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TECHNICAL BULLETIN

Halobutyl Inner Liners II

This report provides results of the supplemental evaluation of Thermax® N990 and Thermax® MFT in a bromobutyl inner liner compound.

Compounds were prepared and mixed at the BFGoodrich (BFG) Laboratory in Akron, Ohio. All testing was performed at BFG, with the exception of the low temperature stiffness testing, which occurred at Akron Rubber Development Laboratory, and the air permeation testing, which was performed at Akron Polymer Laboratory.

The base formulation was from the original Cancarb inner liner study. However, the carbon black was adjusted in three new test compounds noted below. The four compounds are identified with the first digit as A, B, C and D. The D compound utilized a Medium Fine Thermal (MFT) specialty grade from Cancarb.

Test Formulations

	A60 (control)	B8030	C9030	DMFT
Bromobutyl 2030	100.0	100.0	100.0	100.0
N660 Carbon Black	60	30	30	30
Thermax® N990	-	80	90	-
Thermax® MFT	-	-	-	90
Stearic Acid	1.0	1.0	1.0	1.0
Tackifier	4.0	4.0	4.0	4.0
Paraffinic Oil	7.0	7.0	7.0	7.0
MBTS	1.3	1.3	1.3	1.3
Zinc Oxide	3.0	3.0	3.0	3.0
Sulphur	0.5	0.5	0.5	0.5

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The results of the tests performed at BFG are reported as follows:

	A60 (control)	B8030	C9030	DMFT
Thermax® N990	-	80	90	-
Thermax® MFT	-	-	-	90
N660 Carbon Black	60	30	30	30
Mooney Viscosity, 100°C, ASTM D1646-96a				
M_L (1 + 4)	67.59	76.31	78.10	75.23
Mooney Scorch, 137°C, ASTM D1646-96a				
Min Torque	44.63	53.4	55.52	52.70
Minutes to 1 pt. rise	8.75	6.58	6.67	6.08
Minutes to 5 pt. rise	13.67	12.42	12.00	11.83
Minutes to 10 pt. rise	15.75	15.75	15.25	15.25
Minutes to 35 pt. rise	18.33	19.42	18.92	18.83
Monsanto R100 Oscillating Disk Rheometer @ 166°C, 1° arc, ASTM D2084-95				
M_L (dNm)	8.7	9.5	9.8	9.0
M_H (dNm)	19.0	22.7	22.8	21.9
T_c50 (min)	5.2	5.5	5.3	5.2
T_c90 (min)	9.4	10.5	11.1	10.1
T_c95 (min)	13.4	19.8	21.8	21.0
Stress-Strain, Originals @ RT, Cured 30 minutes @ 166°C, ASTM D412-97				
100 Modulus (MPa)	1.1	1.6	1.7	1.4
300% Modulus (MPa)	4.6	5.5	5.6	5.1
Tensile Strength (MPa)	10.3	6.9	6.7	6.6
Elongation (%)	757	573	561	570
Shore A Hardness	60	67	69	70
DeMattia Crack Growth, Originals @ RT, Pierced, Average of 2, mm, ASTM D813-95				
1,000 cycles	2.50	2.50	2.50	2.50
50,000 cycles	2.50	5.00	5.00	3.75
75,000 cycles	2.50	5.00	5.00	5.00
125,000 cycles	5.00	8.75	10.00	7.50
200,000 cycles	6.25	12.50	15.00	12.50
250,000 cycles	7.50	15.00	17.50	15.00
Tear Resistance, Monsanto Tensometer 10, Die C, RT, ASTM D624-91				
kN/m	39.6	36.9	34.0	40.0
Rubber Peel Abhesion Test – 50 mm/min., Instron 1122, Cured 35 minutes @ 166°C, Adhesion to Standard Radial Carcass, Average Load, Average of 2 Samples				
kN per inch width (Measure of peel force per inch of width)	0.346	0.645	0.562	0.55

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Physical Properties

The viscosity was noticeably higher in all of the Thermax® compounds. This was in contrast to an earlier Cancarb study that showed similar viscosity between the control and the thermal black compounds.

The four compounds showed similar scorch properties, as indicated by the T_s10 and T_s35. This was also in contrast to the earlier reported work, which indicated slightly more scorch safety in the Thermax® compounds (but with different amounts of Thermax®). The time to onset of scorch was similar to the earlier work (roughly 18 – 19 minutes).

The Rheometer data indicated similar torque properties and T_c50 and T_c90 results. However, the T_c95 showed substantially longer cure times for the Thermax® compounds. This would be expected with the higher carbon loading, which retards the rate of cure. This could possibly be adjusted with a higher accelerator loading.

Stress-strain properties, although not truly relevant to the inner liner application, were indicative of typical Thermax® compounds, with comparatively lower tensile strength. The elongation was lower and the hardness was higher with the Thermax® compounds. Again, this would likely be due to the higher Thermax® loading.

Crack growth (ASTM D813) and tear resistance (ASTM D624) were tested to measure the fatigue properties. Next to permeability, fatigue resistance is the most crucial property for the inner liner. With the DeMattia Crack Growth test, the control compound showed greater crack growth resistance with the increasing number of cycles. The Thermax® MFT compound had tear resistance properties equal to that of the control, while the Thermax® N990 compounds had slightly lower tear resistance. Whether or not there is sufficient fatigue resistance with the Thermax® compounds is the key issue. This would be subject to the judgment of the tire industry.

Adhesion was measured with BFGoodrich's method, using an Instron 1122 system. There is no ASTM standard for this test but the BFG method is accepted and understood throughout the tire industry. In brief, an inner liner sample is sandwiched between two piles of calendered standard polyester carcass material. The test then measures the peel force per width of sample, the greater the force implying the better the adhesion. The control sample showed a "knotty, uneven tear" with poor adhesion, while the Thermax® samples showed better adhesion and smooth peeling. The best result was provided by the compound with 80 phr Thermax® N990 and 30 phr N660.

Permeability

BFGoodrich does not have permeability testing capability and contracts Akron Polymer Laboratory (APL) to perform this test. APL performs this test according to ASTM D1434, method V, using gas at 48 psi @ 35°C. Four different measurements of permeability are provided in the report, with three samples tested for each compound and then an average is reported. Unfortunately the results were not consistent and both APL and BFG expressed their concern on this.

However, based on the averages, there are two consistent results. The first is that the Thermax® MFT compound (90 phr MFT, 30 phr N660) provided the least amount of permeation. That is, it had the least amount of average permeation in all four values reported. The second is that the control, with only 60 phr of N660, had the greatest amount of permeation. Thus, the results support the theory of greater impermeability, with a note of caution on the results.

The following table provides two of the reported values:

Akron Polymer Laboratory - Air Gas Permeability Results:
48 psi @ 35°C (average of 3)

Sample	$cm^3 \cdot cm/cm^2 \cdot sec \cdot atm$	$cm^3 \cdot mm/m^2 \cdot day \cdot atm$
A60 (control)	1.67^{-08}	144.2
B8030	1.21^{-08}	104.9
C9030	1.47^{-08}	126.7
DMFT	8.17^{-09}	70.6

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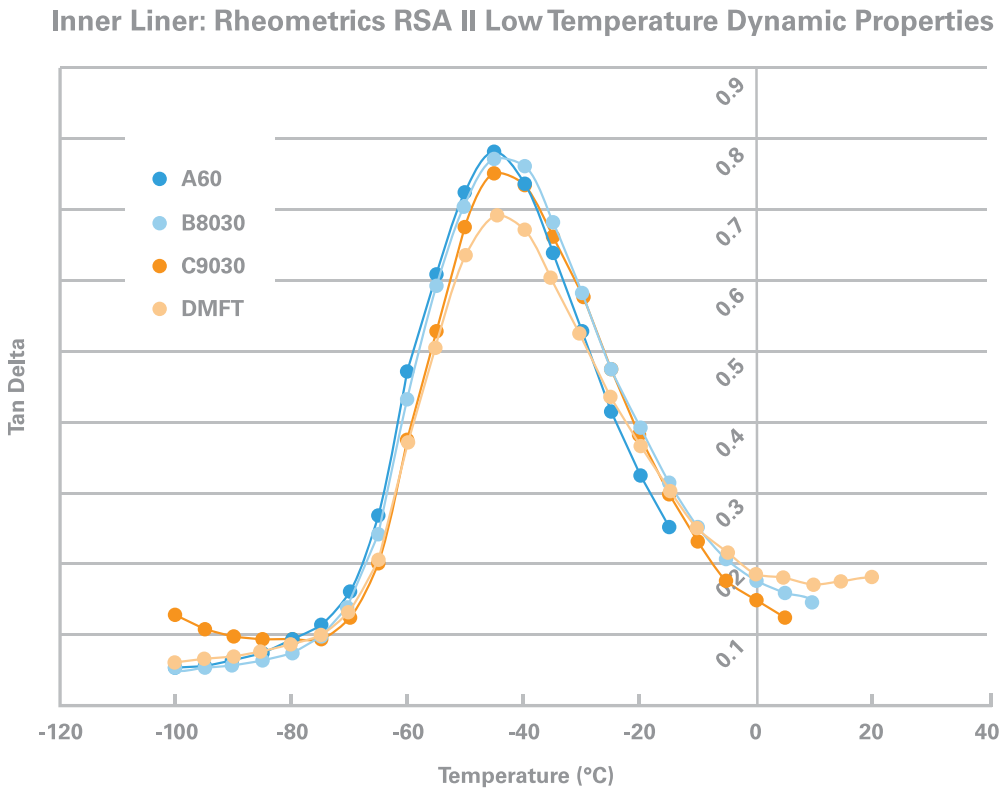
Low Temperature Stiffness

There are two tests to measure low temperature stiffness for the inner liner compound. The first is an evaluation of the dynamic properties, namely tan delta, at a temperature sweep from -100°C to +20°C, using the industry standard Rheometrics RSA II. This test is based on ASTM D5279. The second test is the standard method for stiffening at low temperature: flexible polymers and coated fabrics, as per ASTM D1053-92, method A. This test is often referred to as the Gehman Torsion test as the torsion apparatus was designed by Dr. Gehman of Goodyear. Both of these tests measure the subnormal temperature stiffening of the rubber.

The Rheometrics test measures tan delta over a wide range of temperature. As with other rubber applications and dynamic tests, a low tan delta indicates a higher amount of flexibility and elasticity. The results of this test are shown in the chart below:



A similar trend in the tan delta is shown for the four compounds throughout the temperature sweep. For the control compound, the torque dropped to zero around -10°C, while the Thermax® compounds continued until the +10°C level. The lowest tan delta was shown with the Thermax® MFT compound, and the second lowest with the C9030 compound. Both of these had the highest amounts of Thermax®.

The "Gehman Torsion" low temperature stiffness test was performed at Akron Rubber Development Laboratory according to ASTM D1053-92a. This test provides a measurement, at low temperatures, of the stiffening of the rubber. Briefly, a specimen is mounted onto a torsion head and then twisted 180°. It is then exposed to a certain low temperature for a specified time period. The amount of twist is measured after the time elapses. The angle of the twist, which is inversely related to the stiffness, is plotted versus the specified temperature. A ratio of low temperature to room temperature modulus is developed. This test was performed using methanol chilled by dry ice. The chart below provides the relative modulus temperatures. T_2 refers to twice the room temperature stiffness; T_{100} being 100x the room temperature stiffness.

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Relative Modulus Temperatures, °C

	A60 (control)	B8030	C9030	DMFT
T_2	-18.0	-15.0	-19.0	-10.0
T_5	-37.0	-35.0	-34.5	-33.0
T_{10}	-43.0	-41.0	-41.5	-39.5
T_{100}	-56.0	-59.5	-55.0	-54.5

According to the ASTM standard, the temperatures encompass the transition region between the glassy and rubbery states of the compound. Overall, it appears that the control compound has better low temperature stiffness resistance than the Thermax® compounds. The C9030 compound has a similar relative modulus to the control and the B8030 compound has the highest T_{100} temperature. As seen with the above results and the tan delta data from the dynamic property/low temperature data, the general conclusion may be that Thermax® does not provide significantly better low temperature properties, but it is at least of equal value and is not detrimental to the compound. Therefore, cost reduction via replacing the more expensive halobutyl and better impermeability remain the primary reasons for using Thermax® in the inner liner.