

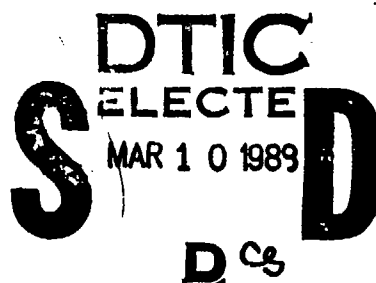
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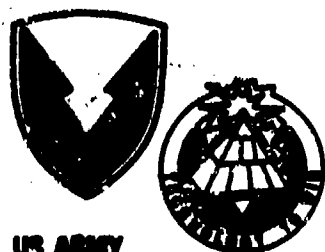
**STANDARD RUBBERS FOR
CHEMICAL DEFENSE RESEARCH
VOLUME I: FORMULATION AND PROPERTIES**

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POLYMER RESEARCH BRANCH



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ABSTRACT

There is need to establish a systematic materials data base, regarding interaction with chemical warfare (CW) liquids, to facilitate development of advanced materiel and to permit better understanding of basic interaction phenomena. Accordingly, the U.S. Army Materials Technology Laboratory (MTL) has conducted detailed CW liquid interaction studies with two sets of rubber materials obtained from commercial sources and designated by MTL as "standard rubbers." Volume I of this series of reports deals with the composition and properties of these rubbers, all of them having the many ingredients typical of commercial materials. *→ kg work →*

From MTL characterization of the two sets of rubbers, the hardness appeared to be related to both the glass transition temperature and the carbon black content. Additional MTL characterization or fingerprinting was performed with the second set of rubbers. Infrared spectroscopy of the pyrolyzates derived from the rubbers illustrated the presence of saturated aliphatic groups in all the rubbers and specific functional groups in many of the rubbers, correlating with the compositions of the known elastomeric components. Thermogravimetric analysis of the rubbers run to 850°C depicted a series of sequential weight loss processes, each one due to a specific group of ingredients, namely the volatiles, polymeric, and carbon black, leaving behind a residue composed of thermally stable inorganics.

The standard rubber compounds will continue to serve as materials for generating useful and systematic interaction data with CW liquids. Results will be described in Volumes II and III of this series of reports.

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INTRODUCTION

Purpose of Standard Rubbers

Materials based upon organic polymers (such as rubbers, plastics, coatings, adhesives, etc.) are found widely in materiel used by the military services. There is a growing need to determine the susceptibility of these materials to the chemical threats anticipated from the modern battlefield. Susceptibility might be defined as lacking one or more of three attributes or desirable characteristics of materials. These can be stated as: (a) resistance to permeation (good barrier), (b) resistance to sorption (low contamination), and (c) resistance to degradation (good stability). For a material to be compatible with a chemical warfare (CW) environment, it must embody a certain combination of these attributes, depending upon its function in the military system. For example, the primary requirement for a tank track pad or an engine hose would be (b), for an emergency shelter or protective cover, it would be (a). Attribute (c) would be important for all materials exposed to large quantities of decontaminating liquids.

To implement the development and acquisition of suitable materiel, Army Regulation 70-71, entitled *Nuclear, Biological, and Chemical Contamination Survivability of Army Materiel*, was issued in 1984. This document focuses on the need for contamination survivability of Army materiel, as defined by:

- a. Decontaminability: Capability of being rapidly decontaminated to reduce the hazard to a negligible level for unprotected personnel.
- b. Hardness: Capability of withstanding the damaging effects of nuclear, biological, chemical (NBC) contamination and subsequent decontamination.
- c. Compatibility: Capability of being operated, maintained, and resupplied by persons wearing the full NBC protective ensemble.

With proper systems analyses, the first two operational needs (decontaminability and hardness) should be directly translatable into material requirements for Army systems, and therefore should govern the assessment and improvement of appropriate materials for specific components.

Even with a knowledge of material requirements, it is difficult for systems developers to choose or to specify appropriate materials for particular end items because of the dearth of systematic data regarding interaction of materials with CW agents and decontaminants. Some preliminary experimental studies have been performed with rubbers and plastics exposed to CW liquids,¹⁻⁵ in order to screen rapidly a wide variety of possible candidate materials for end item use. However, there has been little effort to systematically relate such interactions to material properties and to specimen geometry. Moreover, there is little information at the present time about fundamental equilibrium and kinetic parameters of materials interaction with CW liquids. Therefore, it is not possible to extrapolate known interaction behavior to longer or shorter times, or to materials of somewhat different composition, or to specimens of different geometry, or, indeed, to relate the interaction behavior of one class of materials to another class. The existence of a systematic and detailed materials data base would facilitate the design and development of improved NBC survivable Army materiel.

1. SCHNECK, D., DAASCH, L. W., and BOUCK, J. E. *Corrosion/Compatibility Data on Chemical Agents and Their Decomposition Products*. ARCSL-TM-80003, Chemical Systems Laboratory, Aberdeen Proving Ground, MD, 1980.
2. QUINN, H. M., DAVIS, G. T., and WILCHER, I. *Compatibility of Plastics with Components of Decontaminating Solutions as Determined by Immersion Testing*. ARCSL-TR-80033, Chemical Systems Laboratory, Aberdeen Proving Ground, MD, March 1981.
3. ALBIZO, J. M., DAVIS, G. T., QUINN, H. S., and NITSUMA, B. J. *Compatibility of Plastics with Mustard (HD), Thiodiglycol, VX Hydrolysis Products, DS-2, HTH, and Tetrachloroethylene*. ARCSL-TR-80069, Chemical Systems Laboratory, Aberdeen Proving Ground, MD, February 1981.
4. *NBC Materials Handbook*. ROL 6109-1, Hughes Aircraft Co., Canoga Park, CA, January 1983.
5. *NBC (Nuclear, Biological, Chemical) Contamination Survivability: A Handbook for Development/Management of Material Programs*. Battelle Columbus Laboratories, Sponsored by CRDC, Aberdeen Proving Ground, MD, September 1985.

It is with these shortcomings and limitations in mind that the Polymer Research Branch of the U.S. Army Materials Technology Laboratory (MTL) set out to conduct detailed studies of the interaction of CW liquids with a limited number, but wide variety, of fully compounded rubber materials of known formulation. We have designated these materials as "standard rubbers" for the purpose of this investigation.

By "standard rubbers," we mean compounded rubbers of known formulation and processing, furnished in large enough quantities to provide specimens for all of the expected characterization and testing procedures, with additional amounts available for investigation by other laboratories. This will ensure that all of the tests will be performed with rubbers of fixed and known formulation. Such a standard collection will, therefore, permit meaningful comparisons of results, including various tests with a given rubber, and a given test with various rubbers. This is necessary to generate a consistent body of information which relates liquid interactions with formulation and properties of the rubbers, and which will serve as a prototype data base for rubber materials.

Outline of Topics for Each Volume

The MTL investigations with standard rubbers are described and summarized in this three-volume report, as follows.

Volume I: Formulation and Properties.

This volume lists the members of two sets of standard rubbers obtained by MTL, their recipes, characterization, and selected properties.

Volume II: MTL Immersion Tests with Decontaminants and Simulants.

This volume describes the MTL immersion testing of the rubbers with decontaminants and simulants, including tabulation of derived and estimated quantities of solubility, diffusivity, and permeability. Selected correlations between material interaction and material properties are illustrated. Limited comparisons are presented of MTL simulant immersion tests and prior CRDEC agent immersion tests for generically similar materials.

Volume III: MTL Analysis and Correlation of Contractor-Performed Droplet Permeation Tests.

This volume describes the agent and simulant droplet permeation testing of these rubbers by a contractor. The volume then presents the MTL treatment of the contractor data, leading to empirical expressions for the thickness dependence of breakthrough time, followed by correlations between breakthrough times, glass transition temperatures, and diffusion coefficients, as well as comparisons of simulant and agent breakthrough results.

Sources of Funding for MTL Investigation with the Standard Rubbers

During FY 82, 83, and 84, the MTL investigation with the standard rubbers was funded by the NBC Office at the U.S. Army Tank-Automotive Command, Warren, MI. This funding permitted acquisition of both sets of standard rubbers, MTL immersion testing of the first set, and establishment of the contract for agent and simulant droplet permeation testing.

During FY 85, 86, and 87, the MTL investigation was supported by the Physical Protection Directorate, U.S. Army Chemical Research, Development and Engineering Center, Aberdeen Proving Ground, MD. This funding permitted MTL immersion testing of the second set of standard rubbers, the MTL analysis of the contractor-generated droplet permeation test results, and MTL development of correlations between droplet permeation data, immersion data, and material properties.

REQUIREMENTS FOR STANDARD RUBBERS

As envisioned by MTL, the set of standard rubbers should encompass a wide-ranging series of typical rubbers, representative of those found in commercial and military applications. Because the total number of rubbers should be relatively small in order to maintain a reasonably sized test matrix, it was decided that there would be just one rubber compound based upon each generic type of elastomer or gum stock. In addition, these rubber compounds would each contain the usual large number of ingredients and would not be formulated systematically. Therefore, each rubber compound would represent a typical formulation within its generic class and it would not necessarily be related to the other members of the set. It was decided not to define the rubbers in terms of military specifications because such descriptions do not usually govern the complete formulation of the rubber compound. Consequently, the formulation or the recipe of a given military specification rubber may change over a period of time or might vary with the particular manufacturer.

It was necessary that each rubber compound be produced in thin sheets. This would reduce the time required for attainment of sorption equilibrium during the immersion tests and for attainment of breakthrough during the droplet permeation tests to a manageable level. It was also necessary that each sheet be of uniform thickness, because this dimension is of critical importance in calculating or comparing kinetic parameters of interaction, such as diffusion coefficients and breakthrough times, from test results.

Finally, it was necessary that the sheets of each rubber compound be furnished in several thicknesses. This would permit an empirical investigation into the effect of specimen thickness upon droplet breakthrough times. At present, the effect of specimen thickness upon droplet permeation behavior is not completely formulated or even known, due to the complexity of the process.⁶

Droplet testing is the principal means for determining materials response to CW agents. Droplet testing is employed because of the small quantity of hazardous liquid involved and because field or combat dispersal of agents produces a challenge in the form of droplet contact. Before developers of Army systems can specify types of materials and the geometries necessary to provide improved chemical contamination survivability, a practical knowledge of CW droplet permeation behavior must be available. This information would include the thickness dependence of droplet permeation for many kinds of materials used in protective garments, shelters, and packaging, as well as in components for vehicles and aircraft. These empirical thickness dependencies must be a part of any data base of materials interactions with CW liquids.

Because droplet permeation is so complex and yet is a vital element of materials testing, attempts are being made to develop mathematical models of the process.⁶ Droplet permeation depends upon the solubility and the unknown concentration-dependent diffusivity of the liquid in the rubber sheet. In addition, there are other undetermined parameters, such as the droplet radius (related to contact angle but changing with time) and the coefficients of evaporation of the liquid from the droplet and from the upper surface of the wetted sheet. Furthermore, there is the possibility that diffusion is not classical (Fickian) and that it involves polymer relaxation effects. These parameters and their interplay are specific to each penetrant/rubber combination and will control the overall permeation behavior.

In summary, empirical determinations of thickness dependence must be undertaken before a practical description of droplet permeation can be achieved and before mathematical models of droplet permeation can be verified.

6. ANGELOPOULOS, A. P., MELDON, J. H., and SCHNEIDER, N. S. *Numerical Simulation for the Permeation of Barrier Materials by Neat Liquid Droplets*. U. S. Army Materials Technology Laboratory, MTL TR 88-35, October 1988.

ACQUISITION OF STANDARD RUBBERS

First (Preliminary) Set of Standard Rubbers

A set of 13 fully compounded rubbers in sheet form was purchased from the Irving B. Moore Corporation, Cambridge, MA. As intended by MTL, each rubber compound was based upon a different generic type of elastomer or gum stock. The formulation or compound recipe for each rubber was chosen by I. B. Moore from rubber compounds then in production or development for commercial or military application, but not related to CW considerations, and not dependent upon whether or not any of the rubbers happened to be described by a military specification. The name (but not the number) given to each complete rubber compound was identical to the generic name of the corresponding base elastomer. The recipe or formulation for each rubber compound is listed in Appendix A. As evident, most of the rubber compounds are highly complex mixtures containing many ingredients, e.g., the base elastomer, curing agent, curing accelerator, reinforcing fillers, plasticizer, processing aids, antioxidants, etc. Because these rubbers were based upon a wide range of different elastomer types and were not formulated systematically, each rubber compound was not necessarily related to the other members of the set. Each rubber was to be produced in nominal sheet thicknesses of 0.015, 0.031, and 0.062 inches.

Unfortunately, it is extremely difficult almost beyond the state-of-the-art, to mold thin uniform sheets of conventional rubber compounds in sizes of six inches square or larger. This proved to be a continuing problem for all of the standard rubber compounds obtained to date. In particular, with this first set of rubbers, many of the individual sheets possessed excessive and nonuniform thicknesses, characteristics which magnified the difficulties in conducting and interpreting droplet permeation tests. In spite of these shortcomings and limitations, however, some characterization and testing was undertaken with this set of rubber compounds.

Second (Final) Set of Standard Rubbers

Owing to the problems cited above with the first set of rubber materials, MTL decided to acquire another set of rubber compounds to serve as standard materials. Accordingly, a series of eleven compounded rubbers was ordered from Smithers Scientific Services, Inc., Akron, OH. At MTL's request, the formulations of eight of these rubber compounds were based loosely upon the corresponding materials from I. B. Moore Corp. The formulations of two more of the compounds (natural rubber and SBR), were based upon previous materials made by Smithers for MTL for its track and suspension rubber investigation.⁷ The eleventh rubber, aflas, was formulated by Smithers. Each rubber compound was to be produced in three sheet thicknesses (0.040, 0.020, and 0.010 inch), except for the silicone which was requested in just the 0.040 inch thickness. Each sheet thickness was to be provided in the amount of eight square feet, i.e., 32 pieces, each piece being six inches square. Smithers compounded the uncured materials, sheeted them out on a two-roll mill, cut them into preforms, and then compression molded them in a heated four-sample cavity mold to produce the cured rubber sheet materials. For several reasons, Smithers encountered problems in fabrication of acceptable sheet rubber samples. The principal reason was the extreme thinness of the desired sheets. Standard rubber test sheet thicknesses are about 0.070 inch. The first attempts to mold our thin sheets suffered from inadequate and nonuniform flow of rubber to all parts of the mold before completion of the curing process. This frequently resulted in excessive and non-uniform sheet thicknesses, especially with the 0.010 inch sheets. Another problem was the hardness and rigidity of the rubbers as originally formulated in the specifications given to Smithers, leading to extreme difficulties in molding of the thin sheets, in particular the butyl, aflas, fluorocarbon, natural rubber, and vamac. Some of these were reformulated by Smithers to produce softer and more flexible compounds in the uncured state.

As a possible alternative to molding, Smithers then investigated the use of a cutting machine to provide thin slices from a 0.25-inch thick block of butyl rubber. Although this process produced sheets as thin as 0.018 inch with surprisingly good consistency and uniformity, the overall lack of surface smoothness and the inability to generate thinner sheets led us to reject this approach.

7. WILDE A. P. and MEE, C. P. *NBC-Resistant Track and Suspension Rubbers*. U. S. Army Materials Technology Laboratory, AMMRC TR 85-4, March 1985.

On a trial basis with butyl (one of the difficult formulations), Smithers molded some 0.010 inch-thick sheets by using carefully controlled preform dimensions and weight, as well as a reduced curing temperature and a longer curing time (to permit more flow in the mold before completion of crosslinking). This produced more uniformity in the sheets of butyl, normally difficult to mold. Based upon this approach to the more difficult rubber compounds, Smithers molded the remaining rubber sheet specimens, using the four cavity mold and a similarly altered cure schedule.

The final formulation or recipe for each rubber compound is listed in Appendix B. As before, the name given to each rubber compound was identical to the generic name of the corresponding base elastomer. The four-digit numbers are for procurement purposes only, and have no technical significance. Again, each of the rubber compounds contains many ingredients, whose functions are listed at the right-hand side of the recipe sheets. Owing to the wide range of elastomer types and the absence of systematic formulation, each of these rubber compounds is not necessarily related to the other members of the set.

CHARACTERIZATION OF STANDARD RUBBERS

Description

The various classes of rubbers, as represented by the standard rubber compounds, are briefly described in the following section. The rubbers are grouped in terms of the chemical nature of their elastomeric base material. For general information about rubbers, including formulations, properties, and uses, one can refer to books by Babbitt⁸ and Morton.⁹

Hydrocarbons

As the name implies, these are composed only of carbon and hydrogen and are essentially nonpolar. Consequently, they tend not to have good oil and gasoline resistance.

Natural Rubber - This elastomer is produced commercially from the latex of *Hevea Brasiliensis*, and is composed of isoprene units. The rubber undergoes strain-induced crystallization, leading to high tensile strength. Unsaturation in the isoprene causes susceptibility to oxidation. Natural rubber is used primarily in tires, mechanical goods, and latex products.

SBR - This elastomer is a copolymer of styrene and butadiene and is the most widely used synthetic rubber in the world. Unsaturation in the butadiene component promotes susceptibility to oxidation. Primary uses include tires, footwear, mechanical goods, and foamed products.

Butyl - This elastomer is composed of isobutylene and includes a small amount of isoprene for crosslinking. It is noted for its low permeability to gases and many liquids and for its resistance to weathering and to chemical attack. Primary uses of butyl rubber have been for inner tubes, electrical insulation, and as moisture barriers.

EPDM - This elastomer is a copolymer of ethylene and propylene, with small amounts of a diene monomer added for crosslinking. Its low unsaturation leads to good weather resistance. Its uses have been mainly in the automotive and tire fields and as modifiers for plastics and lubricating oils.

Fluorocarbons

These are produced mainly from fluorinated hydrocarbon monomers. Their partially fluorinated structures lead to polar materials, and the resulting rubbers are highly resistant to oils and lubricants. Their lack of unsaturation provides good resistance to sunlight and ozone.

8. *The Vanderbilt Rubber Handbook*. BABBIT, R.O. ed., R. T. Vanderbilt Co., Norwalk, CT, 1978.

9. *Rubber Technology*. MORTON, M. ed., Second Edition, Van Nostrand Reinhold Co., New York, 1973.

Fluorocarbon - This elastomer is a copolymer of vinylidene fluoride and hexafluoropropylene. Because of their fluid resistance and high temperature stability, fluorocarbon rubbers find application mainly as seals and gaskets in corrosive environments.

Aflas - This elastomer is a copolymer of propylene and tetrafluoroethylene. Aflas rubbers are resistant to heat and chemicals and have found use as gaskets, liners, and diaphragms, particularly where a combination of corrosive chemicals is encountered.

Silicones

These polymers are distinguished by their backbone compositions of alternate silicon and oxygen atoms. The silicon atoms, in addition, contain organic side groups. The backbone structure and lack of unsaturation leads to chemical inertness, resistance to ozone, and good high temperature stability. These rubbers exhibit very low glass transition temperatures, which provides good flexibility but which may also enhance the diffusivity of liquids and gases.

Silicone - This elastomer contains side groups such as methyl, vinyl, and phenyl, attached to the silicon in the backbone. These rubbers are used for insulating, potting, and encapsulation of electrical components, as well as for hoses, seals, and gaskets in high temperature environments.

Fluorosilicone - This elastomer contains fluorocarbon side groups attached to the silicon in the backbone. These rubbers are found in diaphragms, valves, and seals because of their good resistance to most fluids.

Miscellaneous

The polymers listed in this group are unrelated to each other, but they all encompass some degrees of polarity in their structures.

Neoprene - This elastomer is a polymer of 2-chlorobutadiene. It has the unsaturation of butadiene, thus requiring the use of antioxidants in its rubber formulations. The presence of the chlorine substituent leads to a polar material which has good oil resistance. The rubber tends to crystallize when stretched, thus leading to high tensile strength. Neoprene is widely used in electrical insulation, industrial hose, drive belts, footwear, and adhesives.

Hydrit - This elastomer is a polymer of epichlorohydrin. Its lack of unsaturation is responsible for inherent ozone resistance. The highly polar nature of this rubber leads to good oil and fuel resistance. The lack of crystallinity is responsible for relatively small values of low temperature compression set. This rubber is used in adhesives, electrical insulation, hose, and belting.

Vamac - This elastomer is a copolymer of ethylene and methyl acrylate, with a small amount of a cure site monomer which contains carboxylic acid groups. The polar nature of the methyl acrylate provides oil resistance. The lack of unsaturation in the polymer backbone affords good resistance to oxidation and weathering. Vamac is found in electrical insulation for elevated temperature application and is also used in hoses and seals.

Nitrile - This elastomer is a copolymer of butadiene and acrylonitrile. Commercial materials have acrylonitrile contents ranging from 20 to 50%, the higher contents leading to rubbers of greater oil resistance, but decreased resilience and increased stiffness. Due to the unsaturation of the butadiene, nitriles are not inherently ozone resistant. Nitrile rubbers are chosen primarily for their oil resistance, which is due to the polarity of the acrylonitrile component. Nitriles are used in fuel lines and hoses, seals, diaphragms, and oil-resistant clothing.

Hypalon - This is an elastomer of chlorosulfonated polyethylene, being essentially a hydrocarbon with relatively small numbers of chlorine and sulfonyl chloride substituents. Although an amorphous polymer, this material has enough polarity to exhibit good resistance to oil and grease. The lack of unsaturation provides resistance to degradation by weathering and ozone. Hypalon rubber finds application in electrical insulation, hoses for chemical delivery, and in weather resistant coated fabrics.

Urethane - These elastomers are block copolymers which contain hard segments (composed of diisocyanate and diol components) and soft segments (composed of preformed polyether or polyester components). The morphology is characterized by segregated hard phase microdomains dispersed in a continuous soft phase. The polar nature of these rubbers leads to good oil resistance. Polyurethane rubbers are versatile materials, possessing high tensile strength and excellent abrasion resistance. They find such diverse areas of application as solid industrial tires, footwear, and sealing and potting of electrical components.

Basic Characterization of First and Second Sets

Methods

Hardness - The hardness of the rubber compounds was determined by use of a Shore A durometer. Each test specimen consisted of six small pieces of sheet layered horizontally. The hardness values reported were the averages of five separate determinations.

Glass Transition Temperature - As a specimen is heated from a very low temperature, the rigid glassy material will undergo a relatively sudden change to a soft rubbery material at a characteristic temperature, called the glass transition temperature (T_g). Because all of these materials exhibit T_g values well below room temperature ($\approx 295^\circ\text{K}$), they all exist in the rubbery state at room temperature, although to various extents. This diversity of T_g values affects their relative kinetic responses to liquids, as will be evident in later volumes of this report.

To locate the T_g for each rubber, a 10- to 15- milligram specimen was heated at a rate of $20^\circ\text{C}/\text{min}$ in the Perkin-Elmer DSC-2 differential scanning calorimeter from 150 to 300°K (-123 to $+27^\circ\text{C}$). A relatively rapid shift of the baseline in the endothermic direction indicated the temperature at which the glass-to-rubber transition occurred. Each result listed is the average of three separate determinations.

Results for the First Set

Table 1 lists the 13 rubber compounds and presents some basic characterization results obtained by MTL. It is evident that the rubbers of lowest hardness have low values of T_g and/or are filled with SiO_2 . The rubbers of low T_g which also have high hardness (butyl and nitrile) also contain large amounts of carbon black filler. The rubbers with the

Table 1. STANDARD RUBBERS: SET 1
HARDNESS, GLASS TRANSITION TEMPERATURE, AND FILLER CONTENT.
FILLED WITH CARBON BLACK UNLESS OTHERWISE NOTED.

I.B. Moore Rubber Compound	Hardness, Shore A	Glass Transition Temperature		Filler Content, phr*
		$^\circ\text{K}$	$^\circ\text{C}$	
† Silicone S5E	51	157	-116	77**
† Fluorosilicone FS5C	58	203	-70	37**
Butyl B7A	75	206	-67	75
† Natural Rubber R4A	44	210	-63	15
Nitrile N7A	75	211	-62	15
Neoprene C6A	66	212	-61	65
Ethylene-Propylene EP8A	88	224	-49	160
Hydriol Y8E	61	227	-46	25
Styrene-Butadiene G6A	70	228	-45	43**
Vamac M6E	65	240	-33	42**
‡ Hypalon H8E	82	249	-24	80
Urethane P6E	76	250	-23	30
Fluorocarbon V7D	75	255	-18	30

*Parts per hundred parts base elastomer

†Filled with silica

‡Filled with mixture of carbon black and inorganic compounds

**Made from masterbatch of unknown filler content, thus filler content estimated from thermogravimetric analysis by MTL

highest T_g values (urethane and fluorocarbon) are relatively hard, in spite of their low contents of reinforcing filler. The hardest rubbers in this set (hypalon and ethylene-propylene) have moderate values of T_g and large amounts of filler. Therefore it appears that hardness of the rubber compound is related to both the T_g of the rubber and the amount of carbon black filler present in the rubber.

Because of the difficulties encountered with this set of rubbers (mainly from the overly large and irregular sheet thicknesses), MTL decided that this group of materials should be considered only as a preliminary set of standard rubbers. They are mentioned briefly in this report because some of the subsequent empirical relationships regarding liquid interactions tended to duplicate those found later with the second set of standard rubbers and, thus, served to increase the weight of evidence for these empirical correlations.

Results for the Second Set

Table 2 lists the 11 rubber compounds comprising the second set of rubbers and presents some basic characterization data generated by MTL. The rubber of least hardness (silicone) has the lowest T_g value and is filled with SiO_2 . The next softest rubbers (vamac and hydrin) have moderate values of T_g but very low filler contents. The natural rubber, butyl, and nitrile, although having low T_g values, exhibit moderate values of hardness because of very large filler contents. The fluorocarbon has moderate hardness, high T_g value and low filler content. The hypalon and atlas are the hardest rubbers of this set, with high T_g values and moderate filler contents. As with the other set, the hardness of these rubber compounds appears to be related both to the T_g and to the carbon black filler content.

Table 2. STANDARD RUBBERS: SET 2
HARDNESS, GLASS TRANSITION TEMPERATURE, AND FILLER CONTENT.
FILLED WITH CARBON BLACK UNLESS OTHERWISE NOTED.

Smithers Rubber Compound	Hardness, Shore A	Glass Transition Temperature		Filler Content, phr*
		°K	°C	
† Silicone 0003	54	149	-124	60**
Natural Rubber 0010	70	204	-69	75
Butyl 0001	72	208	-65	75
Nitrile 0004	75	217	-56	145
Neoprene 0005	67	218	-55	65
‡ Styrene-Butadiene 0011	75	223	-50	65
Vamac 0007	60	234	-39	35
‡ Hypalon 0006	80	245	-28	55
Hydrin 0008	62	249	-24	25
Fluorocarbon 0002	78	252	-21	30
Atlas 0009	83	263	-10	50

*Parts per hundred parts base elastomer

†Filled with silica

‡Filled with mixture of carbon black and inorganic compounds

**Made from masterbatch of unknown filler content, thus filler content estimated from thermogravimetric analysis by MTL

Ten of the rubbers in Set 2 are based upon the same generic elastomers as are found in Set 1. Of these, aside from the natural rubber and styrene butadiene rubber, eight of the rubber compounds in each set are formulated similarly to their namesake in the other set. As seen in Tables 1 and 2, the hardness and glass transition temperature are generally closely matched for the corresponding compounds. This indicates that with similar recipes, two manufacturers can produce rubbers with comparable physical properties.

Additional Characterization of Second Set

Tensile properties of the second set of standard rubbers were determined by Smithers, using 0.070 inch test sheets molded at the same time as the standard sheet materials. In addition to individual tabulation in Appendix B along with the rubber recipes, the tensile data are collected and summarized in Table 3.

**Table 3. STANDARD RUBBERS: SET 2
MODULUS, TENSILE STRENGTH, AND ELONGATION**

Smithers Rubber Compound	100% Modulus psi	Tensile Strength psi	Breaking Elongation %
Silicone 0003	220	1360	700
Natural Rubber 0010	200	2200	450
Butyl 0001	250	1720	440
Nitrile 0004	580	1500	190
Neoprene 0005	390	2630	330
Styrene-Butadiene 0011	520	1970	240
Vamac 0007	120	1920	650
Hypalon 0006	420	1850	560
Hydriol 0008	220	2380	610
Fluorocarbon 0002	470	1400	340
Aflas 0009	900	1520	200

A wide variety of property values is evident, ranging over a factor of seven for the 100% modulus. In general, there is an inverse relationship between breaking elongation and 100% modulus, showing that the stiffer rubbers are capable of less extension. On the other hand, the tensile strength does not appear to be related to the other two properties for this series of generically diverse rubbers.

Because further evaluation and correlations for standard rubbers were performed mainly with the second set, additional characterization tests are described and reported below for these materials.

A general approach to the characterization of materials involves the process of "fingerprinting." By this is meant the elucidation and recording of a set of characteristics that are peculiar to or unique for a given material. This set of characteristics or fingerprints can then be used for identification and tracking. For example, the fingerprints of an unknown material can be compared to those in a data base to determine if the unknown material is related to or identical to any material previously cataloged. Fingerprinting can be used as a means for quality control of materials during manufacturing processes or a given material can be fingerprinted at intervals throughout its service life or storage life to determine if significant changes occur in its composition or properties. As part of the characterization of the second set of standard rubbers, two kinds of fingerprinting tests were conducted, infrared spectroscopy and thermogravimetric analysis.

Methods

Infrared Spectroscopy (IRS) - With opaque materials, one method for obtaining infrared spectra involves the use of attenuated total reflection (ATR). Here, the IR beam undergoes multiple reflections from the specimen surface before it reaches the detector. However, for compounded rubbers filled with carbon black, this method would present difficulties. The presence of numerous compounding ingredients would lead to very complex spectra. In addition, the carbon black particles would both scatter and absorb most of the incident IR beam, leading to a very weak signal which contains little spectral information.

An alternative method involves very rapid pyrolysis of the rubber specimen to provide a sample for IR examination. Fragments of the elastomeric portion are collected on a salt plate. The low molecular weight additives do not condense on the plate. Nonvolatile materials, such as carbon black, are left behind. The sample to be characterized by IR now contains just the fragmented polymer in a very thin layer, suitable for IR transmission measurements.

The MTL method generally followed the procedure outlined in ASTM D 3677-83. After nitrogen gas purge of the pyrolysis cell, a small chunk of the rubber compound was heated at 600°C for 60 seconds by use of a Foxboro Pyro-Chem apparatus. The pyrolyzate was collected on a potassium bromide salt plate. An infrared spectrum of the pyrolyzate was then obtained with a Perkin-Elmer Fourier transform infrared spectrometer, model 1550.

Thermogravimetric Analysis (TGA) - The MTL procedure generally followed the method described in ASTM E 1131, with examples given in a recent MTL technical report.¹⁰ TGA of the rubber compounds was performed with a Dupont 951 TGA module in conjunction with a Dupont 9900 thermal analyzer. A small piece of 40 mil rubber sheet was sliced perpendicularly to the plane of the sheet to give four or five pieces of specimen each less than 1 mm thick. These were placed randomly in the balance pan and provided total specimen weights ranging from 13 to 24 milligrams.

The specimen was heated at a rate of 10°C/min from room temperature to 850°C, then held isothermally for two minutes at this final value. Up to 550°C, the sample was continuously flushed with argon gas to prevent oxidation. Above 550°C, the sample was continuously flushed with air to permit oxidation and removal of the carbon black filler. Sample weight was automatically and continuously recorded during the run to track the progressive weight loss due to removal of various components from the rubber compound.

Results

Infrared Spectroscopy - The transmission spectra of the rubber pyrolyzates provided fingerprints of each base elastomer in the rubber compounds. The spectra are shown in Figures 1 through 11. Each spectrum is a plot of percent transmission versus wave number (cm^{-1}). Table 4 gives a summary of some of the characteristic absorption bands observed in the spectra. Each band is described in terms of its approximate wave number, its assignment, and the rubber pyrolyzate in which it was detected.

All rubbers exhibited C-H stretch bands, indicating the presence of saturated aliphatic groups. All rubbers except silicone possessed $-\text{CH}_2-$ deformation bands, suggesting that the only saturated aliphatic groups in silicone were $-\text{CH}_3$. The presence of C=C unsaturation was seen mostly for those rubbers which were produced from diene monomers, presumably not all of whose additional double bonds became involved in the subsequent crosslinking process. These are exemplified by butyl and natural rubber (both containing isoprene) and by nitrile and SBR (both containing butadiene). The butyl, aflas, and natural rubber displayed presence of C- CH_3 groupings, as would be expected from their precursors of isobutylene, propylene, and isoprene, respectively. The SBR exhibited bands due to an aromatic constituent, namely styrene.

Additional significant IR absorption bands were due to the presence of functional groups containing atoms other than carbon and hydrogen. Vamac showed presence of the ester side chain by the C=O stretch and C-O stretch. Hydrin displayed the C-O-C stretch due to its ether linkages. The presence of C-F stretch was seen in the fluorinated rubbers (fluorocarbon and aflas). Silicone displayed bands due to Si- CH_3 and Si-O. The presence of C $\equiv$ N stretch was evident in the spectrum of nitrile. The hypalon showed a band which may suggest the presence of the $-\text{SO}_2\text{Cl}$ group, as would be expected from a rubber based upon chlorosulfonated polyethylene. Finally, all of the rubbers except silicone showed evidence of carbonyl groups, suggesting that some oxidation occurred during the pyrolysis process, probably due to traces of air in the pyrolysis cell.

In addition to the band assignments listed in Table 4, there were other bands observed in the MTL spectra that matched those listed in ASTM-D-3677-83 and that were labelled by ASTM as diagnostic for pyrolyzates of particular rubbers. Six of the rubber pyrolyzates identified in ASTM-D-3677-83 were generically the same as those examined by MTL. The following ASTM diagnostic bands (cm^{-1}) were observed by MTL:

- butyl: 1370, 1390, 885, 1250-1220, 727.
- acrylonitrile-butadiene (nitrile): 2220, 962, 1610, 909.
- chloroprene (neoprene): 820, 747, 769, 885, 699.
- chlorosulfonated polyethylene (hypalon): 909, 962, 990, 813, 741, 720, 695.

10. SACHER, R. E., MACAIONE, D. P., and SINGLER, R. E. *Thermal Analysis Characterization of Elastomers and Carbon Black Filled Rubber Composites for Army Applications*. U. S. Army Materials Technology Laboratory, AMMRC TR 85-14, June 1985.

Table 4. SUMMARY OF INFRARED ABSORPTION BANDS OBSERVED
WITH MTL STANDARD RUBBERS: SET 2

Approximate Wave Number of Band, cm^{-1}	Band Assignment*	MTL Rubber Pyrolyzate for which Band was Detected
3000-2900	Saturated Aliphatic C-H Stretch	All Rubbers
1470-1430	-CH ₂ - Deformation	All Rubbers Except Silicone
3130	-CH=CH- CH Stretch and Deformation	Butyl, Nitrile
1640	Nonconjugated C=C Stretch	Butyl, Nitrile, Natural Rubber, Vamac, SBR
1390-1340	C-CH ₃ CH Deformation	Butyl, Aflas, Natural Rubber
1170	$\text{C} \begin{smallmatrix} \nearrow \text{CH}_3 \\ \searrow \text{CH}_3 \end{smallmatrix}$ Skeletal	Butyl
1740	Saturated Ester C=O Stretch	Vamac
1170	Ester C-O Stretch	Vamac
1115	Alkyl Ether C-O-C Stretch	Hydrin
1300-1100	C-F Stretch	Fluorocarbon and Aflas
1260	Si-CH ₃ Deformation	Silicone
800	$\text{Si} \begin{smallmatrix} \nearrow \text{CH}_3 \\ \searrow \text{CH}_3 \end{smallmatrix}$ Rock	Silicone
1100-1000	Si-O Stretch	Silicone
2240	Saturated Alkyl Nitrile C $\equiv$ N Stretch	Nitrile
3030	Aromatic =C-H Stretch	SBR
700	Aromatic C-H Out-of-Plane Deformation	SBR
1600 and 1500	Aromatic C=C Skeletal	SBR
1375	Sulfonyl Chloride (Possibly) C-SO ₂ Cl	Hypalon
1760-1700	Carbonyl Vibrations	All Rubbers Except Silicone. Probably Due to Oxygen Contamination During Pyrolysis.

*Assignments made at MTL, with additional documentation taken from book by Bellamy¹¹

11. BELLAMY, L. I. *The Infrared Spectra of Complex Molecules*. Second Edition, John Wiley and Sons, Inc., New York, 1958.

- isoprene (natural rubber): 885, 1370, 1640.
- styrene-butadiene: 699, 1490, 909, 990, 962.

It was evident that the MTL spectra displayed nearly all of the ASTM diagnostic absorption bands for these six rubber pyrolyzates. The five remaining types of rubber examined by MTL were not addressed by ASTM-D-3677-83.

Thermogravimetric Analysis - A typical TGA record is displayed in Figure 12, showing two traces as a function of temperature. The first trace (specimen weight) runs from upper left to lower right, indicating three principal weight loss processes. These are (a) loss of volatile components, (b) loss of elastomer and other polymeric species, and (c) loss of carbon black (and any other carbonaceous material) due to oxidation by air, leaving the ash or residue at the end of the run. The other trace is the derivative curve (% weight loss per minute), showing three peaks, one for each of the principal weight loss processes, and each occurring at the time of most rapid weight loss.

The TGA records for the 11 standard rubbers are given in Figures 13 through 23. The shapes, magnitudes, and positions of the humps and peaks for each rubber compound serve as the thermal fingerprint for that compound. The weight loss results computed from these curves are summarized in Table 5. Here are listed for each rubber compound the apparent groupings of components according to their volatility during the run. The elastomer base appears to be the predominant component, followed by the carbon black filler (except for the silicone).

Table 5. STANDARD RUBBERS: SET 2
SUMMARY OF MTL THERMOGRAVIMETRIC ANALYSIS WEIGHT LOSS RESULTS

Smithers Rubber Compound	Estimated Weight Percent			
	Volatiles	Elastomer and Other Polymers	Carbon Black	Ash or Residue
Silicone 0003	2.6	60.6	0	36.1*
Natural Rubber 0010	6.3	53.9	36.9	2.7
Butyl 0001	2.9	56.4	36.4	4.2
Nitrile 0004	0.3	43.7	53.3	2.4
Neoprene 0005	1.4	47.6	48.5	2.4
Styrene-Butadiene 0011	7.9	56.0	27.2	8.8†
Vamac 0007	5.1	67.9	26.6	0.5
Hypalon 0006	1.5	62.6	19.9	14.8†
Hydriol 0008	0.9	68.6	25.1	5.0
Fluorocarbon 0002	1.1	63.6	25.6	7.5
Aflas 0009	1.6	62.8	33.1	2.4

*Filled with silica

†Filled with mixture of carbon black and inorganic compounds

For comparison, a similar grouping of rubber compound components is given in Table 6, computed by MTL from the recipe for each rubber. The agreement between TGA analysis and rubber recipe varies noticeably. For example, with neoprene the agreement is not very good, with large discrepancies evident. Conversely, with SBR the agreement is quite satisfactory, with no discrepancy larger than 1.7%. In general, the TGA indicated lower than expected values of volatiles and polymer, and higher than expected values of carbon black. This suggests that loss of volatiles and polymer was partly delayed, with portions of them forming a carbonaceous char which did not escape until subsequent oxidation along with the carbon black filler after introduction of air at 550°C.

For TGA determinations to be as accurate for all of the rubbers as for the SBR, it would be necessary to employ a variety of TGA heating regimens, each one tailored specifically for a particular class of rubber. Such an optimized routine would have to be developed empirically for each rubber by trial and error to permit accurate determination of all the groups of components found in each rubber formulation.

Summary of Fingerprinting Characterization - Although general purpose spectroscopic and thermogravimetric examinations do not necessarily give precise measures of the compositions of such varied and highly complex rubbers,

they can generate useful fingerprints of these materials. The fingerprints can be used to distinguish between various classes of rubbers, to provide a means of quality control of manufactured rubber items, and to investigate the long term stability of these compounds.

Table 6. STANDARD RUBBERS: SET 2
APPROXIMATE COMPOSITION ACCORDING TO THE RUBBER COMPOUND RECIPES

Smithers Rubber Compound	Approximate Weight Percent			
	Non Polymeric Organics	Elastomer and Other Polymeric	Carbon Black	Thermally Stable Inorganics
Silicone 0003	Complete Recipe Not Available			
Natural Rubber 0010	12.5	49.4	38.1	1.9
Butyl 0001	7.4	53.1	38.8	2.7
Nitrile 0004	9.0	38.8	50.4	1.7
Neoprene 0005	11.3	51.7	32.6	4.5
Styrene-Butadiene 0011	9.6	55.3	28.4	8.6*
Vamac 0007	7.8	64.5	27.7	0
Hypalon 0006	8.2	59.0	16.4	16.4*
Hydriol 0008	2.2	75.0	18.4	4.4
Fluorocarbon 0002	0	72.8	21.2	6.0
Aflas 0009	0	70.4	29.6	0

*Filled with mixture of carbon black and inorganic compounds

SUMMARY

Owing to the lack of systematic data regarding the interaction of materials with CW liquids, it is difficult for systems developers to specify appropriate materials for Army application. Accordingly, MTL has been working with a wide variety of "standard rubbers" regarding their properties and their interactions with CW liquids. Volume I of this series of reports deals with the requirements for standard rubbers and describes the formulation and properties of two sets of such rubber materials.

The manufacturers' formulations of these rubbers indicate their complexity, due to inclusion of the many ingredients required for suitable processing, functional, and durability characteristics of commercial materials. Because of difficulties in molding thin sheets, specimens of both sets were subject to irregularities, such as excessive and non-uniform thickness, the second set to a lesser degree than the first set. MTL determined the hardness and the glass transition temperature of each rubber. Results for the two sets indicated that the hardness was related to both the carbon black content and the glass transition temperature.

Additional MTL characterization or fingerprinting was performed with the second set of rubbers. Infrared spectroscopy of the pyrolyzate derived from the rubbers depicted the significant absorption bands of the corresponding elastomeric components. All of the rubbers displayed the presence of saturated aliphatic groups. Unsaturation was evident in those rubbers having diene precursors. For certain rubbers, the presence of corresponding functional groups was noted, i.e., phenyl, ester, ether, fluorine, silicon, nitrile, and sulfonyl chloride. For nearly all of the rubbers, there was evidence of carbonyl groups, apparently due to oxygen contamination during the pyrolysis treatment.

MTL thermogravimetric analysis of the rubbers delineated a series of weight loss processes, indicating the presence of groups of components, such as volatiles, polymeric, carbon black, and thermally stable inorganics in each rubber compound. Quantitative comparison with the rubber formulations showed that agreement was uneven for these groupings of components. This indicated that a given standardized heating regimen is not necessarily optimized for quantitative analysis of each of the widely varied rubber compounds in this set.

These standard rubber compounds have been, and will continue to be, a useful series of materials. Their composition and processing are well defined and documented. They have served MTL well as test specimens for interaction studies with CW liquids, as described in Volumes II and III of this series of reports. These rubbers, in gumstock and in

cured form, have also been made available to other Army laboratories for specialized testing. Such investigations will involve CW liquid interactions with gumstock and with cured rubber, as well as permeation through rubber sheets of various thicknesses.

ACKNOWLEDGMENT

Appreciation is expressed to the following members of the Polymer Research Branch for their contributions to the program: Nathaniel S. Schneider for helpful discussions and suggestions during procurement of the standard rubber sheet materials and during preparation of this report; Emily A. McHugh for the hardness tests and the runs with the differential scanning calorimeter; James M. Sloan for the infrared spectroscopic examinations and the absorption band assignments; and Domenic P. Macaione for the thermogravimetric analyses and the subsequent data reduction.

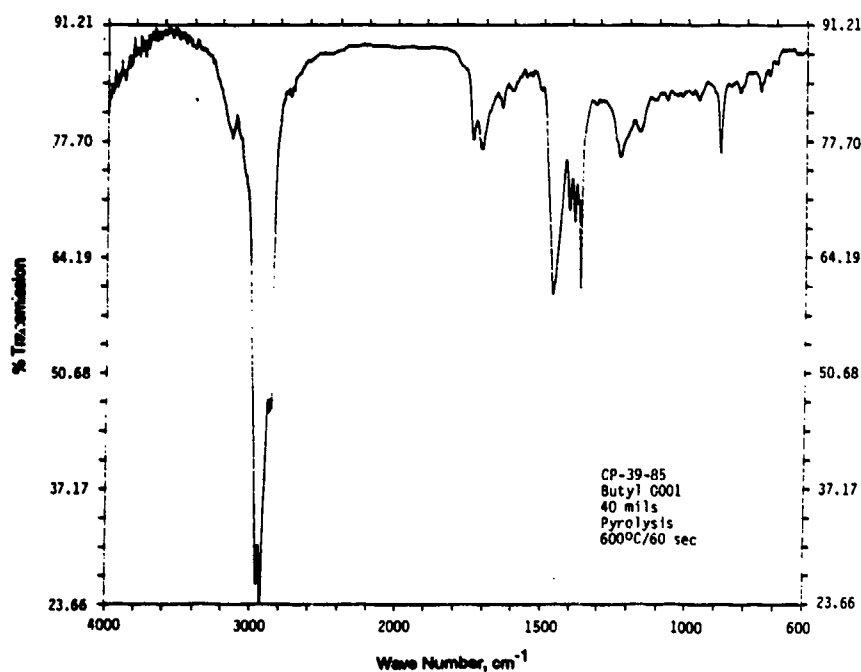


Figure 1. IR spectrum of Butyl 0001 pyrolyzate.

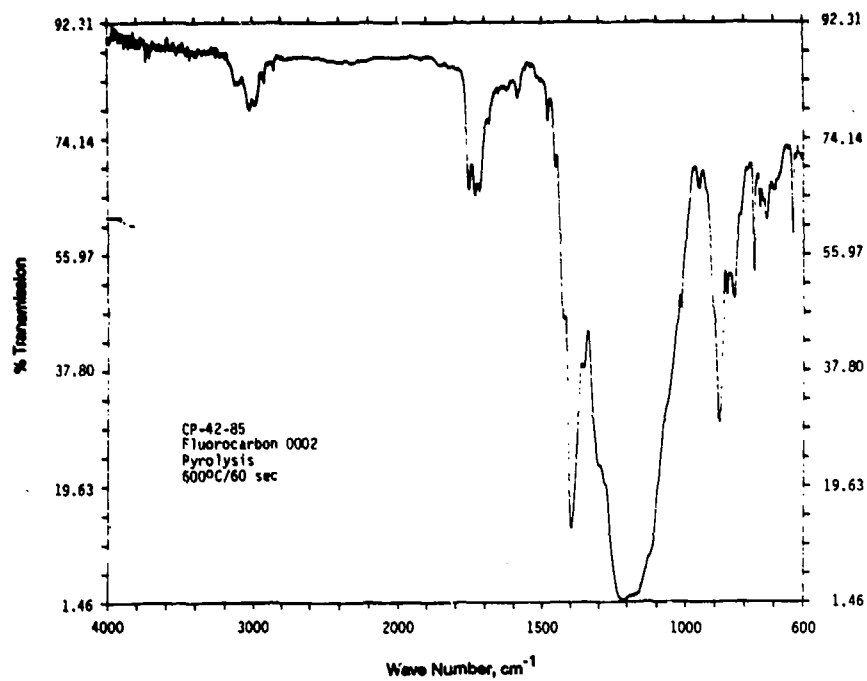


Figure 2. IR spectrum of Fluorocarbon 0002 pyrolyzate.

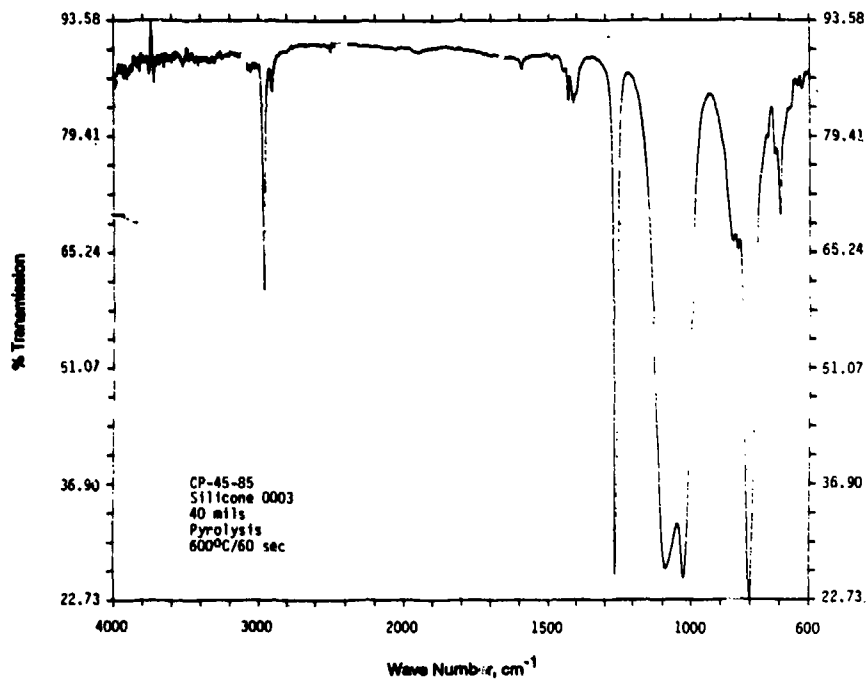


Figure 3. IR spectrum of Silicone 0003 pyrolyzate.

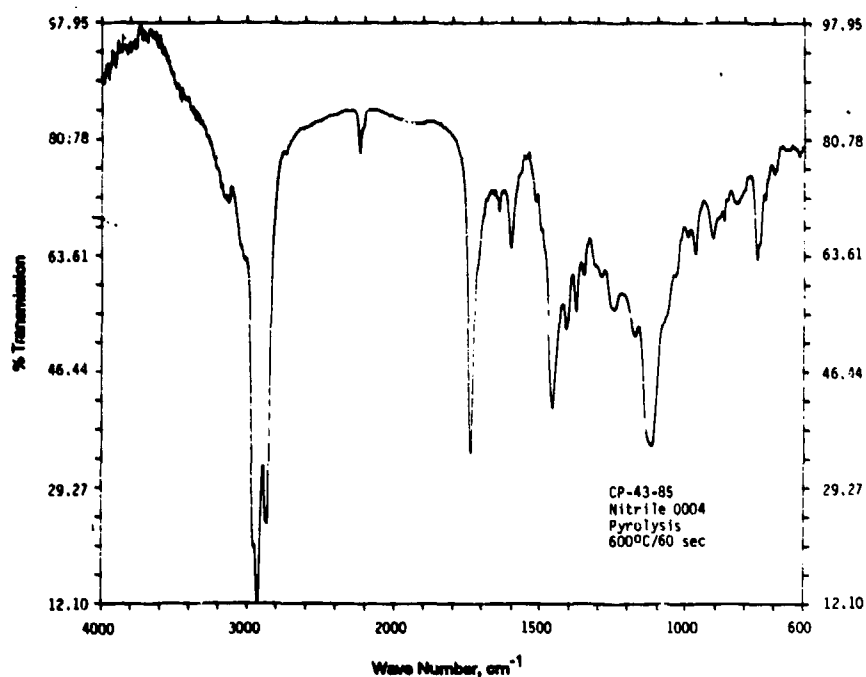


Figure 4. IR spectrum of Nitrile 0004 pyrolyzate.

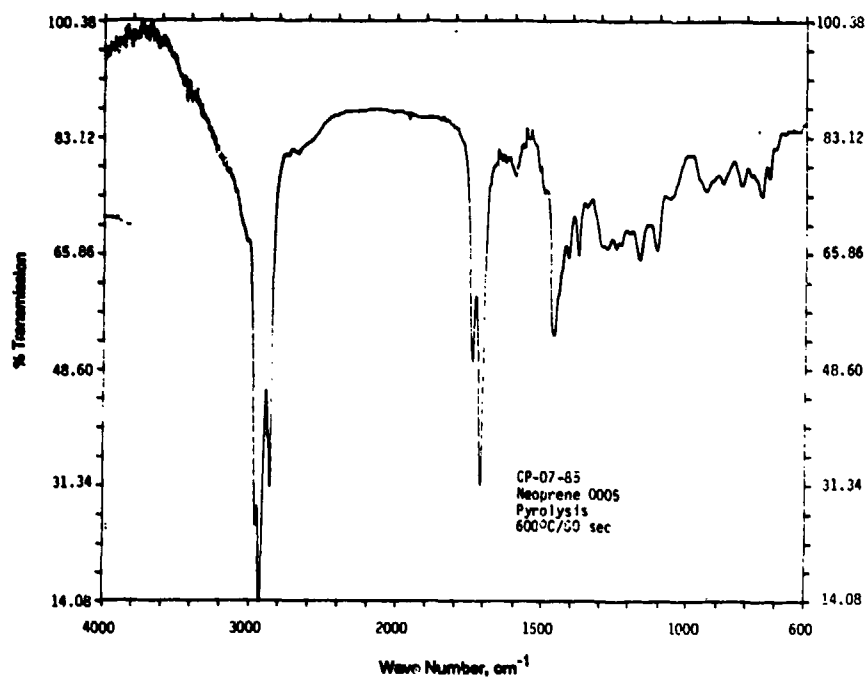


Figure 5. IR spectrum of Neoprene 0005 pyrolyzate.

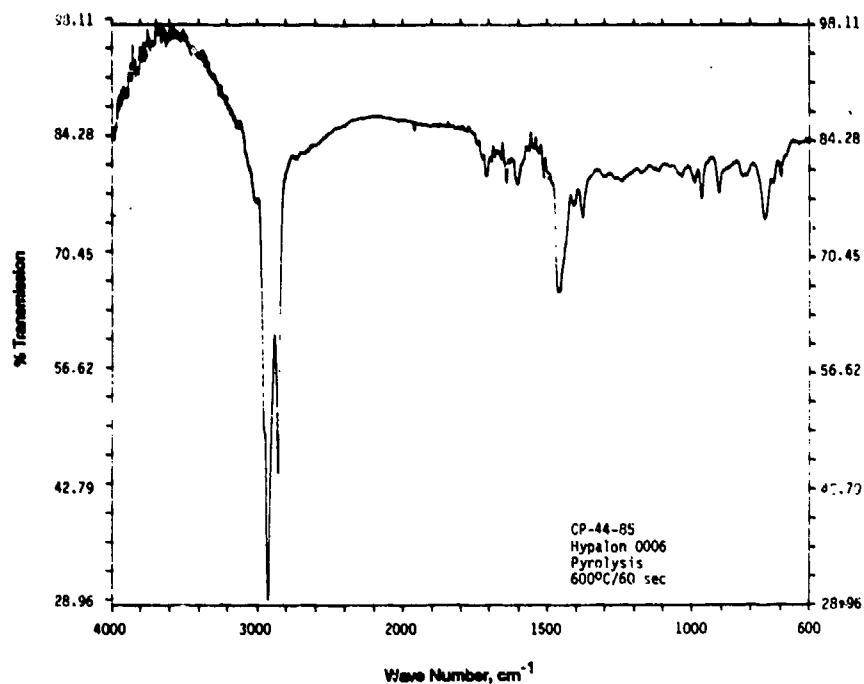


Figure 6. IR spectrum of Hypalon 0006 pyrolyzate.

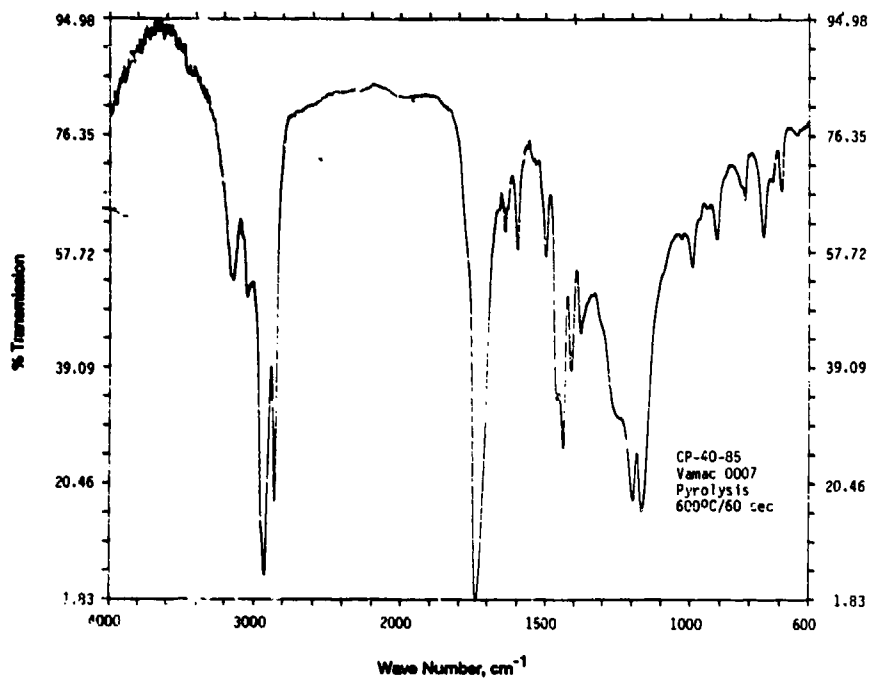


Figure 7. IR spectrum of Vamac 0007 pyrolyzate.

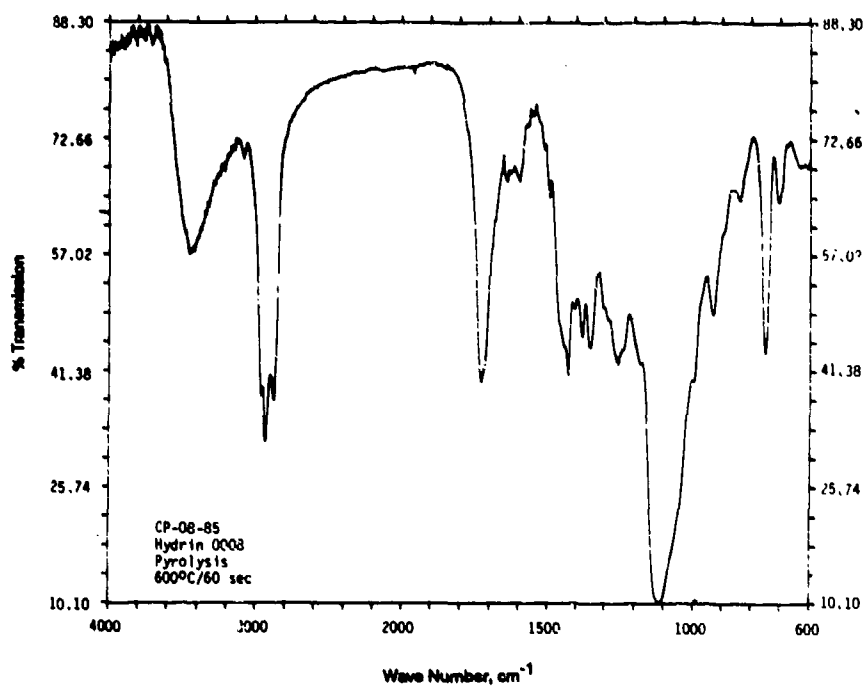


Figure 8. IR spectrum of Hydrin 0008 pyrolyzate.

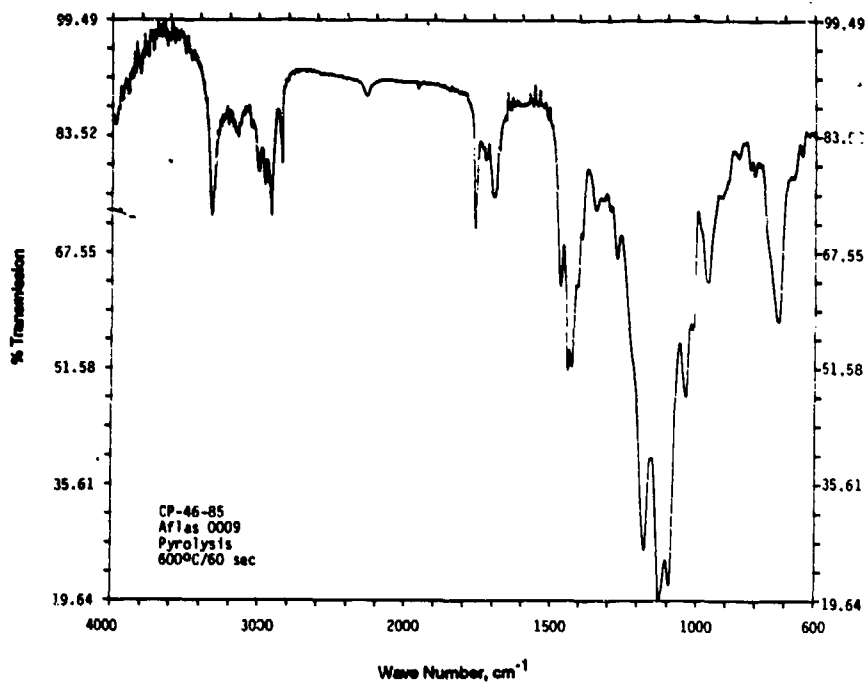


Figure 9. IR spectrum of Atlas 0009 pyrolyzate.

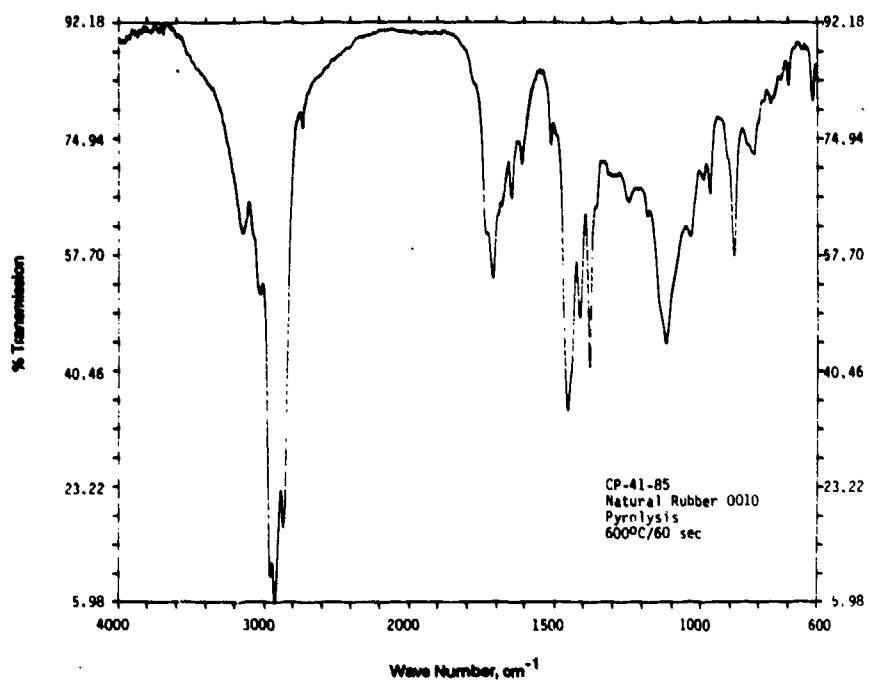


Figure 10. IR spectrum of Natural Rubber 0010 pyrolyzate.

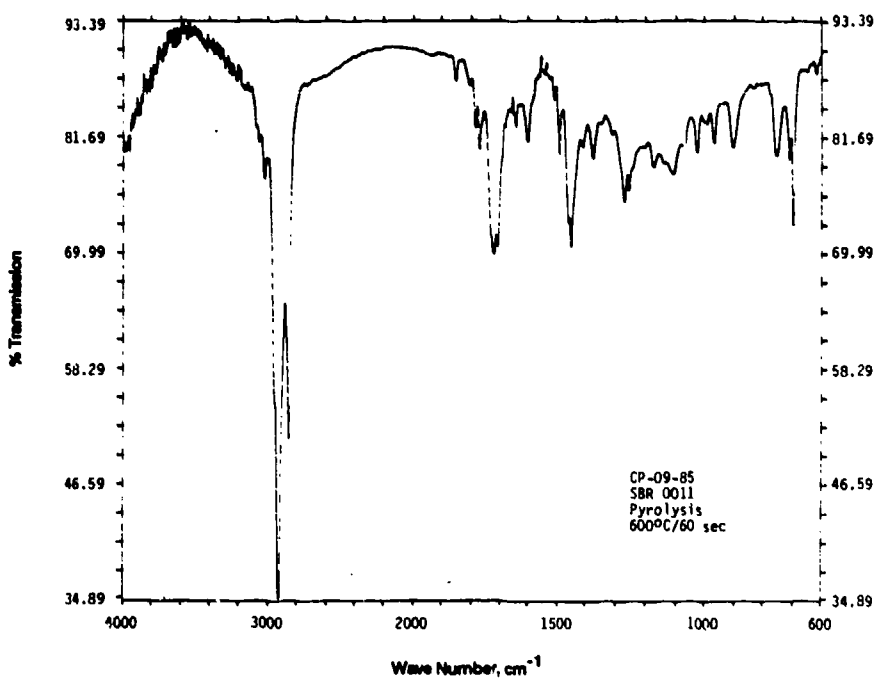


Figure 11. IR spectrum of Styrene-Butadiene Rubber 0011 pyrolyzate.

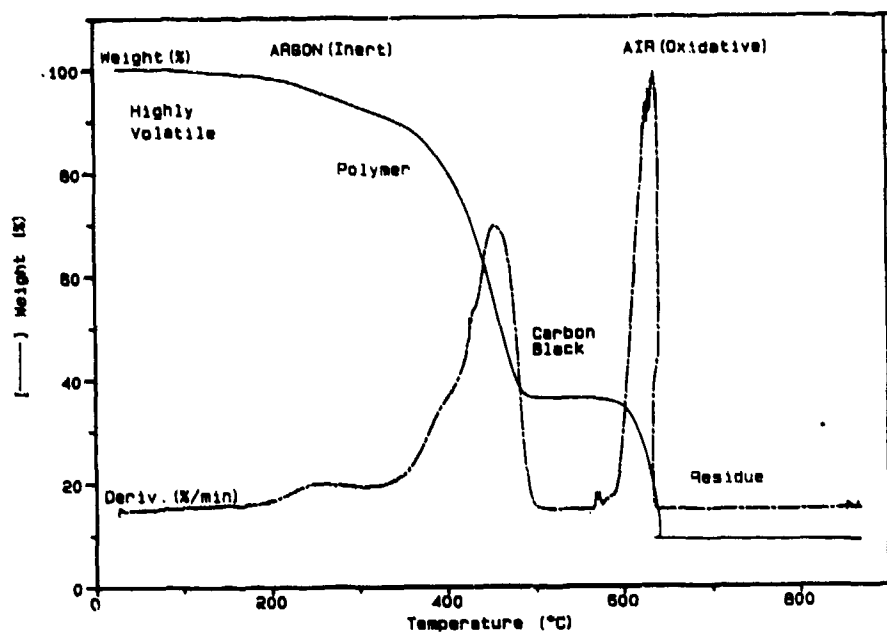


Figure 12. Typical TGA record, indicating three weight loss processes.

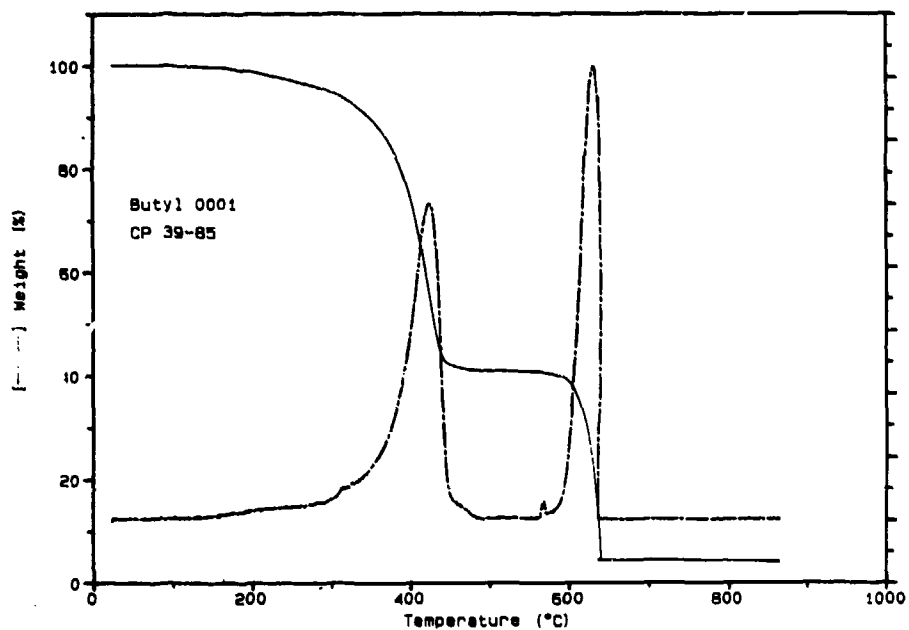


Figure 13. TGA record for Butyl 0001.

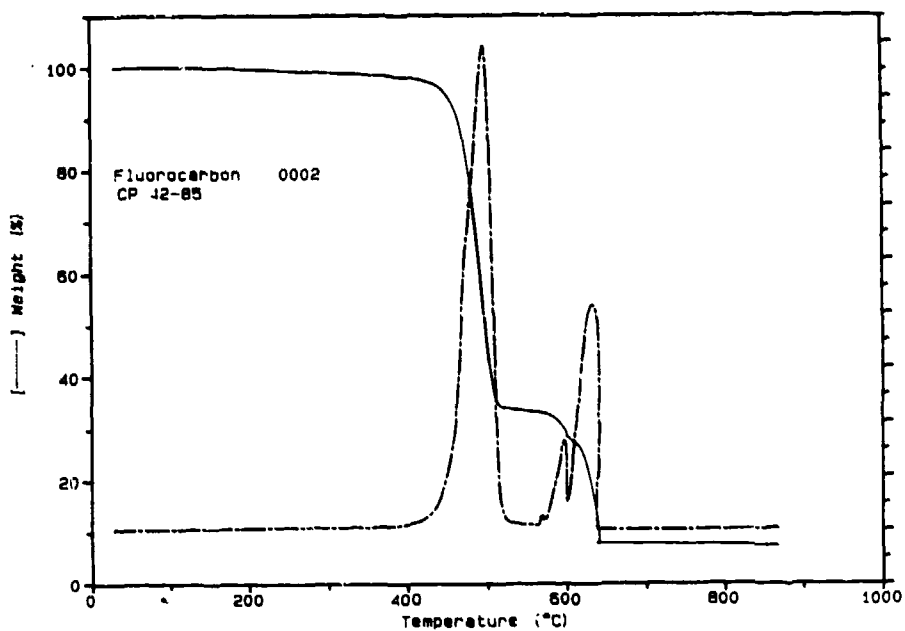


Figure 14. TGA record for Fluorocarbon 0002.

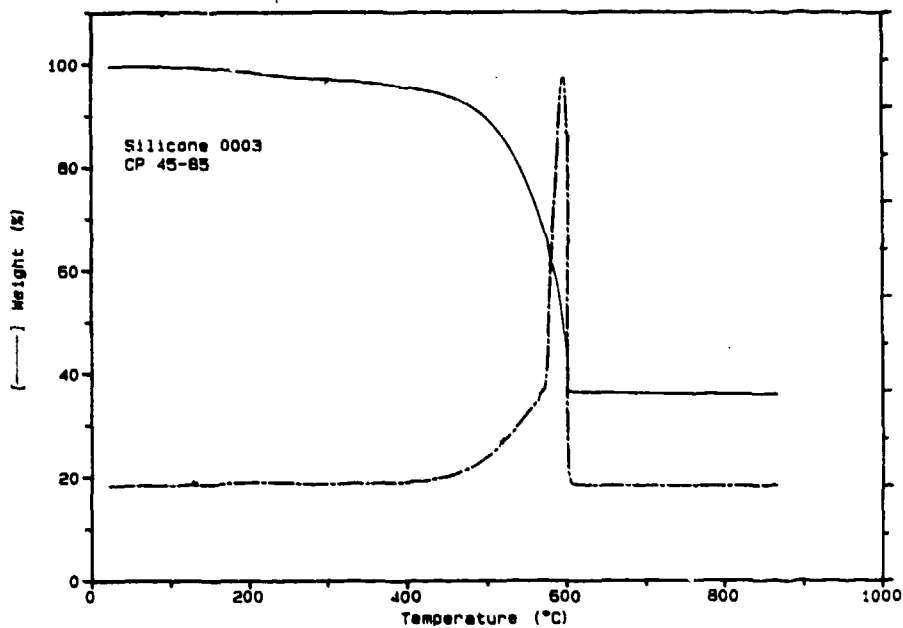


Figure 15. TGA record for Silicone 0003.

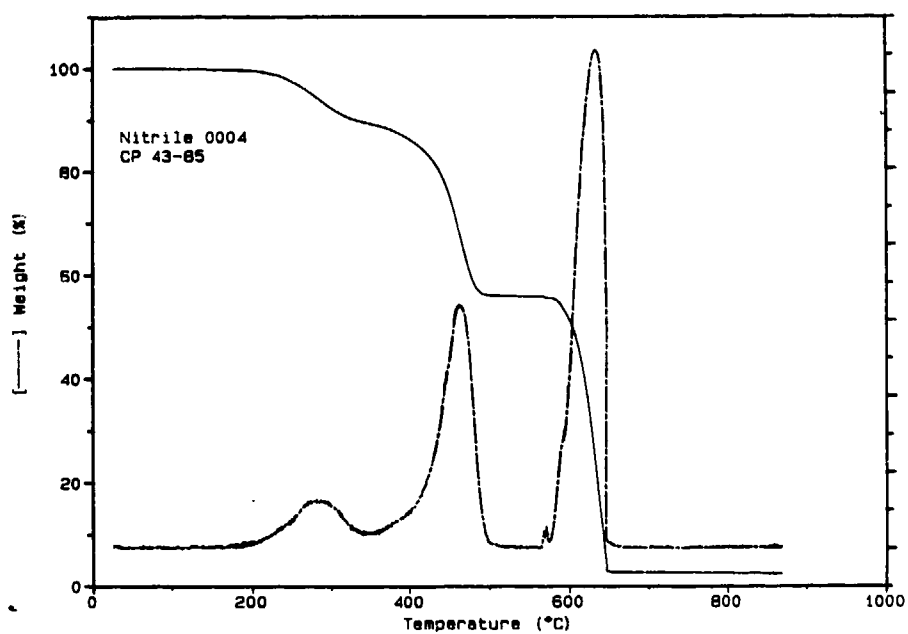


Figure 16. TGA record for Nitrile 0004.

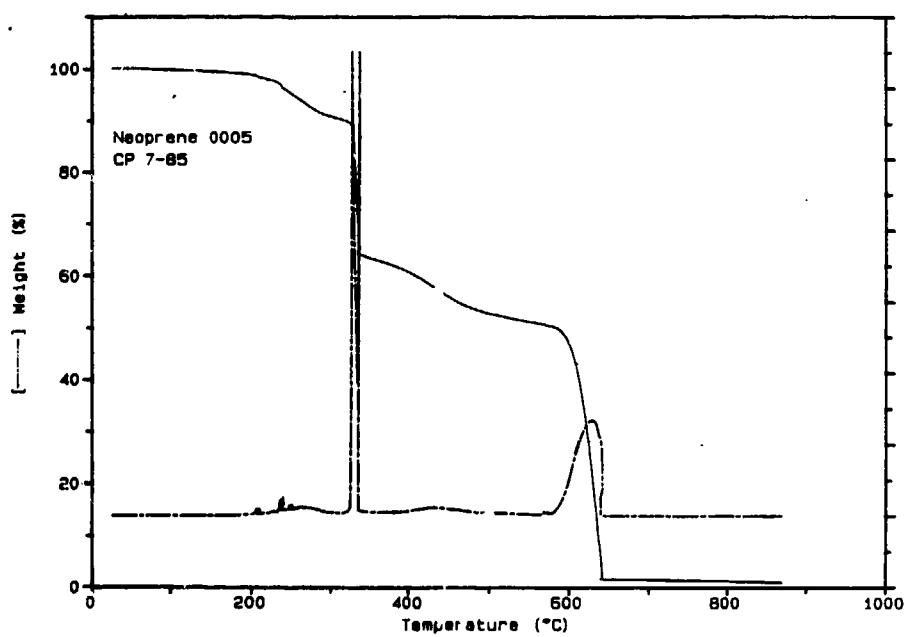


Figure 17. TGA record for Neoprene 0005.

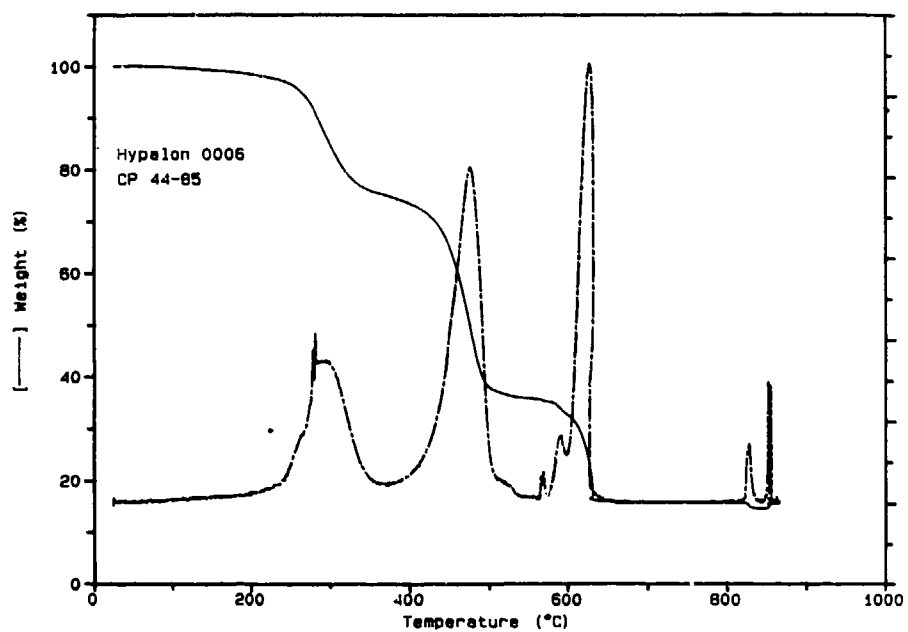


Figure 18. TGA record for Hypalon 0006.

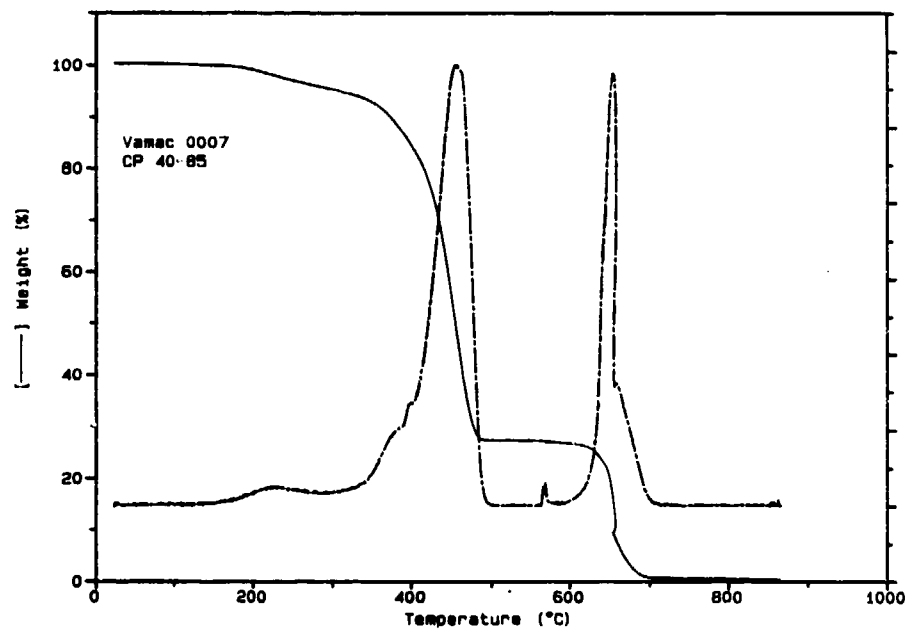


Figure 19. TGA record for Vamac 0007.

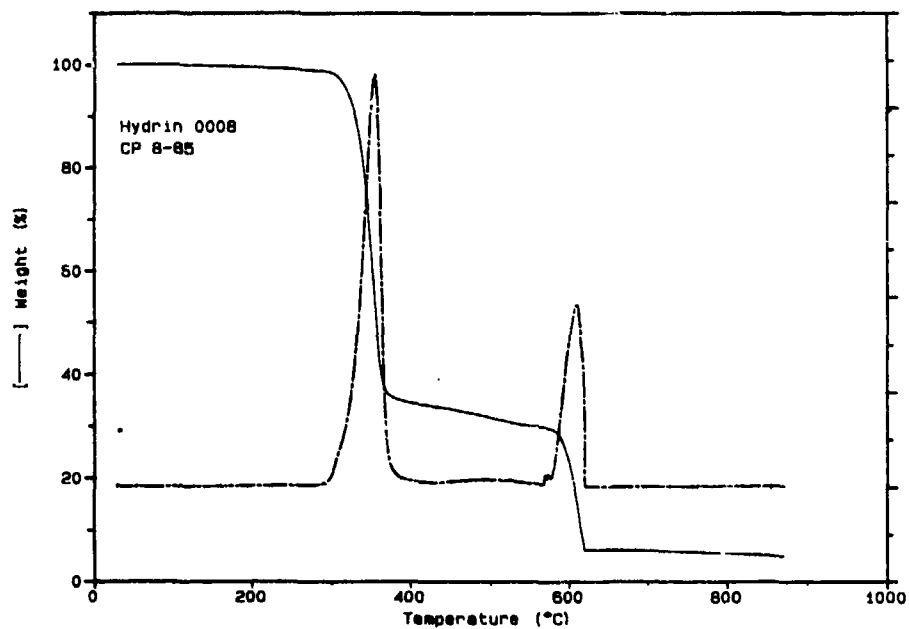


Figure 20. TGA record for Hydrin 0008.

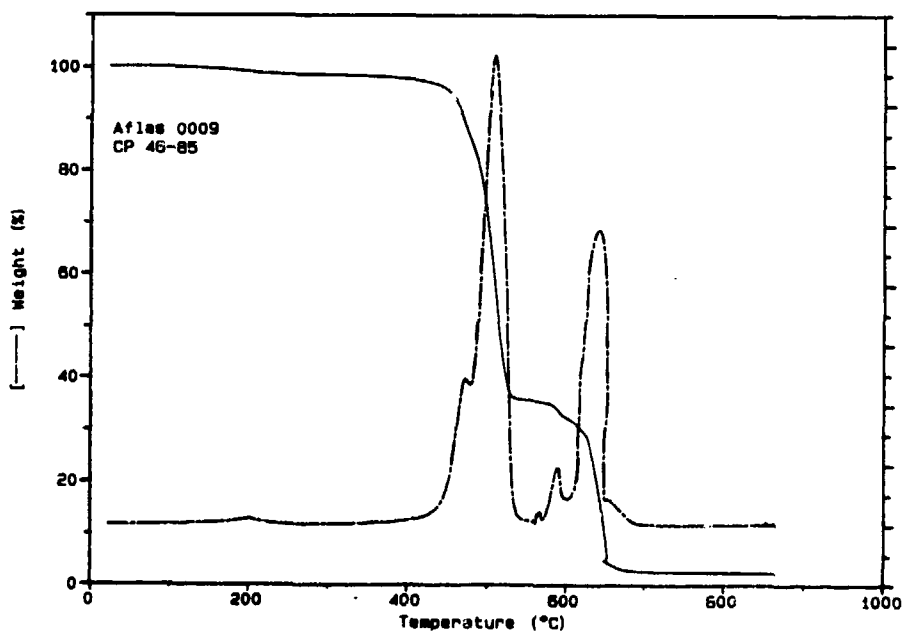


Figure 21. TGA record for Atlas 0009.

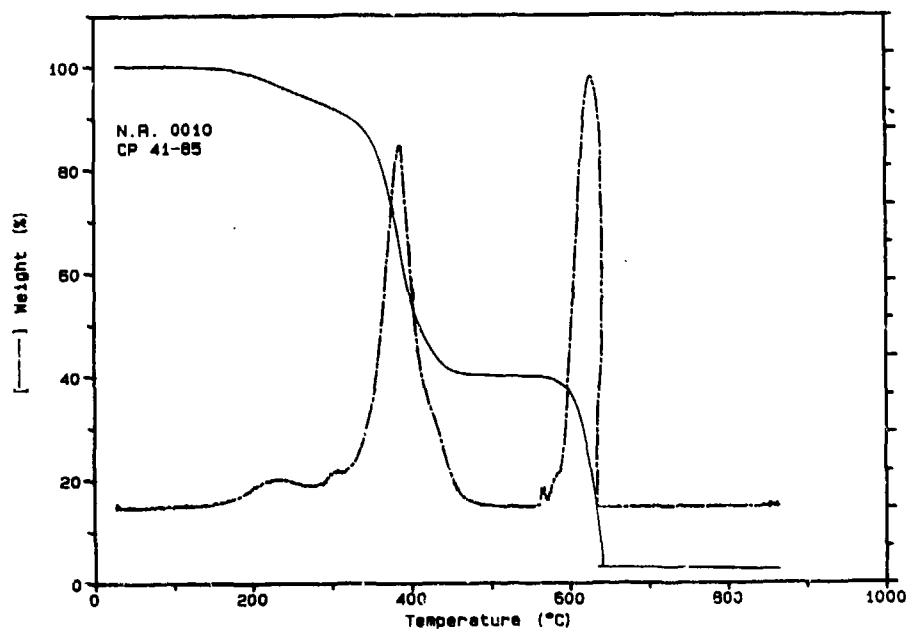


Figure 22. TGA record for Natural Rubber 0010.

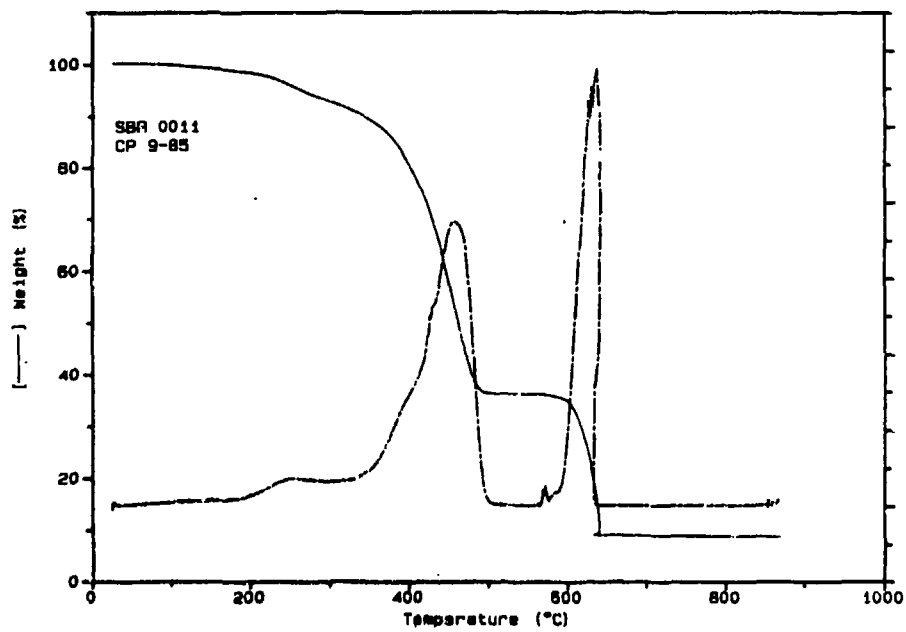


Figure 23. TGA record for Styrene-Butadiene Rubber 0011.

APPENDIX A. RECIPES FOR STANDARD RUBBERS: SET 1*

COMPOUND INGREDIENT INFORMATION

Neoprene CXA	100.0	Neoprene W
	4.0	Scorchguard O
	0.5	Stearic Acid
	2.0	Wingstay 100 Az
	1.0	Agerite Stalite S
	65.0	SRF Carbon
	1.0	Vanwax H
	18.0	Arizona 208
	1.0	IML-1
	1.0	A.C. Polyethylene
	5.0	Zinc Oxide
	0.5	NA-22
	0.5	Methyl Tuads

EPDM EP8A	100.0	Nordel 2744
	5.0	Zinc Oxide
	1.0	Stearic Acid
	1.0	Agerite Resin D
	110.0	FEF Carbon
	50.0	MT Carbon
	30.0	Circosol 4240
	3.0	PPA - 790
	2.0	Sulfur
	1.5	Captax
	0.8	Tetrone A
	0.8	Tellurac 80%
	0.3	Methyl Tuads

Nitrile N7A	100.0	Paracril 18-80
	0.6	Sulfur
	5.0	Zinc Oxide
	1.0	Stearic Acid
	3.0	Agerite Resin D
	50.0	FEF Carbon
	95.0	MT Carbon
	30.0	TP-95
	3.0	Altax
	4.0	Methyl Tuads

* Irving B. Moore Corporation

SBR G6A	162.0	Ameripol 1610
	5.0	Zinc Oxide
	1.5	Stearic Acid
	3.0	Cumar MH 1-1/2
	2.0	Agerite Resin D
	1.0	Santocure
	2.0	Sulfur
	0.2	Methyl Tuads
Vamac M6E	124.0	Vamac B-124
	0.5	Armeen 18D
	2.0	Stearic Acid
	25.0	FEF Carbon
	2.0	Zelec UN
	5.0	Santicizer 409
	1.25	DIAC #1
	4.0	DPG
Hypalon H8E	100.0	Hypalon 40
	1.0	Stearic Acid
	20.0	Mag. Oxide
	3.0	A.C. Polyethylene
	30.0	Suprex Clay
	30.0	XC-72 Carbon
	10.0	Sundex 790
	1.0	NBC
	2.0	Agerite Resin D
	2.0	KP-140
	0.75	Altax
	2.0	Tetrone A
Urethane P6E	100.0	Millathane 76
	30.0	SRF Carbon
	4.0	Altax
	2.0	Captax
	0.5	Cadmium Stearate
	2.0	Sulfur
	1.0	Caytur 4
Fluorocarbon V7D	80.0	Fluorel 2174
	20.0	Fluorel 2145
	3.0	Mag. Oxide
	6.0	Calcium Hydroxide
	30.0	MT Carbon
	0.25	Carnauba Wax

Natural Rubber R4A	100.0	SMR-5L
	1.0	Reogen
	1.0	Stearic Acid
	5.0	Zinc Oxide
	1.0	Vanwax H
	2.0	Santowhite
	15.0	HI SIL 233
	2.0	Sulfasan R
	1.2	Methyl Tuads

Hydrin Y6E	100.0	Hydrin 100
	6.0	Red Lead
	1.0	Zinc Stearate
	1.0	NBC
	25.0	FEF Carbon
	1.0	ZO - 9
	1.5	END - 75

Fluorosilicone FS5C	100.0	LS 422
	3.0	SE 456
	3.0	FK300 DS
	2.0	Ferro Blue Pigment
	1.6	Cadox TS-50

Butyl B7A	100.0	Butyl HT 10-66
	1.0	Stearic Acid
	1.0	Antiox. 2246
	0.25	Maglite D
	60.0	HAF Carbon
	15.0	FEF Carbon
	2.0	Paraffin Wax
	2.0	A.C. Poly.
	5.0	Zinc Oxide
	2.0	Sulfur
	1.0	Methyl Tuads
	1.0	Altax

Silicone S5B

General Electric SE 557U

APPENDIX B. RECIPES FOR STANDARD RUBBERS: SET 2*

MTL COMPOUND 0001--SMITHERS NO. M0235 (CHLOROBUTYL)
(Page 1 of 2)

	<u>PHR</u>	<u>GRAMS</u>
BUTYL HT 1066	100	720
STEARIC ACID	1	7.2
ANTIOXIDANT 2246	1	7.2
N-330 CARBON BLACK	60	432
N-550 CARBON BLACK	15	108
POLYETHYLENE AC617	4	28.8
KENFLEX A	5	36.0
DRAKEOL 35	5	36.0
PARACIN 1	3	21.6
ZINC OXIDE	5	36.0
MAGNESIUM OXIDE	0.5	3.6
ALTAX	2	14.4
METHYL TUADS	1	7.2
SULFUR	1	7.2
	<u>203.5</u>	<u>1465.2</u>

CURING CONDITION: 60 minutes at 293°F

PHYSICAL PROPERTIES:

Monsanto Rheometer - ASTM D2084, 293°F, 100 Range, 60 Minute Motor, MCP Dies,
20 Second Preheat

Minimum Torque	27.6
Maximum Torque	112.6
Scorch, t_2	5.9
90% Cure Time	43.0
100% Modulus, PSI	250
300% Modulus, PSI	1200
Tensile Strength, PSI	1720
Elongation, %	440
Shore A Hardness	71

* Smithers Scientific Services, Inc.

MTL COMPOUND 0001--CHLORCBUTYL
(Page 2 of 2)

List of Ingredients

Ingredient	Supplier	Chemical Description	Function
Butyl HT 1066	Exxon	Chlorobutyl Elastomer 51 Mooney	
Stearic Acid	Emery Ind., et al	Activates Cure	
Antioxidant 2246	American Cyanamid	2,2'-Methylene Bis (4-Methyl-6-t- Butyl Phenol)	Protects Compound Against Degradation
N-300 Carbon Black	Cabot Corp., et al	Incomplete Combustion Product of Petroleum Oil	Reinforcing Filler
N-550 Carbon Black	Cabot Corp., et al	Incomplete Combustion Product of Petroleum Oil	Reinforcing Filler
Polyethylene AC 617	Allied Chemical	Low Molecular Weight Polyethylene	Process Aid, Mold Release
Kenflex A	Kenrich Petro- chemical	Undisclosed	Plasticizer
Drakeol 35	Penreco	Mineral Oil	Plasticizer, Softene
Paracin #1	Cas Chem Inc.		Plasticizer
Zinc Oxide	St. Joe Minerals, et al	ZnO	Same as MgO
Magnesium Oxide	Merck, et al	MgO	Reacts with Chlorine Creates C-C- Crosslinks
Altax	RT Vanderbilt, et al	Benzothiazyl Disulfide	Accelerates Cure
Methyl Tuads	RT Vanderbilt, et al	Tetramethylthiuram Disulfide	Accelerates Cure
Sulfur	Stauffer, et al		Crosslinking Agent

MTL COMPOUND 0002--SMITHERS NO. L0829 (FLUOROELASTOMER)

(Page 1 of 2)

	<u>PHR</u>	<u>GRAMS</u>
FLUOREL 2174	80	1200
FLUOREL 2145	20	300
CARNAUBA WAX	3	45
N-990 CARBON BLACK	30	450
MAGLITE D	6	9.0
CALCIUM HYDROXIDE	2.5	3.75
	<u>135.5</u>	<u>2007.75</u>

Mix Cycle -

0 min. - Fluorel Rubber

30 sec. - 1/2 Black

1 min. 30 sec. - 1/2 Black

2 min. 30 sec. - Carnauba Wax

3 min. 30 sec. - Scorch

5 min. - Dump

Maglite D, Calcium Hydroxide added on mill

CURING CONDITION: 30 minutes at 293°F

PHYSICAL PROPERTIES:

Monsanto Rheometer - ASTM D2084, 293°F, 100 Range, 60 Minute Motor, MPC Dies,
20 Second Preheat

Minimum Torque	18.6
Maximum Torque	121.0
Scorch, t ₂	7.6
90% Cure Time	18.0
100% Modulus, PSI	470
300% Modulus, PSI	1230
Tensile Strength, PSI	1400
Elongation, %	340
Shore A Hardness	73

MTL COMPOUND 0002--FLUOROCARBON
(Page 2 of 2)

List of Ingredients

Ingredient	Supplier	Chemical Description	Function
Fluoral 2174	3M Company	Fluoroelastomer 38 Mooney Viscosity	General Purpose Polymer
Fluoral 2145	3M Company	Fluoroelastomer 28 Mooney Viscosity	Added for Improved Processing Process Air
Carnauba Wax	Akron Chemical, et al		Process Aid Mold Release
N-990 Carbon Black	J.M. Huber	Incomplete Combustion Product of Natural Gas	Semi Reinforcing Filler
Maglite D (Magnesium Oxide)	Merck	MgO	Reacts with chlorine to Form C-C Crosslinks
Calcium Hydroxide		Ca(OH) ₂	Acid Acceptor- Promotes Cure

MTL COMPOUND 0003--SMITHERS NO. M0773AX (SILICONE)
(Page 1 of 1)

	<u>PHR</u>	<u>GRAMS</u> ;
GENERAL ELECTRIC		
SILICONE RUBBER COMPOUND SE5563	100	950.50
DICUP 40KE - PEROXIDE CURATIVE	<u>1</u>	<u>9.51</u>
TOTAL	101	960.01

Curatives added on Mill

CURING CONDITION: 45 Min. at 293°F

PHYSICAL PROPERTIES:

Monsanto Rheometer - ASTM D2084, 100 Range, 60 Minute Motor, MPC Dies,
20 Second Preheat

Minimum Torque	4.5
Maximum Torque	45.8
Scorch, t ₂	5.5
90% Cure Time	34.5
100% Modulus, PSI	220
300% Modulus, PSI	470
Tensile Strength, PSI	1360
Elongation, %	700
Shore A Hardness	51

MTL COMPOUND 0004--SMITHERS NO. MO690 (NITRILE)
(Page 1 of 2)

	<u>PHR</u>	<u>GRAMS</u>
PARACRIL 18-80	100	500
N-550 CARBON BLACK	50	250
N-990 CARBON BLACK	95	475
TP 95	25	125
AGERITE RESIN D	3	15
POLYETHYLENE AC 617	5	25
ZINC OXIDE	5	25
STEARIC ACID	1	5
ALTAX	1.5	7.5
METHYL ZIMATE	0.5	2.5
SULFUR	1.5	7.5
	<u>287.5</u>	<u>1437.5</u>

CURING CONDITION: 30 minutes at 293°F

PHYSICAL PROPERTIES:

Monsanto Rheometer - ASTM D2084, 293°F, 100 Range, 60 Minute Motor, MCP Dies,
20 Second Preheat

Minimum Torque	11.0
Maximum Torque	68.2
Scorch, t ₂	2.8
90% Cure Time	9.7

100% Modulus, PSI	580
300% Modulus, PSI	--
Tensile Strength, PSI	1500
Elongation, %	190
Shore A Hardness	70

MTL COMPOUND 0004--NITRILE
(Page 2 of 2)

List of Ingredients

Ingredient	Supplier	Chemical Description	Function
Paracril 18-80	Uniroyal	Nitrile-Butadiene Elastomer 22% ACN, 65-80 Mooney	
N-550 Carbon Black	Cabot Corp., et al	Incomplete Combustion Prod. of Petroleum Oil	Reinforcing Filler
N-900 Carbon Black	J.M. Huber, et al	Incomplete Combustion Product of Natural Gas	Semi-Reinforcing Filler
TP-95	Morton-Thiokol	Di(Butoxy-Ethoxy-Ethyl) Adipate	Plasticizer
AgeRite Resin D	R.T. Vanderbilt	Polymerized 1,2-Dihydro-2,2,4-Trimethyl Quinoline	Prevents Heat Degradation
Polyethylene AC617	Allied Chemical	Low Molecular Weight Polyethylene	Process Aid Promotes Mill Release
Zinc Oxide	St. Joe Minerals, et al	ZnO	Activates Cure
Stearic Acid	Emery Ind., et al		Activates cure
Altax	R.T. Vanderbilt	Benzothiazyl Disulfide	Accelerates Cure
Methyl Zimate	R.T. Vanderbilt	Zinc Dimethyldithiocarbamate	Accelrates Cure
Sulfur	Stauffer, et al	S	Crosslinking Agent

MTL COMPOUND 0005--SMITHERS NO. L0831 (NEOPRENE)
(Page 1 of 2)

<u>INGREDIENT</u>	<u>PHR</u>	<u>GRAMS</u>
Neoprene W	100	760
Scorchguard O	4	30.4
Stearic Acid	0.5	3.8
Wingstay 100 AZ	2	15.2
Age Rite Stalite S	1	7.6
N-774 Carbon Black	65	494.4
Van Max E	1	7.6
Plasticizer SC-B	18	136.8
Darvan ME	1	7.6
Polyethylene AC617	1	7.6
Zinc Oxide	5	38.0
Akrochem ETU-22 PM	0.67	5.1
Methyl Tuads	0.5	3.8
	<u>199.67</u>	<u>1517.5</u>

CURING CONDITION: 15 minutes at 320°F

PHYSICAL PROPERTIES:

Monsanto Rheometer - 320°F, 3° Arc, 100 CPM, 20 Second Preheat, MCP Dies.
100 Range

Minimum Torque 7.1
Maximum Torque 70.9
Scorch, t_2 , min 2.0
90% Cure Time, min 13.2

100% Modulus, PSI 390
300% Modulus, PSI 2410
Tensile Strength, PSI 2630
Elongation, % 330
Shore A Hardness 63

Specific Gravity 1.363

MTL COMPOUND 0005--NEOPRENE
(Page 2 of 2)

List of Ingredients

Ingredient	Chemical Description	Function
Neoprene W	Polychloroprene	
Scorchguard O	Magnesium Oxide	Crosslink Promoter
Stearic Acid		Activator
Wingstay 100 AZ	Mixed Diaryl p-phenylenediamine	Antidegradant
Age Rite Stalite 5	Mixture of Octylated Diphenyl- amines	Antidegradant
N-774 Carbon Black	Incomplete Combustion product of Petroleum Oil	Reinforcing Filler
Van Wax H	Blend of Paraffin Wax	Antiozonant
Plasticizer SC-B	Diester of Triethylene Glycol	Plasticizer
Darvan ME	Sodium Alkyl Sulfates	Process Aid
Polyethylene AC617	Low Molecular Weight Polyethylene	Processing Aid
Zinc Oxide		Crosslinking Promoter
Akrochem ETU-22-PM	Dispersion of Ethyl Thiourea	Accelerator
Methyl Tuads	Tetramethylthiuram Disulfide	Accelerator

MTL COMPOUND 0006--SMITHERS NO. M0723 (HYPALON)
(Page 1 of 2)

	<u>PHR</u>	<u>GRAMS</u>
HYPALON 40	100	1000
SUPREX CLAY	25	250
N-550 CARBON BLACK	30	300
STEARIC ACID	1	10
POLYETHYLENE AC 617A	3.5	35
SUNDEX 790	10	100
KP 140	3	30
AGERITE RESIN D	2	20
NBC	1	10
MAGLITE D	5	50
ALTAX	0.75	7.5
TETRONE A	1.75	17.5
	<u>183.0</u>	<u>1830.0</u>

Mix Cycle -

0 min. - 110°F - Hypalon, NBC, Resin D

30 sec. - 160°F - Black

2 min. - 260°F - Clay

3 min. - 310°F - Sundex KP 140, Stearic Acid, Polyethylene
(Allow to recover from temperature drop)

4 min. 30 sec. - Scrape

6 min. - Dump

CURING CONDITION: 50 minutes at 293°F

PHYSICAL PROPERTIES:

Monsanto Rheometer - ASTM D2084, 290°F, 100 Range, 60 Minute Motor, MPC Dies,
20 Second Preheat

Minimum Torque	13.2
Maximum Torque	38.2
Scorch, t ₂	3.4
90% Cure Time	31.0

100% Modulus, PSI	420
300% Modulus, PSI	1250
Tensile Strength, PSI	1850
Elongation, %	560
Shore A Hardness	72

MTL COMPOUND 0006--HYPALON
(Page 2 of 2)

List of Ingredients

Ingredient	Supplier	Chemical Description	Function
Hypalon 40	DuPont	Chlorosulfonated Polyethylene Elastomer	
Suprex Clay	J.M. Huber	Hydrated Aluminum Silicate	Semi-Reinforcing Filler
N-550 Carbon Black	Cabot Corp., et al	Incomplete Combustion Product of Petroleum Oil	Reinforcing Filler
Stearic Acid	Emery Ind., et al		Activates Cure
Polyethylene AC617	Allied Chemical	Low Molecular Weight Polyethylene	Processing Aid Promotes Mill Release
Sundex 790	Sun Oil	Aromatic Petroleum Oil	Plasticizer; Softener
KP140	Kenrich Petrochemicals	Tributoxyethyl Phosphate	Plasticizer; Softener
AgeRite Resin D	R.T. Vanderbilt	Polymerized 1,2-Dihydro 2,2,4 Trimethyl-quinoline	Prevents Heat Degradation
NBC	DuPont	Nickel Dibutyldithiocarbamate	Heat Stabilizer
Maglite D	Merck	Magnesium Oxide (MgO)	Promotes C-C Bond Formation
Altax	R.T. Vanderbilt	Benzothiazyl Disulfide	Accelerates Cure
Tetrone A	DuPont	Dipentamethylene Thiuram Hexasulfide	Accelerates Cure

MTL COMPOUND 0007--SMITHERS NO. M0234 (ETHYLENE ACRYLIC)
(Page 1 of 2)

	<u>PHR</u>	<u>GRAMS</u>
VAMAC B-124	100	992
ARMEEN 180	0.4	4
STEARIC ACID	1.6	16
N-550 CARBON BLACK	20.2	200
GAFAC RL-120	0.8	8
SANTICIZER 409	4.0	40
DIAX #1	1.0	10
DPG	3.2	32
	<u>131.2</u>	<u>1302.0</u>

Mix Cycle -

CURING CONDITION: 50 minutes at 293°F

PHYSICAL PROPERTIES:

Monsanto Rheometer - ASTM D2084, 293°F, 200 Range (note), 60 Minute Motor,
MCP Dies, 20 Second Preheat

Minimum Torque	3.0
Maximum Torque	33.4
Scorch, t ₂	4.0
90% Cure Time	48.0

100% Modulus, PSI	120
300% Modulus, PSI	660
Tensile Strength, PSI	1920
Elongation, %	650
Shore A Hardness	51

MTL COMPOUND 0007--VAMAC
(Page 2 of 2)

List of Ingredients

Ingredient	Supplier	Chemical Description	Function
Vamac B124	Dupont	Ethylene/Acrylic Elastomer 85% Polymer; Mooney Viscosity -21	
Armeen 18D	Arnak	Octadecylamine	Amine Curative
Stearic Acid	Emery Ind.		Activates Cure
N-550 Carbon Black	Cabot Corp., et al	Incomplete Combustion Product of Petroleum Oil	Reinforcing Filler
Gafac RL-210	GAF		Curative
Santicizer 409	Monsanto		Plasticizer
Diak #1	DuPont	Hexamethylene Diamine Carbamate	Promotes Crosslinking
DPG	R.T. Vanderbilt	Diphenyl Guanidine	Accelerates Cure

MTL COMPOUND 0008--SMITHERS NO. L0834 (EPICHLOROHYDRIN)
(Page 1 of 2)

<u>INGREDIENT</u>	<u>PHR</u>	<u>GRAMS</u>
Hydrin 100	100	1200
Chem-I-Sorb ERL-06-90	6.67	80
Zinc Stearate	1	12
NBC	1	12
N-550 Carbon Black	25	300
ZO-9	1	12
Akrochem ETV-22 PM	1.5	18
Total	<u>136.17</u>	<u>1634.0</u>

CURING CONDITION: 20 minutes at 320°F

PHYSICAL PROPERTIES:

Mossanto Rheometer - 320°F, 100 CPM, 3° Arc, MCP Dies, 20 Second Preheat,
100 Range

Minimum Torque	11.2
Maximum Torque	58.4
Scorch, t ₂ , min	1.4
90% Cure Time, min	19.0

100% Modulus, PSI	220
300% Modulus, PSI	1050
Tensile Strength, PSI	2380
Elongation, %	610
Shore A Hardness	58

Specific Gravity	1.487
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MTL COMPOUND 0008--EPICHLOROHYDRIN
(Page 2 of 2)

List of Ingredients

Ingredient	Chemical Description	Function
Hydrin 100	Polyepichlorohydrin	
Chem-I-Sorb ERL-06-90	90% Red Lead Oxide in EPR Binder	Curing Agent
Zinc Stearate		Process Aid
NBC	Nickel Dibutyldithiocarbamate	Antidegradant
N-550 Carbon Black	Incomplete Combustion Product of petroleum Oil	Reinforcement Filler
ZO-9	Blend of Metallic Salts of Fatty Acid	
Akrochem ETV-22 PM	75% Dispersion of Ethylene Thiourea	Curing Agent

MTL COMPOUND 0009--SMITHERS NO. M0729D (FLUOROELASTOMER)
(Page 1 of 2)

	<u>PHR</u>
AFLAS 150P	100
N-990 CARBON BLACK	50
CARNAUBA WAX BLACK	2
DIAX #7	8
LUPERCO 101XL	<u>9</u>

CURING CONDITION: 50 minutes at 293°F

PHYSICAL PROPERTIES:

Monsanto Rheometer - ASTM D2084, 293°F, 100 Range, 60 Minute Motor, MCP Dies,
20 Second Preheat

Minimum Torque	16.0
Maximum Torque	80.2
Scorch, t ₂	3.7
90% Cure Time	39.3

100% Modulus, PSI	900
300% Modulus, PSI	--
Tensile Strength, PSI	1520
Elongation, %	200
Shore A Hardness	81

MTL COMPOUND 0009--AFLAS
(Page 2 of 2)

List of Ingredients

Ingredient	Supplier	Chemical Description	Function
Aflas 150P	Xenox Corp - Importers	Tetrafluoroethylene/ Propylene Elastomer Med. Viscosity	
N-990 Carbon Black	J.M. Huber	Incomplete Combustion Prod. of Natural Gas	Semi-Reinforcing Filler
Carnauba Wax	Akron Chemicals, et al		Process Aid Promotes Mill Release
Diak #7	DuPont	Reactive Polyfunc- tional Triazine Derivative	Accelerator
Luperco 101XL	Pennwalt	2,5-Dimethyl-2,5 Bis (t-Butyl Peroxy) Hexane	Crosslinking Promoter

MTL COMPOUND 0010--SMITHERS NO. L0836 (NATURAL RUBBER)
(Page 1 of 2)

	<u>PHR</u>	<u>GRAMS</u>
SMR-L	100	640
ZINC OXIDE	4	25.6
STEARIC ACID	1.5	9.6
WINGSTAY 29	1.5	9.6
SANTOFLEX 77	3	19.2
N-550 CARBON BLACK	75	480
TP 90B	10	64.0
PLASTHALL HA 7A	10	64.0
SANTOCURE	0.9	5.8
UNADS	0.5	3.2
SULFUR	1.2	7.7
	<u>207.6</u>	<u>1328.7</u>

CURING CONDITION: 10 minutes at 320°F (Physical Properties)

PHYSICAL PROPERTIES:

Monsanto Rheometer - ASTM D2084, 320°F (note), 100 Range, 60 Minute Motor,
MCP Dies, 20 Second Preheat

Minimum Torque	17.0
Maximum Torque	55.8
Scorch, t ₂	1.3
90% Cure Time	2.2

100% Modulus, PSI	200
300% Modulus, PSI	1230
Tensile Strength, PSI	2200
Elongation, %	450
Shore A Hardness	54

MTL COMPOUND 0010--NATURAL RUBBER
(Page 2 of 2)

List of Ingredients

Ingredient	Supplier	Chemical Description	Function
SMR-L	Dupont	General Purpose, Light Colored Natural Rubber	
Zinc Oxide	St. Joe Minerals, et al	ZnO	Activates Cure
Stearic Acid	Emery Inc., et al		Activates Cure
Wingstay 29	Goodyear	Diphenylamine Derivative	Antioxidant; Prevents Degradation
Santoflex 77	Monsanto	N,N' - Bis (1,4-Dimethylpentyl)-p-Phenylenediamine	Antiozonant; Prevents Degradation
N-550 Carbon Black	Cabot Corp., et al	Incomplete Combustion Prod. of Petroleum Oil	Filler
TP90B	Morton-Thiokol	Di(Butoxy-Ethoxy-Ethyl) Formal	Plasticizer
Plasthall HA 7A	C.P. Hall		Plasticizer
Santocure	Monsanto	N-Cyclohexyl-2-Benzothiazolsulfenamide	Accelerates Cure
Unads	R.T. Vanderbilt	Tetramethyl Thiuram Monosulfide	Accelerates Cure
Sulfur	Stauffer, et al	S	Crosslinking Agent

MTL COMPOUND 0011--SMITHERS NO. L0873 (SBR)
(Page 1 of 2)

<u>INGREDIENT</u>	<u>PHR</u>	<u>GRAMS</u>
SBR 1503	100	680
Zinc Oxide	4	27.2
Stearic Acid	1	6.8
Age Rite Resin D	1.5	10.2
Santoflex 13	1.5	10.2
Sun Anti Chek Wax	3.03	20.6
DSC-18	0.41	2.8
M-339 Carbon Black	51.9	353.0
HiSil 233	13	88.4
Plasthall P-7092	8	54.4
Struktol WB-300	8	54.4
TMTD	0.5	3.4
Santocure	1.75	11.9
Sulfur	2.0	13.6
Total	<u>196.59</u>	<u>1337.3</u>

CURING CONDITION: 5 minutes at 320°F

PHYSICAL PROPERTIES:

Monsanto Rheometer - 320°F, 100 CPM, 3° Arc, MCP Dies, 20 Second Preheat,
100 Range

Minimum Torque	12.0
Maximum Torque	81.0
Scorch, t ₂ , min	2.1
90% Cure time, min	4.3

100% Modulus, PSI	520
Tensile Strength, PSI	1970
Elongation, %	240
Shore A Hardness	70

Specific Gravity	1.174
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MTL COMPOUND 0011--SBR
(Page 2 of 2)

List of Ingredients

Ingredient	Chemical Description	Function
SBR 1503	Styrene/Butadiene Copolymer 23.5% Styrene	
Zinc Oxide	ZnO	Activator
Stearic Acid		Activator
Age Rite Resin D	Polymerized 1,2-dihydro-2,2,4 trimethylquinoline	Antidegradant
Santoflex 13	N-(1,3-dimethylbutyl)-N'- phenyl-p-phenyldiamine	Antidegradant
Sun Anti Chek Wax	Blended Paraffin Wax	Ozone Protection
DSC-18	Mercapto-Silane Coupling Agent	Reinforcement Promoter for Silica
N-339 Carbon Black	Partial Combustion Product of Petroleum Oil	Reinforcement Filler
HiSil 233	Precipitated Silica	Reinforcement Filler
Plasthall P-7092	Polyester Glutarate	Plasticizer
Struktol WB-300	Blended Fatty Acid Derivatives	Process Aid
TMTD	Tetramethylthiuram Disulfide	Secondary Accelerator
Santocure	N-cyclohexyl-2- benzothiazolesulfenamide	Primary Accelerator
Sulfur	S	Crosslinking Agent

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