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Comprehensive Testing to Measure the Response of Styrene Butadiene Rubber to Hanford Tank Waste Simulant

P. J. Nigrey

Prepared by
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Comprehensive Testing to Measure the Response of Styrene Butadiene Rubber to Hanford Tank Waste Simulant

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Abstract

This report presents the findings of the Chemical Compatibility Program developed to evaluate plastic packaging components that may be incorporated in packaging mixed-waste forms for transportation. Consistent with the methodology outlined in this report, we performed the second phase of this experimental program to determine the effects of simulant Hanford tank mixed wastes on packaging seal materials. That effort involved the comprehensive testing of five plastic liner materials in an aqueous mixed-waste simulant. The testing protocol involved exposing the materials to ~143, 286, 571, and 3,670 krad of gamma radiation and was followed by 7-, 14-, 28-, 180-day exposures to the waste simulant at 18, 50, and 60°C. Styrene Butadiene Rubber (SBR) samples subjected to the same protocol were then evaluated by measuring seven material properties: specific gravity, dimensional changes, mass changes, hardness, compression set, vapor transport rates, and tensile properties. From the analyses, we determined that SBR has relatively good resistance to radiation, this simulant, and a combination of these factors. These results suggest that SBR is a relatively good seal material to withstand aqueous mixed wastes having similar composition to the one used in this study.

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INTRODUCTION

Hazardous and radioactive materials packaging is designed to facilitate the transport and storing of materials without posing a threat to the health or property of the general public. U.S. regulations establish general design requirements for such packagings. While no regulations have been written specifically for mixed waste packaging, regulations for the constituents of mixed wastes, that is, hazardous and radioactive substances, have been codified by the U.S. Department of Transportation (U.S. DOT, 49 CFR 173) and the U.S. Nuclear Regulatory Commission (NRC, 10 CFR 71). The packaging materials and contents must be chemically compatible. Furthermore, Type A [49 CFR 173.412 (g)] and Type B (10 CFR 71.43) packaging design requirements stipulate that there be no significant chemical, galvanic, or other reaction between the materials and contents of the package.

Based on the federal requirements, a Chemical Compatibility Testing Program was developed in the Transportation Technology Department at Sandia National Laboratories/New Mexico (SNL/NM). The program attempts to assure any regulatory body that the issue of certain packaging material compatibility towards hazardous and radioactive materials has been addressed. This program was detailed in a 1993 milestone report¹ submitted to the Department of Energy (DOE). The results of this program were reported to the DOE in various unpublished milestone documents and in a number of externally published papers.²⁻⁶

The milestone report *Chemical Compatibility Test Plan and Procedure Report* (CCTP&PR) describes a program to evaluate plastic transportation packaging components that may be used in transporting mixed waste forms. Consistent with the methodology in the CCTP&PR, the first phase of this experimental program has been completed. This effort involved screening ten plastic materials in four simulant mixed waste types.⁷ All materials that include "rubber" in their names are used as seals; the others are used as liners. These plastics were as follows:

Seals

- butadiene-acrylonitrile copolymer rubber (Nitrile),
- epichlorohydrin rubber (EPI),
- isobutylene-isoprene copolymer rubber (Butyl),
- ethylene-propylene rubber (EPDM),
- fluorocarbon (FKM) rubber, and
- styrene-butadiene rubber (SBR).

Liners

- cross-linked polyethylene (XLPE),
- high-density polyethylene (HDPE),
- fluorocarbon (Kel-F™),
- polytetrafluoroethylene (Generically PTFE or Teflon®),
- polypropylene (PP).

The selected simulant mixed wastes were

- (1) an aqueous alkaline mixture of sodium nitrate and sodium nitrite;
- (2) a chlorinated hydrocarbon mixture;
- (3) a simulant liquid scintillation fluid; and
- (4) a mixture of ketones.

The first phase of testing protocol involved exposing the materials to 286,000 rad (290 krad) of gamma radiation followed by 14-day exposures to the waste types at 60°C. After radiation and chemical exposure, the seal materials or rubbers were tested using Vapor Transport Rate (VTR) measurements, while the liner materials were tested using specific gravity. For these tests, screening criteria of about 1 g/hr/m² for VTR and a specific gravity change of 10% were used. Materials that failed to meet these criteria for all four waste types were judged to have failed the screening tests and were excluded in the next phase of this experimental program. Based on this work, it was concluded that while all seal materials passed exposure to the aqueous simulant mixed waste, EPDM and SBR had the lowest VTRs. In the chlorinated hydrocarbon simulant mixed waste, only FKM rubber passed the screening tests. This means that only FKM rubber would be selected for further testing in the chlorinated hydrocarbon simulant. In both the simulant scintillation fluid mixed waste and the ketone mixture simulant mixed waste, none of the seal materials met the screening criteria. For specific gravity testing of liner materials, the data showed that while all materials passed the screening criteria in the aqueous simulant, Kel-F™, HDPE, and XLPE were found to offer the greatest resistance to the combination of radiation and chemicals.

Following the completion of these screening tests, the next phase of this program (i.e., the comprehensive testing of liner materials in the aqueous simulant mixed waste) began. Since screening tests showed that all liner materials met the screening criteria when exposed to the aqueous simulant mixed waste, the five liner materials were subjected to comprehensive testing. The five materials evaluated consisted of HDPE, XLPE, PP, Kel-F™, and Teflon®. The testing protocol involved exposing the respective materials to approximately 143, 286, 571, and 3,670 krad of gamma radiation followed by 7-, 14-, 28-, 180-day exposures, respectively, to the waste simulant at 18, 50, and 60°C. The radiation exposure values were calculated based on γ -ray dose rate data available to us for the components of a pump submerged in a specific storage tank at Westinghouse Hanford Company. These data indicate a maximum γ -ray dose rate in the range of 750 to 850 R/hour. The maximum dose rate of 850 rad/hour was used in calculating the dose that container materials will receive from a ⁶⁰Co source at Sandia National Laboratories. Using this dose rate, the four doses described above were calculated for 7-, 14-, 28-, 180-day exposures, respectively. From the data analyses, the fluorocarbon Kel-F™ was identified as having the greatest chemical durability after exposure to gamma radiation followed by exposure to the Hanford tank simulant mixed waste. The most striking observation was the extremely poor performance of PTFE when exposed to the higher radiation doses. Even at lower radiation exposures, PTFE exhibited significant losses in performance. These results were reported to DOE.⁸

In this report, we present another part of the second-phase testing. Since all seal materials passed the screening tests in the aqueous simulant mixed waste, all seal materials would be subjected to

comprehensive testing. While earlier studies investigated the response of EPDM and butyl rubber, this second-phase study involved the comprehensive testing of SBR. The results of comprehensive testing of EPDM and butyl rubber had first been reported to the DOE. A synopsis of the comprehensive test results for EPDM and butyl rubber was presented at the Fourth Biennial Mixed Waste Symposium⁹ and at PATRAM '98.¹⁰ The complete results of comprehensive testing of EPDM and butyl rubber have also recently been published.¹¹⁻¹² The comprehensive testing protocol involved exposing SBR to a matrix of four gamma radiation doses (~143, 286, 571, and 3,670 krad), three temperatures (18, 50, and 60°C), four exposure times (7, 14, 28, and 180 days), and the aqueous Hanford tank simulant. It should be mentioned that, while some SBR samples were exposed to only the simulant, other samples were only irradiated, and still others were irradiated and then exposed to the simulant to mimic the action of mixed wastes. Following exposure to these conditions, the SBR samples were evaluated by measuring seven material properties. These properties included specific gravity, dimensional changes, mass changes, hardness, compression set, VTR, and tensile properties.

TEST DESCRIPTION

In this section, we describe the experimental aspects of the comprehensive phase of this testing program for elastomeric materials.

Materials

The selected material, SBR, is an elastomer having known chemical resistance to brake fluids, alcohols, and water. SBR is also known under its old name Buna S or GRS (Government Rubber Styrene). The latter term refers to rubber originally made during World War II in U.S. Government-owned plants as a substitute for natural rubber. Appendix A provides additional information on this material including its properties.

Simulant Preparation

The simulant mixed waste form used in this testing phase was an aqueous alkaline simulant Hanford tank waste. Three-liter (L) batches of the simulant were prepared by dissolving 536 g (6.3 moles) of sodium nitrate and 150 g (2.2 moles) of sodium nitrite in deionized water (1800 mL) using a 4-L beaker. After these salts had completely dissolved, 246 g (6.2 moles) of sodium hydroxide were added, stirred, and slightly heated on a magnetic hotplate (Corning, Model PC-320). To this hot (~70°C), stirred solution, 51 g (0.30 moles) cesium chloride and 48 g (0.29 moles) strontium chloride were added. Finally, 96 g (0.9 moles) of sodium carbonate dissolved in 800 mL of deionized water were added to the solution. This latter addition resulted in the formation of a copious amount of white precipitate. Based on its insolubility, it was believed to be strontium carbonate. To the resulting mixture, another 400 mL of deionized water were added to bring the total volume of water used to 3 L. After cooling to near ambient temperature, the stirred mixture was stored in amber glass bottles (Fisher Scientific, #03-327-6). All chemicals used in the preparation of the waste simulant were American Chemical Society reagent grade chemicals. The above composition produced a mixture with the following chemical concentrations:

- 2.1 Molar (M) sodium nitrate
- 0.7 M sodium nitrite
- 2.1 M sodium hydroxide
- 0.3 M sodium carbonate
- 0.1 M cesium chloride
- 0.1 M strontium chloride

Sample Preparation

Standardized test methods were used to cut, condition, and test the materials. The geometry of the material samples was specified by the test method. The samples were cut using an expulsion press (Part # 22-16-00) and dies manufactured by Testing Machines Inc., Amityville, NY. For example, the rectangular (1 in. x 2 in. x 0.125 in., 2.5 cm x 5.0 cm x 0.318 cm) samples required for specific gravity and hardness measurements were cut in the expulsion press fitted with an Expulsion Straight Edge Die (Part #23-10-06). Rectangular samples (1 in. x 3 in. x 0.125 in.,

2.5 cm x 7.6 cm x 0.318 cm) required for dimensional and mass measurements were cut in the expulsion press fitted with an Expulsion Straight Edge Die (Part #23-10-07). Circular discs (0.5-in. diameter x 0.125 in. thick, 1.3 cm diameter x 0.318 cm thick) required for compression set measurements were cut in the expulsion press fitted with a custom circular cutter from CCS Instruments, Akron, OH. Larger circular discs (2.69-in. diameter x 0.125 in. thick, 6.83 cm diameter x 0.318 cm thick) required for VTR measurements were cut in the expulsion press fitted with an expulsion die (Part #23-00-00) specifically designed for American Society for Testing and Materials (ASTM) Standard Test Method D 814¹³ testing. Similarly, the Type C tensile samples required for tensile testing were cut in the expulsion press fitted with an Expulsion Die (Part # 23-14-08) specifically designed for use in the ASTM D 412-Method A.¹⁴

The use of a press and dies permitted the cutting of multiple samples having uniform dimensions. For identification of samples, an identification code was developed to uniquely indicate the test type, sample number, and testing conditions. The black SBR samples were individually labeled using indelible ink marking pens. As recommended by ASTM D 1349,¹⁵ the plastics were conditioned at a standard temperature of 73.4°F (23°C) and relative humidity of 50% for at least 24 hours prior to the testing process. This was done by storing the cut samples in a desiccator filled with magnesium nitrate hexahydrate (500 g) saturated with water. A humidity/temperature sensor was used to monitor the conditions in the desiccator. Procedures for generating this constant relative humidity environment are described in ASTM E 104.¹⁶ During conditioning, the samples were stacked atop each other and separated from each other using ~1/16-in.-thick (~0.16 cm) metal pins. The required samples for each test were bundled together using plastic cable ties by procedures described in a subsequent section.

Sample Quantities

Some SBR samples were exposed to gamma radiation alone, some to the simulant (chemicals) alone, or to a combination of radiation followed by exposure to the simulant. Since radiation was expected to have the greatest effect on the compression set and tensile properties of SBR, we prepared specific samples for radiation exposure alone. These samples were referred to as “Rad Only” samples. The purpose of exposing certain samples to only gamma radiation, while other samples received exposure to both radiation and chemicals, was to differentiate the effects of radiation alone from those when two environmental conditions (radiation and simulant) were applied.

For “Rad Only” compression set measurements, 48 samples (six specimens per test) were cut for the matrix of four radiation doses, four exposure times, and three exposure temperatures for a total of 288 samples. Tensile property measurements (five specimens per test) for “Rad Only” samples required the preparation of 240 samples. A total of 528 samples was needed for these two measurements. In view of the perceived effect of radiation on compression set and tensile property measurements, the material properties of SBR were measured prior to exposure to either radiation and temperature. The values obtained from these measurements represented the initial material properties. These measurements required the preparation of 11 additional samples.

“Simulant Only” samples, referred to as 0 krad samples (OK in the appendices) in subsequent discussions, were required for each of the seven measurements. For specific gravity and

hardness measurements, a total of 12 samples was required. Dimensional and mass measurements (three per test) required the preparation of nine samples. For VTR measurements (three per test), nine samples were needed. Compression set measurements required 72 samples. Finally, tensile property measurements required 60 samples. Thus, a total of 162 samples was required for all seven “Simulant Only” tests. These samples were exposed to the aqueous simulant for the four time periods at the three temperatures.

We now turn to the samples required for exposure to the combination of radiation and chemicals. For specific gravity and hardness measurements, a total of 48 samples were cut out for the combination of three temperatures, four radiation doses, and four time periods. For dimensional and mass measurements, 36 samples were prepared. Compression set measurements involved 288 samples. VTR measurements involved 36 samples, and tensile testing involved 240 samples. Thus for all seven measurements, 648 samples were prepared for exposure to the three temperatures and four radiation doses. For nondestructive tests such as specific gravity, dimensional, mass, hardness, and VTR measurements, the same samples were reused for the other exposure times, i.e., one sample set was used for 7, 14, 28, and 180-day exposures at each temperature and for each radiation dose. A total of 1349 SBR samples was used to perform the various measurements.

Sample Irradiation

The elastomer samples were irradiated by an underwater ^{60}Co gamma source at SNL/NM. These samples were loaded into a metal basket in the same configuration that was used to condition the samples, i.e., the samples were stacked atop each other and separated by a metal spiral or by metal pins. The basket was then inserted into a watertight, stainless steel canister (volume ~4 L). The canister was sealed and lowered into the pool to a depth of 6 feet, purged with a slow, steady flow (~30 mL/min) of dry air and allowed to come to thermal equilibrium at either ambient (~32°C), 50°C, or 60°C.¹⁷ Once thermal equilibrium was attained within the canister immersed in the pool of water, the canister was lowered into its irradiation location in the pool and exposure was begun to obtain the desired radiation dosage. The highest dose rate currently available at the Low Intensity Cobalt Array (LICA) Facility is ~540 krad/hr. The array that was used for irradiating these samples typically had dose rates of ~81 krad/hr. Thus for an irradiation experiment where a gamma-ray dose of 143 krad was required, the samples were exposed for approximately 1.8 hours. For doses of 286, 571, and 3,670 krad (3.67 Mrad), the corresponding longer exposure times were needed. After the samples received the calculated radiation dosage, the canister was removed from the pool, and the samples were again placed in the conditioning chambers. No more than 24 hours typically elapsed between the time that the samples had been exposed to radiation and when they were exposed to the simulant waste or the test temperatures.

Sample Exposure to Simulant

The general exposure protocol for specific gravity samples involved placing four specimens of the SBR into a container and exposing them to the aqueous simulant at three temperatures and four time periods. The four specimens were bundled together using 7-1/2 in. (19 cm) nylon cable ties. Within each bundle, the specimens were separated with ~1/16-in. (0.16 cm) metal pins used as spacers. This allowed for the ready access of the waste simulant to all surfaces of each specimen. A 2-L plastic container was loaded with the four bundled test specimens and then

filled with 1,600 mL of the test solution. Care was taken to ensure that sufficient simulant waste was present to expose the entire surface area of all the samples. After adding the liquid simulant waste, the plastic lid was attached to the jar and tightened. The jars were placed in respective environmental chambers maintained at 18, 50, and 60°C. The jars were kept in these environmental chambers for 7, 14, 28, and 180 days. Similar procedures were followed for each of the other four testing procedures, i.e., dimensional testing, hardness testing, compression set tests, and tensile tests. In the case of VTR measurements, each of three 1/2 pint (~236 mL) Mason Jars (Kerr Group, Inc., Los Angeles, CA, Part # 70610-3) was filled with approximately 200 mL of the test solution. The SBR discs were loosely attached to the jars with metal bands. The jars were placed in an upright configuration (SBR and band facing up) into the respective environmental chambers. The jars were held at the respective test temperatures for one hour to equilibrate. After sealing and weighing them, the jars were placed in the chambers again in an inverted position and held at the specific test temperature for the required time period.

Experimental Approach

The material properties that should be evaluated to assess the suitability of potential elastomeric materials in mixed waste packaging designs are specific gravity changes, dimensional changes (involving mass and dimensional measurements), hardness, compression set, VTR, and tensile property changes (tensile strength, tensile stress, and ultimate elongation). Since the measurement of all these material properties was expected to be costly and time-consuming, screening tests with relatively severe exposure conditions such as high temperatures and high radiation levels were implemented to quickly reduce the number of possible materials for full evaluation. It is generally recognized that polymeric materials are susceptible to degradation caused by high temperature and high radiation doses. The results of these screening studies have been previously reported at several technical conferences^{2,6} and in a SAND Report.⁷ From the screening study, it was found that all of the selected seal materials had passed the screening criteria in the aqueous simulant mixed waste. This then necessitated the testing of these six materials by exposure to a matrix of four radiation doses, three temperatures, and four exposure times in the simulant waste. In view of the extensive number of materials and exposure conditions, this second phase of the program was referred to as the Comprehensive Testing Phase.

Because of budget constraints imposed on this program, testing was subdivided into comprehensive testing on liner materials and seal materials. Additional funding constraints required still another subdivision of the testing activity that was evaluating individual elastomers. The order of testing for the individual elastomers was established by the degree of response in the aqueous simulant. In other words, the best elastomer was evaluated first while the worst elastomer was evaluated last. From the data given in a previously published report,⁷ the best-to-worst materials in the aqueous simulant were determined to be EPDM, butyl, SBR, Nitrile, Viton®, and EPI rubber. Accordingly, the first material evaluated was EPDM rubber,¹¹ followed by butyl rubber.¹² The next elastomer to be evaluated was SBR.

The evaluation parameters used in this comprehensive testing phase consisted of measuring the specific gravity, dimensions, mass, hardness, compression set, VTR, and tensile properties of the seal materials. Tensile properties for elastomers included tensile strength, ultimate elongation, and tensile stress. These parameters were evaluated using standardized test methods such as

those developed by the ASTM. For specific gravity measurements, ASTM D 792¹⁸ was used. In measuring dimensions and mass, ASTM D 543¹⁹ was used. For hardness measurements, ASTM D 2240²⁰ was used. In evaluating compression set, ASTM D 395 - Method B²¹ was used. However, in using ASTM D 395, six samples (3 sets of 2 samples) of 0.125-in. thickness (3.17 mm) each were held at the three temperatures with a spacer bar thickness of 4.5 mm. For VTR measurements, ASTM D 814 was used. Finally, for evaluating tensile properties, ASTM D412 - Method A was used.

Before describing the results of this study, we will discuss the comprehensive testing strategy used for SBR. This strategy is shown in a flow diagram in Figure 1. The rubber was subjected to four different protocols (Paths A-D). To determine the intrinsic properties of the materials, "Baseline" property measurements (Path A) were made in each of the seven tests. These properties were measured at ambient laboratory conditions. In order to differentiate the effects on the materials by radiation and chemicals, one series of samples was exposed to the simulant alone (Path B), while the other series of samples was exposed to both radiation and the simulant (Path C). The first series of these samples is referred to as "Simulant Only" in the flow diagram, while the latter was referred to as "Radiation and Simulant". It should be noted that both series of samples were exposed for the four time periods (7, 14, 28, and 180 days) at three different temperatures (18, 50, and 60°C). For two testing protocols, tensile testing (Tensile) and compression set (Set), where the effects of radiation and temperature alone could have significant impact on these properties, a series of samples described as "Radiation Only" is shown in the flow diagram (Path D). These samples were irradiated at three temperatures previously given and then exposed for the four previously given time periods at the three respective temperatures. What may not appear obvious from the flow diagram is the large number of samples being tested in this comprehensive testing phase of the program. A total of 2,126 measurements on 1,349 samples was analyzed (Appendices B-J).

Figure 1. Comprehensive seal testing strategy.

RESULTS

Specific Gravity

Specific gravity measurements, also known as relative density measurements, measure the densities of materials under different conditions. A decrease in density of the material can indicate leaching or swelling, and swelling can lead to increases in permeability. Such indications of increases in permeability in the material will also be confirmed by VTR measurements. Increases in density are caused by absorption of the test liquid, which leads to increases in mass of the material and can also lead to higher permeability of the test liquid.

In Figure 2, the effects of exposure time, gamma radiation dose, and exposure temperature of the aqueous simulant on SBR are shown.

Figure 2. Specific gravity (S. G.) changes in SBR after exposure to ~0, 143, 286, 571, and 3,670 krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60 °C.

These three-dimensional bar graphs provide a plot of radiation dose, exposure temperature, and the average percent specific gravity change in the x, y, z directions, respectively. Where the radiation dose is indicated as 0 krad, the samples received no gamma radiation and were only exposed to the simulant (i.e., these samples are the "Simulant Only samples discussed earlier). It should be noted that the scale for these specific changes is rather small, e.g., from 0 to 1%, and either positive or negative. In Figure 2 and all subsequent figures, negative changes can be recognized by the dark bar tops in the x-y plane. These bars project into the negative portion of the graph. The sign of the specific gravity indicates whether specific gravity has increased or decreased when compared to the pristine material, i.e., the specific gravity of SBR at ambient conditions. Therefore, changes in the magnitude and the sign of specific gravity values indicate changes in this property. The greater the absolute values of the changes, the more the materials are affected by the specific set of environmental conditions. Since properly engineered packaging components are not expected to be affected by contents of the package, such as aqueous mixed wastes, elastomers exhibiting the smallest changes in specific gravity should be selected as packaging components. From an overall perspective, the data in Figure 2 show that temperature of the simulant, the radiation dose, and the exposure time have some slight effects on the specific gravity of SBR. With increased temperatures, increased radiation exposure, and longer exposure time, increased specific gravity changes were observed. Radiation exposure alone appears not to exhibit a significant effect on the specific gravity SBR. However, since these changes are only at the 1% levels, these results are consistent with the known chemical resistance of this elastomer and demonstrates that SBR is a suitable elastomer for use under these conditions if specific gravity is the determining package design criterion. While the precise specific gravity values are not obvious from the data in the previous figure, their actual values can be found in Appendix B of this report.

In the following section, we will present the results of the effects of only the simulant waste and a combination of radiation and the simulant on the dimensional properties of SBR.

Dimensional Properties

Similar to specific gravity measurements, or density changes, the dimensional property measurements can provide important information about the effects of different environmental conditions on materials. Specifically, the swelling of the material or leaching of components of the material may be manifested by increases or decreases in the dimensions of the material. The dimensional properties measured and reported in this section include changes in length, width, and thickness of the material. Since the standard test method (ASTM D 543) used to measure dimensional properties includes the determination of mass as part of the test, this property was also measured. Dimensional changes are described by evaluating the product of these changes, i.e., volume (length x width x thickness). The technical justification for using this approach is that, while length and width changes have generally been much smaller than thickness changes, the product of these changes encompasses individual components into one general dimensional property, the volume of the materials. The effect of the different environmental conditions on sample mass changes will be presented first.

To measure the effect of exposure time and exposure temperature of the aqueous simulant on SBR, the mass of the samples was measured before and after exposure to only the surrogate waste. The results are given in Figure 3 (a) - (d) above in the 0 krad data field. Similar to data shown in the previous section, the scale for average % mass change is very small, e.g., from 0 to 3%. The sign of the mass changes indicates whether the mass of the material has increased or decreased when compared to the pristine material, i.e., the material's mass at ambient conditions. Therefore, changes in the magnitude and the sign (positive or negative) of % mass change values can vary in these measurements. The greater the absolute values of these changes, the more the material is affected by this set of environmental conditions. Since properly engineered packaging components are not expected to be effected by contents of the package, i.e., the mixed wastes, materials exhibiting the smallest changes in mass should be selected as packaging components. From an overall perspective, the data in Figure 3 show that temperature of the simulant and exposure time have a slight effect on the changes in mass of the material. For both of these environmental conditions, a slight but perceptible decrease in mass was observed. After 180-day exposures, mass losses in excess of 1% were observed at 50 and 60°C. At higher temperatures, the greater mass losses were observed. While the exact mass values are not obvious from the data in the previous figure, their precise values can be found in Appendix C of this report.

Figure 3. Mass changes in SBR after exposure to ~0, 143, 286, 571, and 3,670 krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60°C.

In Figure 4 (a) - (d), the average % volume changes of SBR exposed to the four gamma radiation doses followed by exposure to the aqueous simulant waste at 18, 50, and 60°C for 7, 14, 28, and 180 days is given. SBR had volume changes of less than ~1.7% under these conditions. In most cases these volume changes were much smaller, i.e., in the ~0.8% range. At elevated temperatures, small decreases in volume were observed. The largest (>1%) of these changes was found at 60°C. These cases are recognized by the darkly colored top surface of the bar graph. With increased exposure time and exposure temperature, there is a decrease in the sample volume, i.e., SBR appears to shrink when exposed to these environmental conditions. A general trend suggests that, with increasing exposure time, most of the SBR samples shrank. The notable exception is for samples exposed at 18°C. Most of these samples exhibited slight swelling, i.e., increases in volume, with increasing exposure time. The greatest volume changes can be seen in Figure 4d, where SBR exhibited the greatest decrease in volume of nearly 2% at 50 and 60°C. It should be noted, however, that these decreases are only at the 1 - 2% levels. While the exact volume values are not obvious from the data in the previous figure, their specific values can be found in Appendix D of this report.

Figure 4. Volume (Vol.) changes in SBR after exposure to ~0, 143, 286, 571, and 3,670 krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60°C.

Hardness Properties

The measurement of changes in the hardness of materials can provide important clues about the effects of environmental conditions on the material. If the hardness of the material is decreasing, swelling of the material may have occurred. Alternatively, the polymeric constituents of the elastomer may have substantially degraded. Conversely, if the hardness of the material is increasing, additional cross-linking of the polymer may have resulted. The results of these measurements, in addition to providing important data, may complement other measurements such as specific gravity, dimensional, and tensile properties.

The measurement of hardness involves the use of a standard instrument manufactured by Shore Instrument Company known as a Shore durometer. The degree of hardness that the plastic material exhibits will dictate the type of durometer used. For elastomers, which in relative terms tend to be rather soft, a Type A durometer is used. Similar to the approach used for the previously described property measurements, the initial hardness values (~63 Shore A hardness points) were determined for pristine samples, i.e., samples not exposed to anything. Using these initial hardness values, % hardness changes were measured for samples that were exposed to the simulant alone (see 0 krad data field in Fig. 5 (a) - (d) at the three temperatures and four exposure times) and to a combination of radiation and simulant at these temperatures and exposure times. We will now present the results of these measurements.

To measure the effect of exposure time and exposure temperature of the aqueous simulant on SBR, hardness testing was performed on the materials exposed to the surrogate waste alone at the three temperatures and four time periods. The results of these measurements are given in Figure 5 (a) - (d) in the 0 krad data field. The sign of the hardness changes indicates whether the hardness of the material has increased or decreased when compared to the pristine material. Decreasing hardness indicates that the material has become softer as a consequence of the exposure conditions. As was previously mentioned, properly engineered plastic packaging components are not expected to be affected by the packaging contents. An elastomer exhibiting the smallest changes in hardness should be considered a good candidate as a packaging component.

Figure 5. Hardness (Hard.) changes in SBR after exposure to ~0, 143, 286, 571, and 3,670 krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60°C.

An inspection of the results in Figures 5 (a) - (d) reveals that, in general, the hardness of SBR essentially remained unchanged for exposures up to 28 days. Under these conditions, some of the samples became harder, and other samples exhibited decreases in hardness, i.e., became somewhat softer. The notable exceptions were samples exposed at 18°C. These samples consistently became slightly harder with increasing radiation doses. When SBR samples were exposed for 180 days (Figure 5d), all the samples which received a combination of radiation and chemical exposure exhibited a 2 to 3% increase in hardness. The hardness changes were not strongly affected by radiation dose or temperature. The largest hardness change was observed in samples that received radiation doses of 571 krad and 180 day exposures to the 60°C simulant. These results suggest that prolonged exposure to this oxidizing simulant results in some hardening of SBR. Since decreases in volume under these conditions (Figure 4) were observed, this hardening appears to be caused by contraction of the material. An alternative explanation for the increasing hardness is the loss of the plasticizer component in the rubber. Since decreases in

mass were also observed under these conditions (Figure 3d), the increasing hardness could possibly also be explained by losses in elasticity of the material. While the actual hardness values are not obvious from the data in the previous figure, their precise values can be found in Appendix E of this report.

Compression Set

Compression set tests are used to measure the ability of elastomers to retain elastic properties after prolonged action of compressive stresses. Set is usually determined in air and reported as the percent of deflection by which the elastomer fails to recover after a fixed time and under a specified compression and temperature. In these experiments, the time period of compression was 22 hours at 18, 50, and 60°C. A complete return by the elastomer to its original thickness, after the compressive stresses are removed, results in a calculated set of 0%. For a situation where the elastomer does not return to its original thickness but remains at the thickness under compression (in this case a 4.5 mm deflection), a set of 100% is calculated. These values, therefore, represent a percentage of the original deflection. The practical aspect of such a situation is that the elastomer just contacts the matting surface of the device that contains the elastomer. In the case of O-rings seals, this situation could lead to seal failure since the elastomeric seal makes minimum contact with the sealing surface. It should be obvious from the previous discussions that materials having a low set value are desirable. To measure set, we have used the standardized test method, ASTM D 395. Using this method, the SBR samples were held in the compression set device at three temperatures. Figure 6 shows an example of the experimental configuration used for the compression set tests.

In order to understand effects of radiation alone on set values, a portion of the SBR samples were exposed to only the four radiation doses (i.e., no chemical exposure), the three temperatures, and the four exposure times. Similarly, the effects of the simulant alone were studied under these conditions. Finally, samples exposed to a combination of radiation and simulant were studied. We will now proceed to discuss the results of these different measurements. It is important to mention that the results shown in Figures 7 and 8 plot changes in compression set versus radiation dose and temperature. These changes represent the difference in the set observed under the specific environmental conditions and the set of pristine SBR samples. Because set values are expressed in percent, the change in set is the difference of these values in percent.

Figure 6. Compression set fixture: (a) a partly assembled fixture with the 4.5 mm spacer bars and butyl rubber samples, and (b) an assembled fixture with SBR samples.

In Figure 7 (a) - (d), the set changes for SBR samples exposed to four gamma ray doses followed by 7-, 14-, 28-, and 180-day exposures at the three temperatures are provided. The data in Figure 7 represents a situation where SBR has only been exposed to gamma radiation. The important point to keep in mind for these measurements is that the set was measured after being held for 22 hours at the three temperatures. Another way to express this important experimental detail is that only the samples themselves saw the different radiation doses, exposure times, and exposure temperature. The compression set measurements were performed at one time and three temperatures. These compression set measurements on SBR were thus different from the measurements on EPDM and butyl rubber where measurements were performed at one time and one temperature (ambient).

The results show that while several samples exhibited an increase of the set in the range of ~ 0 to 0.2%, most samples had decreases of the set in the range of ~ 0 to 3%. Decreases for changes in the set indicate that the samples after exposure to these environmental conditions had a smaller set than the unexposed samples, i.e., these samples were more elastic than the unexposed samples. Most decreases in set were observed in samples with larger gamma radiation dose exposures and/or longer exposure times. However, systematic trends in samples exposed to increasing radiation doses, exposure time, and exposure temperatures are not very pronounced. These results suggest that these set changes are not very large and possibly within the experimental variability of the test results. However, an overall decrease in set was detected with increasingly severe environmental conditions of dose, time, and temperature.

Figure 7. Compression set (C. S.) changes in SBR after exposure to ~143, 286, 571, and 3,670 krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days at 18, 50, and 60°C.

Figure 8 (a) - (d) shows the set changes for SBR samples exposed to a combination of the four gamma ray doses followed by 7-, 14-, 28-, and 180-day exposures to the aqueous simulant at the three temperatures. Similar to the samples exposed to gamma radiation alone, the set changes exhibited by samples exposed to a combination of radiation and the aqueous simulant were minimal, i.e., in the range of -2 to 2%. The set in SBR samples exposed to only the simulant (0 krad data field) generally also decreased with increased temperature and increased exposure time. The combination of radiation followed by exposure to the simulant has a beneficial effect by resulting in lower compression sets, i.e., the samples became more elastic. The greatest retention of deformation was noted in samples exposed to radiation and the simulant for 180 days at 18°C.

Figure 8. Compression set (C. S.) changes in SBR after exposure to ~0, 143, 286, 571, and 3,670 krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60°C.

To summarize, SBR is not affected greatly by radiation, the simulant, or a combination of these two environmental conditions. While the precise set values are not obvious from the data in the previous figure, the specific values can be found in Appendix F of this report.

Vapor Transport Rates

VTR measurements provide a measure of the permeability of various chemical agents in elastomers. The rate of transmission of a liquid through an elastomer that acts as a barrier is important in elastomer seal performance. This transmission is referred to as vapor transmission since the liquid diffuses through the elastomer in a molecular sense and escapes into the surrounding atmosphere in vapor form. This type of testing provides a steady-state measure of the rate of vapor and liquid transmission through relatively thin elastomers. While the calculated values of VTR cannot be directly converted to traditional permeability values, the VTR values can be used to provide an indication of permeability.

The VTR measurements were performed at three temperatures and four exposure times. In these experiments, one set of SBR samples was exposed to only the simulant aqueous waste, while the remaining samples were exposed to a combination of radiation and the simulant. To measure VTR, we have used the standardized test method, ASTM D 814. Using this method, the SBR samples were sealed to a ground-glass surface using a metal screw band. It should be noted that VTR experiments by this method with SBR samples exposed to only gamma radiation are not possible since the testing method requires the presence of a chemical agent. Figure 9 shows a group of cells used for VTR measurements.

For VTR measurements, it is not possible to describe these data in terms of VTR changes. The reason for this inability to determine VTR change is that it is not possible to determine VTR on pristine SBR. The pristine property value of the material was used to calculate changes in that property. For example, in the previous section the compression set value of SBR that had not been exposed to radiation, the simulant, or the combination of radiation and simulant was used to define the compression set of pristine SBR, i.e., its initial value. In VTR measurements, however, a similar VTR value for pristine SBR is not possible since its determination will require exposure to a simulant. However, one could show VTR changes between SBR samples exposed to only the simulant and samples exposed to the combination of radiation and simulant. Because all the previously described measurements included data for SBR samples exposed to the simulant alone, we retained this data format here. We will provide the VTR values, in g/hr/m^2 , rather than changes in VTR.

Figure 9. VTR cells.

Figure 10 (a) - (d) shows VTR for SBR samples exposed to a combination of the four gamma ray doses followed by 7-, 14-, 28-, and 180-day exposures to the aqueous simulant at the three temperatures.

Figure 10. VTR for SBR after exposure to ~0, 143, 286, 571, and 3,670 krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60°C.

The data shown for the 0 krad dose represents samples exposed only to the simulant for the four exposure times and temperatures. All samples exhibited VTR values below 1 g/hr/m². In fact, most of the VTRs for these samples are below 0.3 g/hr/m². The SBR samples that had only been exposed to the simulant generally exhibited higher VTR at all temperatures than did samples exposed to the radiation. These effects are easily seen for the 60°C data points in Figure 10 a - d. It can also be seen that the VTR for SBR samples generally increased with increased temperature and decreased with increasing exposure time. The effects of increasing radiation dose on VTR are less clear. At both 18 and 60°C, minimum VTR is observed for SBR samples that received gamma-ray doses between 286 and 571 krad. While the specific VTR values are not obvious from the data in the figure, their precise values can be found in Appendix G of this report.

Tensile Properties

Tensile properties, also known as mechanical properties, are the properties associated with a material's response to mechanical forces. A quantity more useful than force is the engineering stress (σ), which is the ratio of the magnitude of a force to the magnitude of the originally undeformed area of the body upon which it is acting. True stress is therefore defined as $\sigma = F/A$, where A is the cross-sectional area at the time that force (F) is applied. The most common engineering units of stress are pounds of force per square inch (lb/in.² or psi). These units may be converted to the corresponding SI unit, the Pascal (Newton/meter) by multiplying the psi value by 6895. However, we are always calculating the % changes in properties, and the units are irrelevant. The actual values in Mega Pascal (MPa) are reported in Appendix H.

Another important tensile property to consider is strain. A stressed material undergoes deformation or strain (ϵ), defined quantitatively as either the incremental deformation divided by the initial dimension or the percent of the original dimension. Since strain is a dimensionless quantity, the precise choice of units is not important. In this study, a 1-inch gage length was used, and the units of strain are therefore inch/inch. Two fundamentally different types of strain are observed. The first type is elastic strain or elastic deformation, where strain is recoverable upon the release of stress. In other words, when a causal stress is removed, the resultant strain vanishes, and the original dimensions of the object are recovered. A practical example of this type of strain is the stretching of a rubber band. Since SBR is a specific type of rubber, this material exhibits this type of strain. The second type of strain is plastic strain. This occurs when stress is increased, and a value is eventually reached where permanent deformation of the object has occurred. An example of this property is the bending of wire with the fingers. Note that "plastic strain" does not necessarily mean that the deformed material is a plastic.

For many plastic materials that might be suitable as packaging components such as seals and liners, high strengths and high strains are expected from the material. The strains exhibited should also be elastic in nature. In certain instances, however, other specific tensile properties are desirable, i.e., high strength and low strain. It was the purpose of this study to determine the tensile properties of the pristine material and then determine the effects of radiation alone, the simulant alone, and a combination of these environmental conditions on the tensile properties of SBR.

Tensile Strength

The tensile or ultimate strength of a material is calculated by dividing the observed maximum load placed on the material during the tensile test by the original cross-sectional area of the material. While many polymeric materials exhibit stress-strain curves having an initial maximum followed by lower stresses, this is not the case for elastomers. The maximum load value in elastomers is typically observed at the break point of the material.

The measurement of tensile properties involves the use of tensile testing equipment that can apply controlled tensile loads to test specimens. The equipment is capable of varying the speed of load (stress) and accurately measuring the forces (strains) and elongation applied to the specimens. In this study, an Applied Test System, Inc. Universal Testing Machine, Series 1400, was used. This computer-controlled testing equipment was able to perform the required tests with user-developed testing methods. These methods prescribe the strain rates and breaking points along with many other experimentally important variables. The selection of these

experimental variables was based on the standard test method ASTM D 412. For the determination of the tensile strength of elastomers, the use of a high elongation extensometer and high rates of grip separation (50 mm/min, 20 in./min) were used. The acquired data were analyzed with software developed by this manufacturer. The software calculates numerous tensile properties. The data discussed in this subsection require a determination of tensile strength. This can be calculated as described previously, using peak loads and cross-sectional area. In addition, the software also calculates ultimate elongation and tensile stress values. In this subsection, tensile strength values were of interest.

Since an understanding of the effect of mixed waste environments is not possible without understanding the effects of radiation and simulant alone, the latter experimental conditions were also investigated. The results of tensile strength changes in the materials exposed to gamma radiation at the three temperatures and four exposure times are provided in Figure 11. In Figure 11 (a) - (d), the average % tensile strength changes of SBR exposed to gamma radiation alone at 18, 50, and 60°C for 7, 14, 28, and 180 days is shown. Similar to previous property measurements, these % changes were determined by measuring the change in tensile strength from that of the pristine material (12.6 MPa). When the change in tensile strength (expressed as a percentage) was a positive value, the material tensile strength had increased under the specific exposure conditions. Negative values indicate decreases in tensile strength.

Figure 11. Tensile strength (T. S.) changes in SBR after exposure to ~143, 286, 571, and 3,670 krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days at 18, 50, and 60 °C.

From a general perspective, Figure 11 shows no significant effect on the tensile strength for most radiation doses, exposure times, and exposure temperatures tested. A notable exception is SBR samples exposed to 571 and 3,670 krad at higher temperatures and longer exposure times. Under these conditions, the changes in tensile strength for these samples were in the range of 10% to 20%. However, for most of the other exposure conditions, many of these changes appear to be in the range of $\pm 1 - 7\%$. At the longest exposure time (180 days, 50°C) and the highest radiation dose, the tensile strength of SBR appears to have decreased considerably ($\sim 20\%$).

Figure 12 shows the average values of percentage of tensile strength changes of SBR exposed to the four gamma radiation doses followed by exposure to the aqueous simulant waste at 18, 50, and 60°C for 7, 14, 28, and 180 days. SBR samples that were only exposed to the simulant waste (0 krad data field) only showed a decrease in tensile strength at the higher temperatures and longest exposure time (180 day). For SBR samples exposed to both radiation and the simulant, the general trend is that neither of these environmental factors have a significant effect on the tensile strength of the material.

Figure 12. Tensile strength (T. S.) changes in SBR after exposure to ~0, 143, 286, 571, and 3,670 krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60°C.

With increasing radiation dose, the tensile strength of SBR did not increase or decrease by more than 12%. Only at the highest radiation dose (3,670 krad) did the tensile strength decrease to 11% with increasing exposure time and increasing temperature. From these results, it appears that elevated temperatures and exposure times of more than 28 days also have slight effects on the tensile strength of SBR. After 180 days of exposure, most of the SBR samples only decreased in the range of 1 - 7%. These results indicate that the tensile strength of SBR is not affected by either radiation, the simulant of the combination of both conditions. While the specific tensile strength values are not obvious from the data in the figure, their precise values can be found in Appendix H of this report.

Elongation at Break or Ultimate Elongation

As discussed previously, the stress-strain diagrams of linear polymers exhibit an initial maximum stress value. This maximum stress value occurs at the yield point of the material. At this point, deformation starts to localize in the material, forming a “neck”, and the material is said to undergo “necking”. However, since elastomers are extremely elastic, necking is not observed in these materials. The determination of elongation at yield in elastomeric materials, as opposed to thermoplastic materials, is not possible. In elastomers, the maximum stress value occurs at the break point of the material. The amount of elongation that the material exhibits at this point is known as the elongation at break. For elastomers, the term ultimate elongation is used rather than elongation at break. The ultimate elongation of an elastomer is defined by Eq. 1 as

$$\text{Ultimate Elongation} = [(L_f - L_o)/L_o] \times 100 \quad \text{Eq. 1}$$

where L_o is the initial gage length (1 in. in this study), and L_f is the gage length at the break point. These ultimate elongation values are expressed as a percent. It should be clear that increasing values of ultimate elongation equate to increasing elasticity in the material, while decreasing values represent decreasing elasticity. The data presented in the following sections will describe the change in elongation. These values were obtained by subtracting the ultimate elongation of the pristine material (251%) from the ultimate elongation observed in the material at the specific environmental conditions. As in previous measurements, positive and negative values for changes in ultimate elongation are possible. Values are provided in Appendix I.

In Figure 13 (a) - (d), the average changes in ultimate elongation of SBR exposed to the four gamma radiation doses followed by exposure at 18, 50, and 60°C for 7, 14, 28, and 180 days are given. It should be mentioned that the scale for ultimate elongation changes is considerably larger than shown in previous figures. In this section, the scale ranges from -100% to 100%. Since the pristine elongation of SBR is 251%, this range corresponds to an ultimate elongation range of ~151% to 351%. While the scale is larger than in previous property measurements, it is still less than observed^{7,8} for thermoplastics. For the latter thermoplastic materials, the scales ranged from -600% to 1000%. These results suggest that the necking observed in thermoplastics play an important role for accommodating deformation.

Figure 13. Ultimate Elongation (U. EL.) changes in SBR after exposure to ~143, 286, 571, and 3,670 krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days at 18, 50, and 60°C.

The data generally show a 4% decrease in ultimate elongation. With increasing exposure time, there is a net decrease in ultimate elongation, but as a whole the ultimate elongation remained unchanged. At the longest exposure time of 180 days, the greatest changes in ultimate elongation were observed. For this exposure time, decreases in ultimate elongation of 10 to 24% were observed. With increasing temperatures, decreases in ultimate elongation in the 10% range were observed. No general trends can be detected from the data for SBR exposed to increasing radiation doses. These results indicate that the elasticity of SBR is not strongly affected by gamma radiation.

Figure 14 shows the average changes in ultimate elongation for SBR exposed to the four gamma radiation doses followed by exposure to the aqueous simulant waste at 18, 50, and 60°C for 7, 14, 28, and 180 days. Similar to the SBR exposed only to gamma radiation, there is a general decrease in ultimate elongation with increased exposure time. The decreases in ultimate elongation are in the 1 to 11% range. The SBR samples exposed to only the aqueous simulant (0 krad radiation dose) also follow this trend but to a lesser degree. These results suggest that the simulant alone does not result in a strong decrease in ultimate elongation in SBR, i.e., SBR is

relatively resistant to the chemical effects of this simulant. With increasing exposure times, SBR exhibited slight decreases in ultimate elongation values. However, these decreases are only in the range of 10%. Upon exposure for 180 days, most SBR samples showed a net loss in ultimate elongation. These results indicate that when SBR receives a longer exposure to the simulant, its elasticity decreases somewhat. The specific ultimate elongation values are provided in Appendix I of this report.

Figure 14. Ultimate elongation (U. EL.) changes in SBR after exposure to ~0, 143, 286, 571, and 3,670 krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60°C.

Tensile Stress or 100% Modulus

For most materials, the initial portion of a stress-strain diagram is linear. This implies that strain is proportional to stress. The proportionality constant (slope of this linear region) is called the modulus of elasticity. The modulus of elasticity, or Young's modulus, is a property of the stressed material. In fact, the magnitude of the modulus can be related to the nature of the chemical bonds existing in the material. Therefore, the modulus provides a measure of the bonding strength in the material being investigated. High values of modulus indicate that strong

bonding is present in the material. As one might surmise from the previous discussion, materials having strong covalent bonding have the highest modulus values. Thus, the larger the value for modulus, the stronger the bonding is expected to be in the material. Modulus has the same units as stress (psi or MPa). The rubber industry also refers to the modulus of a compound. They give it a specific designation such as 100% modulus or 300% modulus. This is due to the fact that the value generated is not an engineering modulus as mentioned previously, but rather is the stress required to obtain a given strain. Therefore, the 100% modulus, is simply the stress (σ) required to elongate the elastomer to twice its reference gage length. Rather than representing the slope of a region in a stress-strain curve, the 100% modulus represents a single data point on the curve. The 100% modulus or tensile stress of an elastomer has identical units as the engineering modulus. Since we are interested in measuring changes in the tensile stress of the exposed material from that of unexposed or pristine material (3.51 MPa), we will discuss the % change in tensile stress of the materials. This is calculated from the relationship given in Eq. 2:

$$\% \text{ Change in Tensile Stress} = (\sigma_f - \sigma_o) / \sigma_o \times 100 \quad \text{Eq. 2}$$

where σ_f is the measured tensile stress under the specific environmental conditions at 100% elongation, and σ_o is the tensile stress of the pristine material at 100% elongation. The 100% modulus changes can be a positive or negative value depending on the magnitude of either σ_f or σ_o . Positive changes in % tensile stress indicate that the material of interest required greater application of stress to elongate the elastomer 100% than for pristine material. Negative values indicate that the material of interest required less application of stress than the pristine material. Appendix J provides the actual tensile stress values of SBR under the different environmental conditions and the % change.

In Figure 15 (a) - (d), the average % change in tensile stress of SBR exposed to the four gamma radiation doses followed by exposure at 18, 50, and 60°C for 7, 14, 28, and 180 days is given. Increasing exposure to gamma radiation doses does not result in large increases or decreases in tensile stress. Similarly, increasing the exposure temperatures has no dramatic effect on the tensile stress of SBR. The changes ranged from -17% to just over 19%. Even though some of the samples exhibited a slight decrease in tensile stress, other samples had slight increases in tensile stress. At higher radiation dose and temperatures, there was a general trend toward decreasing tensile stress. Consistent with previous tensile property measurements, the larger changes were observed for samples exposed to the highest radiation dose (3,670 krad). Over the four exposure times the decreases in tensile stress ranged from 3 to 17%. These trends are generally consistent with softening in the material, i.e., increased plasticity of the polymer chains. The latter observation is in agreement with a decrease in the rubbery qualities of the material, which has been confirmed by decreases in hardness (Figure 5), and decreases in tensile strength (Figure 12), and slightly increasing ultimate elongation (Figure 13).

(a)

(b)

(c)

(d)

Figure 15. Tensile stress (T. ST.) changes in SBR after exposure to ~143, 286, 571, and 3,670 krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days at 18, 50, and 60°C.

Figure 16 (a) - (d) shows the average percentage of change in tensile stress of SBR exposed to the four gamma radiation doses followed by exposure at 18, 50, and 60°C to the aqueous simulant waste for 7, 14, 28, and 180 days. Similar to the "Rad Only" data in Figure 15, the larger changes in tensile stress are observed in samples exposed to the highest (3,670 krad) radiation doses. The largest of these are on the order of ~ -25%. At this radiation dose, the tensile stress values ranged from ~ -3% to -25% of the pristine SBR's tensile stress values. Comparing the results of Figures 15 and 16, SBR exposed to both radiation and the simulant waste had slightly lower tensile stress values at the higher radiation doses. Since tensile stress is a measurement of the stiffness²³ of the elastomer, the combination of radiation and chemical exposure has resulted in a slight decrease in the stiffness of the material.

(a)

(b)

(c)

(d)

Figure 16. Tensile stress (T. ST.) changes in SBR after exposure to ~0, 143, 286, 571, and 3,670 krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60 °C.

These results suggest that the simulant has counteracted the embrittling action of radiation, i.e., the simulant appears to cause some plasticizing action. It should be mentioned that plasticizing agents are compounding ingredients in rubber formulations that contribute to the rubbery properties of elastomers. The action of the simulant under these environmental conditions appears to result in some plasticization of SBR. With increasing exposure time, the tensile stress values appear to decrease somewhat initially and then increase somewhat. This points to an initial loss in stiffness of the elastomer followed by an increase in the stiffness. The results in Figure 16 also show that increased exposure temperatures generally do not have any significant effect on tensile stress. It appears that SBR is remarkably resistant to the effects of low radiation doses, the simulant, and both environmental conditions. However, at the highest radiation doses of 3,670 krad, some loss of tensile stress in SBR was evident.

DISCUSSION

The purpose of the Chemical Interaction Program, previously referred to as the Chemical Compatibility Program, is to provide a scientifically defensible methodology for measuring the chemical interactions of polymeric liner and seal materials with hazardous wastes. These polymeric materials are those that may be used in current and future container designs for the transportation of hazardous and mixed wastes throughout the DOE complex.

With the completion of the screening phase of the program several years ago, the comprehensive phase of this program has been in progress. Since all seal and liner materials passed the screening tests when exposed to the simulant Hanford tank waste, ten materials needed to be subjected to the test matrix. This results in an extremely large sample and test set. In view of manpower and budget constraints, the comprehensive testing phase of the program was subdivided into the testing of liner materials and seal materials. The results of liner testing is the subject of a previous milestone⁷ report. Because of further funding constraints, the comprehensive testing of seal materials was subdivided into the testing of individual elastomers. In previous reports,^{11,12} we discussed the results from testing of the first two elastomeric seal materials, EPDM and butyl rubber. This report describes the testing results from the next elastomeric seal material, SBR. Subsequent reports will discuss the testing results for a fluorocarbon elastomer exposed to simulant Hanford tank waste. We will now proceed to discuss the results obtained for each of the properties measured starting with specific gravity and ending with tensile stress of SBR.

From an overall perspective, the specific gravity data show that simulant temperature, radiation dose, and the exposure time have slight effects on the specific gravity of SBR. These results are consistent with the known chemical resistance of this elastomer and demonstrate that SBR is a suitable elastomer for use under these conditions if specific gravity is the determining design criterion for selection of packaging seal components. Increasing exposure times, radiation doses, and exposure temperatures generally caused increases in specific gravity of less than 1%. The greatest changes were noted for the samples exposed to the most extreme exposure conditions, i.e., at 3,670 krad and 60°C. SBR samples that were not irradiated in most sometimes displayed smaller increases in specific gravity than samples exposed to both radiation and the simulant. However, with longer exposure times, specific gravity values similar to combination conditions were observed. These results indicate that SBR may be affected to a greater extent by the combination of radiation and chemical exposure and that SBR is less resistant to radiation exposure.

The mass of SBR decreased slightly after exposure to the simulant or the combination of radiation and simulant at increasing exposure times and exposure temperatures. These decreases in mass were observed only at the elevated temperatures of 50 and 60°C. At 18°C, SBR exhibited slight (~0.2%) weight gains. The largest decreases in mass of ~1.3% were observed after 180-day exposures. Since the mass changes are not very large, the slight increases in specific gravity noted earlier could be caused by a combination of mass and dimensional changes. Specifically, the volume of the SBR could decrease to a greater degree than its increases in mass, resulting in a net increase in specific gravity. In fact, this is exactly what was observed. For most sample volumes, decreases in volume were observed. As will be recalled,

the volume of the samples was calculated through multiplication of its dimensions. A further inspection of the changes in dimensions revealed that, while the length and width of most samples decreased, the sample thickness generally increased. Since thickness measurements are more accurate than measurements of sample length or width, increasing thickness indicates slight swelling of the material. It should, however, be kept in mind that this swelling is relatively small (~0 to 0.3%). Since these dimensional changes are not isotropic in the rectangular geometry of the samples, large changes in one of the sample dimensions may dominate changes in volume. In actual packaging, seals are in the form of O-rings. O-rings with their circular geometry may exhibit more isotropic behavior. Thus the relatively small anisotropic changes in dimension may be even smaller in O-rings where isotropic behavior is expected. The practical implication of these results is that SBR O-rings, even when directly exposed to a Hanford Tank waste under similar conditions as used in this study, are not expected to change dimensionally very much.

As was previously discussed for dimensional property changes, the hardness of SBR increases with increasing exposure time to both radiation and the simulant. For samples exposed to only the simulant, the hardness does not change significantly. Additionally, certain radiation levels appear to exert a beneficial effect on the retention of hardness by SBR. This was found to be especially true for the case where SBR was exposed for 180 days. Under these conditions, most of the SBR samples for all radiation doses have become harder after previously becoming softer at the lower exposure times. Since SBR contracted slightly under these conditions, the observed hardening of the material at these severe conditions could be ascribed purely to a shrinkage phenomenon. Hardening in the presence of shrinkage could be due to chain cross-linking in the polymer. Confirmation of this process will require additional tests. The observation that radiation in combination with chemical exposure may have a beneficial effect on hardness suggests that the cross-linking and chain scission (breaking) processes are occurring simultaneously. Possibly the combination of processes leads to a polymeric structure having generally shorter polymer segments that are cross-linked by radiation and chemical reactions.

Compression set measures the retention of elastic properties of material after exposure to compressive stresses. When SBR samples were exposed to only gamma radiation, set changes in the range of -3.4 to 0.16% were found. For samples exposed to both radiation and the simulant, the compression set change range of values increased slightly from -1.8 to 3.2%. The combined effects of radiation and chemical exposure resulted in increased compression sets, i.e., the material became more deformed, or less rubbery. These deformations are most pronounced in samples to the simulant at 18°C. Compared with the hardness results, the SBR became slightly harder at the longest exposure time. Its elastic properties, however, appear to have suffered under these conditions. As previously discussed, perhaps the competition between cross-linking and chain scission reactions could offer an explanation. However, other factors such as the interaction of the other constituents of elastomers (oils, vulcanizing aids, fillers, etc.) at these environmental conditions would need to be considered.

The VTR of SBR changed very little when exposed to radiation and the aqueous simulant. Most of the VTRs for SBR samples were less than 0.3 g/hr/m^2 . These results are not unexpected since the permeation of water molecules through this polymeric network is expected to be slow. That this process is temperature dependent is confirmed by the results. As temperature is increased, the VTRs also increased. The interesting aspect of the results is that the VTRs appear to decrease with increased exposure time. A possible explanation is that the presence of the inorganic salts, especially the precipitate found in the simulant, may clog microscopic pores in the SBR, thereby reducing the transport of water vapor.

The tensile strength of SBR also exhibits small changes when exposed to radiation, the simulant, and both radiation and simulant. For samples exposed to only radiation, decreases in tensile strength of less than 18% were noted. Many of these samples decreased less than 7% in tensile strength. The combination of high radiation doses and chemical exposure resulted in only 11% decreases in tensile strength. At the highest radiation dose, the scission of polymeric chains may dominate as the determining process that leads to lower strength. While the material's strength may be retained at the less severe environmental conditions, the material's elasticity decreased. This is supported by the data for ultimate elongation discussed below.

The elastic property of materials can be measured by evaluating their degree of ultimate elongation. For SBR samples exposed to only gamma radiation, a general decrease in elongation was observed with increasing exposure time. These results indicate that the material is more elastic at shorter exposure times and less elastic at longer exposure time. However, because most SBR samples were still rather elastic, i.e., stretching more than $\sim 200\%$, a change of $\sim 10\%$ still results in rather elastic material. For SBR exposed to both radiation and simulant, while there is a general decrease in elongation, the material still retained similar elasticity compared with samples only exposed to radiation. These results again point to the plasticizing effects of the simulant.

Finally, the tensile stress properties of SBR will be discussed. Tensile stress or 100% modulus measurements provide a measure of the stiffness of the elastomer. A greater tensile stress value indicates that the elastomer is more likely to recover from localized forces and thereby resist extrusion. The effect of radiation on the tensile stress values of SBR did not increase by more than 20%, nor did they decrease by more than 17%, i.e., tensile stress increases of 20% and decreases of 17% were observed. The effects of radiation alone on SBR resulted in slightly stiffer material. For samples exposed to only the simulant or to a combination of both radiation and simulant, decreases in tensile stress of up to 9% and 25%, respectively, were observed. These results suggest that the simulant somewhat counteracted the effect of radiation to reduce the stiffness. Similar to previous observations, the simulant appears to act as a plasticizing agent. Alternatively, the combination of radiation and simulant exposure resulted in SBR becoming less elastic and assuming a more plastic character. The observation that decreases in hardness, tensile strength, ultimate elongation, and tensile stress were observed under radiation and chemical exposures, while compression set increased, suggests that SBR indeed has lost some of its elastic properties and become more plastic. SBR exposed to higher radiation doses may exhibit increased plasticity, i.e., the material's original dimensions under an applied load are not recovered when the load is removed.

In summary, the measurement of changes in specific gravity, mass, volume, hardness, compression set, and VTR indicated that SBR is remarkably resistant to the effects of radiation and the aqueous simulant at the temperatures and exposure times tested. From the tensile testing results, consisting of tensile strength, ultimate elongation, and tensile stress measurements, SBR appears to be more susceptible to the effects of radiation and the aqueous simulant. When compared to the previously obtained tensile testing results for EPDM rubber and butyl rubber, SBR appears to be comparable to EPDM rubber and somewhat more resistant than butyl rubber under these environmental conditions. The beneficial effect of both radiation and the simulant in helping to reduce the changes in some material properties was noted. These results suggest that this particular aqueous mixed waste has relatively small effects on SBR.

CONCLUSIONS

Sandia has developed a program for studying the chemical interactions of plastic packaging components that may be used in packaging for transporting mixed waste forms. Consistent with the methodology outlined in this report, the second phase of this experimental program was performed to determine the effects of simulant Hanford Tank mixed wastes on a packaging seal material, SBR. This effort involved the comprehensive testing of SBR with an aqueous mixed waste simulant. The testing protocol involved exposing the respective materials to ~143, 286, 571, and 3,670 krad of gamma radiation followed by 7-, 14-, 28-, 180-day exposures to the waste simulant at 18, 50, and 60°C. From the analyses performed, we determined that SBR has very good resistance to radiation, this simulant, and a combination of both environmental factors. These results suggest that SBR is a relatively good seal material to withstand aqueous mixed wastes having similar composition to the one used in this study. SBR appears to respond comparably to EPDM rubber when exposed to the simulant used in this study. Both of these elastomers appear to be superior in performance when compared to butyl rubber.

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APPENDIX A

SBR Material Information

Material Supplier: Parker Seal Group^a
 O-Ring Division
 2360 Palumbo Drive
 P.O. Box 11751
 Lexington, KY 40512
 (606) 269-2351

<u>Measurement</u>	<u>Initial Value</u>	<u>Cure Date^b</u>	<u>Batch Number</u>
Specific Gravity	1.195	CD 1Q95	B316566
Dimensional	NA	CD 1Q95	B316566
Mass NA	CD 1Q95	B316566	
Hardness (Shore A)	62.7 points	CD 1Q95	B316566
Compression Set	5.67	CD 1Q96	B320139
Vapor Transport Rates	NA	CD 1Q95	B316566
Tensile Property		CD1Q95	B316566
		CD1Q96	B320139
Tensile Strength	12.6 MPa		
Ultimate Elongation	251%		
Tensile Stress	3.51 MPa		

- a. Procured from Parker Seal Group (G0244-70) through Southwest Seal and Supply, 1413 1st Street NW, Albuquerque, NM 87102-1533, (505) 247-0265. This material was obtained in the form of molded sheets. The 12" (30.5 cm) square sheets were ~0.125" (0.317 cm) thick. **Cost: ~\$46/ft² (~ \$0.049/cm²)**
- b. Cure Date (CD) nomenclature indicates the quarter and year in which the rubber was prepared. For example, 1Q95 represents material prepared during the first quarter of 1995.

APPENDIX B

SBR Specific Gravity Data*

AVERAGE SPECIFIC GRAVITY (SP.GR.) AND % CHANGE: SBR									
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	SP. GR.	SP. GR.	% CHANGE	SP. GR.	% CHANGE	SP. GR.	% CHANGE	SP. GR.	% CHANGE
OK	1.1960	1.1950	-0.08	1.1971	0.09	1.1980	0.17	1.1972	0.10
143K	1.1960	1.1965	0.04	1.1966	0.05	1.1983	0.19	1.1983	0.19
286K	1.1950	1.1980	0.25	1.1975	0.21	1.1983	0.28	1.1987	0.31
571K	1.1961	1.1974	0.11	1.1972	0.09	1.1983	0.18	1.1982	0.18
3670K	1.1963	1.1981	0.15	1.1988	0.21	1.1989	0.22	1.1988	0.21
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	SP. GR.	SP. GR.	% CHANGE	SP. GR.	% CHANGE	SP. GR.	% CHANGE	SP. GR.	% CHANGE
OK	1.1947	1.1976	0.24	1.1981	0.28	1.1979	0.27	1.1980	0.28
143K	1.1961	1.1965	0.03	1.1975	0.12	1.1972	0.09	1.1953	-0.07
286K	1.1957	1.1975	0.15	1.1972	0.13	1.1979	0.18	1.1977	0.17
571K	1.1961	1.1976	0.13	1.1971	0.08	1.1978	0.14	1.1970	0.08
3670K	1.1949	1.1978	0.24	1.1982	0.28	1.1985	0.30	1.1974	0.21
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	SP. GR.	SP. GR.	% CHANGE	SP. GR.	% CHANGE	SP. GR.	% CHANGE	SP. GR.	% CHANGE
OK	1.1957	1.1982	0.21	1.1983	0.22	1.1980	0.19	1.1996	0.34
143K	1.1965	1.1956	-0.08	1.1973	0.07	1.1973	0.07	1.1981	0.13
286K	1.1944	1.1977	0.28	1.1974	0.25	1.1977	0.28	1.1985	0.34
571K	1.1931	1.1974	0.36	1.1970	0.33	1.1982	0.43	1.1981	0.42
3670K	1.1903	1.1982	0.66	1.1985	0.69	1.1989	0.72	1.1984	0.68

* In all appendices, the data in the "OK" represents samples exposed to only the simulant (i.e., *Simulant Only* samples) while the other data represents samples that received both radiation and simulant exposure.

APPENDIX C

SBR Mass Data

MASS (g) AND % CHANGE: SBR									
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	WEIGHT	WEIGHT	% CHANGE	WEIGHT	% CHANGE	WEIGHT	% CHANGE	WEIGHT	% CHANGE
OK	7.0711	7.0711	0.00	7.0732	0.03	7.0755	0.06	7.1344	0.90
143K	7.5511	7.5528	0.02	7.5550	0.05	7.5608	0.13	7.5924	0.55
286K	7.4729	7.4764	0.05	7.4756	0.04	7.4822	0.12	7.5252	0.70
571K	7.5246	7.5275	0.04	7.5276	0.04	7.5348	0.14	7.5657	0.55
3670K	7.5232	7.5280	0.06	7.5271	0.05	7.5333	0.13	7.5864	0.84
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	WEIGHT	WEIGHT	% CHANGE	WEIGHT	% CHANGE	WEIGHT	% CHANGE	WEIGHT	% CHANGE
OK	7.5008	7.4922	-0.11	7.4823	-0.25	7.4728	-0.37	7.4716	-0.39
143K	7.6323	7.6116	-0.27	7.6024	-0.39	7.5963	-0.47	7.5431	-1.17
286K	7.4578	7.4435	-0.19	7.4259	-0.43	7.4193	-0.52	7.3686	-1.20
571K	7.4565	7.4410	-0.21	7.4286	-0.37	7.4204	-0.48	7.3571	-1.33
3670K	7.3741	7.3625	-0.16	7.3453	-0.39	7.3377	-0.49	7.2768	-1.32
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	WEIGHT	WEIGHT	% CHANGE	WEIGHT	% CHANGE	WEIGHT	% CHANGE	WEIGHT	% CHANGE
OK	7.6707	7.6325	-0.50	7.6143	-0.74	7.5907	-1.04	7.5427	-1.67
143K	7.3638	7.3209	-0.58	7.2976	-0.90	7.2825	-1.10	7.2328	-1.78
286K	7.1222	7.0866	-0.50	7.0739	-0.68	7.0636	-0.82	7.0298	-1.30
571K	7.1293	7.0778	-0.72	7.0632	-0.93	7.0529	-1.07	7.0096	-1.68
3670K	7.3871	7.3537	-0.45	7.3442	-0.58	7.3319	-0.75	7.3032	-1.14

APPENDIX D

SBR Dimensional Data

VOLUME (mm ³) AND % CHANGE: SBR									
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	VOLUME	VOLUME	% CHANGE	VOLUME	% CHANGE	VOLUME	% CHANGE	VOLUME	% CHANGE
OK	5757	5799	0.73	5793	0.63	5779	0.38	5823	1.15
143K	6181	6209	0.45	6182	0.02	6176	-0.08	6225	0.71
286K	6145	6115	-0.49	6119	-0.42	6129	-0.26	6158	0.21
571K	6157	6170	0.21	6200	0.70	6175	0.29	6221	1.04
3670K	6163	6177	0.23	6177	0.23	6175	0.19	6211	0.78
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	VOLUME	VOLUME	% CHANGE	VOLUME	% CHANGE	VOLUME	% CHANGE	VOLUME	% CHANGE
OK	6160	6125	-0.57	6113	-0.76	6106	-0.88	6094	-1.07
143K	6263	6248	-0.24	6220	-0.69	6227	-0.57	6217	-0.73
286K	6118	6086	-0.52	6094	-0.39	6086	-0.52	6068	-0.82
571K	6132	6091	-0.67	6123	-0.15	6082	-0.82	6080	-0.85
3670K	6045	6015	-0.50	6008	-0.61	6009	-0.60	5957	-1.46
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	VOLUME	VOLUME	% CHANGE	VOLUME	% CHANGE	VOLUME	% CHANGE	VOLUME	% CHANGE
OK	6278	6256	-0.35	6237	-0.65	6216	-0.99	6171	-1.70
143K	6033	5993	-0.66	5968	-1.08	5955	-1.29	5941	-1.52
286K	5839	5786	-0.91	5797	-0.72	5789	-0.86	5771	-1.16
571K	5831	5788	-0.74	5794	-0.63	5764	-1.15	5771	-1.03
3670K	6067	6037	-0.49	6032	-0.58	6031	-0.59	6023	-0.73

LENGTH (mm) AND % CHANGE: SBR									
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	LENGTH	LENGTH	% CHANGE	LENGTH	% CHANGE	LENGTH	% CHANGE	LENGTH	% CHANGE
OK	75.28	75.31	0.04	75.33	0.07	75.31	0.04	75.38	0.13
143K	75.43	75.45	0.03	75.41	-0.03	75.44	0.01	75.47	0.05
286K	75.47	75.46	-0.01	75.45	-0.03	75.44	-0.04	75.50	0.04
571K	75.39	75.41	0.03	75.41	0.03	75.37	-0.03	75.49	0.13
3670K	75.36	75.38	0.03	75.35	-0.01	75.33	-0.04	75.48	0.16
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	LENGTH	LENGTH	% CHANGE	LENGTH	% CHANGE	LENGTH	% CHANGE	LENGTH	% CHANGE
OK	75.34	75.29	-0.07	75.27	-0.09	75.16	-0.24	74.99	-0.46
143K	75.27	75.15	-0.16	75.11	-0.21	75.07	-0.27	74.84	-0.57
286K	75.34	75.23	-0.15	75.17	-0.23	75.07	-0.36	74.87	-0.62
571K	75.38	75.26	-0.16	75.24	-0.19	75.14	-0.32	74.95	-0.57
3670K	75.30	75.21	-0.12	75.15	-0.20	75.09	-0.28	74.88	-0.56
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	LENGTH	LENGTH	% CHANGE	LENGTH	% CHANGE	LENGTH	% CHANGE	LENGTH	% CHANGE
OK	75.35	75.16	-0.25	75.07	-0.37	74.96	-0.52	74.73	-0.82
143K	75.29	75.09	-0.27	75.00	-0.39	74.88	-0.54	74.67	-0.82
286K	75.29	75.07	-0.29	74.98	-0.41	74.89	-0.53	74.70	-0.78
571K	75.39	75.17	-0.29	75.09	-0.40	74.93	-0.61	74.84	-0.73
3670K	75.38	75.18	-0.27	75.08	-0.40	75.00	-0.50	74.94	-0.58

APPENDIX D

SBR Dimensional Data (continued)

WIDTH (mm) AND % CHANGE: SBR									
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	WIDTH	WIDTH	% CHANGE	WIDTH	% CHANGE	WIDTH	% CHANGE	WIDTH	% CHANGE
OK	25.17	25.29	0.48	25.27	0.40	25.24	0.28	25.29	0.48
143K	25.19	25.25	0.24	25.21	0.08	25.17	-0.08	25.28	0.36
286K	25.22	25.20	-0.08	25.21	-0.04	25.20	-0.08	25.25	0.12
571K	25.20	25.25	0.20	25.30	0.40	25.24	0.16	25.33	0.52
3670K	25.22	25.24	0.08	25.27	0.20	25.26	0.16	25.34	0.48
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	WIDTH	WIDTH	% CHANGE	WIDTH	% CHANGE	WIDTH	% CHANGE	WIDTH	% CHANGE
OK	25.31	25.15	-0.63	25.14	-0.67	25.15	-0.63	25.06	-0.99
143K	25.22	25.21	-0.04	25.16	-0.24	25.18	-0.16	25.13	-0.36
286K	25.19	25.19	0.00	25.21	0.08	25.19	0.00	25.13	-0.24
571K	25.26	25.21	-0.20	25.26	0.00	25.16	-0.40	25.19	-0.28
3670K	25.18	25.17	-0.04	25.15	-0.12	25.13	-0.20	25.04	-0.56
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	WIDTH	WIDTH	% CHANGE	WIDTH	% CHANGE	WIDTH	% CHANGE	WIDTH	% CHANGE
OK	25.25	25.18	-0.28	25.17	-0.32	25.12	-0.51	25.01	-0.95
143K	25.22	25.14	-0.32	25.10	-0.48	25.08	-0.56	25.02	-0.79
286K	25.21	25.13	-0.32	25.13	-0.32	25.13	-0.32	25.08	-0.52
571K	25.17	25.13	-0.16	25.11	-0.24	25.05	-0.48	25.07	-0.40
3670K	25.25	25.20	-0.20	25.19	-0.24	25.17	-0.32	25.14	-0.44

THICKNESS (mm) AND % CHANGE: SBR									
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	THICKNESS	THICKNESS	% CHANGE	THICKNESS	% CHANGE	THICKNESS	% CHANGE	THICKNESS	% CHANGE
OK	3.04	3.04	0.00	3.04	0.00	3.04	0.00	3.05	0.33
143K	3.25	3.26	0.31	3.25	0.00	3.25	0.00	3.26	0.31
286K	3.23	3.22	-0.31	3.22	-0.31	3.22	-0.31	3.23	0.00
571K	3.24	3.24	0.00	3.25	0.31	3.25	0.31	3.25	0.31
3670K	3.24	3.25	0.31	3.24	0.00	3.25	0.31	3.25	0.31
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	THICKNESS	THICKNESS	% CHANGE	THICKNESS	% CHANGE	THICKNESS	% CHANGE	THICKNESS	% CHANGE
OK	3.23	3.23	0.00	3.23	0.00	3.23	0.00	3.24	0.31
143K	3.30	3.30	0.00	3.29	-0.30	3.29	-0.30	3.31	0.30
286K	3.22	3.21	-0.31	3.22	0.00	3.22	0.00	3.23	0.31
571K	3.22	3.21	-0.31	3.22	0.00	3.22	0.00	3.22	0.00
3670K	3.19	3.18	-0.31	3.18	-0.31	3.18	-0.31	3.18	-0.31
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	THICKNESS	THICKNESS	% CHANGE	THICKNESS	% CHANGE	THICKNESS	% CHANGE	THICKNESS	% CHANGE
OK	3.30	3.31	0.30	3.30	0.00	3.30	0.00	3.30	0.00
143K	3.18	3.17	-0.31	3.17	-0.31	3.17	-0.31	3.18	0.00
286K	3.08	3.07	-0.32	3.08	0.00	3.08	0.00	3.08	0.00
571K	3.07	3.06	-0.33	3.07	0.00	3.07	0.00	3.08	0.33
3670K	3.19	3.19	0.00	3.19	0.00	3.19	0.00	3.20	0.31

APPENDIX E

SBR Hardness Data

AVERAGE HARDNESS (TYPE A) AND % CHANGE: SBR									
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	HARDNESS	HARDNESS	% CHANGE	HARDNESS	% CHANGE	HARDNESS	% CHANGE	HARDNESS	% CHANGE
OK	62.9	63.1	0.3	63.0	0.2	63.0	0.2	62.3	-1.0
143K	62.7	63.4	1.1	63.1	0.6	62.9	0.3	64.4	2.7
286K	62.8	63.4	1.0	63.5	1.1	63.1	0.5	64.2	2.2
571K	62.5	63.2	1.1	63.1	1.0	62.8	0.5	63.7	1.9
3670K	62.6	63.7	1.8	63.5	1.4	63.3	1.1	64.0	2.2
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	HARDNESS	HARDNESS	% CHANGE	HARDNESS	% CHANGE	HARDNESS	% CHANGE	HARDNESS	% CHANGE
OK	62.5	62.5	0.0	62.5	0.0	62.8	0.5	61.8	-1.1
143K	63.3	62.3	-1.6	62.7	-0.9	62.9	-0.6	64.6	2.1
286K	62.6	62.2	-0.6	62.8	0.3	62.6	0.0	63.9	2.1
571K	62.9	62.5	-0.6	62.9	0.0	62.8	-0.2	64.4	2.4
3670K	63.4	61.9	-2.4	63.2	-0.3	63.2	-0.3	64.3	1.4
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	HARDNESS	HARDNESS	% CHANGE	HARDNESS	% CHANGE	HARDNESS	% CHANGE	HARDNESS	% CHANGE
OK	62.1	62.2	0.2	62.2	0.2	62.3	0.3	62.5	0.6
143K	62.8	61.8	-1.6	62.7	-0.2	62.5	-0.5	64.7	3.0
286K	63.0	62.5	-0.8	62.7	-0.5	62.5	-0.8	64.4	2.2
571K	62.4	62.3	-0.2	62.5	0.2	62.4	0.0	64.4	3.2
3670K	62.6	62.3	-0.5	62.6	0.0	62.3	-0.5	63.7	1.8

APPENDIX F

SBR Compression Set Data

COMPRESSION SET (SET, %) AND CHANGE: SBR									
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	SET	SET	CHANGE	SET	CHANGE	SET	CHANGE	SET	CHANGE
OK	5.67	7.07	1.40	6.55	0.88	7.46	1.79	8.89	3.22
143K	5.67	5.94	0.27	5.80	0.13	7.54	1.87	4.61	-1.06
286K	5.67	6.20	0.53	7.07	1.40	6.78	1.11	7.35	1.68
571K	5.67	5.15	-0.52	5.96	0.29	5.76	0.09	6.54	0.87
3670K	5.67	6.02	0.35	5.59	-0.08	5.80	0.13	6.70	1.03
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	SET	SET	CHANGE	SET	CHANGE	SET	CHANGE	SET	CHANGE
OK	5.67	6.75	1.08	6.06	0.39	5.32	-0.35	6.12	0.45
143K	5.67	6.32	0.65	5.14	-0.53	6.25	0.58	4.16	-1.51
286K	5.67	6.11	0.44	5.60	-0.07	5.54	-0.13	5.16	-0.51
571K	5.67	4.43	-1.24	4.46	-1.21	4.59	-1.08	5.29	-0.38
3670K	5.67	4.49	-1.18	6.08	0.41	5.04	-0.63	4.78	-0.89
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	SET	SET	CHANGE	SET	CHANGE	SET	CHANGE	SET	CHANGE
OK	5.67	5.24	-0.43	5.10	-0.57	3.97	-1.70	6.54	0.87
143K	5.67	5.45	-0.22	3.87	-1.80	4.51	-1.16	3.88	-1.79
286K	5.67	5.91	0.24	4.89	-0.78	3.99	-1.68	4.68	-0.99
571K	5.67	4.08	-1.59	4.19	-1.48	4.53	-1.14	4.81	-0.86
3670K	5.67	4.28	-1.39	5.15	-0.52	4.60	-1.07	4.93	-0.74
18 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	SET	SET	CHANGE	SET	CHANGE	SET	CHANGE	SET	CHANGE
143K	5.67	5.4	-0.27	4.69	-0.98	5.44	-0.23	3.13	-2.54
286K	5.67	5.82	0.15	5.20	-0.47	5.83	0.16	5.06	-0.61
571K	5.67	4.25	-1.42	4.96	-0.71	4.99	-0.68	5.23	-0.44
3670K	5.67	5.22	-0.45	4.44	-1.23	4.52	-1.15	5.09	-0.58
50 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	SET	SET	CHANGE	SET	CHANGE	SET	CHANGE	SET	CHANGE
143K	5.67	4.21	-1.46	3.56	-2.11	3.82	-1.85	3.39	-2.28
286K	5.67	5.15	-0.52	4.30	-1.37	4.01	-1.66	3.48	-2.19
571K	5.67	4.01	-1.66	3.69	-1.98	3.44	-2.23	4.10	-1.57
3670K	5.67	3.69	-1.98	4.87	-0.80	4.17	-1.50	4.27	-1.40
60 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	SET	SET	CHANGE	SET	CHANGE	SET	CHANGE	SET	CHANGE
143K	5.67	4.07	-1.60	2.75	-2.92	3.92	-1.75	2.27	-3.40
286K	5.67	4.47	-1.20	3.55	-2.12	2.86	-2.81	3.19	-2.48
571K	5.67	3.9	-1.77	3.29	-2.38	3.62	-2.05	4.44	-1.23
3670K	5.67	3.5	-2.17	4.11	-1.56	3.75	-1.92	3.69	-1.98

APPENDIX G

SBR Vapor Transport Rate Data

VAPOR TRANSMISSION RATE (g/hr/m ²): SBR				
18 C	7 DAYS	14 DAYS	28 DAYS	180 DAYS
RADIATION DOSE	VAPOR TRANSMISSION	VAPOR TRANSMISSION	VAPOR TRANSMISSION	VAPOR TRANSMISSION
OK	0.1462	0.1150	0.0910	0.0325
143K	0.0961	0.1225	0.0846	0.0246
286K	0.0406	0.0277	0.0189	0.0063
571K	0.0081	0.0074	0.0095	0.0054
3670K	0.0961	0.0717	0.0501	0.0168
50 C	7 DAYS	14 DAYS	28 DAYS	180 DAYS
RADIATION DOSE	VAPOR TRANSMISSION	VAPOR TRANSMISSION	VAPOR TRANSMISSION	VAPOR TRANSMISSION
OK	0.3803	0.2409	0.1475	0.0725
143K	0.3641	0.2375	0.1495	0.0723
286K	0.7010	0.3959	0.2247	0.0671
571K	0.0947	0.0724	0.0572	0.0378
3670K	0.1705	0.1123	0.0758	0.0411
60 C	7 DAYS	14 DAYS	28 DAYS	180 DAYS
RADIATION DOSE	VAPOR TRANSMISSION	VAPOR TRANSMISSION	VAPOR TRANSMISSION	VAPOR TRANSMISSION
OK	0.6564	0.6909	0.5657	0.2601
143K	0.4128	0.2646	0.1770	0.1203
286K	0.2463	0.1712	0.1303	0.0892
571K	0.2883	0.2226	0.1563	0.1034
3670K	0.4764	0.2971	0.2020	0.0988

APPENDIX H

SBR Tensile Strength Data

TENSILE STRENGTH (TENS. STR., MPa) AND % CHANGE: SBR									
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	TENS. STR.	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE
OK	12.6	12.7	0.5	12.7	0.5	13.7	8.2	13.7	8.7
143K	12.6	12.8	1.6	13.7	8.7	13.7	8.2	13.3	5.5
286K	12.6	13.0	3.3	12.9	2.2	12.9	2.2	13.4	6.0
571K	12.6	13.0	3.3	13.4	6.6	13.4	6.6	12.7	0.5
3670K	12.6	13.2	4.4	13.0	3.3	13.4	6.0	12.5	-0.5
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	TENS. STR.	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE
OK	12.6	12.3	-2.2	12.2	-3.3	13.3	5.5	12.5	-0.5
143K	12.6	13.2	4.4	13.1	3.8	12.5	-1.1	11.9	-6.0
286K	12.6	12.2	-3.3	12.9	2.2	13.3	5.5	12.8	1.1
571K	12.6	12.3	-2.7	13.2	4.4	13.2	4.4	13.1	3.8
3670K	12.6	12.2	-3.3	12.3	-2.7	11.7	-7.3	11.8	-6.5
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	TENS. STR.	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE
OK	12.6	12.8	1.6	13.2	4.9	13.3	5.5	12.4	-1.6
143K	12.6	12.1	-3.8	13.0	2.7	13.4	6.6	12.4	-1.6
286K	12.6	12.6	0.0	13.0	2.7	13.4	6.0	12.5	-0.5
571K	12.6	12.8	1.1	12.5	-0.5	13.3	5.5	12.8	1.6
3670K	12.6	12.1	-3.8	11.7	-7.3	11.4	-9.3	11.2	-11.2
18 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	TENS. STR.	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE
143K	12.6	13.0	3.3	12.8	1.1	13.3	5.5	12.7	0.5
286K	12.6	13.3	5.5	11.8	-6.6	13.0	3.3	13.0	3.3
571K	12.6	13.2	4.9	13.4	6.0	14.3	13.1	13.9	10.4
3670K	12.6	13.0	3.3	12.4	-1.6	13.5	7.1	13.0	2.7
50 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	TENS. STR.	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE
143K	12.6	13.5	7.1	12.7	0.5	13.3	5.5	12.8	1.1
286K	12.6	13.4	6.0	12.3	-2.7	13.3	5.5	11.9	-6.0
571K	12.6	12.6	0.0	12.3	-2.7	13.0	2.7	10.3	-18.0
3670K	12.6	13.2	4.4	12.6	0.0	11.7	-7.1	10.5	-16.8
60 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	TENS. STR.	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE	TENS. STR.	% CHANGE
143K	12.6	12.3	-2.7	13.3	5.5	14.1	11.5	13.0	3.3
286K	12.6	14.1	11.5	14.3	13.7	13.8	9.3	13.9	10.4
571K	12.6	12.8	1.1	12.7	0.5	14.1	11.5	12.4	-1.6
3670K	12.6	11.9	-6.0	12.4	-1.6	11.4	-9.7	11.0	-12.8

APPENDIX I

SBR Ultimate Elongation Data

ULTIMATE ELONGATION (ELONG., %) AND CHANGE: SBR									
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	ELONG.	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE
OK	251	235	-6	239	-5	252	0	273	9
143K	251	253	1	267	6	248	-1	248	-1
286K	251	269	7	248	-1	243	-3	259	3
571K	251	248	-1	257	2	271	8	239	-5
3670K	251	256	2	262	4	254	1	242	-4
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	ELONG.	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE
OK	251	234	-7	243	-3	260	4	236	-6
143K	251	267	6	244	-3	239	-5	232	-8
286K	251	250	0	249	-1	246	-2	234	-7
571K	251	250	0	261	4	247	-2	229	-9
3670K	251	267	6	257	2	264	5	255	2
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	ELONG.	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE
OK	251	257	2	248	-1	264	5	226	-10
143K	251	243	-3	258	3	242	-4	223	-11
286K	251	240	-4	247	-2	247	-2	236	-6
571K	251	253	1	249	-1	246	-2	229	-9
3670K	251	263	5	263	5	259	3	233	-7
18 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	ELONG.	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE
143K	251	250	0	261	4	242	-4	236	-6
286K	251	247	-2	232	-8	237	-6	248	-1
571K	251	260	4	250	0	268	7	254	1
3670K	251	262	4	258	3	278	11	284	13
50 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	ELONG.	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE
143K	251	260	4	246	-2	254	1	239	-5
286K	251	256	2	247	-2	254	1	213	-15
571K	251	242	-4	242	-4	236	-6	191	-24
3670K	251	268	7	263	5	257	2	225	-10
60 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	ELONG.	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE
143K	251	245	-2	248	-1	249	-1	230	-8
286K	251	251	0	255	2	238	-5	225	-10
571K	251	240	-4	236	-6	256	2	213	-15
3670K	251	280	12	278	11	279	11	251	0

APPENDIX J

SBR Tensile Stress Data

TENSILE STRESS (STRESS, MPa) AND % CHANGE: SBR									
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	STRESS	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE
OK	3.51	3.75	6.88	3.84	9.4	3.74	6.68	3.25	-7.3
143K	3.51	3.43	-2.36	3.45	-1.57	3.56	1.57	3.65	3.93
286K	3.51	3.23	-8.06	3.38	-3.73	3.61	2.95	3.43	-2.36
571K	3.51	3.45	-1.6	3.41	-2.9	3.33	-5.1	3.52	0.4
3670K	3.51	3.38	-3.7	3.16	-10.0	3.33	-5.1	3.48	-0.8
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	STRESS	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE
OK	3.51	4.19	19.45	3.77	7.47	3.20	-8.84	3.40	-3.14
143K	3.51	3.15	-10.22	3.63	3.34	3.52	0.20	3.46	-1.38
286K	3.51	3.17	-9.63	3.36	-4.13	3.54	1.0	3.58	2.0
571K	3.51	3.28	-6.5	3.26	-7.1	3.74	6.5	3.92	11.8
3670K	3.51	3.01	-14.1	3.03	-13.6	2.99	-14.7	3.14	-10.6
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	STRESS	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE
OK	3.51	3.33	-5.11	3.41	-2.75	3.23	-7.9	3.95	12.57
143K	3.51	3.12	-11.20	3.26	-7.1	3.62	3.14	3.85	9.63
286K	3.51	3.74	6.68	3.43	-2.2	3.56	1.4	3.61	2.8
571K	3.51	3.29	-6.3	3.41	-2.9	3.39	-3.5	3.81	8.6
3670K	3.51	2.63	-25.0	2.70	-23.0	2.76	-21.4	3.19	-9.0
18 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	STRESS	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE
143K	3.51	3.64	3.73	3.28	-6.68	3.81	8.6	3.96	13.0
286K	3.51	3.73	6.29	3.86	10.02	3.82	8.84	3.66	4.32
571K	3.51	3.31	-5.70	3.66	4.32	3.54	0.79	3.68	4.72
3670K	3.51	2.93	-16.5	3.11	-11.4	3.11	-11.4	3.27	-6.9
50 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	STRESS	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE
143K	3.51	3.65	4.1	3.63	3.5	3.49	-0.6	3.79	7.86
286K	3.51	3.70	5.30	3.85	9.82	3.71	5.70	4.11	17.09
571K	3.51	3.31	-5.70	3.55	1.18	3.92	11.59	4.14	17.88
3670K	3.51	3.00	-14.5	3.07	-12.6	3.10	-11.8	3.42	-2.6
60 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	STRESS	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE
143K	3.51	3.48	-1.0	3.63	3.3	3.74	6.48	3.97	13.2
286K	3.51	3.88	10.61	3.65	4.13	3.77	7.47	4.20	19.65
571K	3.51	3.55	1.18	3.69	5.11	3.50	-0.20	3.96	12.77
3670K	3.51	2.96	-15.7	3.10	-11.8	3.24	-7.7	3.41	-2.9

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