330	470	440	550
	M_2^2	580	490		670**	# M E O
	M ₃					
	H	78	81	85	86	. 88
81GH327	T	720	680	610	620	680
	E	300	320	190	190	170
	\mathtt{M}_1	320	290	420	450	520
	M_2^-	500	480		я ев	
	M_3		670*			
	н	77	80	83	85	85



THE EFFECT OF 158°F. GAMMA RADIATION ON THE 158°F. PHYSICAL PROPERTIES OF PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS

			Cobalt 60	Radiation	Dose (MR)	
Compound	Property	00	10	20	40	70
				_		
81GH328	${f T}$	730	600	600	600	630
	E	370	360	290	220	170
	\mathbf{M}_{1}	260	430	340	390	500
	M_2	380	570	520	550	
	M_3	570			co 10 40 44	
	Н	78	81	82	82	87
81GH357	т	400	240*	250*	120*	140*
	E	340	210*	170*	120*	60*
	\mathbf{M}_1	80	110*	150*	120*	
	M_2	170				
	M_3	300				
	M_4					
	H	45	48	52	60	65
81GH359	${f T}$	440	360*	280*	230*	150*
	${f E}$	420	340*	220*	1 7 0*	70*
	M_1	90	90*	100*	140*	
	M ₂	170	170*	€ 50 € D	8000	
	M_3	260	290*		000	
	M_4		P			
	H	42	44	45	55	62
81GH360	T	310	250*	270*	260*	200*
	${f E}$	320	220*	200*	190*	80*
	M_1	90	130*	160*	170*	
	M_2	200		* - * +	~ = = =	0
	M_3^2	310		*		
	. M ₄					
	Η	47	49	50	51	60
81GH358	T	530	630*	550*	430*	540*
	E	290	260*	190*	140*	100*
	M_1	100	150*	220*	320*	540*
	M_2	310	490*			
	M_3^2					
	M_4^3					
	H	48	53	56	60	69

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			Cobalt 60	Radiation	Dose (MR)	
Compound	Property	0	10	20	40	70
81GH365	T	440	(204	/ 0 0 .1		
01GH305	E	640	620*	600*	580*	550*
		320	280*	200*	200*	150*
	M ₁	80	140*	200*	200*	330*
	M ₂	230	330*	7000		
	M ₃	580*				
	M_4		= = n			
	H	46	49	51	55	64
81GH366	T	830	830*	770*	7 30*	610*
	${f E}$	330	260*	200*	180*	130*
	\mathbf{M}_{1}	100	170*	230*	300*	460*
	M_2	330	530*	9		
	M_3^2	690	æ≅ான⊓	0 C 8 =		
	M_4			• • • •		
	н	50	52	55	58	65
81GH386	${f T}$	1490	1610	1550	1470	1370
	Ē	380	300	200	110	60
	М ₁	250	370	490	1190	
	M_2	730	1080	1550	1170	
	M ₃	1170		****		
	H	65	66	70	76	85
81GH391	T	1280	1420	1300	1330	1270
	E	360	330	200	150	80
	$\mathbf{M_1}$	200	260	480	860	
	M ₂	630	780			***
	M ₃	1060	1310			
	H	65	65	67	71	78
81GH393	T	1480	1570	1490	1530	1320
	${f E}$	400	290	250	170	90
	\mathbf{M}_{1}	230	290	510	780	
	M_2	600	880	1260		
	M_3^2	1060		en en en		
	Н	65	65	69	7 0	76



			Cobalt 60	Radiatio	n Dose (Mi	<u> </u>
Compound	Property	0	10	20	40	70
<u>John John Land</u>						
81GH387	т	1450	1510	1390	1440	1260
010,1100.	E	310	250	180	140	80
	M ₁	220	380	550	830	
	M_2^1	860	1190			
	M_3^2	1360			□ → F ←	
	н	5 7	61	65	.70	7 5
01611205	T	1320	1360	1300	1250	1400
81GH395	E	310	300	200	180	120
		150	180	400	490	1150
	${f M}_1 \\ {f M}_2$	700	930	1200	- , -	
		1180	, 50 		M 5	
•	M ₃ H	55	60	61	64	70
	11	55	•			
81GH397	${f T}$	1550	1570	1610	1610	1480
61011371	E	360	310	260	200	150
	M_1	160	230	270	500	890
	M ₂	730	850	1160	6 B (1) P	₩ 43 € 57
	M ₃	1310	1520			
	H	55	60	60	62	68
		1510	1/00	1520	1530	1780
81GH420	$\frac{\mathbf{T}}{}$	1510	1690	1530 100	80	70
	E	150	110	1530		
	M ₁	730	1550	1550		
	M ₂					
	M ₃	71	75	78		
	H	71	15	10		
81GH423	${f T}$	1550	1630	1540	1560	1620
	E	160	120	110	100	80
	\mathbf{M}_1	7 50	1320	1420		~ ~ ~ ~
	M_2		a 			
	M_3^2					7.0
	н	70	74	75		



			Cobalt 60	Radiati	on Dose (M	IR)
Compound	Property	0	10	20	40	70
81GH425	Т	1460	1620	1600	1490	1/00
	Ē	150	130	110	90	1680
	$\widetilde{\mathbf{M}}_1$	680	1180	1340	90	90
	M_2		1100	1340		
	M ₃					
	H	71	7 5	75		
01677401						
81GH421	$\frac{\mathbf{T}}{-}$	1330	1350	1290	1230	1260
	E	320	250	200	140	90
	M_1	420	630	620	930	
	M_2	930	1210	1270		
	M_3	1320		17 EP ## ##		
	H	73	7 5	7 5		
81GH427	T	1160	1240	1140	1160	1180
	E	380	300	250	180	110
	M_{1}	310	440	480	670	1080
	M_2	700	900	980		
	M_3^2	1040		J = = =	0 D == =	
	м ₃ Б	70	70	71		
81GH429	T	1250	1200	1220	1140	1230
	E	310	200	180	100	80
	Μį	440	680	750	1100*	# W = H
	M_2^{1}	890				
	M ₃	1220				
	H	76	75	78		



TABLE XXXII

THE EFFECTS OF GAMMA RADIATION ON PERCENT RETAINED ELONGATION OF PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS

Method I

Compound Number	Radiation Flux (ergs/gram DC/hr.)	Dose required to reduce percent retained elongation by 50% (ergs/gram DC)	Reduction in retained elo after indica (ergs/gram	ongation ted dose
17.5			58.7×10^8	126. $\times 10^8$
81 GH311	2.33×10^{7}	2.94×10^{9}	80	92
81 GH317	2.33×10^{7}	3.86×10^9	66	90
81 GH31 8	2.33×10^{7}	2.94×10^9	68	90
01 (711212	2.60×10^{7}	6.80×10^{9}	44	72
81 GH313 81 GH327	2.60×10^{7}	14.7×10^9	32	47
81 GH328	2.60×10^{7}	7.05×10^9	46	70
81 GH357	2.52×10^{7}	1.51×10^9	94	96
81 GH359	2.52×10^{7}	2.52×10^{9}	88	92
81 GH360	2.52×10^{7}	2.85×10^9	73	93
81 GH358	2.52×10^{7}	2.18×10^{9}	80	88
81 GH365	2.52×10^{7}	4.03×10^9	65	75
81 GH366	2.52×10^{7}	4.62×10^9	57	76
81 GH386	2.52×10^{7}	1.51×10^{9}	84	96
81 GH391	2.52×10^{7}	3.44×10^{9}	72	85
81 GH 3 93	2.52×10^{7}	4.87×10^9	56	7 5
81 GH387	2.52×10^{7}	1.93×10^{9}	75	90
81 GH 39 5	2.52×10^{7}	4.87×10^{9}	57	76
81 GH397	2.52×10^7	5.46 x 10 ⁹	53	68
81 GH420	2.52×10^{7}	5.03×10^9	54	62
81 GH423	2.52×10^{7}	4.70×10^9	54	58
81 GH425	2.52×10^7	5.79×10^9	50	55
81 GH421	2.52×10^{7}	3.70×10^{9}	64	85
81GH427	2.52×10^{7}	3.61×10^{9}	68	86
81 GH429	2.52×10^{7}	2.44×10^9	80	88
	To	otal 10.3 x 10 ¹⁰	1560	1905
	Avera	age $43 \times 10^8/\text{CPD}$	65.0	79.3

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TABLE XXXIII

THE EFFECTS OF GAMMA RADIATION ON PERCENT RETAINED ELONGATION OF PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS

Method II

Compound Number	Radiation Flux (ergs/gram DC/h	r.) r	ose required to educe percent etained elongation y 50% (ergs/gram DC)	Reduction in percent retained elongation after 58.7 x 10 ⁸ ergs/ gram DC
81GH311	2.33×10^{7}		3.02×10^{9}	60
81 GH317	2.33×10^{7}		2.44×10^9	70
81 GH318	$\frac{2.33 \times 10^{7}}{2.33 \times 10^{7}}$		1.26×10^9	87
	-			
81 GH 31 3	2.33×10^{7}		5.87×10^9	48
81GH327	2.33×10^{7}		$5.87 \times 10^9 \ (-\Delta^4)$	14%) 44
81 GH 328	2.33×10^{7}		4.62×10^9	52
81 GH 357	2.52×10^{7}	1	.13 - 1.17 x 10 ⁹	00 00
81 GH359	2.52×10^{7}			90-90
•	_		$51 - 1.63 \times 10^9$	88-88
81 GH360	2.52×10^7	2,	$43 - 2.35 \times 10^9$	86-82
81GH358	2.52×10^{7}	2.	$06 - 1.80 \times 10^9$	74-73
81 GH365	2.52×10^{7}		18 - 1.89 x 10 ⁹	72-69
81 GH366	2.52×10^{7}		$48 - 2.81 \times 10^9$	68-64
01 011500	2.52 x 10	2.	40 * 2,01 X 10	00-04
81GH386	2.52×10^{7}		2.02×10^9	78
81 GH 391	2.52×10^{7}		2.27×10^9	77
81 GH393	2.52×10^{7}		3.28×10^9	66
	7			
81 GH387	2.52×10^{7}		2.27×10^9	68
81 GH395	2.52×10^{7}		3.61 x 10 ⁹	62
81GH397	2.52×10^{7}		4.78×10^9	57
	7		. 0	
81 GH420	2.52×10^{7}		3.36×10^9	55
81 GH423	2.52×10^{7}		5.87×10^9	50
81 GH425	2.52×10^{7}		5.87 x 10 ⁹ (-Δ4	!7%) 47
01 (11/2)	2.52×10^{7}		2.68×10^{9}	72
81 GH421 81 GH427	2.52×10^{7} 2.52×10^{7}		4.28×10^{9}	64
	$\frac{2.52 \times 10^{4}}{2.52 \times 10^{7}}$		2.68×10^{9}	72
81 GH429	2,52 X 10		4.00 X 10'	1 6
		Total	7.77×10^{10}	1607
		Average	$32.4 \times 10^8 / \text{CPD}$	67.0



TABLE XXXIV

THE EFFECTS OF GAMMA RADIATION ON PERCENT RETAINED ELONGATION OF PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS

Method III

Compound Number	Radiation Flux (ergs/gram DC/hr.)	Dose required to reduce percent retained elongation by 50% (ergs/gram DC)	Reduction in percent retained elongation after 58.7 x 10 ⁸ ergs/ gram DC
81 GH311	2.33×10^{7}	1.76×10^{9}	84
81 GH317	2.33×10^{7}	1.93×10^{9}	74
81 GH318	2.33×10^{7}	1.01×10^9	84
81 GH313	2.33×10^{7}	4.03×10^9	54
81 GH 327	2.33×10^{7}	4.20×10^9 (- $\triangle 4$	3%) 43
81 GH 328	2.33×10^{7}	4.87×10^9	54
81.GH357	2.52×10^{7}	1.68×10^{9}	82
81GH359	2.52×10^{7}	2.10×10^9	82
81 GH360	2.52×10^{7}	3.19×10^9	66
01/711250	2.52×10^{7}	2.94×10^{9}	65
81 GH358 81 GH365	2.52×10^{7}	4.87×10^9	53
81 GH 366	2.52×10^{7}	3.44×10^9	60
81 GH 300	2, 32 x 10	J. 44 X 10	00
81GH386	2.52×10^{7}	1.76×10^{9}	84
81GH391	2.52×10^{7}	2.27×10^{9}	78
81 GH393	2.52×10^7	2.35×10^9	78
81 GH387	2.52×10^{7}	2.44×10^{9}	7 5
81 GH395	2.52×10^{7}	3.36×10^9	61
81 GH397	2.52×10^7	3.95×10^9	58
81 GH420	2.52×10^{7}	4.87×10^9	53
81 GH423	2.52×10^{7}	5.87×10^9 (- $\triangle 4$	
81 GH425	2.52×10^{7}	$5.87 \times 10^9 (-\Delta 4)$	• •
	7	Q	
81 GH421	2.52×10^{7}	2.52×10^9	71
81 GH427	2.52×10^{7}	3.02×10^9	61
81 GH429	2.52×10^7	1.68×10^9	74
	Tota	7.63 \times 10 ¹⁰	1582
	Averag	ge 31.8 x 10 ⁸	65.8



TABLE XXXV

PROTECTION RATIOS FOR PRACTICAL AIRCRAFT COMPOUNDS WITH AND WITHOUT POTENTIAL ANTI-RADS PRESENT

Compound	Potential Anti-R	ad	Protection Ra	t10
			Method	
		<u>I</u>	II	III
81GH311	None (Control)	1.0	1.0	1.0
81GH31 7	Akroflex C	1.3	0.8	1.1
81GH318	Quinhydrone	1.0	0.4	0.6
81GH313	None (Control)	1.0	1.0	1.0
81GH327	Akroflex C	2.2	1.0	1.0
81GH328	Quinhydrone	1.0	0.8	1.2
81GH357	None (Control)	1.0	1.0	1.0
81GH359	Akroflex C	1.7	1.2	1.3
81GH360	Quinhydrone	1.9	1.9	1.9
81GH358	None (Control)	1.0	1.0	1.0
81GH365	Akroflex C	1.8	1.1	1.7
81GH366	Quinhydrone	2.1	1.2	1,2
81GH386	None (Control)	1.0	1.0	1.0
81GH391	Akroflex C	2.3	1.1	1.3
81GH393	Quinhydrone	3.2	1.6	1.3
81GH387	None (Control)	1.0	1.0	1.0
81GH395	Akroflex C	2.5	1.6	1.4
81GH39 7	Quinhydrone	2.8	2.1	1.6
81GH420	None (Control)	1.0	1.0	1.0
81GH423	Akroflex C	0.9	1.8	1.2
81GH4 2 5	Quinhydrone	1.2	1.8	1.2
81GH421	None (Control)	1.0	1.0	1.0
81GH427	Akroflex C	1.0	1.6	1.2
81GH429	Quinhydrone	0.7	1.0	0.7
		Totals 35.6	29.0	27.9



GENERAL PROCEDURE FOR EVALUATION OF DOSIMETER RESULTS

Dosimetry Run	In Tire Surface Location							
Number	1	2	3	4	4'	5	6	7
1	0.173	0.185	0.185	0.160	0.126	0.105	0.125	0.133
2	0.179	0.166	0.182	0.153	0.120	0.107	0.123	0.128
Average	0.176	0.186	0.184	0.156	0.123	0.106	0.124	0.130
Deviation	(±0.003)	(±.0005)	(±.0015)	(±.0035)	(±0.003)	(±0.001)	(±0.001)	(±.0025)
Deviation (%)	1.7%	0.3%	0.8%	2.2%	2.4%	0.9%	0.8%	1.9%

Average	Effective Flux	(MR/hr) with	Reversal
0.176	0.186	0.184	0.156
0.133	0.24	0.106	0.123
(0.309)	(0.310)	(0.290)	(0.279)
0.154	0.155	0.145	0.140
(Bead)	(Sidewall)	(Shoulder)	(Tread)

The average overall flux, (0.154+0.155+0.145+0.140)/4, is thus 0.148 ± 0.006 megaroentgens/hour, showing a 4.1% average deviation. Since the limits of experimental error in this study must exceed 4.1%, it is satisfactory to state that all parts of the tire will be exposed to a gamma radiation flux of 0.148 MR/hr as of August 8, 1957. (One megaroentgen is equivalent to 83.9×10^6 ergs per gram of carbon.)



TABLE XXXVII

RADIATION EXPOSURE DATA FOR THE FIRST IRRADIATED TIRE

A. Identification of Tire:

N80 - 5289 40813 BA02 - 2 - 57 A-15E C3593-3 E11695-3

Type VII, $26^{\circ\prime} \times 6.6^{\circ\prime}$, 14 ply rating, tubeless with nylon cord

B. Average Effective Flux with Reversal (MR/hr):

Time	Bead	Sidewall	Shoulder	\underline{Tread}
Before	0.154	0.155	0.145	0.140
After	0.102	0.108	0.100	0.100

C. Equations Used to Calculate Exposure Times:

Flux =
$$8.09 \times 10^7 t^{-1.21}$$
 r/hr

Dose =
$$1.94 \times 10^9 \int_{t_1}^{t_2} t^{-1.21} dt$$

E. General Irradiation History:

Date	Action				
8-8-57	Pre-insertion ceric sulfate dosimetry completed				
8-13-57	Irradiation of first tire started				
8-29-57	Canister assembly removed and turned over				
8-30-57	Assembly reinserted for second half of exposure				
9-16-57	Assembly removed and irradiation ended; total irradiation time: 781.8 hours				



TABLE XXXVIII

RADIATION EXPOSURE DATA FOR THE SECOND IRRADIATED TIRE

A. Identification of Tire:

N80 - 5289 40812 BA02-2 - 57 A-15E C3593-4 E11695 - 3

Type VII, $26" \times 6.6"$, 14 ply rating, tubeless with nylon cord

B. Average Effective Flux with Reversal (MR/hr):

Time	Bead	Sidewall	Shoulder	$\underline{\mathtt{Tread}}$
Before	0.162	0.165	0.154	0.152
After	0.101	0.103	0.104	0.096

C. Equations Used to Calculate Exposure Times:

Flux = 1.30 x 19⁹ t^{-1.73} r/hr
Dose = 3.12 x
$$10^{10} \int_{t_i}^{t_k} t^{-1.73} dt$$
 r

D. Total Integrated Exposure: 9.37 x 10⁷ r

E. General Irradiation History:

Date	Action
9-30-57	Pre-insertion ceric sulfate dosimetry completed
10-4-57	Irradiation of second tire started
10-19-57	Canister assembly removed and turned over
10-25-57	Assembly reinserted for second half of exposure
11-12-57	Assembly removed and irradiation ended; total irradiation time: 752.0 hours



TABLE XXXIX

RADIATION EXPOSURE DATA FOR THE THIRD IRRADIATED TIRE

A. Identification of Tire:

N80 - 5290 653948A02 - 3 - 57 A-15E C3593 - 3 E11695 - 3

Type VII (plus anti-rad), $26^{11} \times 6.6^{11}$, 14 ply rating, tubeless, nylon cord

B. Average Effective Flux with Reversal (MR/hr):

Time	Bead	Sidewall	Shoulder	Tread
Before	0.096	0.098	0.099	0.091
After	0.058	0.059	0.057	0.054

C. Equations Used to Calculate Exposure Times:

Flux =
$$1.037 \times 10^{10} t^{-2.13} r/hr$$

Dose = 2.482 x
$$10^{11} \int_{t_1}^{t_2} t^{-2.13} dt$$
 r

D. Total Integrated Exposure: 1.09×10^8 r

E. General Irradiation History:

Date	Action
11-12-57	Pre-insertion ceric sulfate dosimetry completed
11-13-57 to 11-17-57	Loaded rubber test specimens, cis-PBN dosimeters, and pressure-tested canister
11-18-57	Irradiation of third tire started
12-14-57	Canister assembly removed and turned over
12-17-57	Assembly reinserted for second half of exposure
1 - 21 - 58	Assembly removed and irradiation ended; total irradiation time: 1464 hours
1-23-58	Final ceric sulfate dosimetry completed



TABLE XL

DYNAMOMETER LANDING TEST RESULTS FOR THE AIRCRAFT TIRES

High Speed Landings

Tire	Number of Landings	Total Revolutions	Miles of Roll	Temperature, ^o F. before/after
Standard		m = = :		059/2109 m 1
Control #1 Antirad	25	8528	35.5	95 ⁰ /210 ⁰ Tread
Control #1	25	8500	35.4	95 ⁰ /210 ⁰ Tread
Irradiated				0 -
Standard #	1 18	6255	26.	85 ⁰ /120 ⁰ Contained Air
Standard				0=0 (0) =0 == 1
Control #2	25	8827	37.	97 ⁰ /215 ⁰ Tread
Irradiated	2 25			
Standard #	2 25			

Low Speed Landings

Tire	Number of Landings	Total Revolutions	Miles of Roll	Temperature, °F. before/after
Standard				
Control #1	76	36, 395	119	95/160 Contained Air
Antirad				
Control #1	204	97,700	282	95/215 Tread
Standard				
Control #2	147	67, 412	221	95/165 Contained Air
Irradiated				
Standard #2	1			·

Cause of Failure

Tire Standard Control #1	Remarks Failed on the 76th landing with a ply separation in the shoulder. This tire would not qualify under Air Force specifications.			
Antirad Control #1	Testing stopped after two blisters showed severe ply separation.			
Irradiated Standard #1	Failed during the 18th high speed landing with a bead to bead blowout and a small break in the side wall.			
Standard Control #2	Testing stopped after a slight blister on the shoulder and severe bead chafing.			

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TABLE XLI
STRESS-STRAIN DATA FOR IRRADIATED COMPOUNDS OF FIRST TIRE

Compound	Physical								
Number	Property	<u>C</u>		2	3	4	5	<u>6</u>	_7
	TS(PSI)	3360	2020	2260	2120	2880	1940	3000	2000
86TC103	UE (%)	480	190	240			180	340	200
	100 % Mod.		780	760			800	500	೬00
	TS (PSI)	3380	2780	2360	1580	1780	2760	2800	2560
678 PK 196	UE (%)	550	34 0	260	160	. 220	350	450	340
	100 % Mod.	220	440	640	720	580	600	360	520
	TS (PSI)	3820	2760	2440			2760	2560	2140
881 PK 59	UE (%)	560	450	270			400	360	200
	100 % Mod.	200	320	600	640	500	380	400	800
	TS (PSI)	2400	1380	1720	1180	_	1840	1940	1740
693PK 20	UE (%)	190	80	110	60	100	130	110	110
	100 % Mod.	960		1500	_	1740	1340	1580	1280
	TS(PSI)	1900	2120	2400	2480	2260	1800	2440	2040
3 00SX4 1	UE (%)	120	80	80	90	100	70	70	80
	100 % Mod.	1560		_	_	2260	_	_	_
	TE (DOI)	100 0	(0.0	′ = 0	(2 .	S= (
	TS (POI)	100.0	60.0		63.1	85.6		89.2	59.5
ob TC103	UE (POI)	100.0	39.6	50.0	50.0	66.6	37.5	70.9	41.6
	100 % Mod.		390	360	280	280	400	250	400
0.70 DIC10/	TS (POI)	100.0	82.1	69.9		52.6	81. ó		75.6
878 PK 196	UE (POI)	100.0	61.9	47.3	29.1	40.0	63.6	-	61.9
	100 % Mod.		200	291	328	2ó 4	273	164	236
881 PK 59	TS (POI)	100.0	72.3	63.9	69.6	79.1		67.0	56.0
	UE (POI)	100.0	80.4	48.2	51.8	71.4		64.4	35.7
	100 % Mod.	100.0	160	300	320	250	190	200	400
COOPTION	TS (POI)	100.0	57.5		49.1	72.5		80.9	72.5
893PK20	UE (POI)	100.0	42.1	57.9	31.6	52.6	68.5	57.9	57.9
	100 % Mod.	100.0	_	156	_	181	140	164	133
	TS(POI)	100.0	111.7	126.2			94.6		107.3
300 SX 41	UE (POI)	100.0	66.7	66.7	75.0		58.4	58.4	66.7
	100 % Mod.	100.0	_		_	145	_		_

POI is the abbreviation for percent of initial property value.



TABLE XLI (Continued)

STRESS-STRAIN DATA FOR IRRADIATED COMPOUNDS OF FIRST TIRE

Compound Number	TS (POI)	rage Physical Property UE (POI)	Value 100% Mod (POI)		
86TC 103	68.9 <u>+</u> 10.5	50.9 <u>+</u> 10.2	340 <u>+</u> 60		
878PK 196	70.2 <u>+</u> 12.0	55.1 <u>+</u> 14.0	251 <u>+</u> 44		
881 PK 59	68.6 <u>+</u> 5.4	60.5 <u>+</u> 13.1	260 <u>+</u> 69		
89 3PK 20	68.7 <u>+</u> 8.8	52.6 <u>+</u> 9.0	155 <u>+</u> 15		
300SX 41	116.8 <u>+</u> 10.5	67.9 <u>+</u> 6.4	145 (One Value)		
Average of First Four Compounds*	69.1 <u>+</u> 0.5	54.8 <u>+</u> 3.0	251 <u>+</u> 48		

^{*} The data for compound 300SX 41 are omitted, because it appeared to undergo cure during irradiation (cf. TS values).

Uniformity of Dose - Matrix Examination

Compound	Location in Canister						
Number	1	2	3	4	5	6	7
86TC 103	3	5	4	6	1	7	2
878 PK 196	6	3	1	• 2	5	7	4
881 PK 59	6	2	4	7	5	3	1
893PK 20	2	3	1	4	6	7	5
300SX 41	3			4	_1	_6	
Average:	4	, 3, 6	3.4	4.6	3.6	6	2.8

^{*} The numbers in the columns are simply numerical ratings of the percent of initial tensile strength retained after 10^8 r irradiation in ordering of increasing value from 1 through 7.

POI is the abbreviation for percent of initial property value.

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APPENDIX IV

ILLUSTRATIONS

The illustrations for this report are all collected in this appendix for ease of reference. The explanations appear above in the text of the report. The illustrations are listed in the index and include graphs, sketches, and photographs which were obtained during the course of this research.



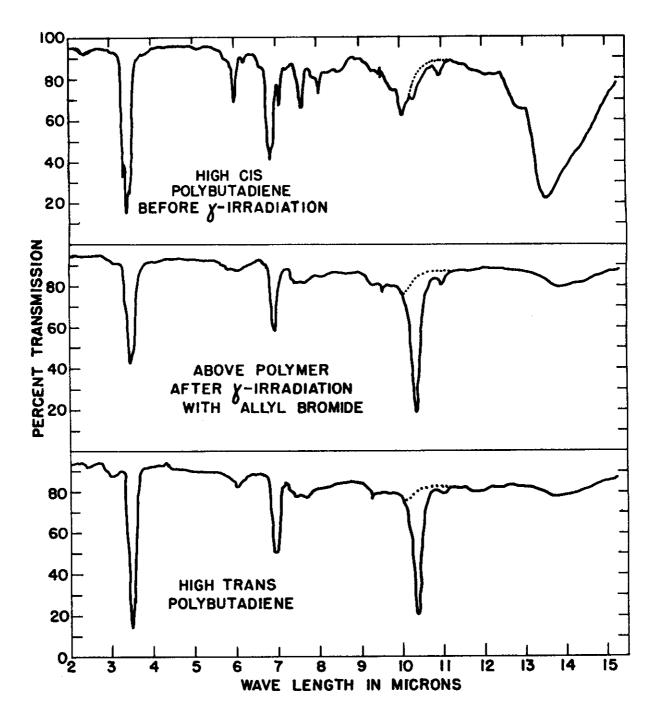


Figure 1. Infrared Absorption Spectra of Cis-Polybutadiene with Allyl Bromide Sensitizer Before and After Irradiation Compared with the Spectrum of a High Trans Polybutadiene Polymerized Directly.



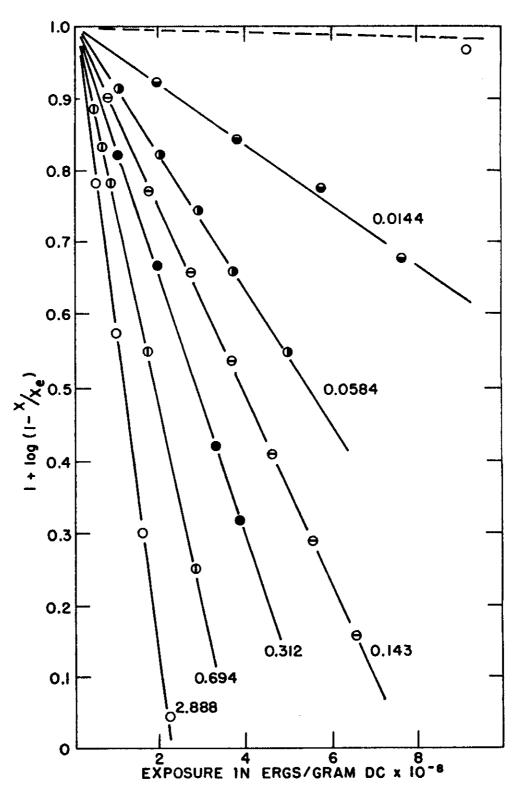


Figure 2. Isomerization Rate of Sensitized Polybutadiene

Concentrations of allyl bromide are given in moles per liter. The dotted line represents the unsensitized isomerization in solution.



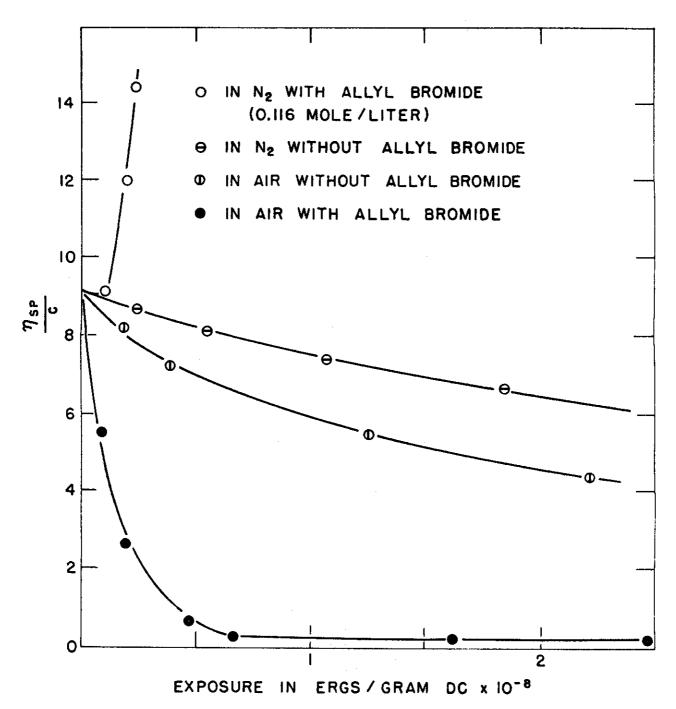


Figure 3. Change in Reduced Viscosity of a Benzene Solution of Polybutadiene at 20°C. Induced by Gamma Radiation.

Initially, the concentration of polymer was 5.30 grams per liter, and the intrinsic viscosity was 3.2.

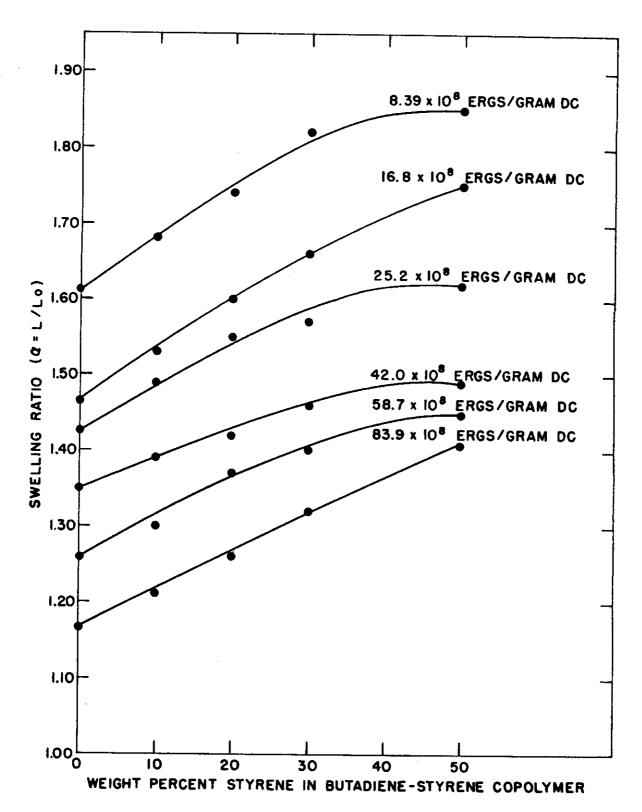


Figure 4. Effect of Styrene Content of SBR on Radiation-Induced Crosslinking.



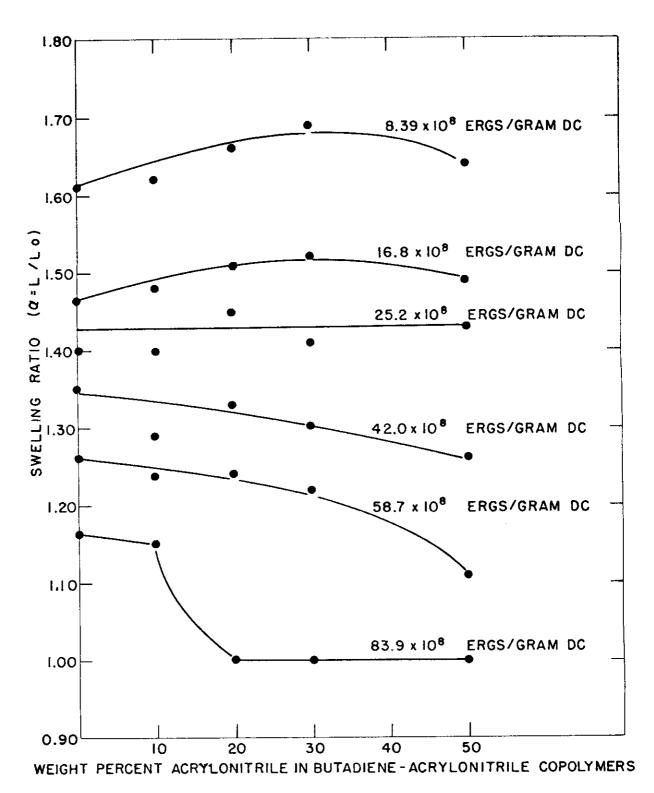


Figure 5. Effect of Acrylonitrile Content of NBR on Radiation-Induced Crosslinking.

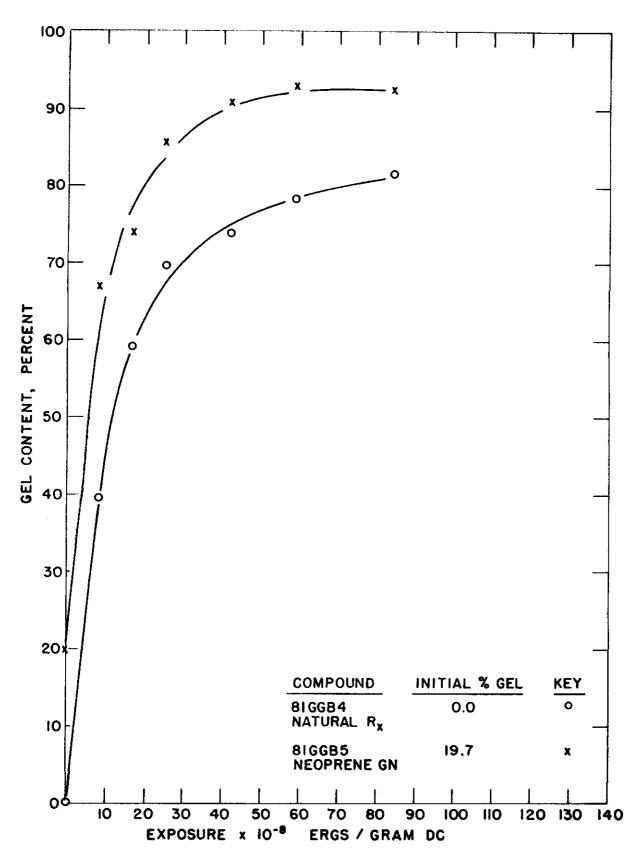


Figure 6. Gamma Radiation-Induced Gelation of the Pure Polymers. I



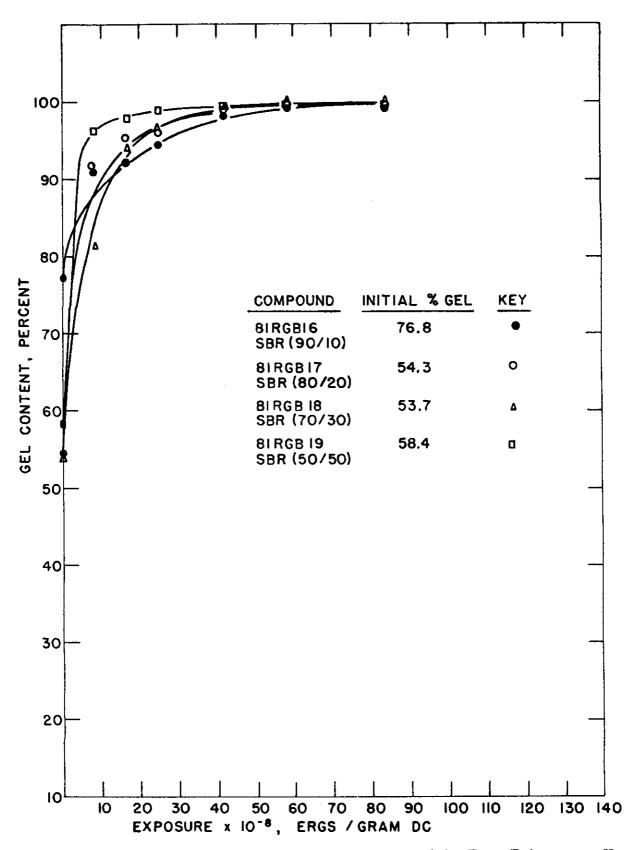


Figure 7. Gamma Radiation-Induced Gelation of the Pure Polymers. II.

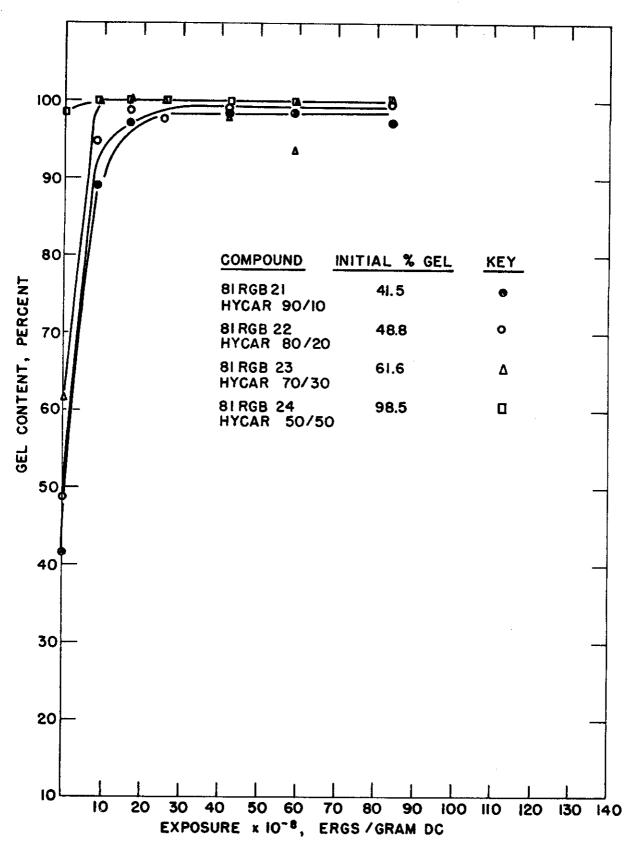


Figure 8. Gamma Radiation-Induced Gelation of the Pure Polymers. III

Contrails

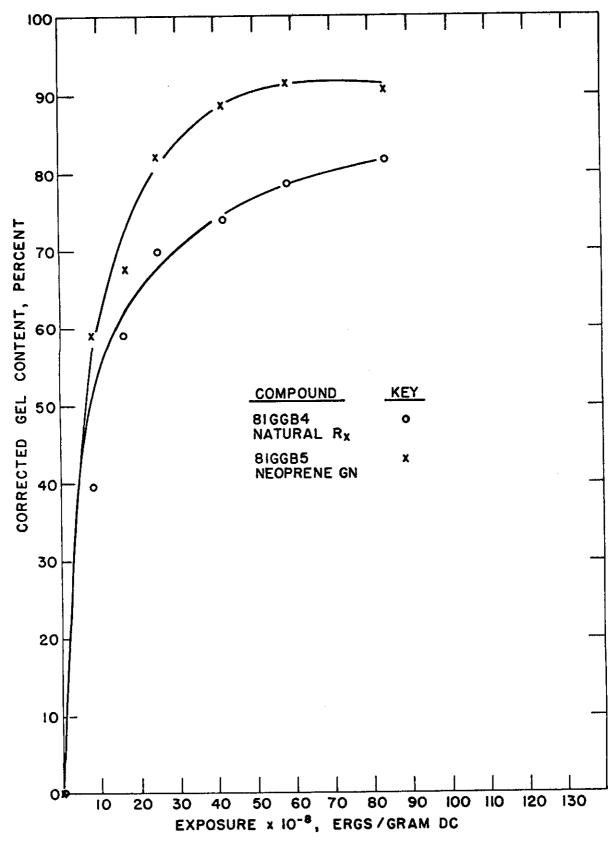


Figure 9. Corrected Gel Contents of Irradiated Pure Polymers . I
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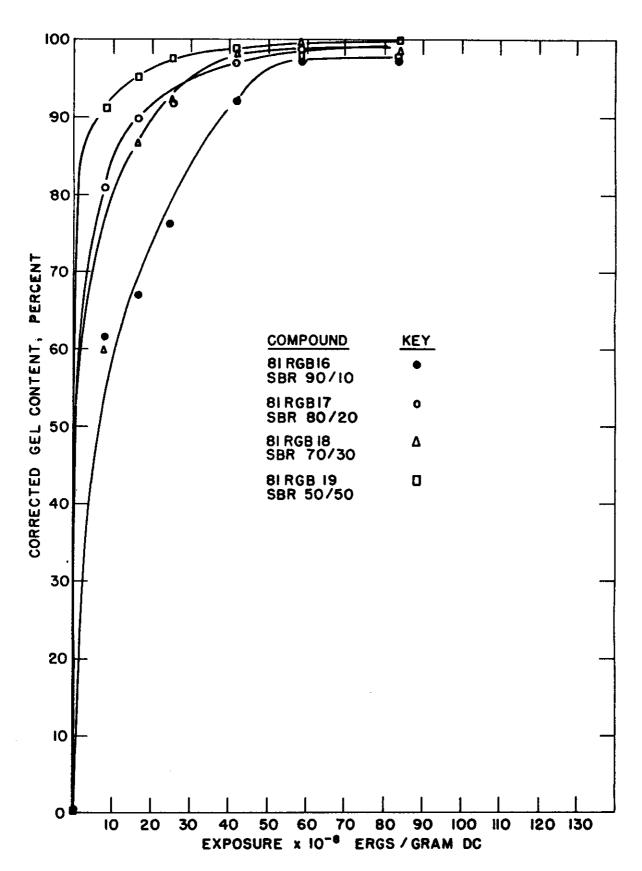


Figure 10. Corrected Gel Contents of Irradiated Pure Polymers. II

Contrails

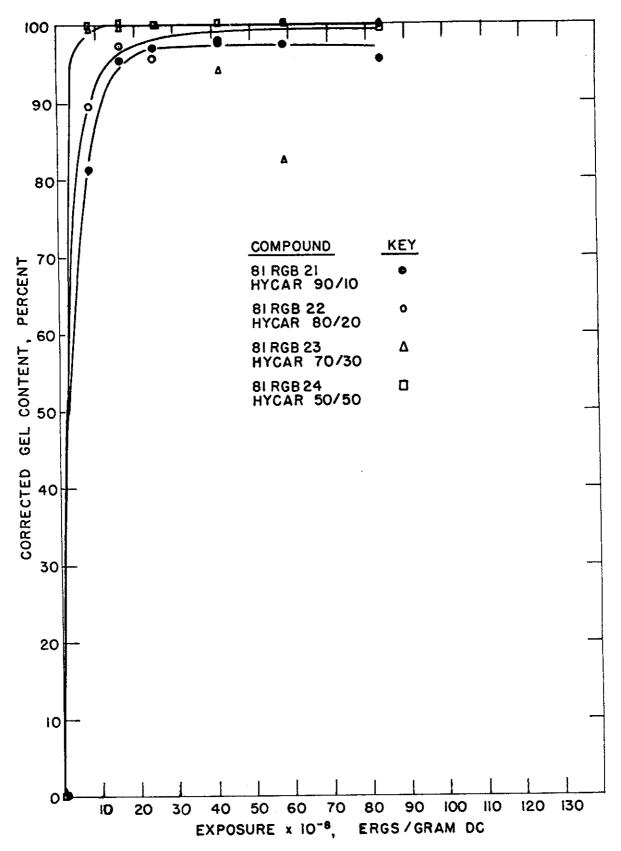


Figure 11. Corrected Gel Contents of Irradiated Pure Polymers. III

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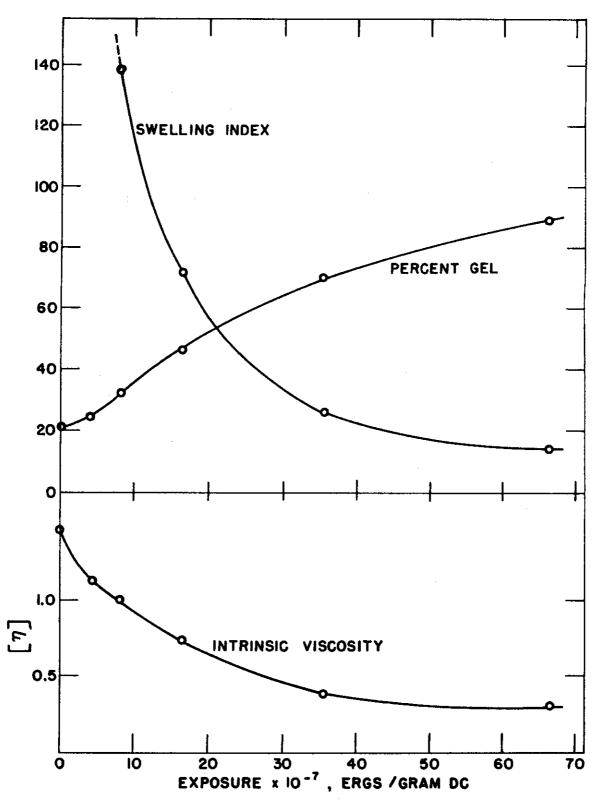


Figure 12. Radiation-Induced Changes in Butadiene-Acrylonitile Copolymers.



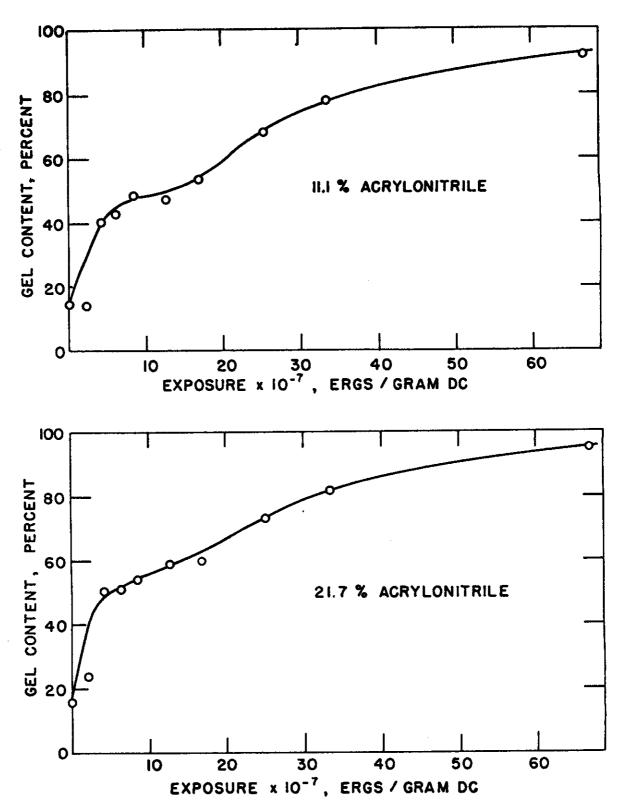


Figure 13. Gelation of Butadiene-Acrylonitrile Copolymers by $\sqrt[4]{}$ - Radiation.

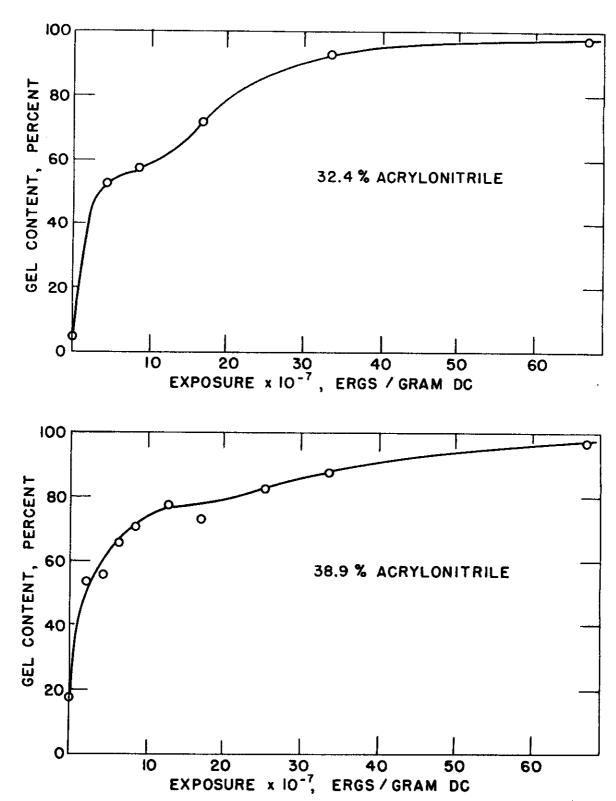


Figure 14. Gelation of Butadiene-Acrylonitrile Copolymers by $\sqrt{-}$ Radiation.



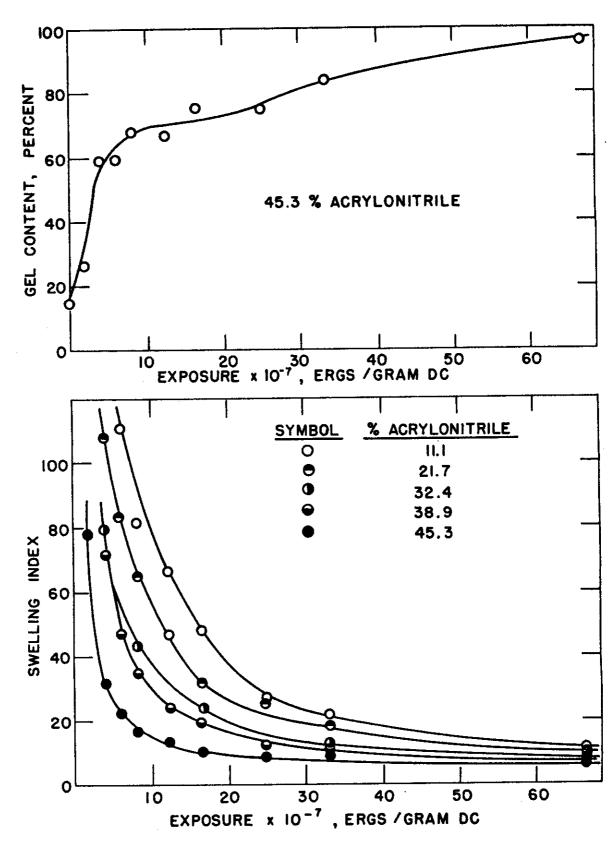


Figure 15. Gelation of Butadiene-Acrylonitrile Copolymers; Changes in Swelling Index with Radiation Exposure.



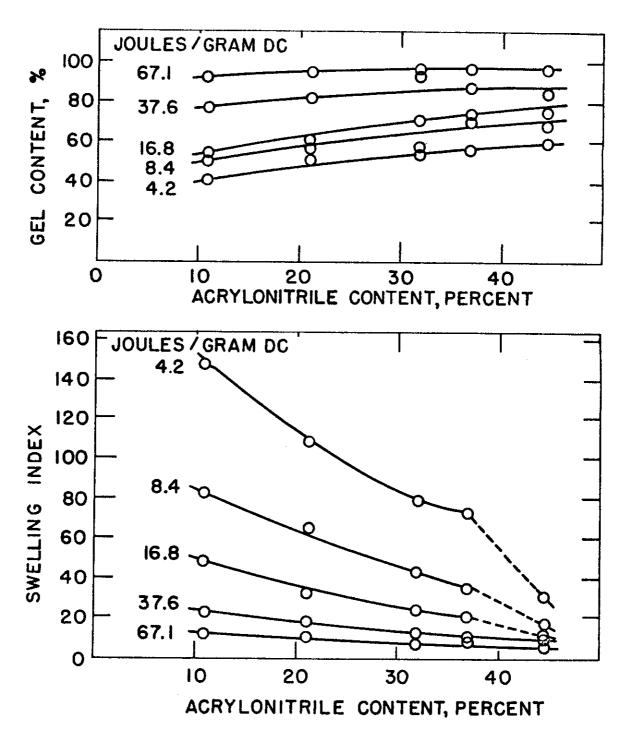


Figure 16. Effect of Acrylonitrile Content on Radiation-Induced Changes in Percent Gel and Swelling Index.

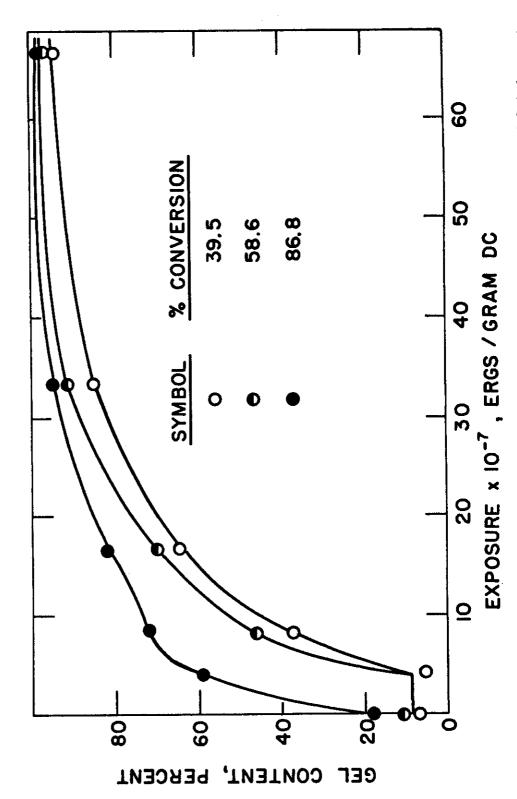
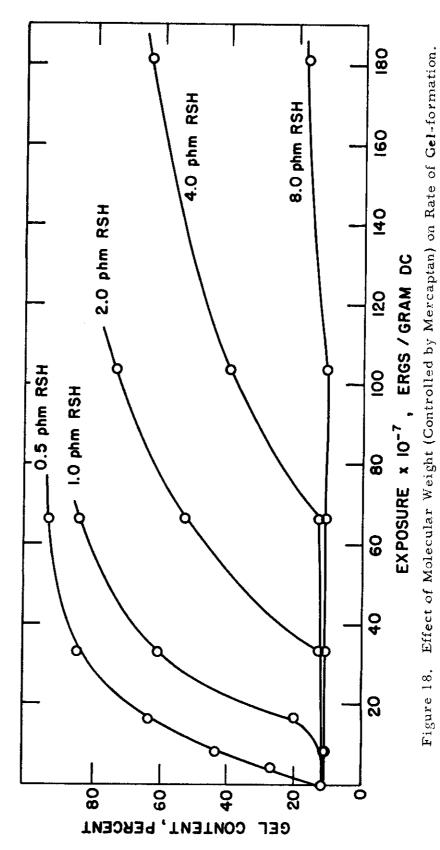


Figure 17. Effect of Molecular Weight (controlled by conversion) on Rate of Gel-formation.



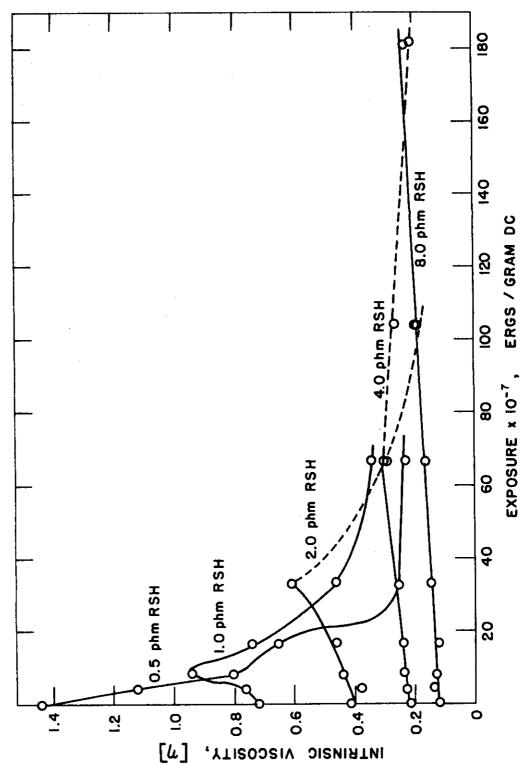


Figure 19. Variation of Intrinsic Viscosity with Radiation Dose for Various Initial Molecular Weights (Controlled by Mercaptan),



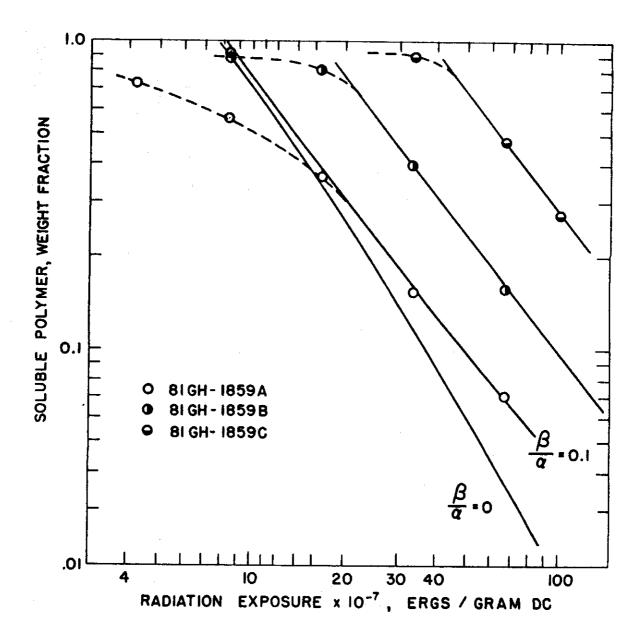
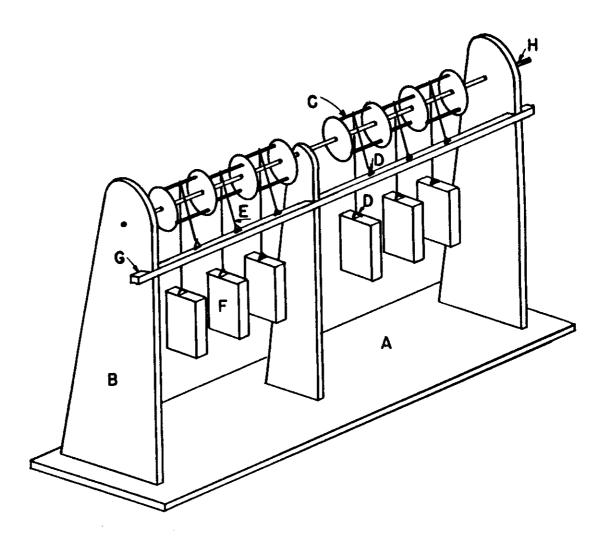


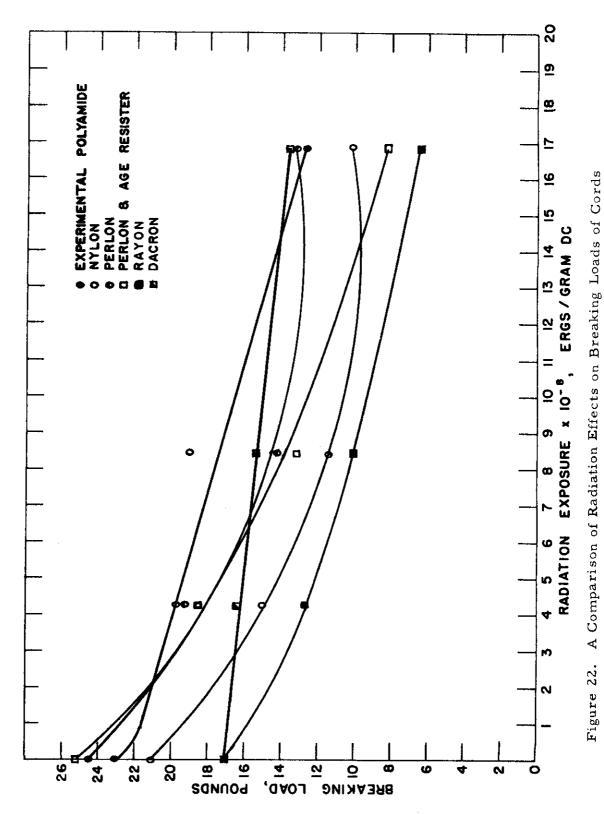
Figure 20. Decrease in Soluble Fraction with Radiation Exposure; Comparison of Data with Theoretical Slopes.

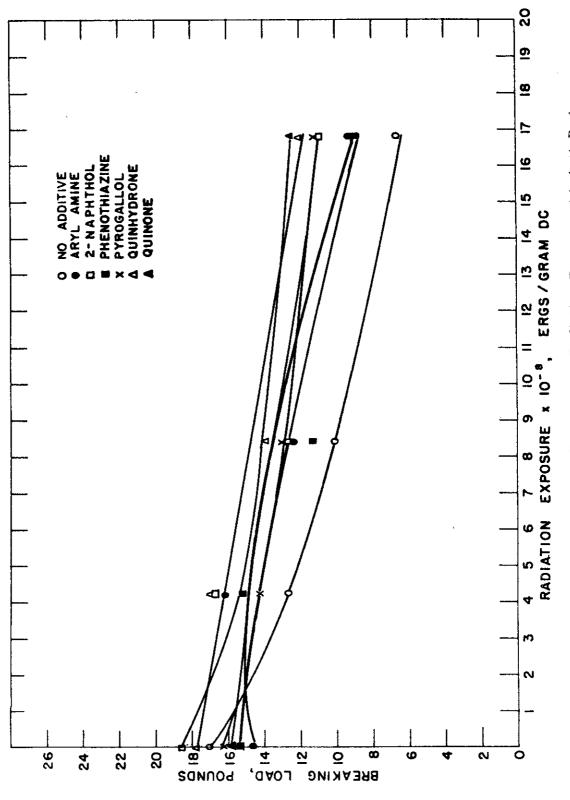




- A. STATIONARY BASE
- B. RIGID SUPPORTS
- C. ROTATING FRAME ROD
- D. CORD SAMPLE CLAMP H. MOTOR DRIVEN ROD
- E. TEXTILE CORD SAMPLE
 - F. WEIGHTS
- G. REMOVABLE BAR

Figure 21. Cord Flex - Testing Apparatus





Protection of Rayon Cords From Radiation Damage with Anti-Rads Figure 23.

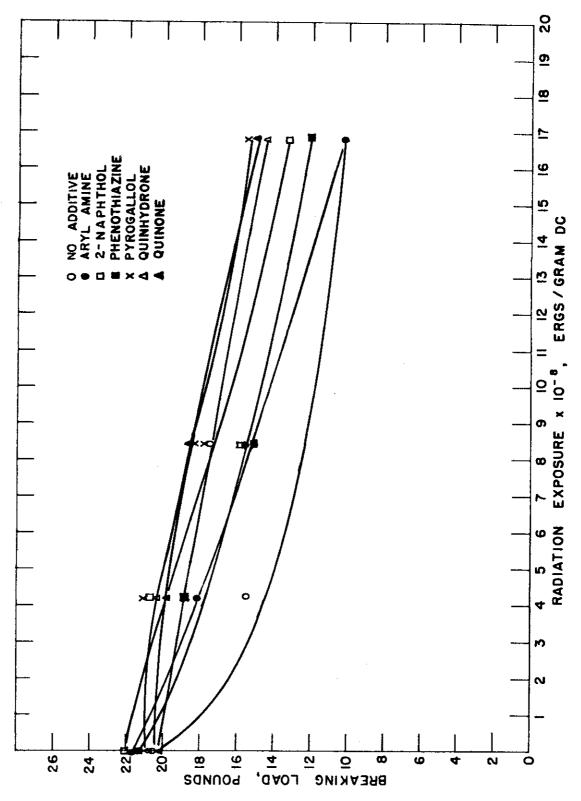


Figure 24. Protection of Nylon Cords from Radiation Damage with Anti-Rads

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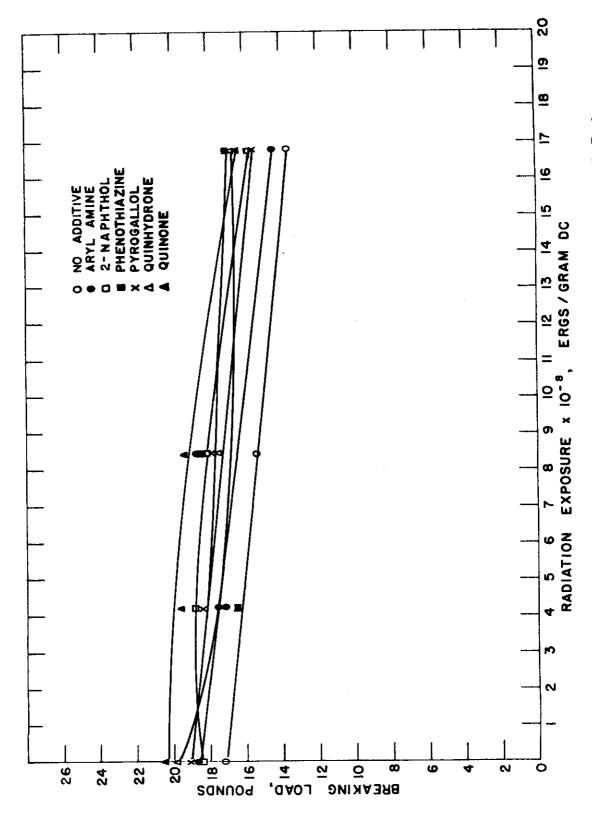
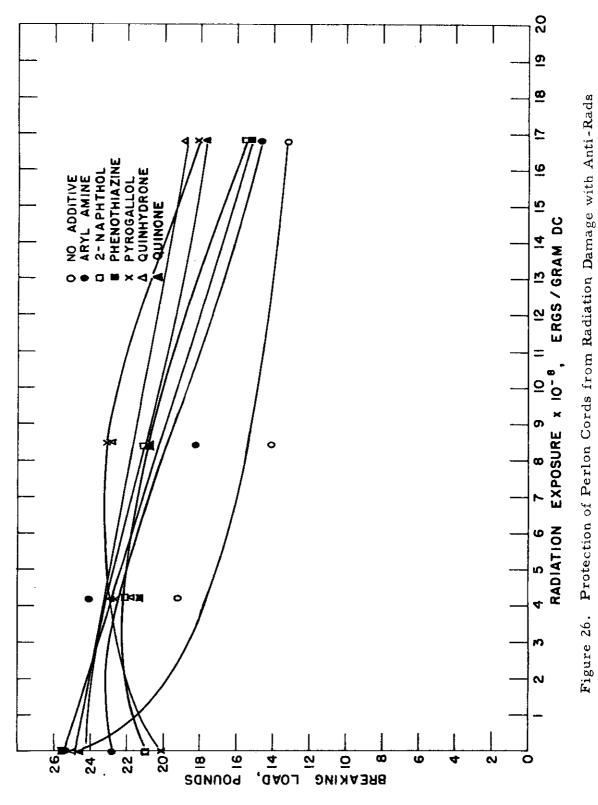


Figure 25. Protection of Dacron Cords from Radiation Damage with Anti-Rads



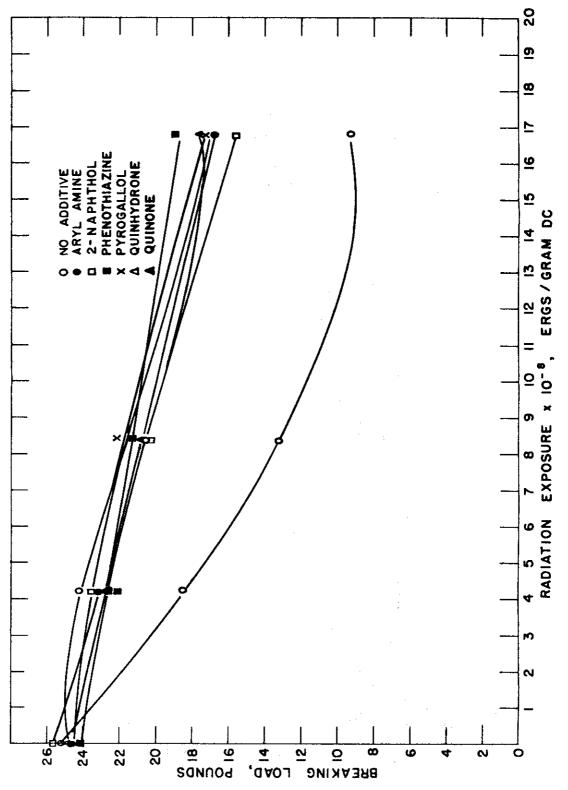
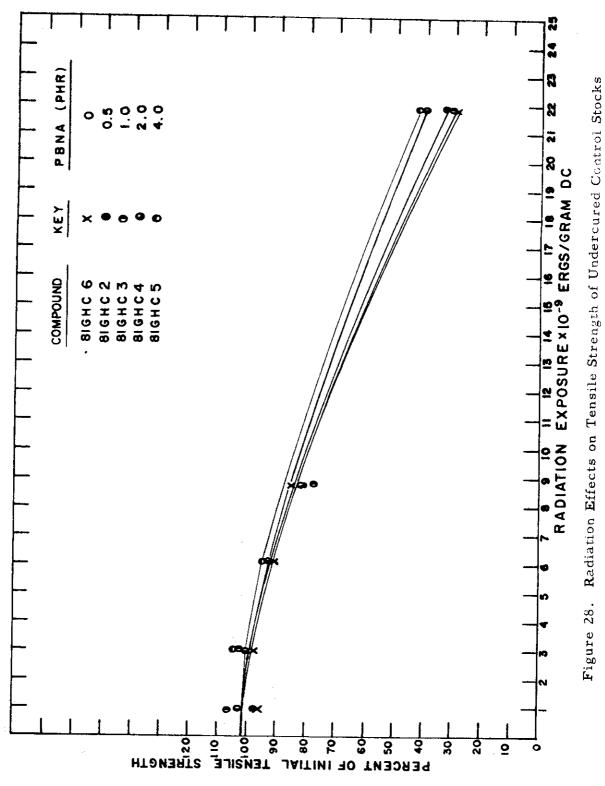


Figure 27. Protection of Perlon-AR Cords From Radiation Damage with Anti-Rads

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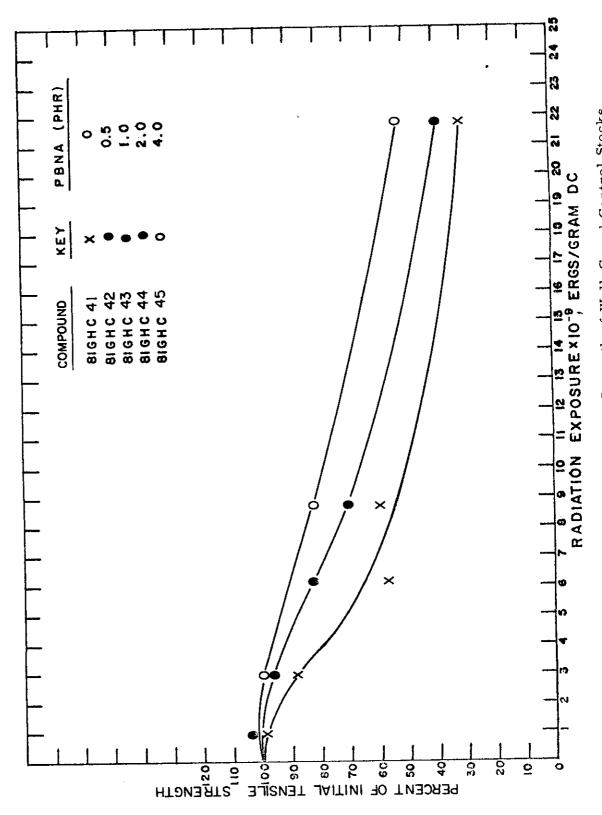
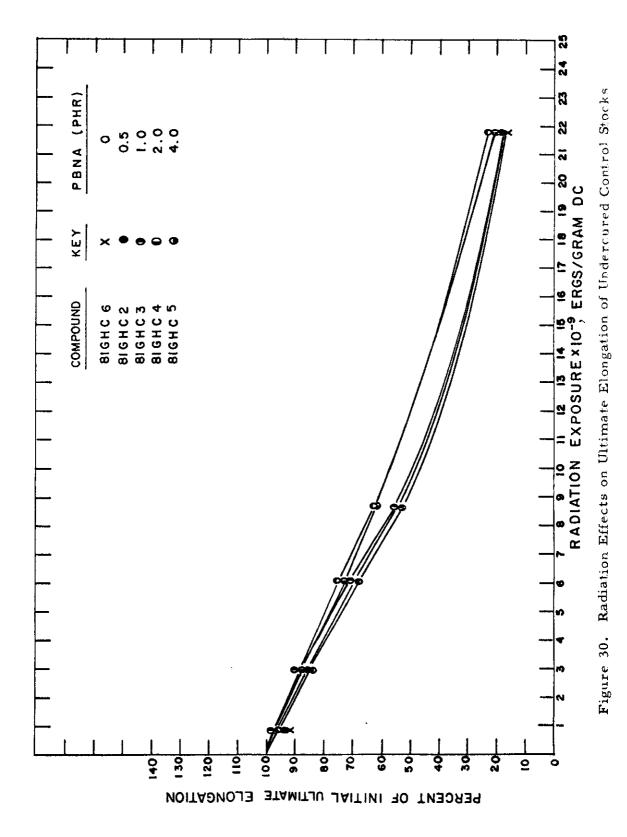


Figure 29. Radiation Effects on Tensile Strength of Well-Cured Control Stocks

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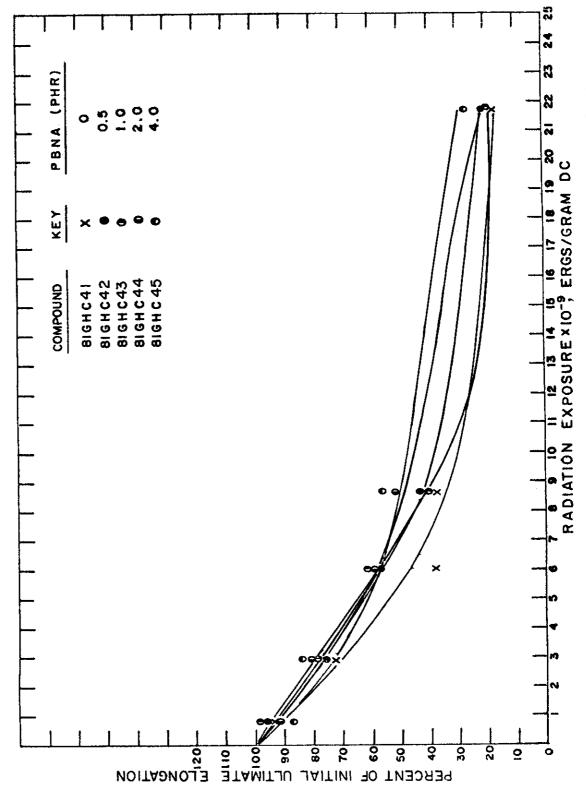
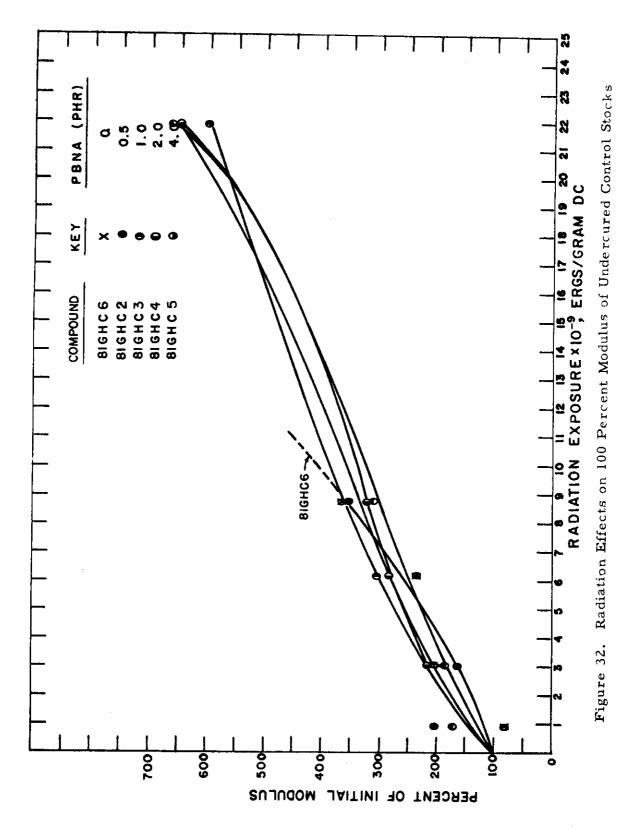
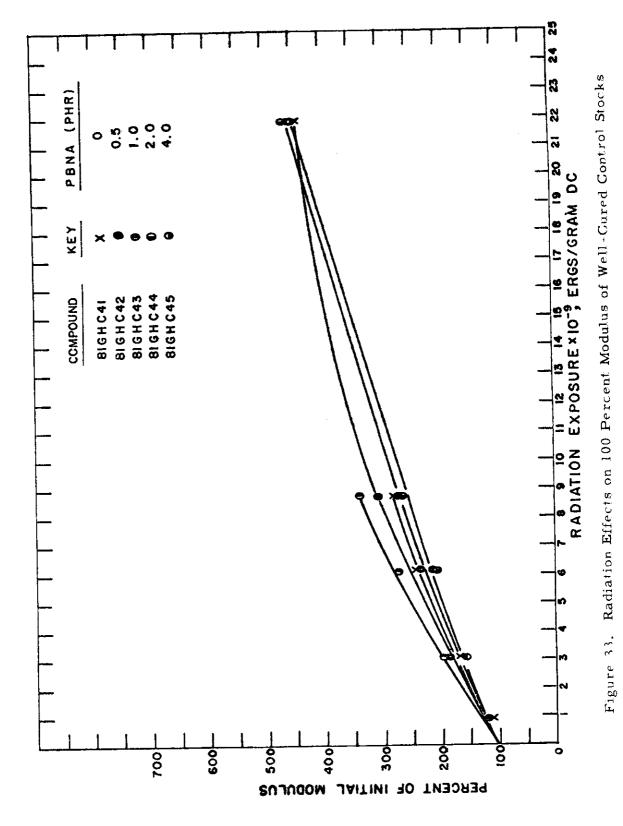
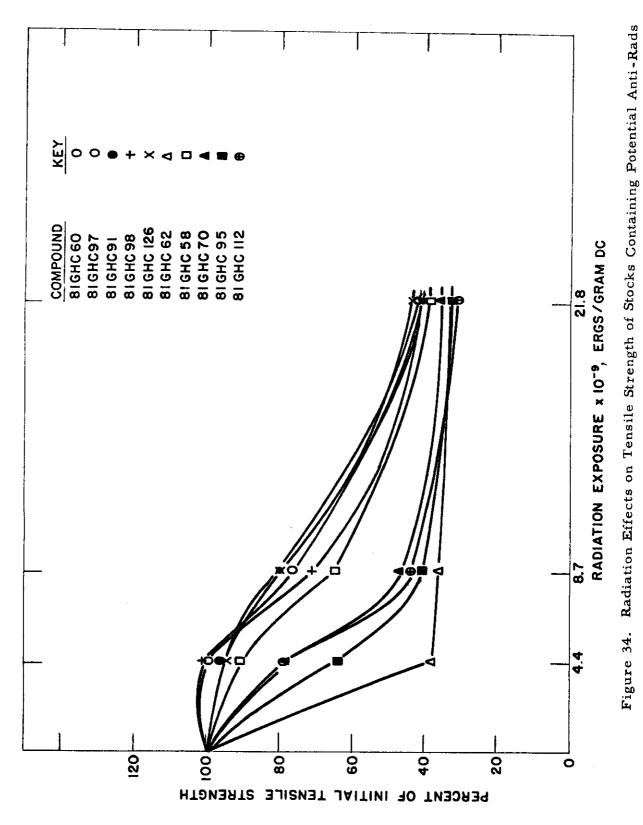


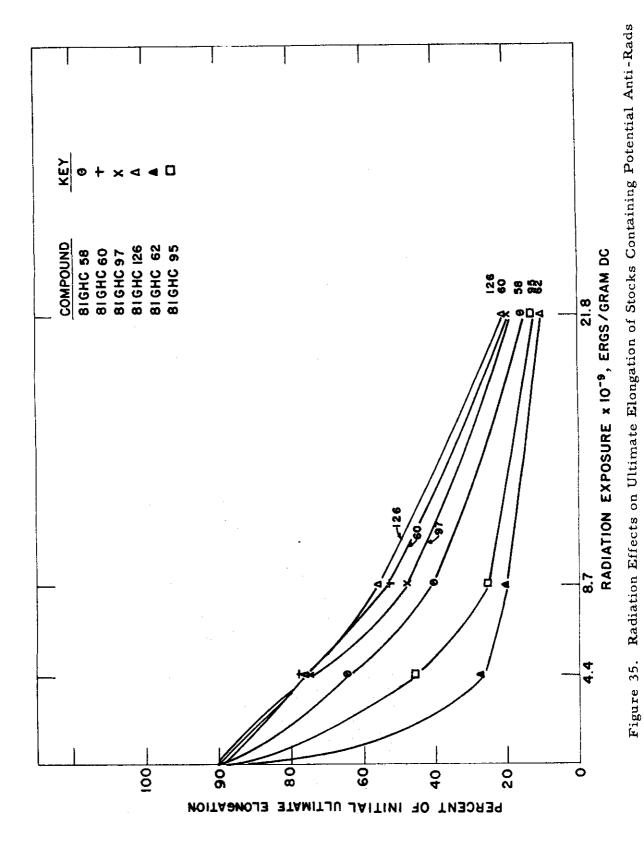
Figure 31. Radiation Effects on Ultimate Elongation of Well-Cured Control Stocks

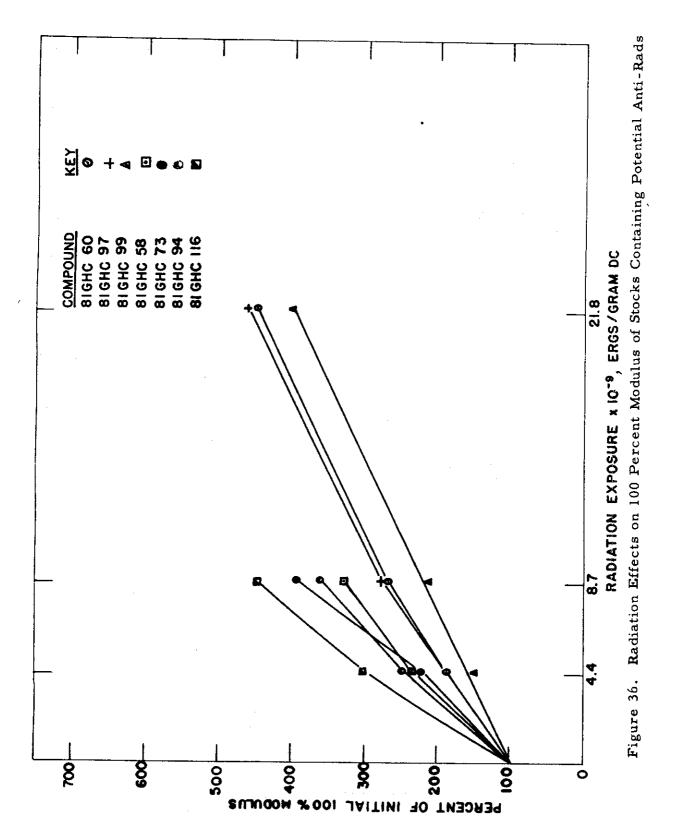




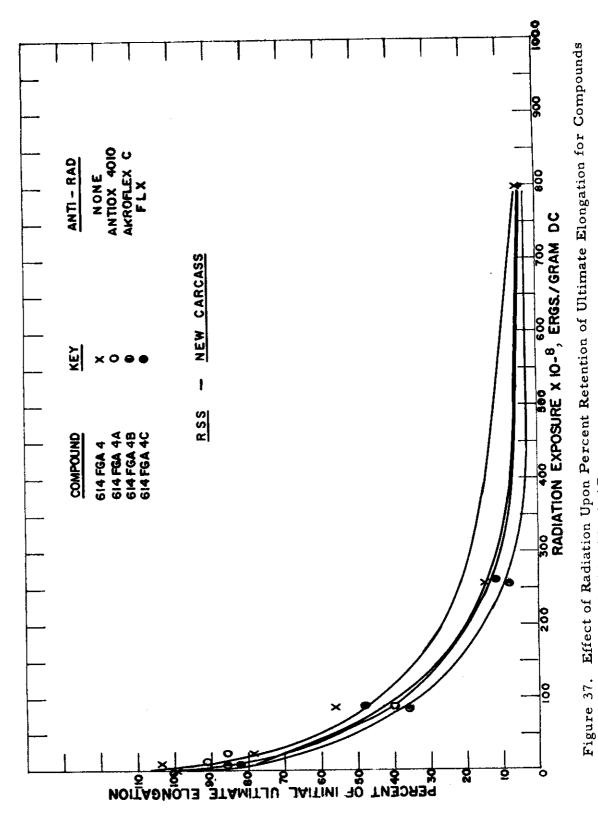


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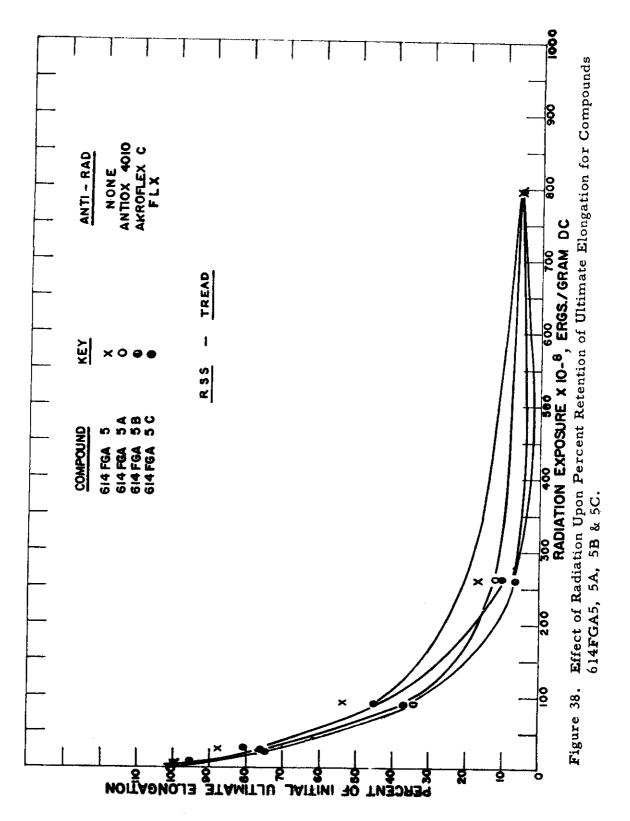


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614FGA4, 4A, 4B, & 4C.

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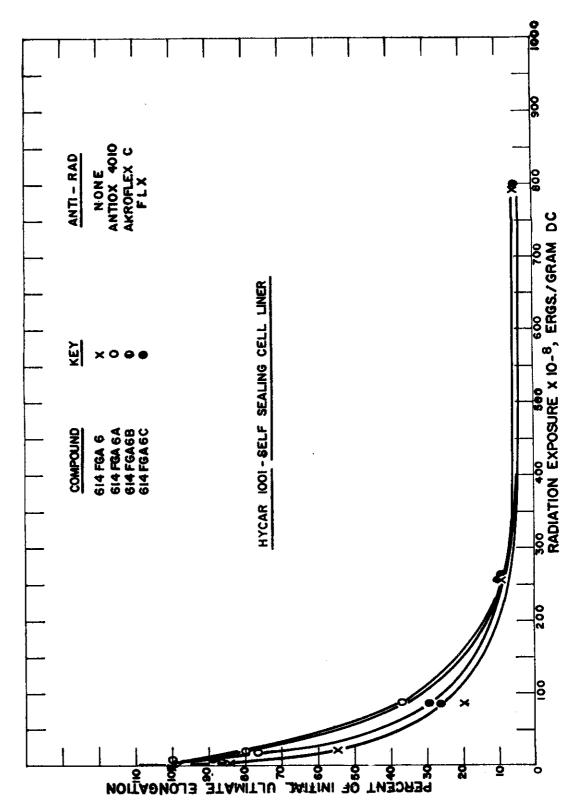
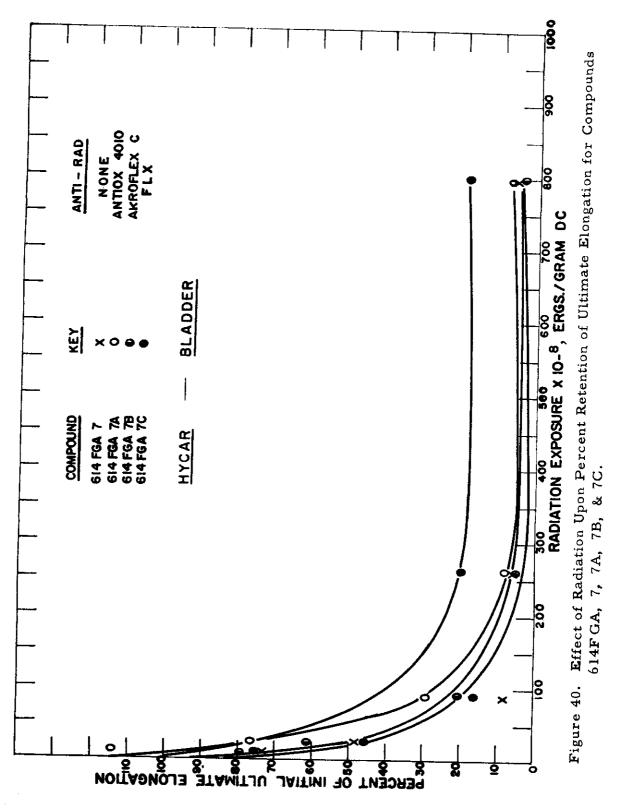
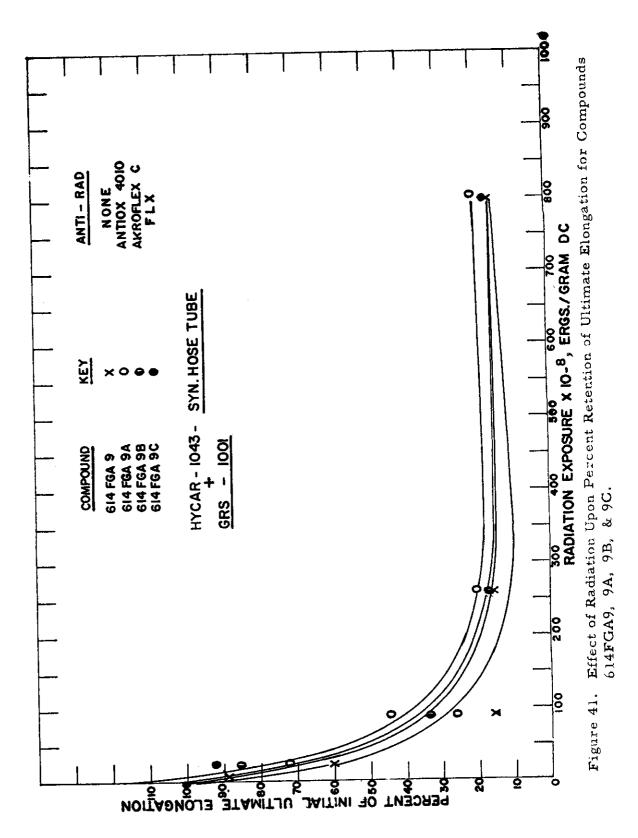
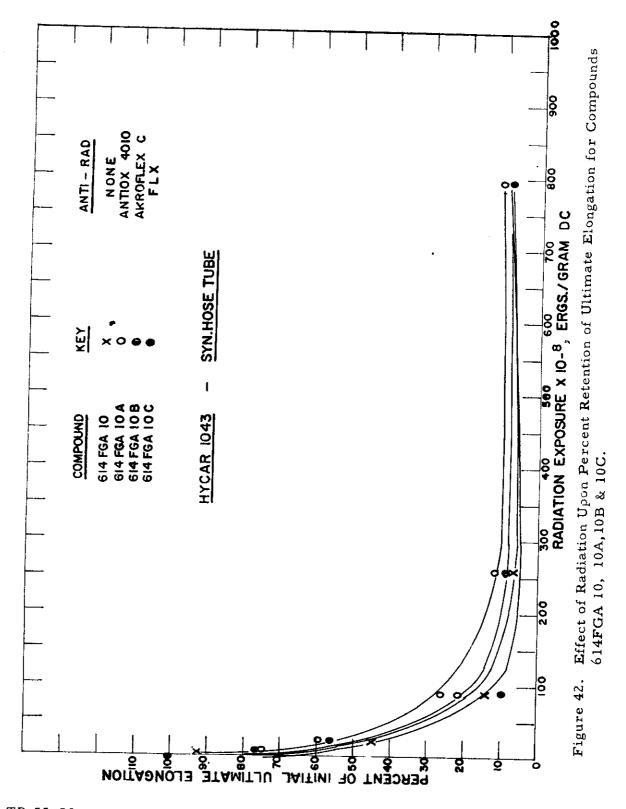


Figure 39. Effect of Radiation Upon Percent Retention of Ultimate Elongation for Compounds 614FGA6, 6A, 6B, & 6C.





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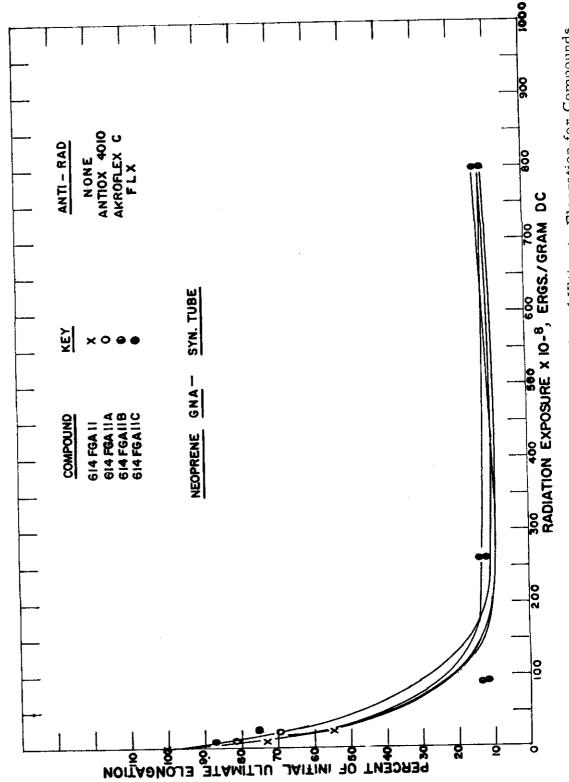
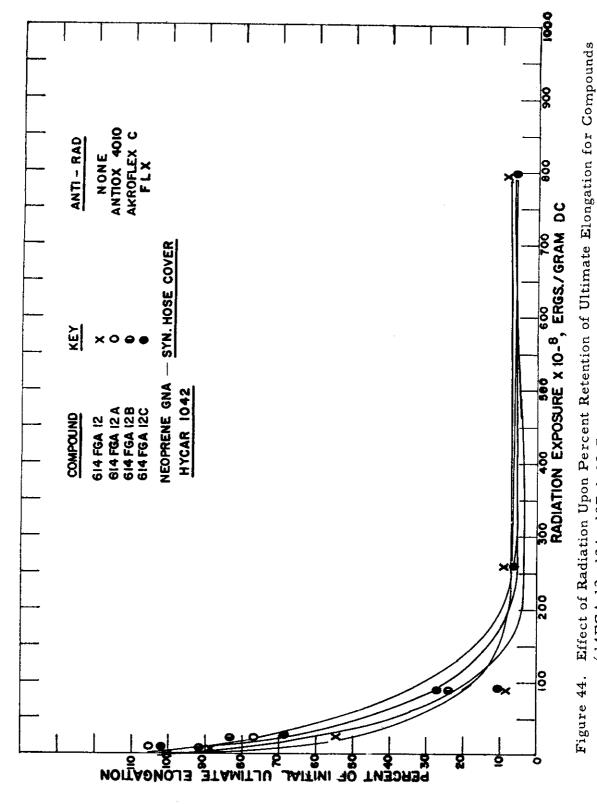
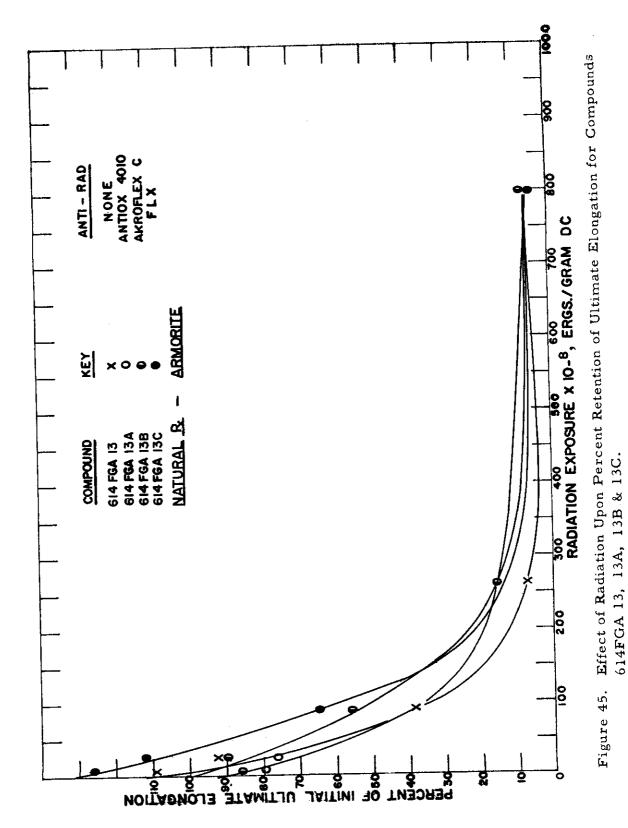


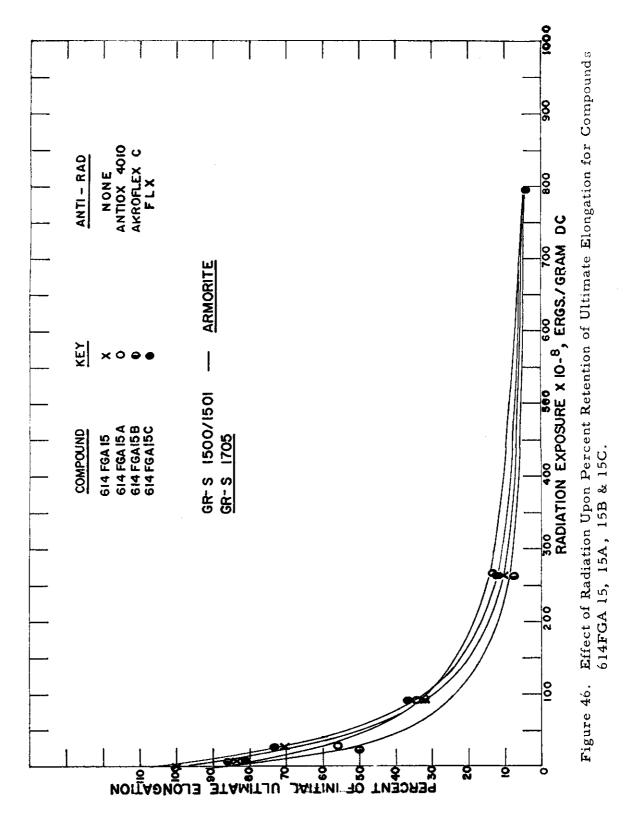
Figure 43. Effect of Radiation Upon Percent Retention of Ultimate Elongation for Compounds 614FGA 11, 11A, 11B & 1 LIC.



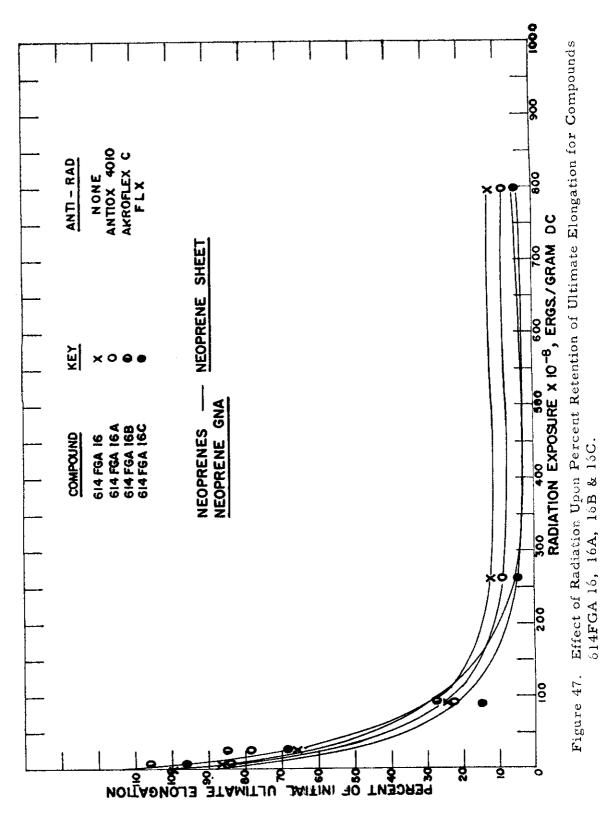
614FGA 12, 12A, 12B & 12 C.

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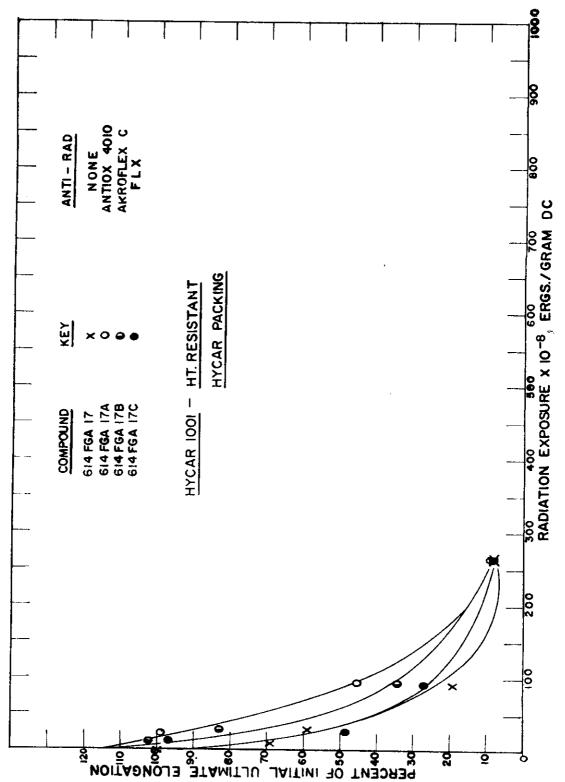




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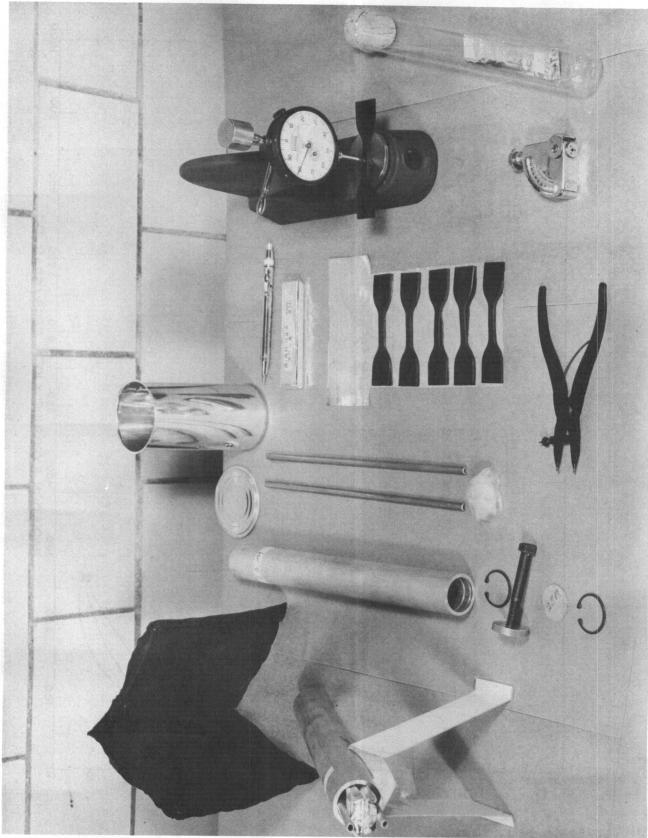


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Effect of Radiation Upon Percent Retention of Ultimate Elongation for Compounds 614FGA 17, 17A, 17B & 17C. Figure 48.

Contrails



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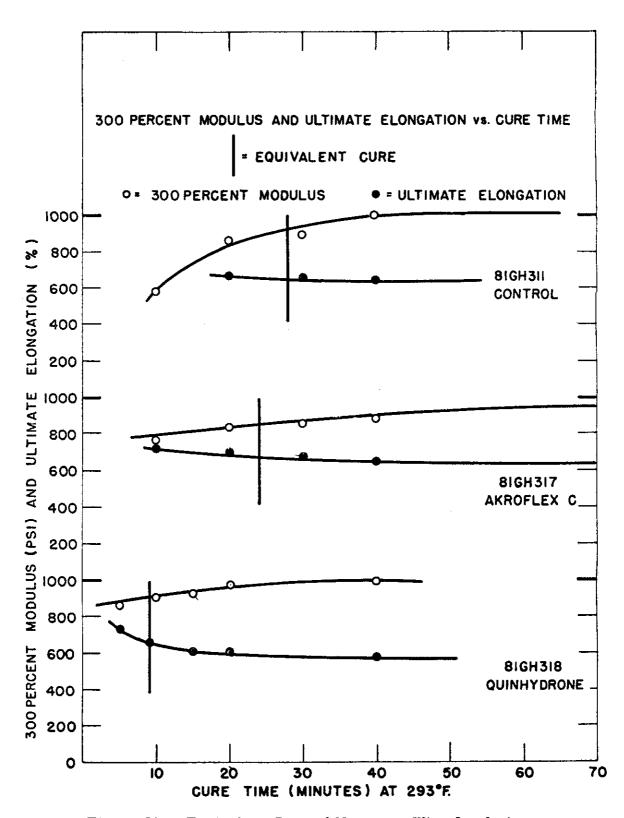


Figure 50. Equivalent Cure of Neoprene Wire Insulation



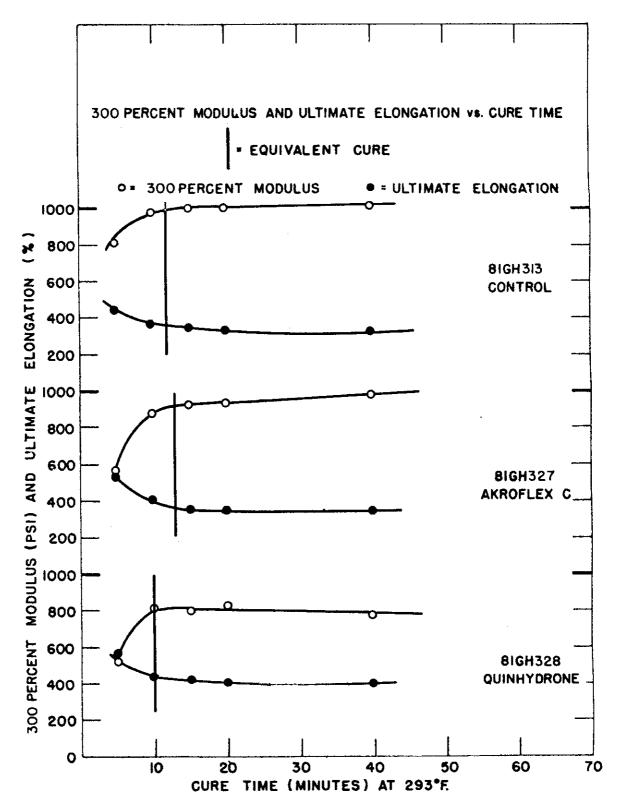


Figure 51. Equivalent Cure of SBR Wire Insulation



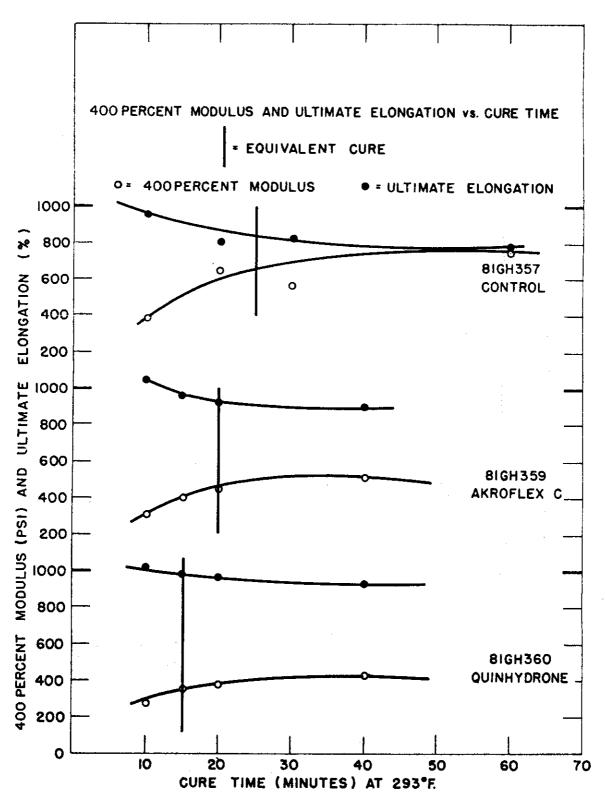


Figure 52. Equivalent Cure of Neoprene Packing Compound

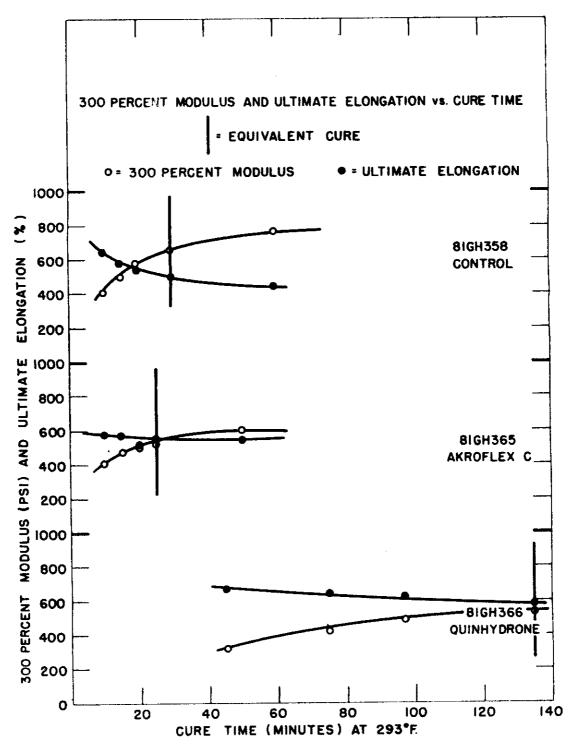


Figure 53. Equivalent Cure of Hycar Packing Compound

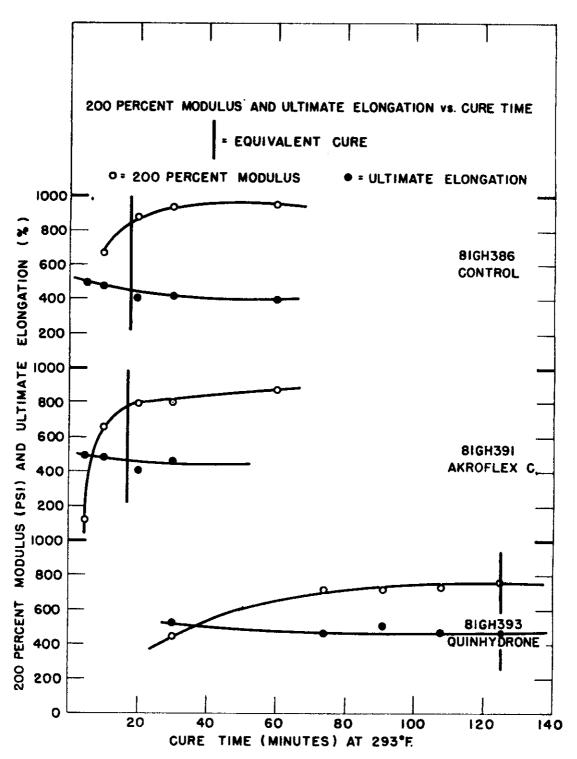


Figure 54. Equivalent Cure of Hycar/Vinylite Bladder Stock.



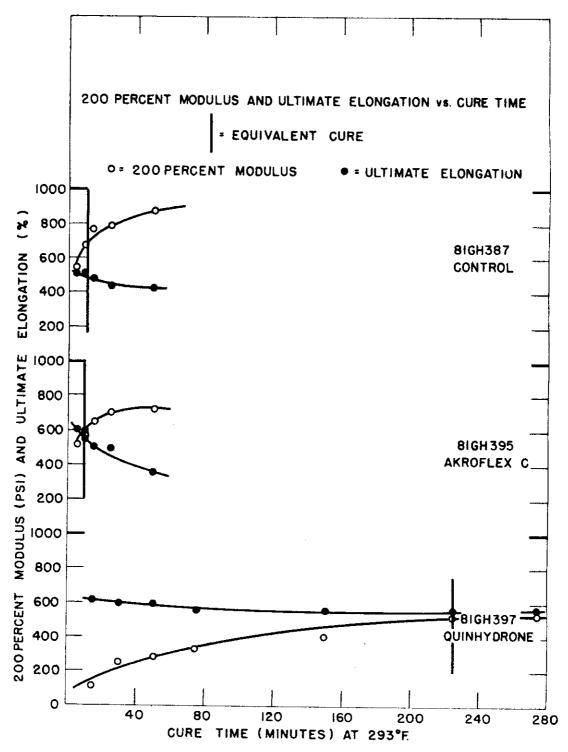


Figure 55. Equivalent Cure of Hycar Self-Sealing Liner Stock.



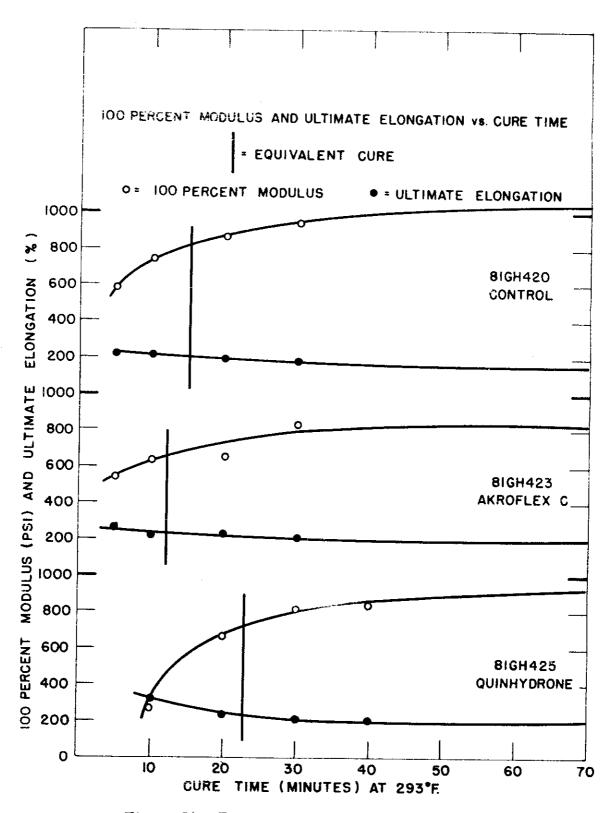


Figure 56. Equivalent Cure of Hycar/SBR Hose



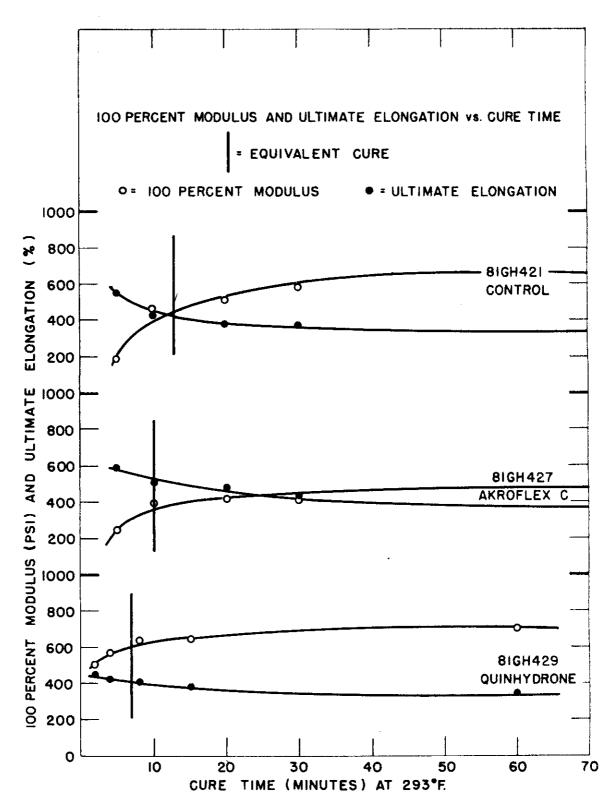


Figure 57. Equivalent Cure of Neoprene Hose



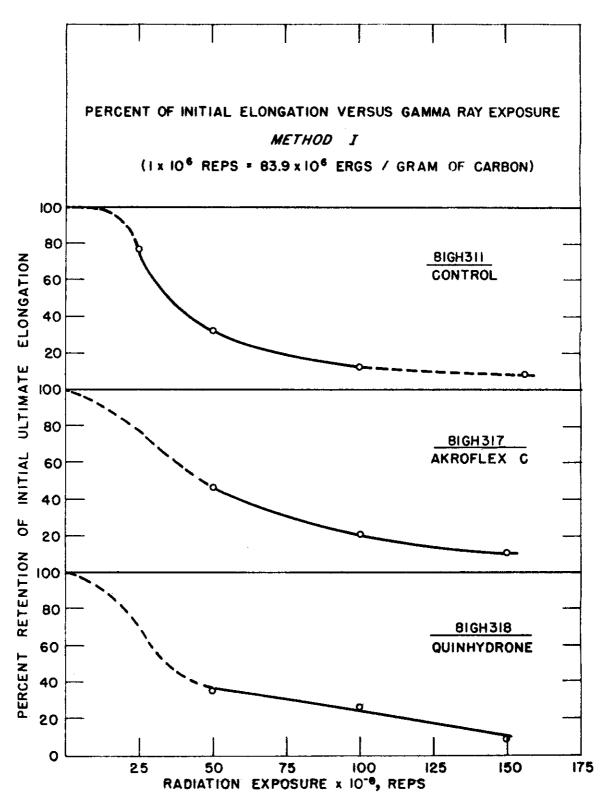


Figure 58. The Effect of Gamma Radiation on Neoprene Wire Insulation.



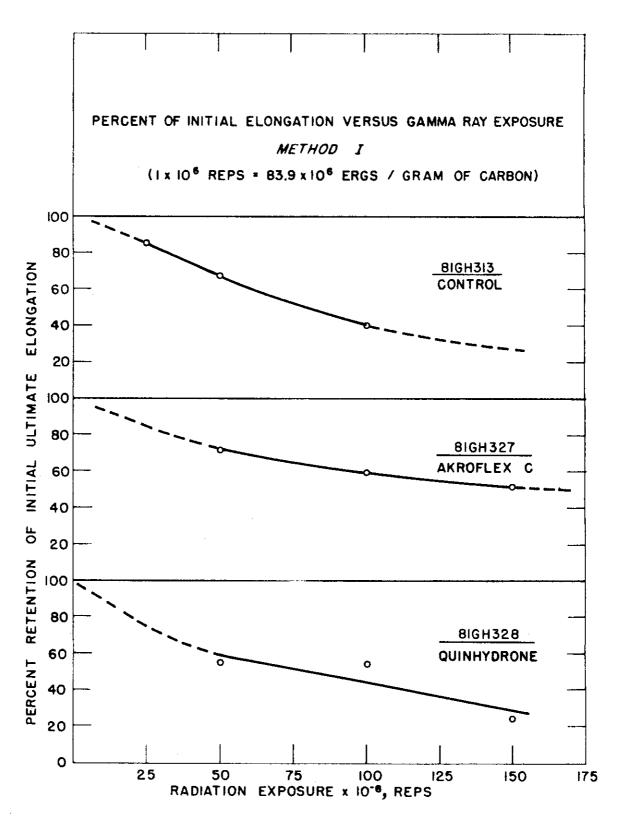


Figure 59. The Effects of Gamma Radiation on SBR Wire Insulation



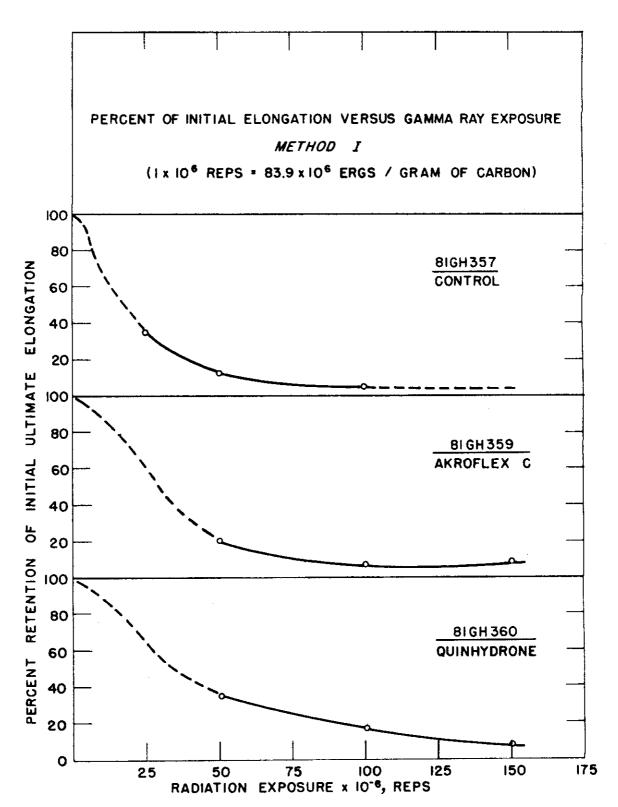


Figure 60. The Effects of Gamma Radiation on Neoprene Packing Compound



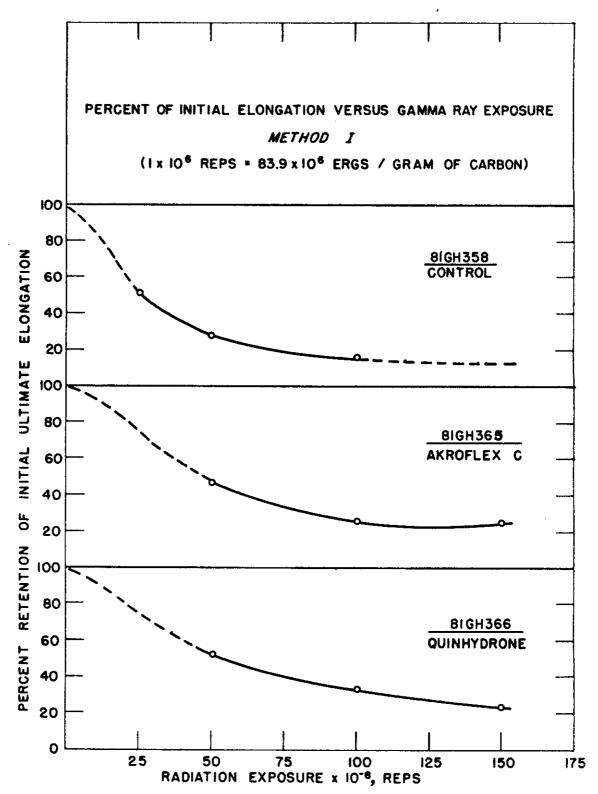


Figure 61. The Effects of Gamma Radiation on Hycar Packing Compound



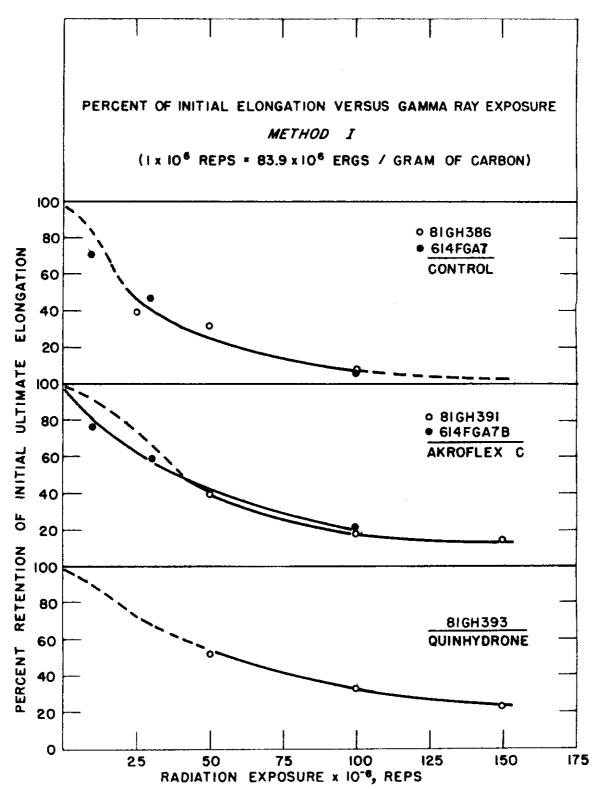


Figure 62. The Effects of Gamma Radiation on Hycar/Vinylite Bladder Stock



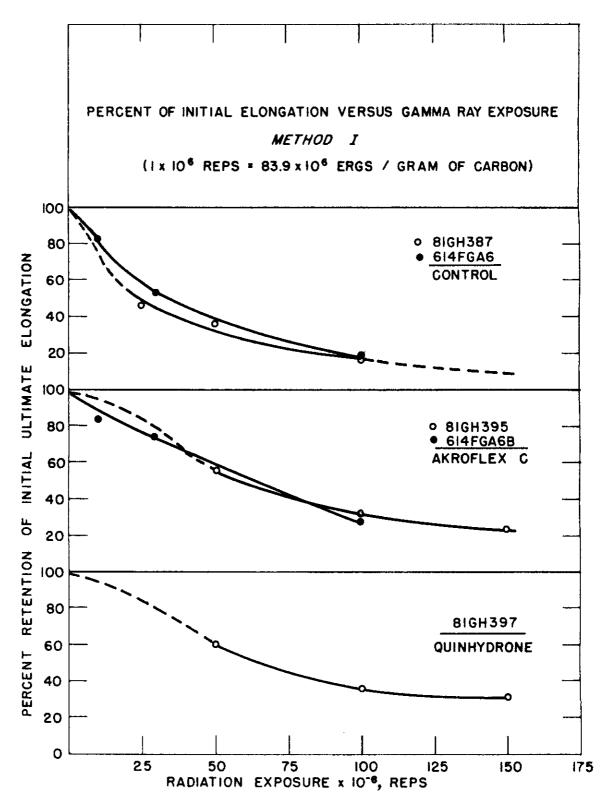


Figure 63. The Effects of Gamma Radiation on Hycar Self-Sealing Liner Stock



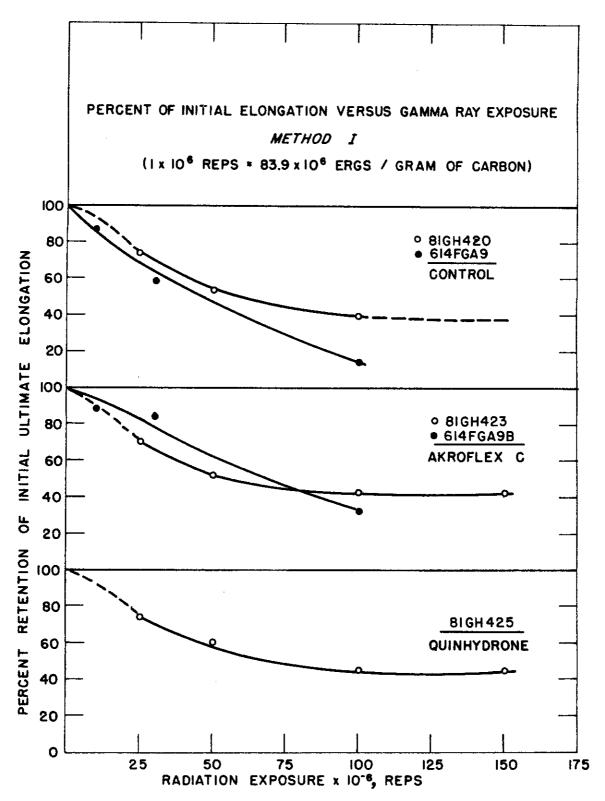


Figure 64. The Effects of Gamma Radiation on Hycar/SBR Hose



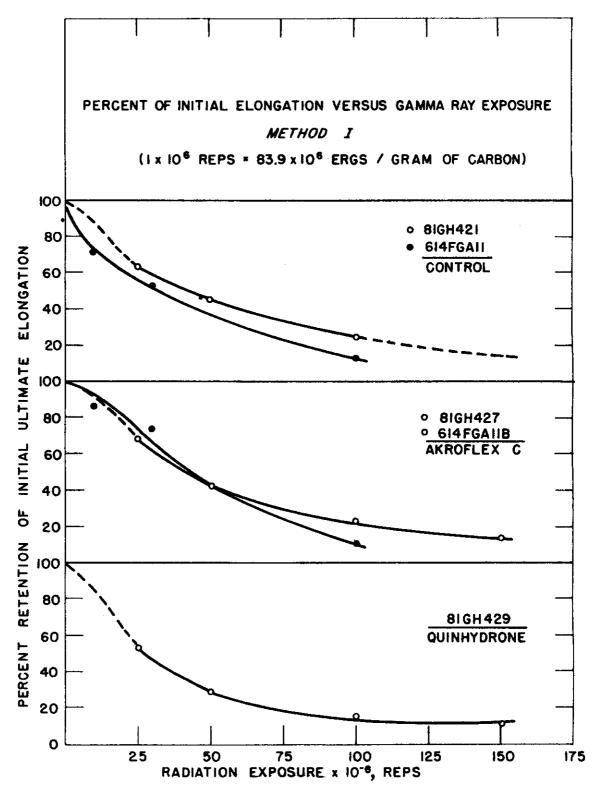


Figure 65. The Effects of Gamma Radiation on Neoprene Hose



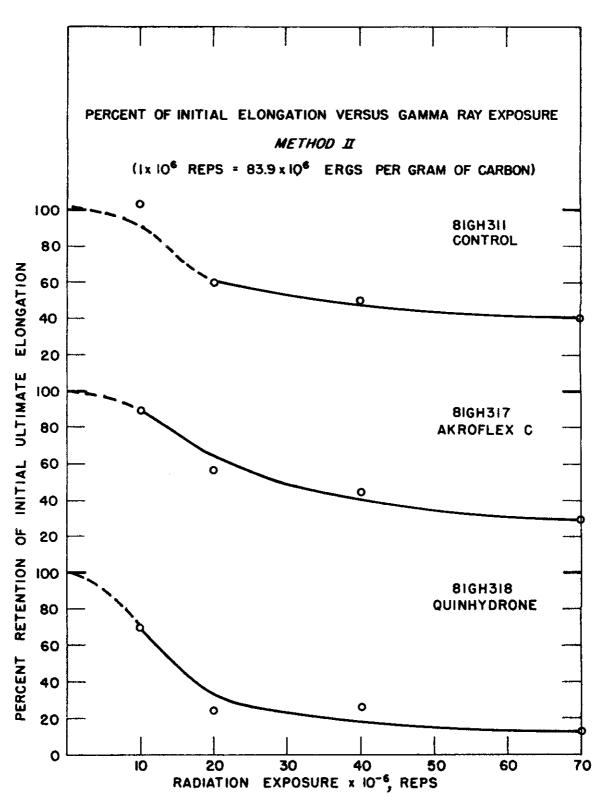


Figure 66. The Effect of Gamma Radiation on Neoprene Wire Insulation



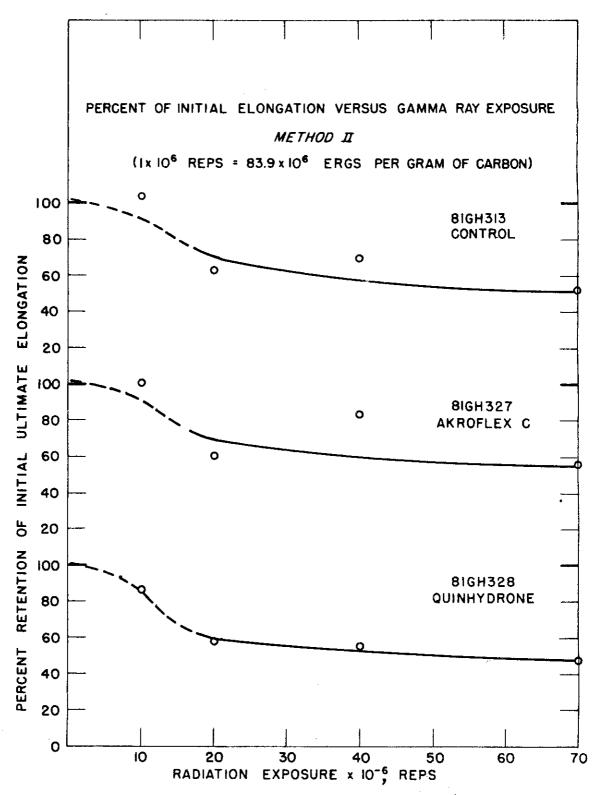


Figure 67. The Effect of Gamma Radiation on SBR Wire Insulation



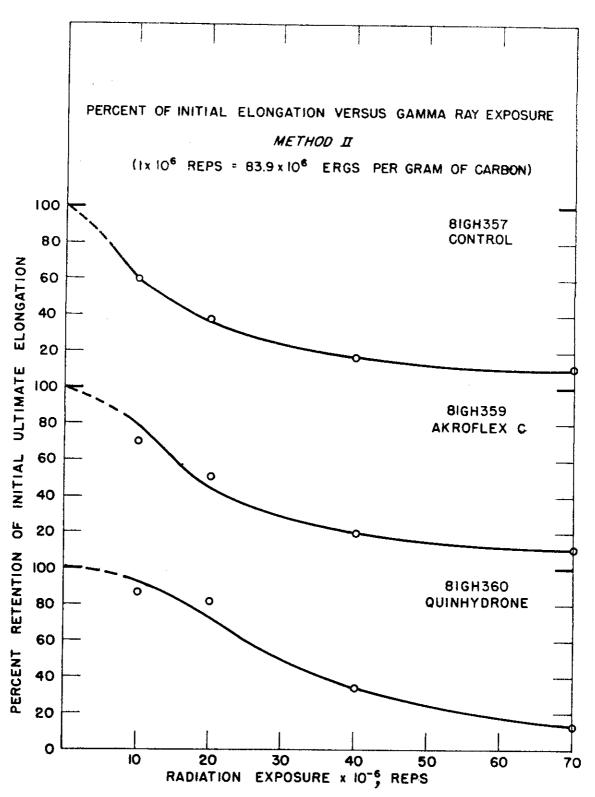


Figure 68. The Effect of Gamma Radiation on Neoprene Packing Compound



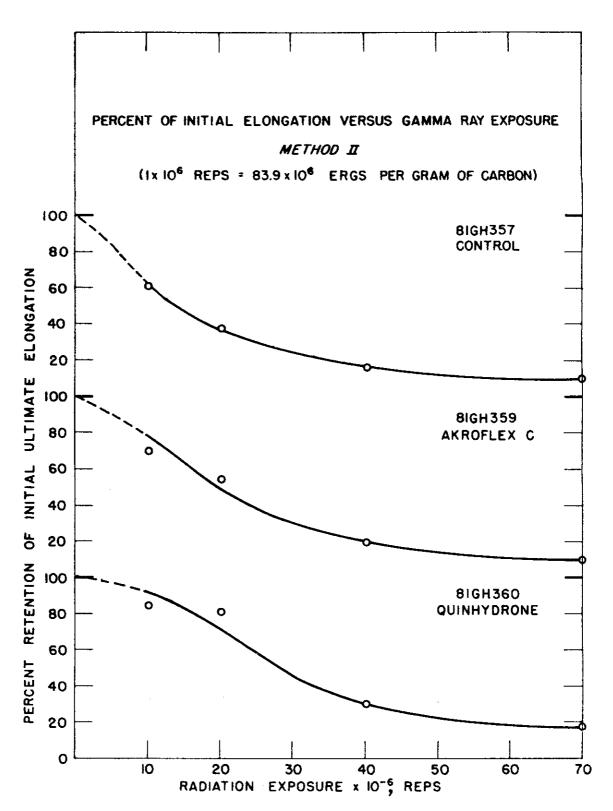


Figure 59. The Effects of Gamma Radiation on Neoprene Packing Compound. (Duplication Experiment)

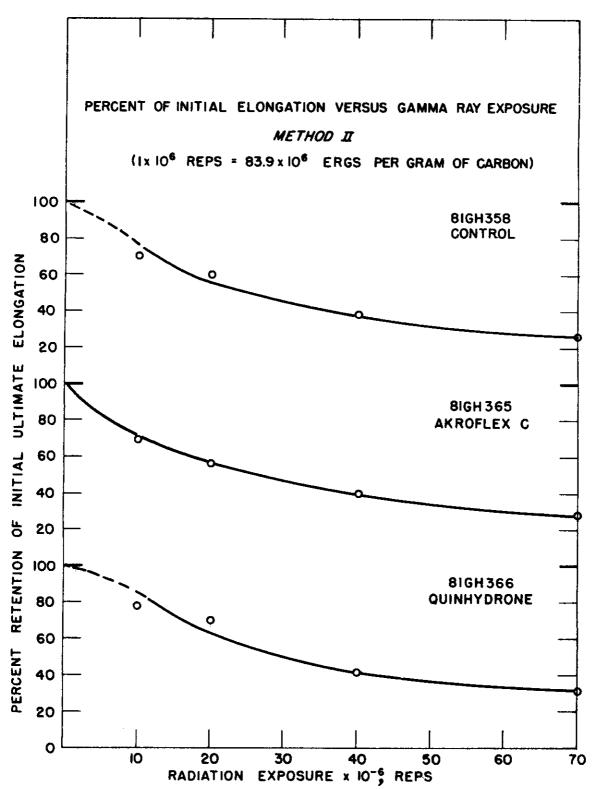


Figure 70. The Effects of Gamma Radiation on Hycar Packing Compound



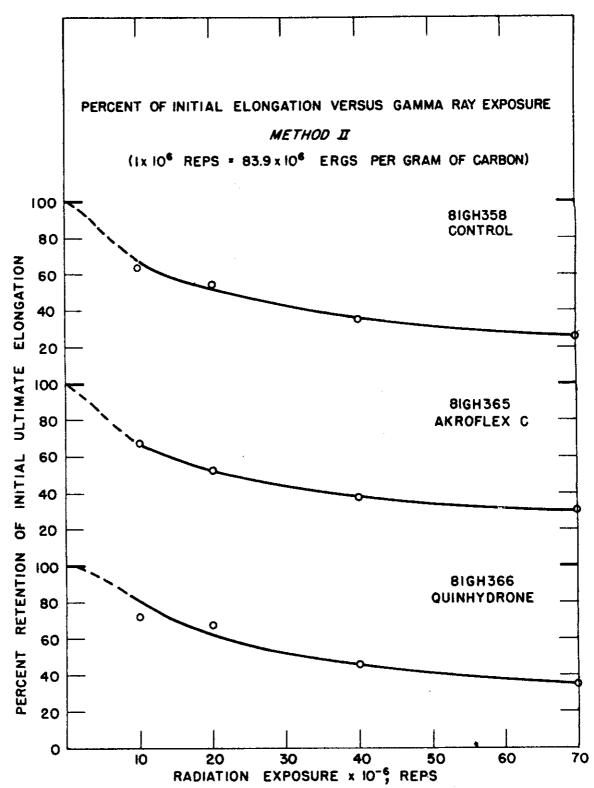


Figure 71. The Effects of Gamma Radiation on Hycar Packing Compound. (Duplication Experiment)

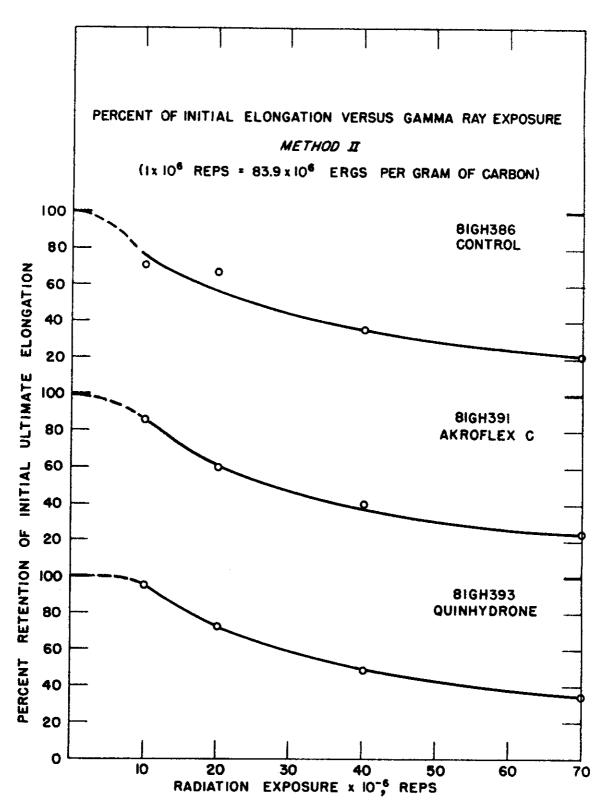


Figure 72. The Effects of Gamma Radiation on Hycar/Vinylite Bladder Stock



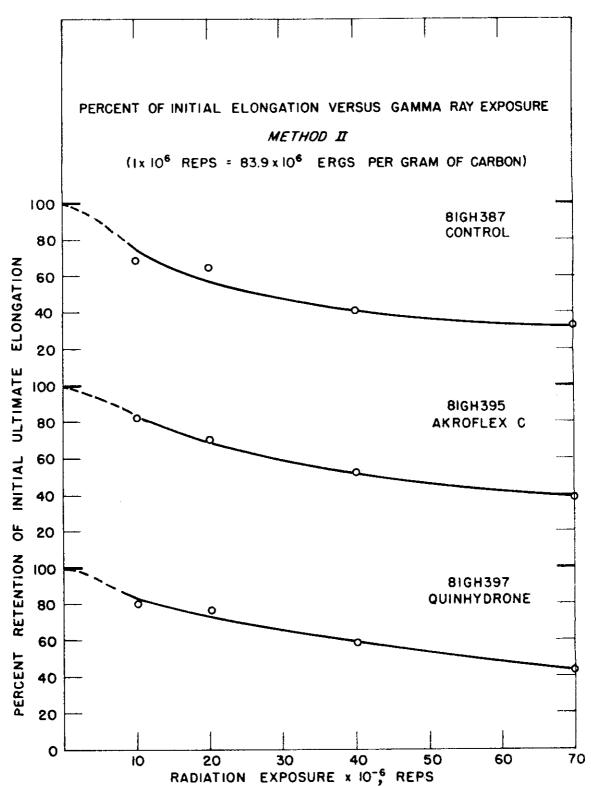


Figure 73. The Effects of Gamma Radiation on Hycar Self-Sealing Liner Stock



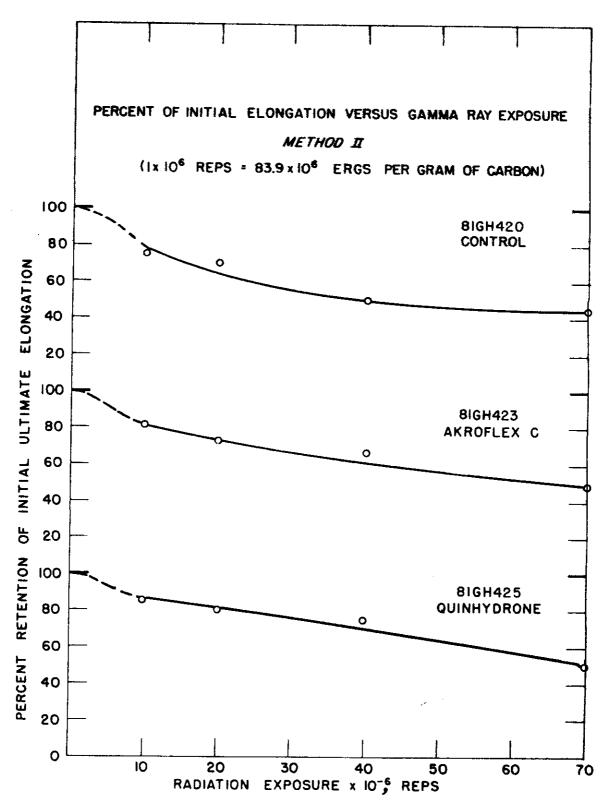


Figure 74. The Effects of Gamma Radiation on Hycar/SBR Hose



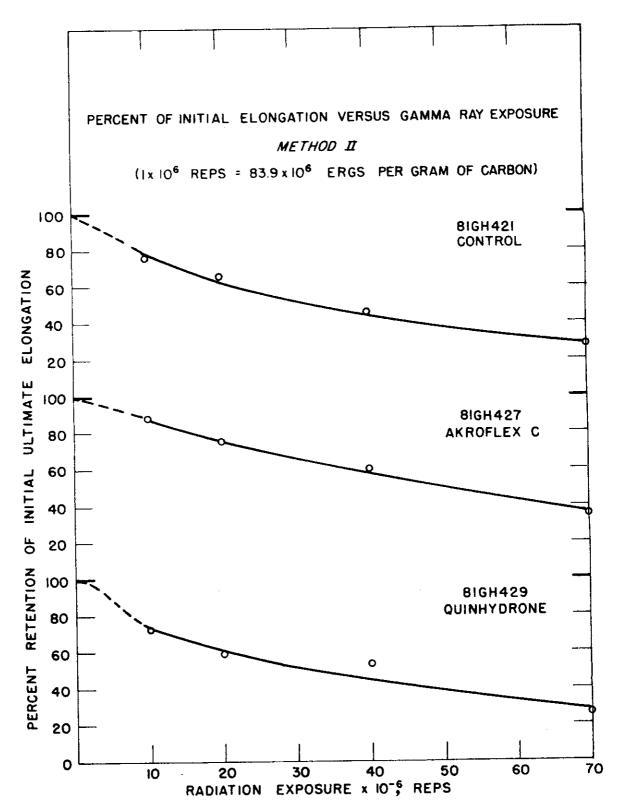


Figure 75. The Effects of Gamma Radiation on Neoprene Hose



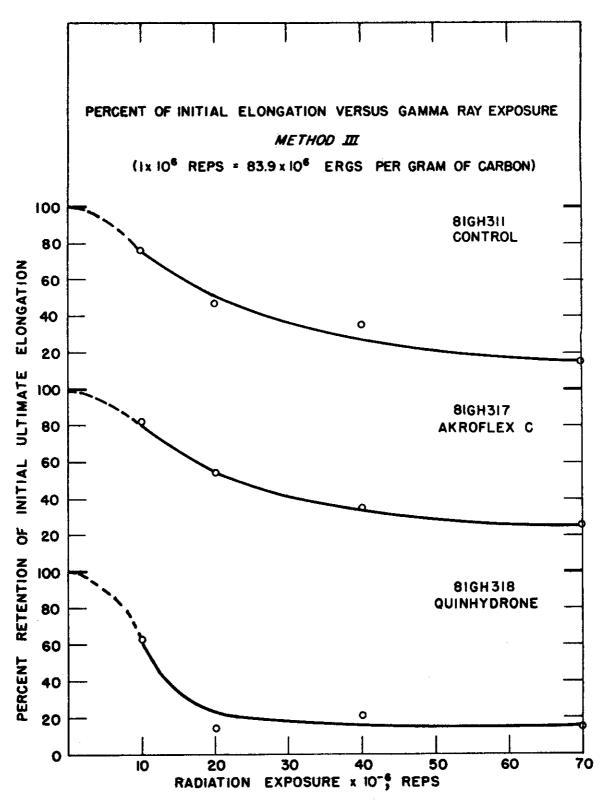


Figure 76. The Effects of Gamma Radiation on Neoprene Wire Insulation



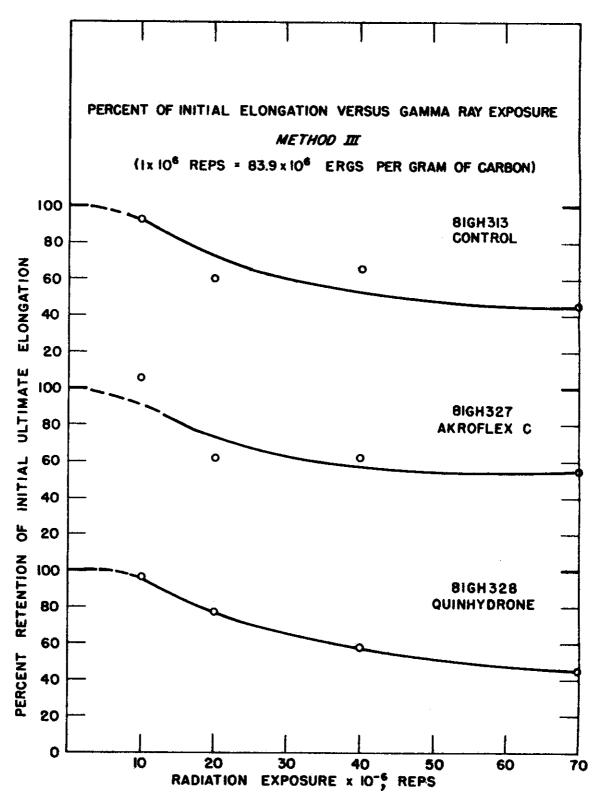


Figure 77. The Effects of Gamma Radiation on SBR Wire Insulation



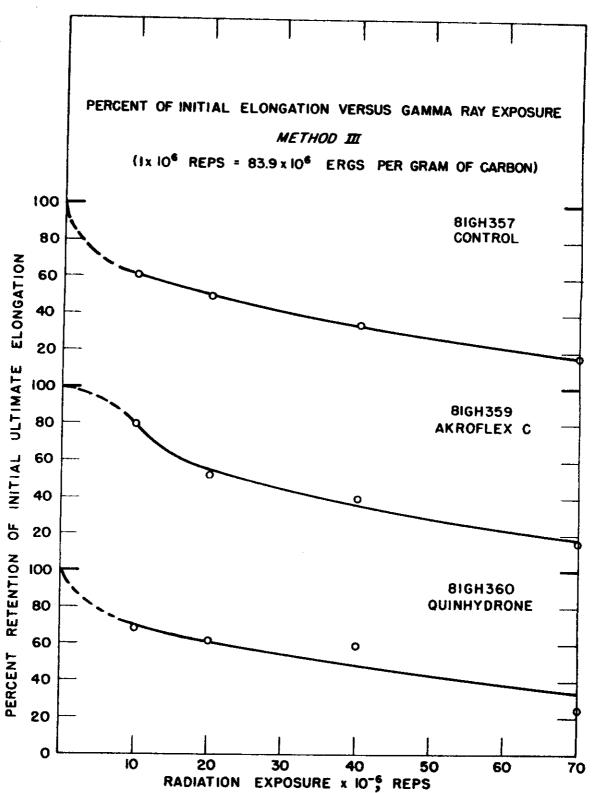


Figure 78. The Effects of Gamma Radiation on Neoprene Packing Compound



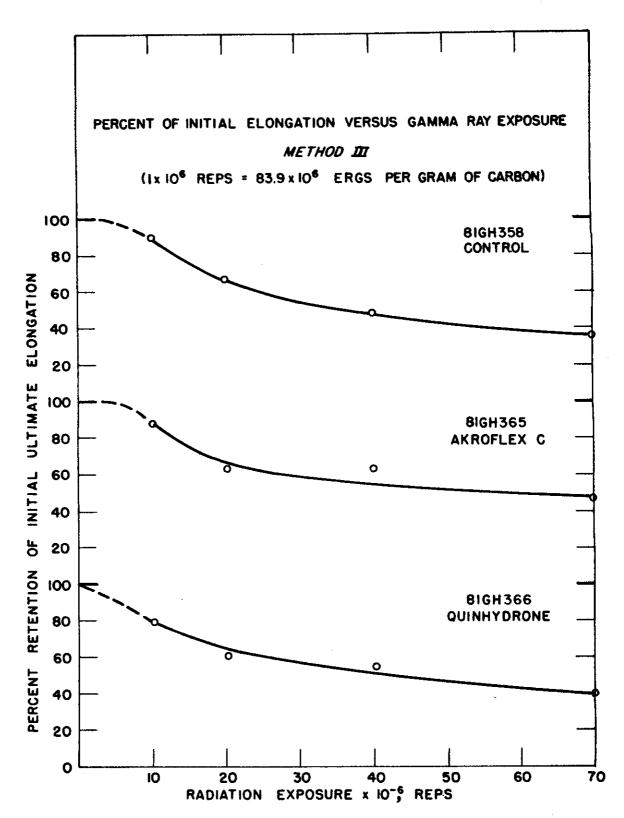


Figure 79. The Effects of Gamma Radiation on Hycar Packing Compounds



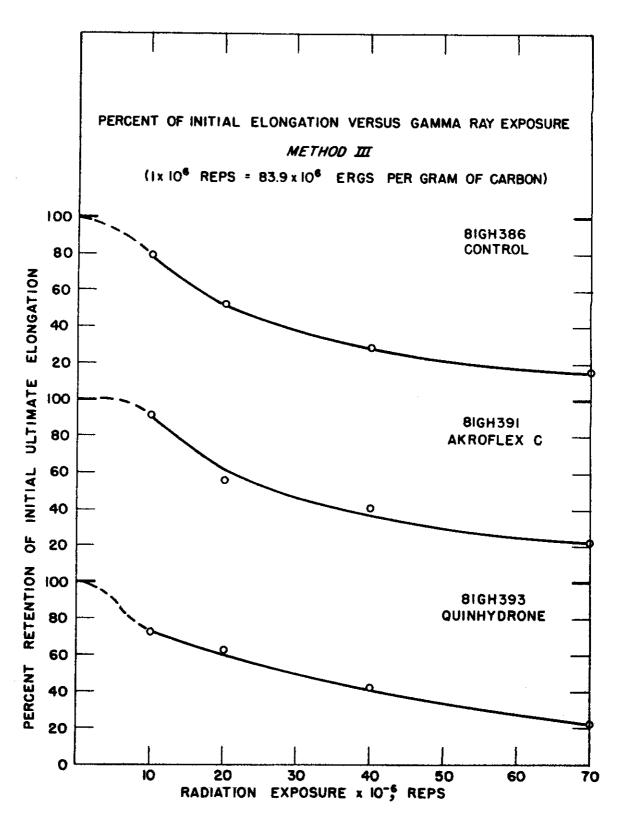


Figure 80. The Effects of Gamma Radiation on Hycar/Vinylite Bladder Stock



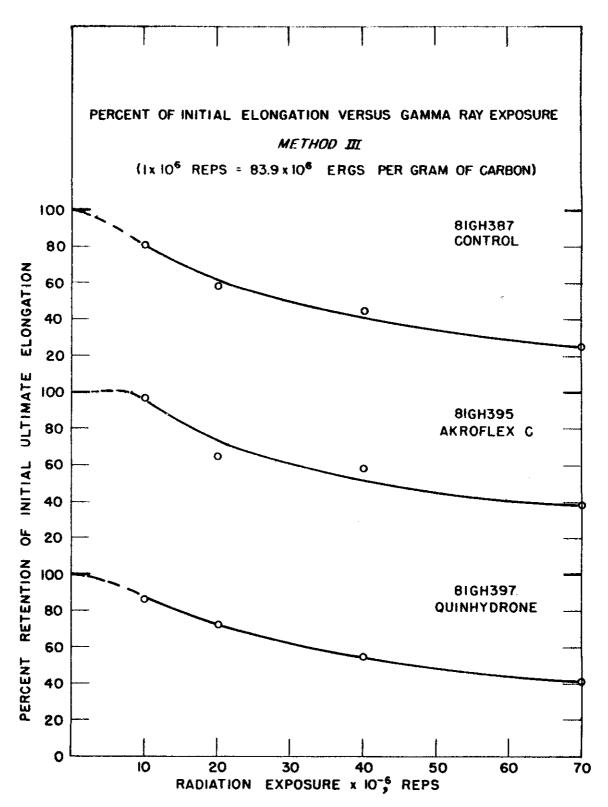


Figure 81. The Effects of Gamma Radiation on Hycar Self-Sealing Liner Stock



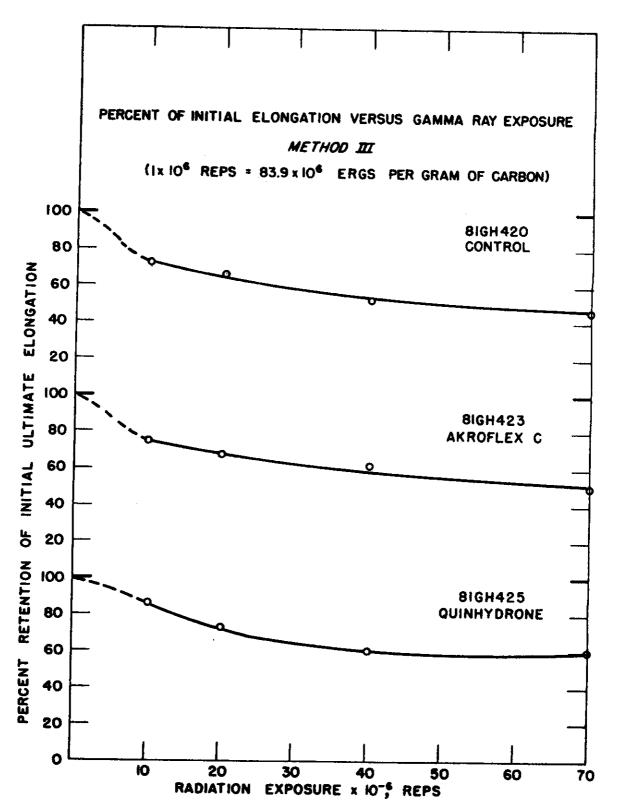


Figure 82. The Effects of Gamma Radiation on Hycar/SBR Hose



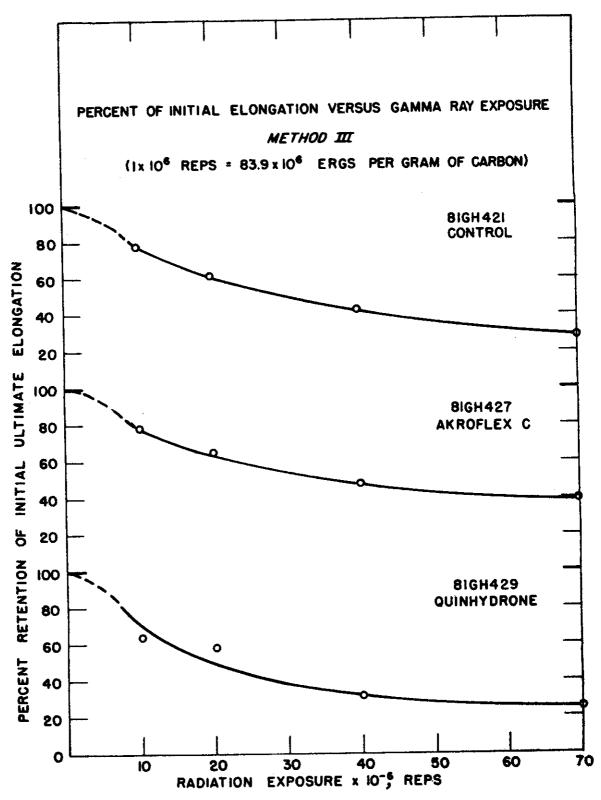
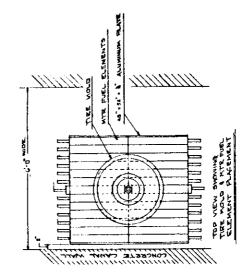
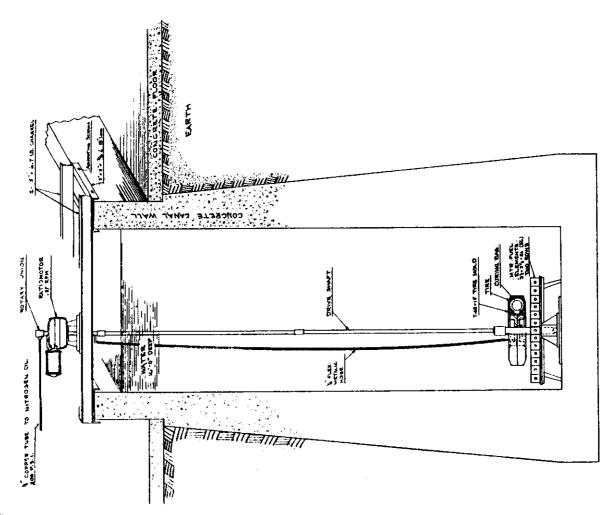


Figure 83. The Effects of Gamma Radiation on Neoprene Hose





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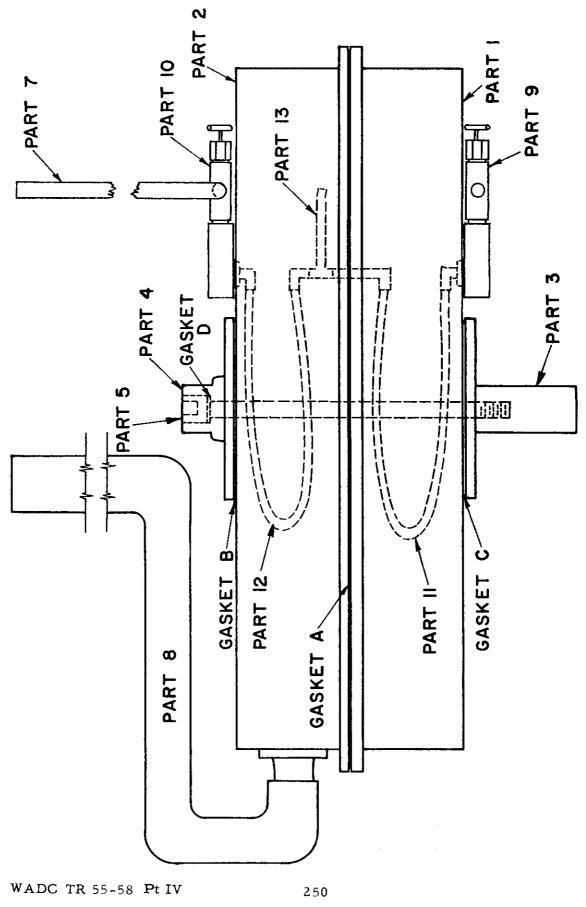


Figure 85. Location of Parts of the Canister Assembly.

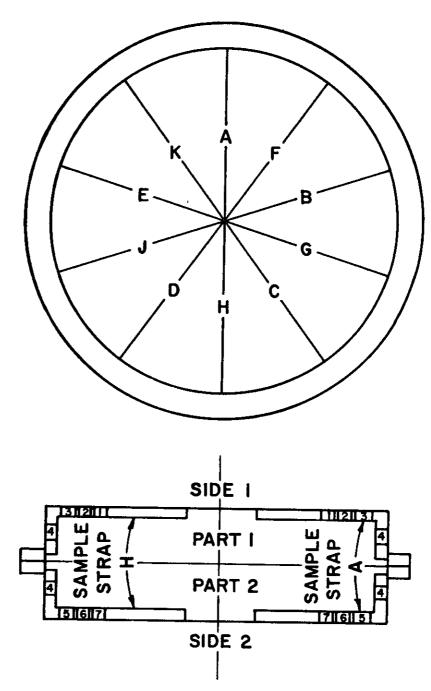


Figure 86. Location of 35 Rubber Samples.

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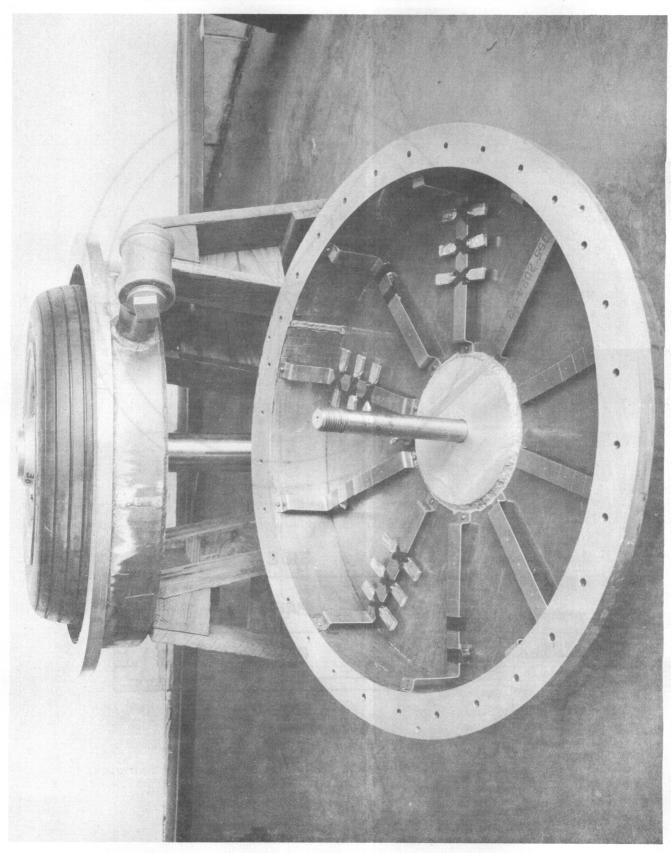
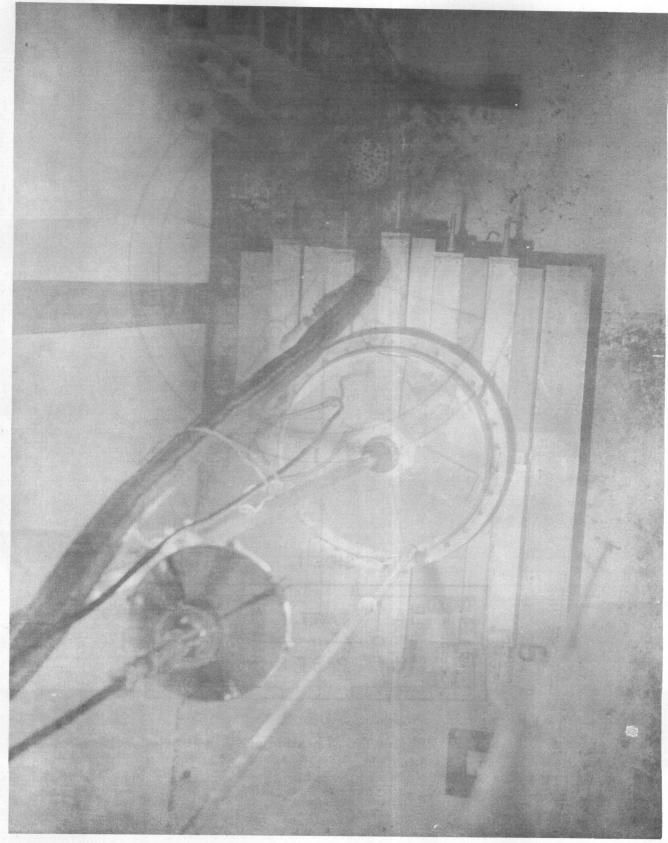


Figure 87. Completed Aircraft Tire Irradiation Canister

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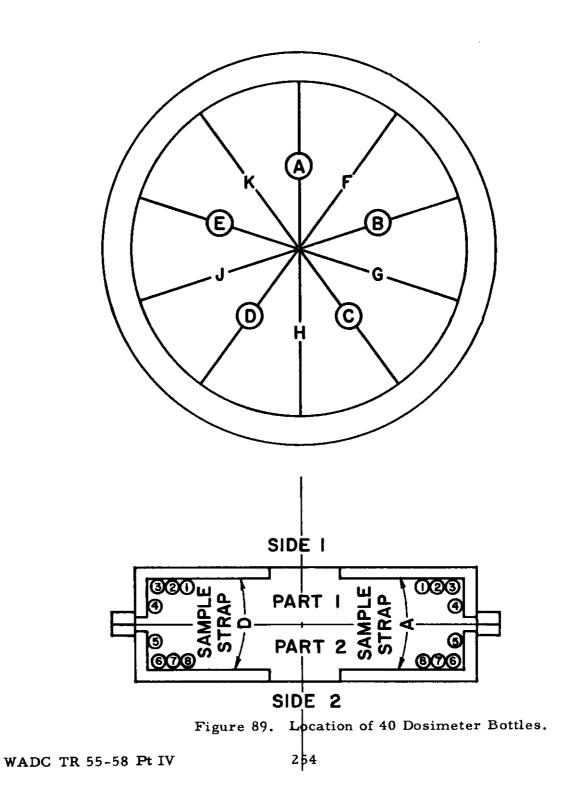
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The Assembled Aircraft Tire Irradiation Equipment in the MTR Gamma Facility Figure 88.

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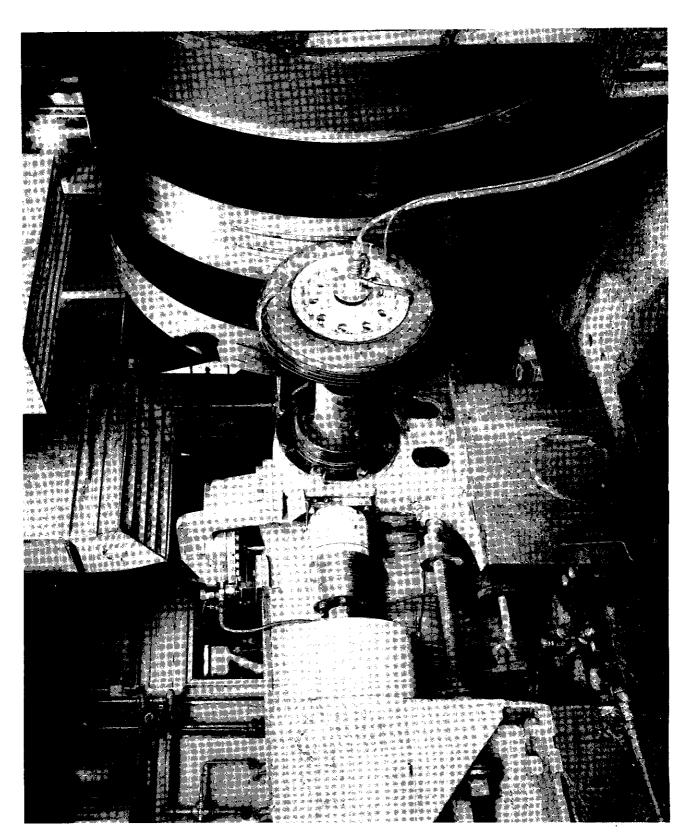
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Approved for Public Release



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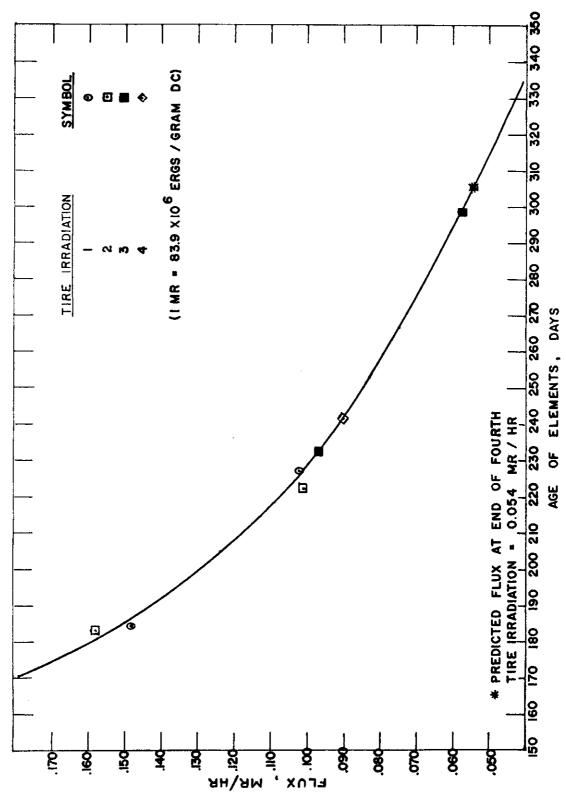
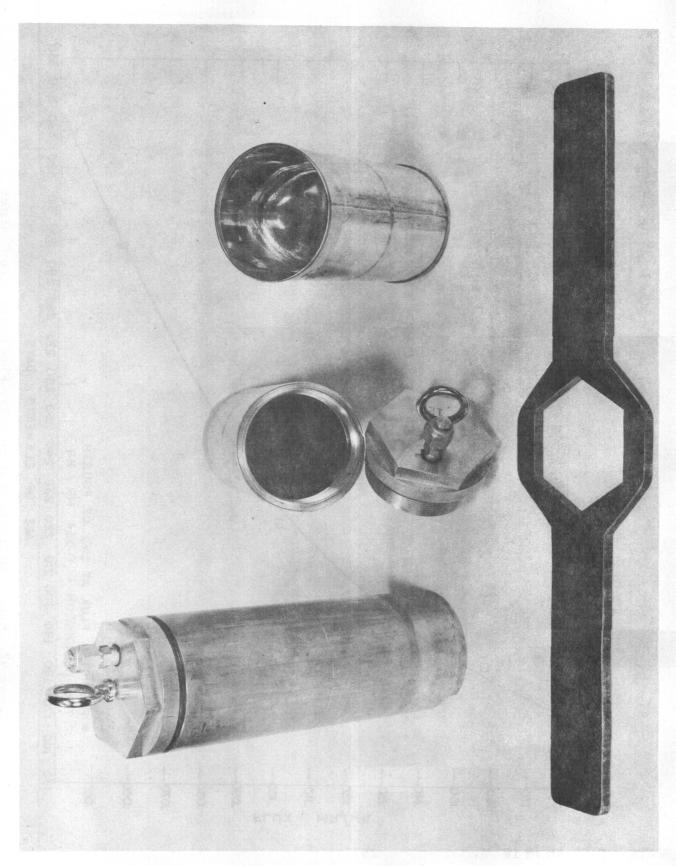


Figure 92. Rate of Decrease of Gamma Ray Flux Inside Tire Canister



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igure 93. Sample Containers for MTR Gamma Irradiation.



APPENDIX V

METHOD OF PREDICTING THE REQUIRED TIRE IRRADIATION PERIOD

The lack of empirical data on the rate of decrease in gamma flux inside a container such as the tire canister required original calculations of fluxes and exposure times in advance of each tire irradiation. Such calculations were necessary in order to insure uniform exposure of each tire to 83.9 x 10^8 ergs per gram of carbon (100 megareps). The steps were as follows:

Development of a Flux Equation

Ceric sulfate dosimetry at various critical locations inside the canister defined the radiation fluxes at the start and end of each irradiation. An empirical equation for the flux "decay rate" of a typical spent fuel element (21), namely flux, $f(r/hr) = 4.2 \times 10^7 t^{-1.21}$, where t is the age of the fuel element in days,

Of course, the use of a 20-element array and the absorption and reflection of the radiations by the equipment made modification of the latter equation necessary. The original average flux value was used to adapt the equation to the new situation. It was reasoned that, since the canister was aluminum and the wheel was magnesium and a part of the radiation path was through air inside the canister, the absorption and geometry were very nearly the same for the tire irradiation as for the original case described in Reference (21).

As an example, consider the first tire irradiation. The average measured flux at the start was 1.48×10^5 r/hr. This value was substituted into the general equation $f = ct^{-1} \cdot 21$ r/hr to give $f = 8.09 \times 10^7$ t $^{-1} \cdot 21$ r/hr.

Prediction of the Required Radiation Exposure Period

The latter flux equation permitted the calculation of the time required for any given exposure, D, by integration. That is, $D = \int_{t}^{t} 2 f \, dt$, where t_1 is the start and t2 is the end of the exposure. The t values are the average ages of the 20 elements. Age is defined as the number of days after the fuel element was discharged from the reactor. Since the total integrated dose was always applied in two equal amounts, one to each side of the tire, the equation became as follows:

$$D = D_1 + D_2 = \int_{t_1}^{t_2} f \, dt + \int_{t_3}^{t_4} f \, dt , \text{ where}$$

$$D_1 = 5 \times 10^7 \, r = 1.94 \times 10^9 - \int_{189}^{t_2} t^{-1.21} \, dt , \text{ and}$$

$$D_2 = 5 \times 10^7 \, r = 1.94 \times 10^9 - \int_{(t_2+1)}^{t_4} t^{-1.21} \, dt.$$



APPENDIX V, (Continued)

That is,

$$D_{1} = 5 \times 10^{7} \text{ r} = -9.24 \times 10^{9} \quad \begin{bmatrix} t & -0.21 & \\ t & & \end{bmatrix} \begin{bmatrix} t_{2} & \\ 189 & \\ t_{2} & \\ t_{3} & \\ t_{4} & \\ t_{2} & \\ t_{4} & \\ t_{2} & \\ t_{3} & \\ t_{4} & \\ t_{2} & \\ t_{3} & \\ t_{4} & \\ t_{2} & \\ t_{4} & \\ t_{2} & \\ t_{3} & \\ t_{4} & \\ t_{2} & \\ t_{3} & \\ t_{4} & \\ t_{2} & \\ t_{3} & \\ t_{4} & \\ t_{2} & \\ t_{3} & \\ t_{4} & \\ t_{2} & \\ t_{4} & \\ t_{2} & \\ t_{3} & \\ t_{4} & \\ t_{2} & \\ t_{4} & \\ t_{2} & \\ t_{3} & \\ t_{4} & \\ t_{2} & \\ t_{3} & \\ t_{4} & \\ t_{2} & \\ t_{4} & \\ t_{2} & \\ t_{3} & \\ t_{4} & \\ t_{2} & \\ t_{5} & \\ t_{5}$$

When the last two equations were solved, the following values were obtained: $t_2 = 205 \text{ days}$, $t_3 = 206 \text{ days}$, and $t_4 = 223 \text{ days}$.

Thus, the first half of the exposure took place between the 189th and the 205th days and the second half, between the 206th and the 223rd days.



APPENDIX VI

LIMITS OF ERROR IN TESTING

Often the evaluation and interpretation of results is made difficult by a lack of information about the limits of testing error. The following limits of error are estimated on the basis of our experience to avoid this difficulty. The figures in parentheses indicate the degree of confidence concerning these limits, expressed in percent. The "confidence" figure signifies the percent of a large number of measurements whose error falls within the stated limits.

Tensile Strength (90%): ± 9%

Ultimate Elongation (90%): ± 14%

Modulus (90%): ±8%

Percent Gel (90%): ± 2%

Swelling Index (95%): \pm 5%

Intrinsic Viscosity (80%): ± 1%

Swelling Ratio (100%): + 2%

Breaking Load (95%): ± 10%

Tenacity (95%): ± 10%

The above figures for tensile strength, ultimate elongation, and modulus are based on a research report by Mr. A.G. Veith which deals in part with this matter. The figures apply to well-cured carbon black-reinforced compounds of the common elastomers. In the case of the newer elastomers the corresponding limits of error should probably be doubled, principally because of uncertainty of optimum cure. Also, the limits of error for tensile strength of Hycar compounds are $\pm 13\%$ for the degree of confidence stated.



APPENDIX VII

SOURCE OF SUPPLY OF THE TWENTY-FIVE BEST COMMERCIAL ANTI-RADS

This list has been made in answer to requests for the names of suppliers of the best anti-rads. The chemicals are listed in order of decreasing effectiveness in inhibiting radiation damage to a standard ASTM natural rubber tread stock. The names of the suppliers are indicated by number code. The code key appears at the end of the list.

- 1. Antiox 4010 (N-cyclohexyl-N'-phenyl-p-phenylene diamine): (4)
- 2. Akroflex C (35% diphenyl-p-phenylene diamine plus 65% phenyl-alphanaphthyl amine): (2)
- 3. Stabilite FLX (N-phenyl-N'-o-tolylethylene diamine): (5)
- 4. Beta-naphthol: (3)
- 5. Beta-naphthyl amine: (3)
- 6. Pyrogallol: (3)
- 7. Phenylhydroquinone: (3)
- 8. UOP-88 (N, N'-dioctyl-p-phenylene diamine): (7)
- 9. Anthraquinone: (3)
- 10. Flectol H (condensation product of acetone and aniline): (6)
- 11. 1, 4-Naphthoquinone: (3)
- 12. Diphenylethylene diamine: (3)
- 13. Phenothiazine: (3)
- 14. Calco 2246 [2, 2'-methylene-bis (4-methyl-6-tertiary-butylphenol)]: (1)
- 15. p-Quinone: (3)
- 16. 2, 5-Ditertiary butyl hydroquinone: (3)
- 17. Quinoline: (3)
- 18. N.B.C. (nickel dibutyldithiocarbamate): (2)
- 19. Hydroquinone monobenzyl ether: (3)



APPENDIX VII, (Continued)

- 20. AgeRite Resin (aldol-alpha-naphthyl amine): (8)
- 21. Diphenyl amine: (3)
- 22. Thermoflex (p, p'-dimethoxydiphenyl amine): (2)
- 23. p-Methoxyphenol: (3)
- 24. 2, 5-Ditertiarybutyl-p-quinone: (3)
- 25. Acridine: (3)

CODE TO SUPPLIERS

The following are not necessarily the sole suppliers of the above chemicals. Also, this list does not necessarily imply that a chemical prepared by one company is superior in any respect to the same chemical from another company.

- (1) American Cyanamid Company Calco Chemical Division Rubber Chemicals Department Bound Brook, New Jersey
- (2) E. I. duPont de Nemours and Company, Inc. Rubber Chemicals Department Wilmington 99, Delaware
- (3) Eastman Organic Chemicals Department Eastman Kodak Company Rochester 3, New York
- (4) Naftone, Inc.
 515A Madison Avenue
 New York, New York
 (American distributors for Farbenfabriken-Bayer A.G.)
- (5) C.P. Hall Company 414 South Broadway Akron, Ohio
- (6) Monsanto Chemical Company Rubber Service Department Akron, Ohio



APPENDIX VII, (Continued)

- (7) Universal Oil Products Company 310 South Michigan Avenue Chicago 4, Illinois
- (8) R.T. Vanderbilt Company, Inc. 133 East Center Street Akron, Ohio

NOTE:

Identification of the commercial rubber chemicals by chemical compound names has been made as well as our information permits. It is conceivable that in some instances our information may be inaccurate. In the majority of cases, however, the identifications which appear in Compounding Ingredients for Rubber, Compiled by the Editors of India Rubber World, New York, New York, Second Edition 1947.

The above list is based upon work done under United States Air Force Contract AF33(616)-2308, which is reported in WADC Technical Report 55-58 Part III, December, 1956.

APPENDIX VIII

SOURCE OF SUPPLY OF THE THIRTEEN BEST ANTI-RADS

This list has been made to answer requests for the names of suppliers of the best anti-rads. The chemicals are listed in order of decreasing effectiveness in inhibiting radiation damage to a standard ASTM natural rubber tread stock. The code number in the parentheses following the additive identifies the supplier. The supplier-code number relationship appears at the end of the list.

- 1. 2.2.4-Trimethyl-6-(α,α-dimethylbenzyl)-1.2-dihydroquinoline: (4)
- 2. "Santowhite": (6)
- 3. "Antioxidant 4010" (N-Cyclohexyl-N'-phenyl-p-phenylene diamine): (7)
- 4. Cyclohexyl aniline: (4)
- 5. "Tinopal PCR+ PCRP": (2)
- 6. 2-tert-Butyl-4-methoxy phenol: (10)
- 7. $2-(\alpha naphthylthio) quinone: (4)$
- 8. "Vultac #3": (9)
- 9. N-Phenyl-beta-naphthylamine: (3)
- 10. "Aminox" (Diphenylamine acetone reaction product): (8)
- 11. "Peptone 22": (1)
- 12. "Bright Tone Indo Maroon MV-6623": (5)
- 13. "Thiosa Fast Red MV-6613": (5)

CODE TO SUPPLIERS

The following are not necessarily the sole suppliers of the above chemicals. Also, this list does not necessarily imply that a chemical prepared by one company is superior in any respect to the same chemical from another source.

(1) American Cyanamid Company Calco Chemical Division Rubber Chemicals Department Bound Brook, New Jersey

APPENDIX VIII, (Continued)

- (2) Geigy Industrial Chemicals P.O. Box 430 Yonkers, New York
- (3) The B. F. Goodrich Chemical Company 3135 Euclid Avenue Cleveland 15, Ohio
- (4) The B.F. Goodrich Company Research Center Brecksville, Ohio
- (5) Harmon Colors
 The B. F. Goodrich Chemical Company
 P. O. Box 14
 Hawthorne, New Jersey
- (6) Monsanto Chemical Company Rubber Service Department Akron 11, Ohio
- (7) Naftone, Inc.
 515 Madison Avenue
 New York 22, New York
 (American distributors for Farbenfabriken-Bayer A. G.)
- (8) Naugatuck Chemical Company
 Division of U.S. Rubber Company
 Naugatuck, Connecticut
- (9) Sharples Chemical, Inc.1100 Widner Bldg.Philadelphia 7, Pennsylvania
- (10) Universal Oil Products Company 30 Algonquin Road Des Plaines, Illinois
- NOTE: Identification of the commercial rubber chemicals by chemical names has been made as well as our information permits. It is conceivable that in some instances our information may be inaccurate.

Addresses of suppliers came from the Oil Paint & Drug Reporter "Green Book" Buyer's Directory 1955-56, and from private sources.

The above list is based upon work done under United States Air Force Contract AF 33(616)-1738, which is reported in WADC Technical Report 55-58, Part IV.

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APPENDIX IX

DYNAMOMETER LANDING TEST PROCEDURE FOR AIRCRAFT TIRES

Break-In Landing:

One low-speed landing (see below) is used as a break-in step prior to the actual test cycles.

High - Speed Landing Test:

The tire is landed against a flywheel rotating at an initial peripheral speed of 200 mph. The flywheel speed is then decreased until a roll distance of 7,000 ± 250 feet has been covered, at which time the tire is unlanded. During the high-speed landings the dynamometer is decelerated at a controlled rate. Therefore, the tire does not absorb the full kinetic energy of the flywheel. The average deceleration rate is 7.5 feet per second between 200 and 100 mph and is unspecified during the remainder of the test cycle. The load is increased to 5,000 pounds within 5 to 6 seconds after landing and then further increased linearly with time to a total load of 10,000 pounds at 29 to 30 seconds after landing. The load is then maintained at 10,000 pounds until the end of the test cycle. Twenty-five (25) cycles of the above test are required. The average high speed landing requires about 320 revolutions.

Low - Speed (Fatigue) Landing Test:

Following the 25 high speed landing cycles, low-speed (90 to 0 mph) landings are conducted until the tire ultimately fails. During each low-speed landing cycle the tire absorbs the full 887,000 foot pounds of kinetic energy which had been developed by the flywheel. The average low-speed landing requires about 470 revolutions. The values for low-speed landings are apt to deviate more from the average than are those for high-speed landings.

The high and low-speed landings are made at one-half hour intervals. This schedule allows the tire to cool approximately to room temperature.