Butyl Rubber: Compound Development and Characterization

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Abstract

This report is intended to summarize the research efforts at the U.S. Army Research Laboratory (ARL) and at the Materials Technology Laboratory, currently the Weapons and Materials Research Directorate (WMRD), to develop the standard butyl rubber compound. The strategy of this work was to compound- and compression-mold high-quality, uniform butyl rubber experimental sheets and to evaluate their cure properties, mechanical properties, and—most importantly—their protective barrier properties, as determined by the MIL-282 method 204 droplet breakthrough test performed at the Edgewood Research, Development, and Engineering Center, Product Assurance Directorate (ERDEC-PAD). The test was performed at ERDEC with chemical agents in a qualified laboratory environment and with chemical agent simulants at ARL in Watertown, MA.

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1. Introduction

Butyl rubber is among the most widely used commercially available synthetic elastomers in use today. The consumption of the various grades of butyl rubber is estimated to be over 500,000 tons. Approximately 80% of this volume is utilized in inner liners for tubeless tires and in inner tubes. It is particularly useful in instances where low rates of gas permeability and improved chemical resistance are important. The Army uses this material extensively for their chemical protective clothing. However, at this time, a thorough understanding of the relationship between composition and permeation properties has not been investigated. Experiments were conducted to characterize the permeation properties of a series of butyl rubber compounds. We have formulated and cured a number of butyl rubber compounds varying in carbon black content, curing system, compounding, and curing variables. The goal of this work is to study the effect of these variables on permeation properties.

2. Project Overview

This project was undertaken with the goal of developing a butyl rubber compound that has a reliable and reproducible permeation breakthrough when challenged with chemical agents or chemical simulants. The developed butyl rubber compound was a candidate for use as a material standard for quality assurance testing of production materials to be used to make chemical protective gloves. The Engineering Directorate, Surety Team, Chemical Evaluation Laboratory, U.S. Army Edgewood Research, Development, and Engineering Center (ERDEC) funded this research effort.

This report is intended to summarize the research efforts at the U.S. Army Research Laboratory and the Materials Technology Laboratory, currently the Weapons and Materials Research Directorate (WMRD), to develop the standard butyl rubber compound. The strategy of this work was to compound- and compression-mold high-quality, uniform butyl rubber experimental sheets and to evaluate their cure properties, mechanical properties, and—most

importantly—their protective barrier properties, as determined by the MIL-282 method 204 droplet breakthrough test performed at ERDEC-PAD. The test was performed at ERDEC with chemical agents in a qualified laboratory environment.

3. Material Overview

Butyl rubbers are copolymers of isobutylene with a small amount of isoprene added to allow for vulcanization. Figure 1 shows the chemical structures that are present in butyl rubber. The very low unsaturation of these elastomers exhibits excellent resistance to chemical and oxidative degradation. The tightly packed linear paraffin chains result in very low permeation rates of gases [1, 2] through the cured elastomer. These properties make butyl rubber the material of choice for many chemical agent protective clothing applications.

In general, the most important single property of butyl rubber is impermeability to air. This, of course, accounts for its use as inner tubes. Pharmaceutical closures, football and basketball bladders, and air-tight components in many other types of equipment also require this property. The heat resistance of butyl is used to advantage in tire curing bags and bladders and in a variety of mechanical goods. Resistance to ozone, weather, and moisture is utilized to a greater degree in roofing and reservoir membranes, electrical insulation, and automotive components.

The very low resilience of butyl rubber makes it the preferred material in applications calling for vibration damping—for example, in body mount systems for cars and trucks. Butyl rubber is resistant to swelling and degradation in animal and vegetable fats and oils and in some ester-based lubricants. It is also resistant to acids, bases, and oxidizing agents.

Butyl rubber is commercially vulcanized in two basic ways [3]: (1) accelerated sulfur vulcanization and (2) vulcanizing with sulfur donating compounds such as tetramethyl thiuram disulfide (TMTD). Accelerated sulfur-cured compounds are similar to the cure system utilized for natural rubber, where the reactive isoprene groups are equivalent. The major difference is that butyl rubber has an isoprene concentration of 1–3%, where natural rubber is composed of
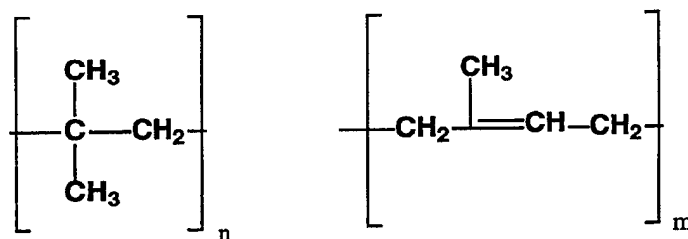
Butyl Rubber Structures

Figure 1. Chemical Structure of Butyl Rubber.

100% isoprene groups. This cure system forms cross-linked elastomers, which can vary in the number of mono-, di-, tri-, and tetra-sulfidic cross-links. Generally, accelerated sulfur cures are much faster than their sulfur donor counterparts. The tri- and tetra- cross-links have a tendency to undergo mechanical reversion, which is a result of unzipping the polymer cross-links at elevated temperatures [4, 5]. However, it also produces a homogeneous and uniform final end product.

Sulfur donor cures are carried out without the presence of elemental sulfur. Sulfur donor-cured compounds utilize curing agents that are primarily composed of mono- and di-sulfide groups, which replace elemental sulfur. These agents promote the formation of simpler monosulfidic cross-links. Hence, the resultant compound exhibits superior aging and low-compression set properties.

Figure 2 shows the chemical structures formed during the vulcanization process for the two curing systems studied. Analysis of the structures of thiuram vulcanizates show that they contain a variety of cross-links [6], carbon-carbon, and mono- and di-sulfide cross-links. These are responsible for good thermal and aging characteristics as well as high strengths in the final vulcanization.

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SULFUR CURE
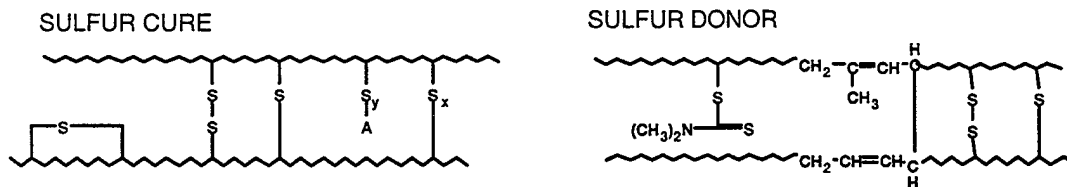
SULFUR DONOR

Figure 2. Chemical Cross-Linking Structures for Accelerated Sulfur-Cured and Sulfur Donor-Cured Butyl Rubber Compounds.

The most common butyl rubber cure system is the accelerated sulfur cures. This system uses a combination of zinc oxide and elemental sulfur with a highly active thiuram accelerator.

Generally, the cross-link strengths and thermal stability exhibit the following trend (in descending order).

-C-C- >> -C-S-C- > -C-S-S-C-

The accelerated sulfur cure system promotes formulation of polysulfidic cross-link structures. These tend to be thermally unstable and can sever to form pendant side groups. The severing of the **S-S** cross-links is the basis of the reversion process:

The physical, chemical, and compounding properties of butyl rubber will vary with the molecular weight, amount of unsaturation, and the degree of branching or chemical modifications occurring in the polymer. Polymers of this type have very little tendency to crystallize, like natural rubber, and depend on molecular entanglements or cross-linking for their strength. The completely amorphous character of butyl rubber allows a greater amount of chain mobility to occur, which results in a highly flexible shock-resistant end material. Their low glass transition temperature ($-60°$ C) yields an elastomer with very good low-temperature properties.

Because of its low unsaturation, butyl rubber is not compatible with other sulfur-curing elastomers of high unsaturation nor with high unsaturated plasticizers and fatty acids. For this

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reason, low unsaturation oils and waxes are used as softeners to improve the low-temperature properties. Fillers, most commonly carbon black, and minerals are used to improve processing and vulcanization properties.

Network degradation and consequent loss of elasticity can result from the level of unsaturation of the base polymer. Hence, a slightly higher level of unsaturation may be required where long-term flexibility in needed. However, the increase in unsaturation of the base polymer makes the butyl rubber more susceptible to ozone cracking, a fact long known for the higher unsaturated rubbers.

Unpigmented butyl rubber will degrade quite rapidly when exposed to direct sunlight in the presence of air. Studies have shown that degradation occurs at the polymer surface when nonpigmented formulated butyl rubber is weathered. When butyl rubber is compounded with carbon black, the black can act as an extremely efficient absorber of ultraviolet light. In fact, no surface deterioration is observed in ordinary carbon black reinforced vulcanizates. Such compounds are completely immune to sunlight and rain for all practical purposes.

For maximum chemical resistance, vulcanizates should be formulated with high filler loadings, high reinforcement, and maximum state of cure. Chemically reactive fillers such as calcium carbonate should not be used.

For materials that require improved air and vapor permeability, the state of cure has little effect. This is because the gas molecules are small compared to size and space between cross-links. Filler particles are impermeable and are to be maximized. Plasticizers increase polymer chain mobility and hence decrease the gas permeability.

Butyl rubber remains flexible down to the region of the glass transition temperature but tends to be somewhat leathery or "dead" at temperatures ranging from $-70°$ C to $-10°$ C. When butyl rubber was first introduced, this behavior caused problems in inner tube performance under

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winter conditions. Then the problem was solved when Polysar developed base polymers with increased molecular weight (MW), a development that has become the industry standard. The higher MW analogs allow incorporation of higher amounts of plasticizers and hence improved rubber retraction at lower temperatures.

4. Experimental

4.1 Materials. The butyl rubber gum stock used was an Exxon 268 purchased from Exxon Chemicals, Linden, NJ, which contained 1.5% unsaturation along the polymer backbone. This base polymer was chosen because of its wide use in existing glove materials and high molecular weight. It allows for a more rapid rate of cure and higher filler loadings and provides more compounding latitude as well as better ozone protection. The carbon black was an N-339 (NIST 378). The black levels were varied between 0 to 55 parts per hundred parts rubber (pphr) as indicated. A zinc oxide/stearic acid combination was added to serve as the accelerator/activator. The sulfur donor cure system consists of TMTD and dithio dimorpholine (DTDM). This combination yielded elastomers with excellent scorch times as well as good thermal and aging characteristics. For the sulfur cure system, elemental sulfur was added with N-t-butyl-2-benzothiayole Sulfenimide (TBSI), an effective sulfur activator to carry out vulcanization. Zinc oxide serves as the activator for the cure. Stearic acid or other fatty acid compounds were not required for the vulcanization of butyl rubber compounds but was utilized in our formulations as a processing aid.

4.2 Compounding. The compounds were mixed in a Rheocord System 40 internal mixer using dual cam style four-wing rotors. The length of the mix cycle was determined by the amount of reinforcing filler (carbon black) added. The size and design of the mixer prohibited the rapid incorporation of the carbon black. In order to minimize the mix time, the rotor speed was kept at 40 rpm until all the components were thoroughly blended. Afterward, the rotor speed was increased to 60 rpm and held for 5 minutes to ensure adequate dispersion.

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Batch-to-batch uniformity was controlled by using the unit work concept. By integrating the torque-vs.-time cure curve, a totalized torque value was obtained. The totalized torque is an indication of the power used in mixing and can be directly correlated to the amount of batch-to-batch dispersion. It can also be used in scaleup procedures.

In order to prevent scorch, the compound was mixed in two stages. In the first stage, all components were added. The starting temperature was selected so that at the end of the mix, when the internal temperature was raised due to the energy of mixing, the final temperature would not exceed the recommended temperature but was high enough to ensure an adequate viscosity for good dispersion. In the first minute of the mixing process, the base polymer was added. In the second minute, the zinc oxide was added. The carbon black was then added in three increments to ensure proper dispersion. Other mixing methods were attempted, but the carbon black was not dispersed properly. The stearic acid was then added. The curatives were added in the second stage after the first stage was allowed to cool. The temperature was kept below 110° C at the end of this mixing cycle to prevent the onset of cure. Table 1 shows the compound formulations.

Table 1. Butyl Rubber Formulations

Components	Sulfur Cure	Sulfur Donor
Exxon Butyl 268	100	100
Carbon Black	0-55	15, 25, 35
Zinc Oxide	5.0	5.0
Stearic Acid	1.0	1.0
Sulfur	1 - 2	
TBSI	0.5,1,2	
DTDM		2.0
TMTD		2.0

4.3 Molding. Compression molding was selected for processing because of the ease of cure. The primary problem with compression molding is the difficulty in creating specimens with a

uniform thickness throughout the molded form. However, with a little practice, usable uniform specimens could be created.

The samples of a predetermined weight were placed in a heated two-roll mill and allowed to form a band. The thickness gap setting was adjusted until a sheet of the desired thickness was obtained. The sheet was rolled several times to ensure uniform thickness throughout the specimen. The final sheet was removed and placed in a preform for use in the compression molding press. The preform with specimen was compression-molded in an ASTM 6- × 6-in stainless steel mold at 165° C. The press used was a Tetrahedron MTP-250. The press was programmed with a set of four one-minute bump cycles and held at constant pressure until done. The bump allows for trapped air bubbles to be released.

4.4 Cure Monitoring. Test sheets (14 mil thick) were vulcanized in a press, and the maximum torque rise (t_{max}) was estimated from the rheographs obtained using a Monsanto R-100 ODR rheometer operating at 165° C.

4.5 Breakthrough Characterization. Breakthrough times were measured by using the MIL-STD-282 droplet test. This test was carried out using an acid amplifying dye embedded in Congo red filter paper. A visually observable red to blue color change of the paper was used for detection of the diffusing simulant [7, 8].

Figure 3 shows a schematic for the placement of the sample. The O-ring that was used to separate the top glass cover from touching the liquid droplet was made from a felt material. Figure 4 shows the box into which these samples were placed. The box was constructed from plexiglass. The test was run at 37° C.
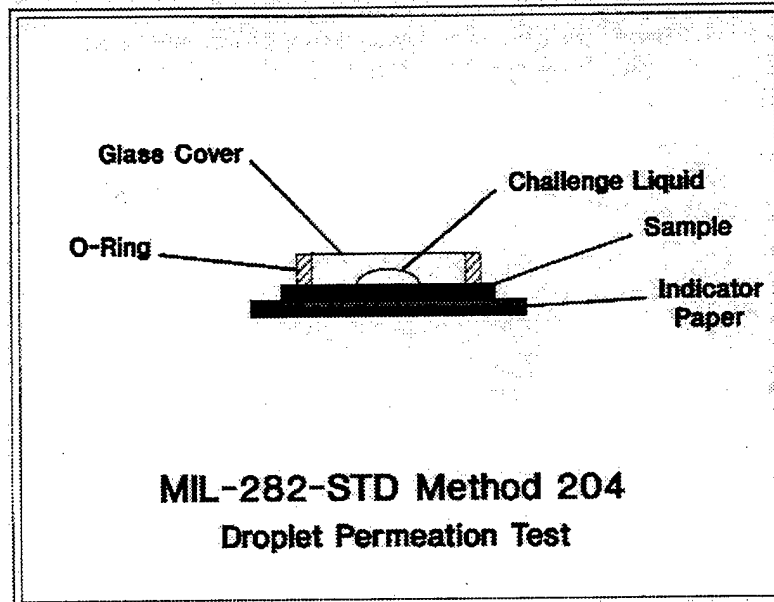
Figure 3. Schematic of the Sample Assembly for Breakthrough Evaluation.
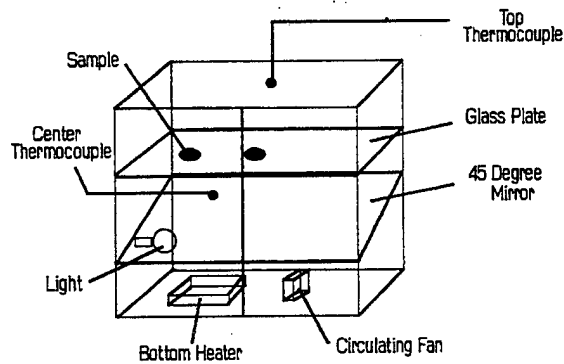
Figure 4. Schematic of the Sample Chamber.

5. Results and Discussion

5.1 Mechanical Properties. A mechanical spectrometer (rheometer) measures the increase or decrease in stiffness of a material over a specific time or temperature range. Cure

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characteristics of an elastomer can be monitored by following the increase in stiffness (known as modulus) over time at a specified temperature. The increase in modulus is a direct measure of the amount of cross-linking taking place in the compound as the three-dimensional network is forming. Figure 5 shows the rheometer cure curves for six butyl rubber compounds with various carbon black loadings, cured with the sulfur donor, TMTD. When cured, the sample containing no carbon black exhibits the smallest increase in modulus. It also exhibits the longest scorch time (i.e., time before initial increase in modulus is detected) of all the samples studied. Upon the addition of carbon black, a gradual increase in the maximum modulus is detected. This phenomena is also accompanied by a decrease in the cure times. Samples with greater than 45% carbon black show no more improvement in modulus.
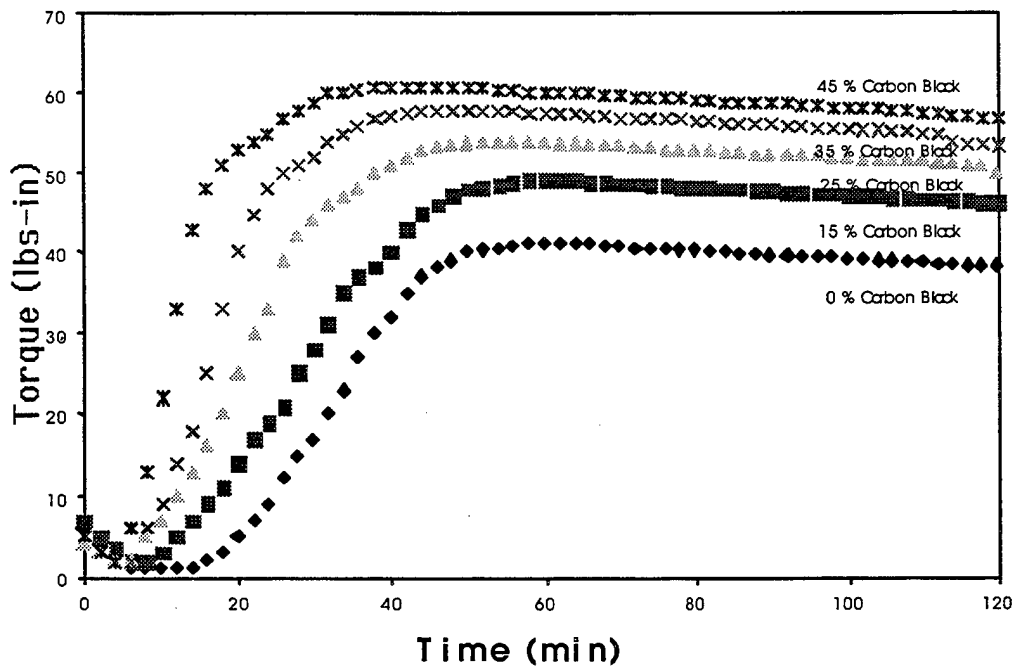

Figure 5. Rheometer Cure Curves for Six Butyl Rubber Compounds Cured With a Sulfur Donor Cure System.

5.2 Sulfur vs. Sulfur Donor Cure System. There are three main critical parameters related to vulcanization, the time elapsed before cure begins, the rate at which cure occurs, and the

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extent to which cure has taken place. Enough time must be allowed for the rubber components to mix, form, and mold. The scorch time is defined as the time before the onset of vulcanization. Hence, a processing time window would be established. Vulcanization is defined as the formation of the cross-linked network. This step should be rapid and end at the extent of cure.

Carbon black can act as both a reinforcing filler and as a thermal conductor. In fact, it is not uncommon for particulate fillers such as carbon black to increase the strength of vulcanized rubbers more than tenfold. It is hardly surprising that relatively few applications of elastomers utilize an unfilled polymer. The increase in modulus (or vulcanizate strength) is a result of the reinforcement effect of the carbon black and can be seen in Figure 5. This figure compares the cure curves for six butyl rubber compounds varying in carbon black content. The compounds all utilize a sulfur donor cure system. The modulus (G') increases with time as the three-dimensional cross-link network is formed. This increase is directly related to the interaction between rubber and carbon particles. The scorch time (processing window) can also be measured from this graph. The shorter cure time with increasing carbon black loading is the result of the ability of the carbon black particles to conduct heat into the bulk of the butyl rubber more efficiently, therefore obtaining a more rapid and complete cure.

Figure 6 shows the rheometer curve for butyl rubber cured with an accelerated sulfur system. This figure shows three different loading levels of carbon black. As with the previous figure, the higher carbon black levels give rise to a larger storage modulus.

Comparison of the accelerated sulfur cure system with the classic sulfur-cured system yields the observation that both cure systems undergo vulcanization in a similar manner. Both the maximum storage and scorch times are almost identical for compounds with comparable amounts of curatives and carbon black. Figure 7 shows a comparison of the cure time (as measured by the time required to achieve the maximum storage modulus). In the compounds studied, the accelerated sulfur cure system exhibited faster cure times. Clearly, the incorporation of accelerators to catalyze the cure reaction plays a vital role in forming sulfur cross-links.
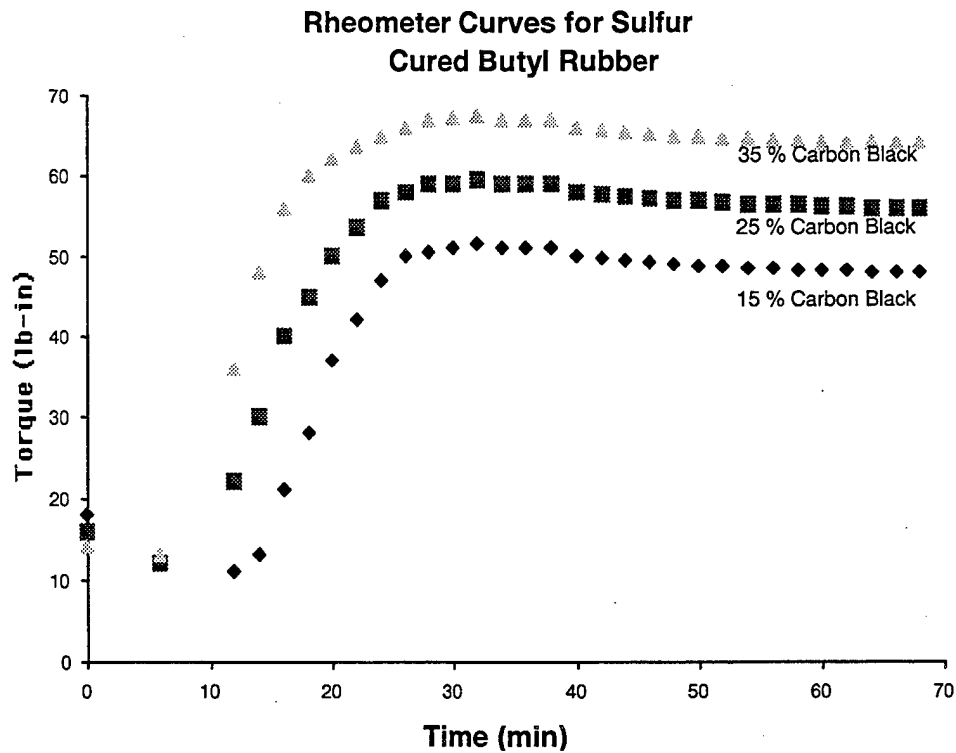
**Rheometer Curves for Sulfur
Cured Butyl Rubber**

Figure 6. Rheometer Cure Curves for Three Butyl Rubber Compounds Cured With an Accelerated Sulfur System.

**Effect of Total Cure Time
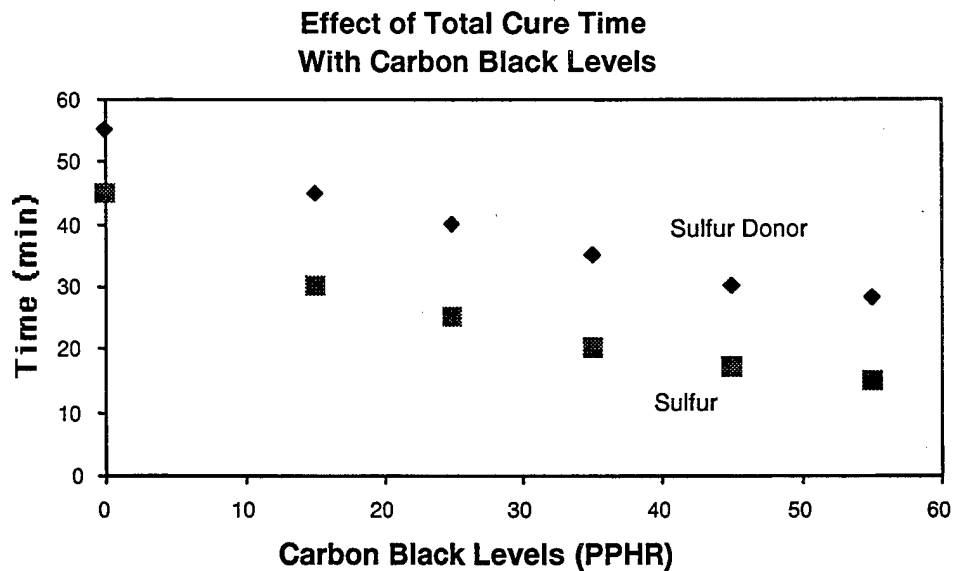With Carbon Black Levels**

Figure 7. Comparison of Cure Times for Accelerated Sulfur and Sulfur Donor Cure Systems.

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Another notable difference between cure systems is the amount of reversion or loss of mechanical properties. The amount of reversion can be observed by following the decrease in the torque after the maximum torque is attained. We can determine the amount of reversion occurring after 90 minutes by the following equation:

$$\%R = \frac{G'_{max} - G'_{90}}{G'}.$$

In this equation, G'_{max} is the maximum torque value obtained during the cure process, and G'_{90} is the torque value after 90 minutes.

Table 2 summarizes the results of the mechanical spectrometer for these two cure systems. The table compares two samples with 35% carbon black content.

Table 2. Rheometer Curve Summary

System	Cure Time (min)	Max. Modulus	Reversion (%)	Scorch Time (min)
Sulfur	22	85	14	4.5
Sulfur Donor	24	82	8	5.0

Reversion is postulated to occur when the initially formed mono-, di-, and polysulfidic cross-links [9] degrade through a process known as desulfurization. When desulfurization is faster than the cross-linking reaction, reversion occurs [6, 10]. This causes physical and mechanical properties such as tensile strength, stiffness, and tear resistance to deteriorate. Previous reports have demonstrated that elastomers containing isoprene units in the backbone are particularly susceptible to reversion. Butyl rubber contains 1–3% isoprene groups; thus, it has the capability of undergoing reversion.

To understand how the cure system affects the reversion process, an analysis of the structure of vulcanizates obtained from each cure system should be done.

Another method to characterize butyl rubber vulcanizates is by monitoring the amount of swelling that occurs when the butyl rubber is exposed to oil. The swelling index can be used as an indicator of relative cross-link density.

Figure 8 shows the swelling index data for three butyl rubber compounds. An increase in the swelling index indicates a lower cross-link density. The data demonstrates that the swell for the sulfur donor-cured butyl rubber vulcanizates is significantly less than that of the normal sulfur-cured system. The top two lines are normal sulfur cures where the amount of sulfur and accelerator were varied. The sulfur 2/2 contains two parts sulfur/two parts accelerator, and the sulfur 1/2 corresponds to one part sulfur/two parts accelerator. When the amount of sulfur is increased, more di-sulfide and tri-sulfide cross-links are formed. This has the effect of a less tightly formed cross-linked matrix.

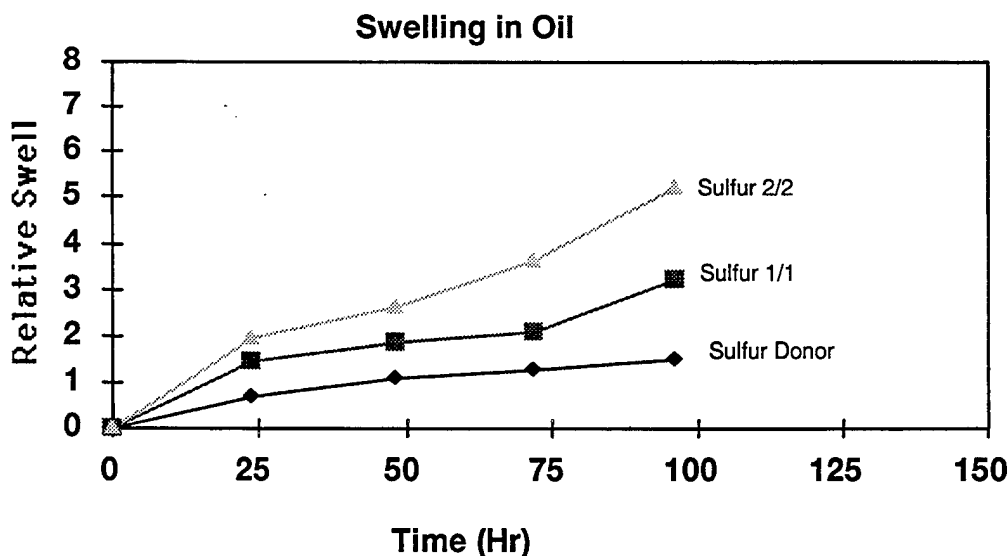**Figure 8. Swelling Index in Paraffin Oil for Three Butyl Rubber Vulcanizates.**

The cross-links formed by the sulfur donor system tend to be shorter (primarily monosulfidic), therefore, limiting the ability of oil diffusion into the rubber matrix. The normal sulfur-cured vulcanizate cross-links tend to be longer, resulting in a looser network structure, and allow transport of the oil into the rubber.

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Figure 9 shows the effect of the addition of sulfur on the swelling index. In general, keeping the sulfur content low and the accelerator content high yields butyl rubber vulcanizates with the best performance.

Effect of Sulfur Addition on Swelling
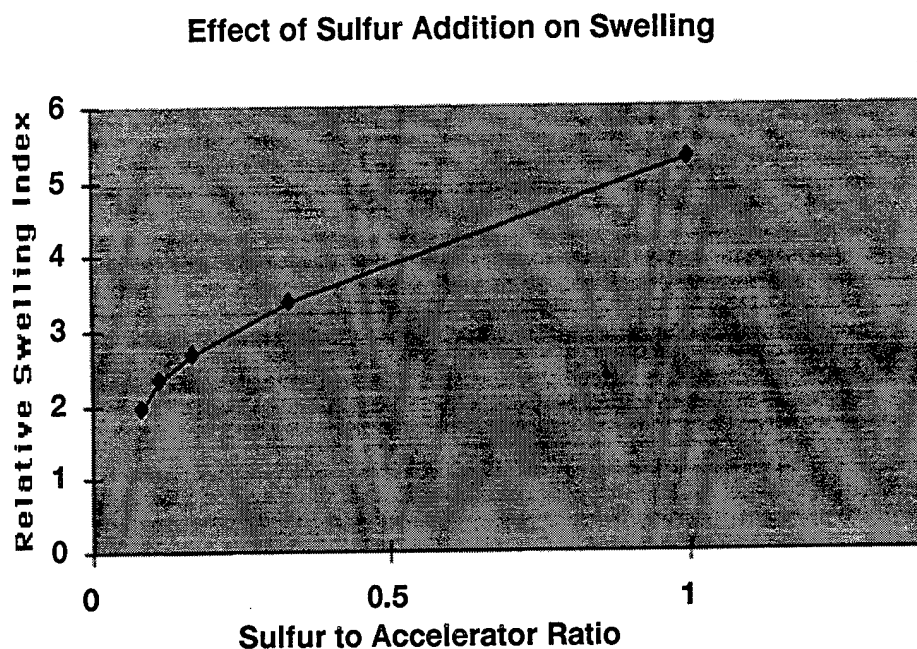
Figure 9. Effect of Sulfur-to-Accelerator Ratio on Swelling Index in Paraffin Oil.

5.3 Permeation.

5.3.1 Background. Permeation is defined as a process by which a diffusion molecule (permeant) is transported through a barrier. The mechanism of permeation is usually described as a three-step process that involves: (1) the sorption of the permeant on the outside surface, (2) diffusion through the bulk membrane, and (3) desorption of permeant from the opposite surface into a collecting medium [9].

5.3.2 Permeation Properties. To evaluate the protective properties of butyl rubber, the MIL-STD-282 droplet test was performed. For our studies, we used the chemical simulant

2-chloroethylethyl sulfide (CEES) or 2-chloroethyl-methyl sulfide (CEMS) as a replacement for the actual chemical agent, mustard gas (HD).

Table 3 gives a comparison of the physical constant for two simulants, CEES and CEMS, and the agent HD.

Table 3. Comparison of Physical Constants for Simulants and Mustard

	HD	CEES	CEMS
Molecular Weight	159	125	111
Density @ 20° C (g/cm³)	1.27	1.07	1.11
Molar Volume (cm³/mole)	125	117	100
Vapor Pressure (mmHg) @ 25° C	0.07	3.4	6.8
Boiling Point (Celsius)	217	157	55

After examination of Table 3, it was concluded that CEES would be a better choice for diffusion studies than CEMS, as the physical constants of CEES more closely resembled the physical constants of HD.

5.3.3 Effect of Challenge Liquid Concentration. Figure 10 demonstrates the dependence of the breakthrough time on the size of the droplet. The data are sulfur-cured butyl rubber specimens and are the same compounds taken on different days.

As expected, the breakthrough time decreases significantly with an increase in the amount of challenge liquid. Since the breakthrough time depends on the diffusion coefficient of the liquid in polymer and the diffusion coefficient is in most cases concentration dependent, the breakthrough time should, therefore, depend on the amount of challenge liquid.
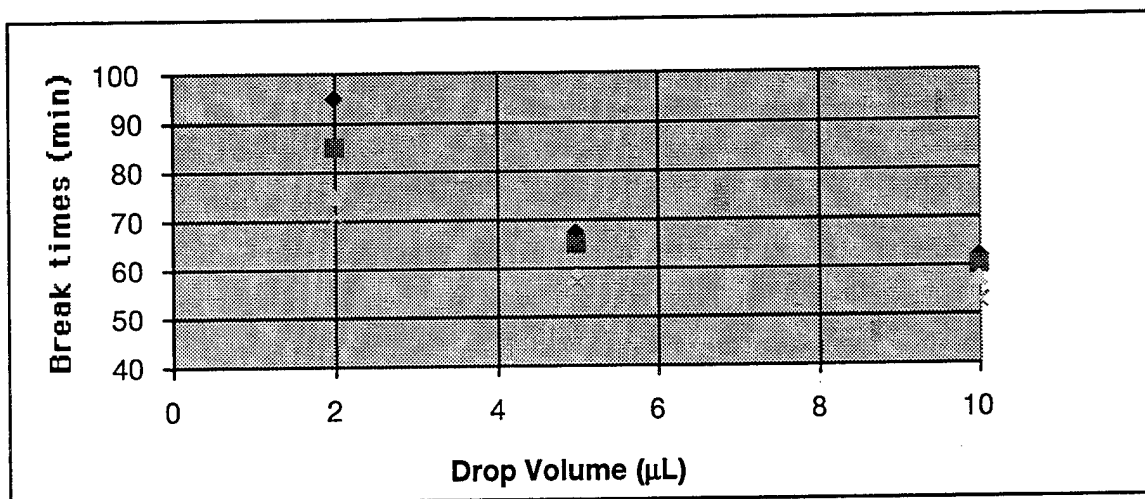
Figure 10. Effect of Drop Volume on Breakthrough Time.

An additional trend is also demonstrated in Figure 10, which is the scatter in the droplet data. Four experiments were run at each droplet volume. As much as a 20% difference was measured for experiments at any specific droplet volume. The scatter in droplet data was found in all droplet experiments.

In an attempt to identify the cause of this scatter, many experimental parameters were varied. Purification of the penetrants, activators, and extraction of the butyl rubber was performed to determine the cause of this scatter. No clear reason was found.

5.3.4 Effect of Carbon Black Levels. Figure 11 shows the effect of carbon black addition on the breakthrough times for both cure systems. A general trend exists where the addition of carbon black decreases the break time in a linear fashion for all samples formulated. This is not a surprising result, as this trend has been reported previously.

This graph also demonstrates that sulfur-cured butyl rubber has a tendency to exhibit shorter break times than does sulfur donor-cured rubber. This result is likely due to the cross-link network of each system. As shown in Figure 3, the sulfur donor-cured rubber has a tighter network structure. This is a direct result of the increase in C-C and monosulfidic cross-links.

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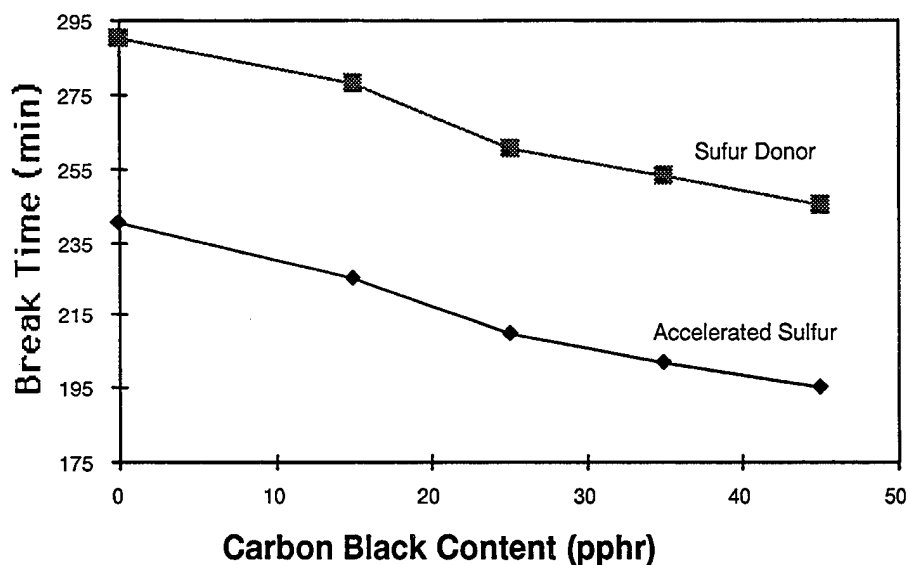
Figure 11. Effect of Carbon Black Addition on the Breakthrough Times for Both Cure Systems.

This, in turn, creates a more tortuous path for a diffusing molecule to pass through. In the more loosely cross-linked network—the accelerated sulfur-cured rubber—molecules will diffuse more easily. The net result is shorter break times.

Another parameter that plays an important role in the protectiveness of barrier materials is the membrane's thickness. Figure 12 shows this trend for four samples. All samples show an increase in break time with an increase in thickness. This increase follows the observed power-law dependence of break time with thickness [11, 12] as follows:

$$t_b = kL^n,$$

where t_b is the breakthrough time, k is a constant, L is the sample thickness, and n is an experimental factor.

Additionally, the four samples in Figure 12 show the effect of toluene solvent extraction on the thickness trend. The bottom curve is the thickness profile for the sulfur donor compound.

Figure 12. Variation in Break Times With Sample Thickness; Comparison of Extracted vs. Unextracted.

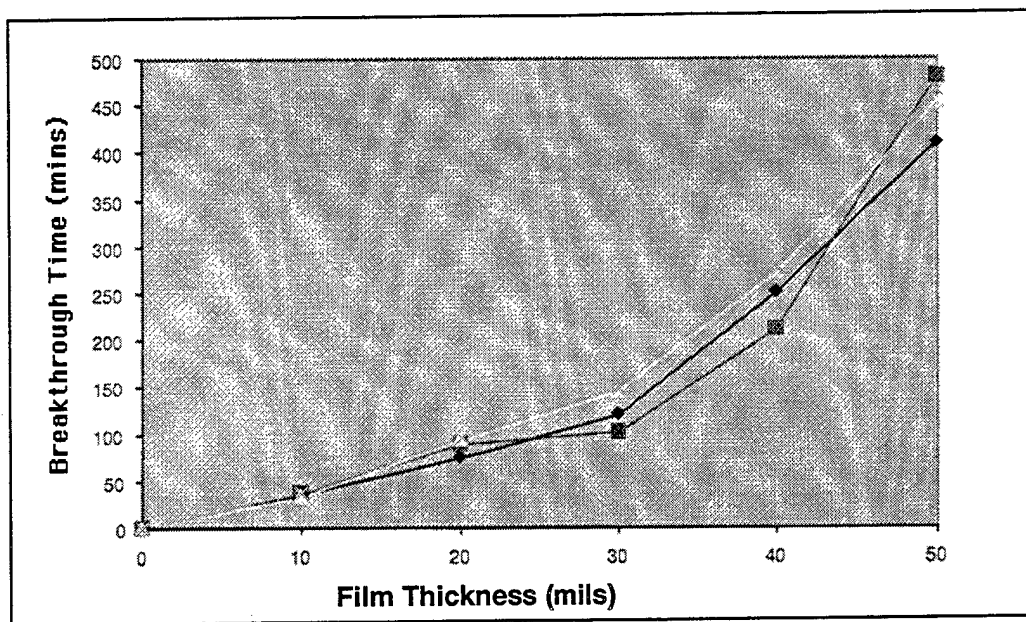This sample has the shortest break time of all samples tested. However, when the specimen is extracted, a dramatic increase in break time is observed. It has been shown that, when extracted, a premature break time results. This is clearly due to the TBSI curing agent, which contains enough sulfur to activate the acid indicator paper, thus creating a false endpoint. When these components are extracted in toluene and the extracted sheet is tested with the MIL-STD-282, the break time approaches those of the accelerated sulfur donor compounds.

In the case of the accelerated sulfur-cured compounds, no real difference in break time is observed after extraction with toluene. This is a clear indication that only the cross-linking network is the driving force for the simulant diffusion.

Finally, a comparison between the breakthrough times for agent HD and the simulant CEES can be made by comparing data supplied by the Engineering Directorate, Surety Team, Chemical Evaluation Laboratory, ERDEC. Table 4 compares the break times for the four compounds, which include two extracted and two unextracted. As observed in Figure 12, unextracted

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Table 4. Comparison of HD Agent and Simulant Break Times for Butyl Rubber

Batch	Thickness (mils)	HD (min)	CEES (min)
P	23	112 ± 12	43 ± 8
R	23	> 420	220 ± 35
25 L	21	> 420	200 ± 25
25 K	13	300 ± 40	145 ± 20

samples of sulfur donor compounds exhibit much smaller break times than unextracted. This is observed when both HD and CEES are used as test liquids. The accelerated sulfur compound samples show no differences.

A general trend exists where break times observed for CEES are approximately half those observed for HD. This is true for all samples tested. Presumably, differences in polarity and gyration radius between HD and CEES are responsible for these differences.

6. Summary and Conclusions

This study has compared different cure systems for butyl rubber. The ultimate goal was to develop butyl rubber compounds that can be utilized as standard reference materials for quality assurance testing. The cure characteristics for the cure systems evaluated were found to be almost identical when comparing scorch times, maximum modulus, and overall cure time. However, some difference in the amount of reversion occurring is evident. It has been shown that the accelerated sulfur-cured compounds show a tendency to undergo this process more than sulfur donor-cured compounds. Permeation testing has demonstrated that the sulfur donor compounds give erroneous break times when evaluated using MIL-STD-282. Clearly, this is a result of curing agents and/or accelerators prematurely blooming to the surface and yielding false endpoints. Generally, sulfur donor-cured compounds after extraction exhibited longer break times, most likely a result of a more tightly cross-linked network.

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This report is intended to summarize the research efforts at the U.S. Army Research Laboratory (ARL) and at the Materials Technology Laboratory, currently the Weapons and Materials Research Directorate (WMRD), to develop the standard butyl rubber compound. The strategy of this work was to compound- and compression-mold high-quality, uniform butyl rubber experimental sheets and to evaluate their cure properties, mechanical properties, and—most importantly—their protective barrier properties, as determined by the MIL-282 method 204 droplet breakthrough test performed at the Edgewood Research, Development, and Engineering Center Product Assurance Directorate (ERDEC-PAD). The test was performed at ERDEC with chemical agents in a qualified laboratory environment and with chemical agent simulants at ARL in Watertown, MA.

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