

TECHNICAL Bulletin

Subject: Halobutyl Inner Liners II

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MEDIUM THERMAL BLACK N990 IN TIRE INNER LINERS

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

Compounds were prepared and mixed at the BFGoodrich Laboratory in Akron, Ohio during the fall of 1998. 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 the three new test compounds and is 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" development grade of thermal black.

Test Formulations

	Control A60	B8030 – N990	C9030 – N990	DMFT 9030
Bromobutyl 2030	100.0	100.0	100.0	100.0
GPF N660	60	30	30	30
Thermax® N990	-	80	90	-
Cancarb 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

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

	A60	B8030	C9030	DMFT
Thermax® N990 Carbon Black	-	80	90	-
Cancarb MFT Carbon Black	-	-	-	90
GPF N660 Carbon Black	60	30	30	30
Mooney Viscosity, 100°C, ASTM D 1646 – 96a				
ML (1 + 4)	67.59	76.31	78.10	75.23
Mooney Scorch, 137°C, ASTM D 1646 – 96a				
Min Torque	44.63	53.4	55.52	52.70
ts1	8.75	6.58	6.67	6.08
ts5	13.67	12.42	12.00	11.83
ts10	15.75	15.75	15.25	15.25
ts35	18.33	19.42	18.92	18.83
Monsanto R100 Oscillating Disk Rheometer @ 166°C, 1° arc, ASTM D 2084 - 95				
ML dNm	8.7	9.5	9.8	9.0
MH dNm	19.0	22.7	22.8	21.9
Tc (50) min	5.2	5.5	5.3	5.2
Tc (90) min	9.4	10.5	11.1	10.1
Tc (95) min	13.4	19.8	21.8	21.0
Stress-Strain, Originals @ RT, Cured 30 minutes @ 166°C, ASTM D 412 - 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 D 813 – 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 D 624 - 91				
kN/m	39.6	36.9	34.0	40.0
Rubber Peel Adhesion 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	0.346	0.645	0.562	0.55
(Measure of peel force per inch of width)				

Physical Properties

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

The four compounds showed similar scorch properties, as indicated by the Ts10 and Ts35. This was also in contrast to the earlier reported work, which indicated slightly more scorch safety in the thermal black compounds (but with different amounts of thermal black). The time to onset of scorch was similar to the earlier work (roughly 18 – 19 minutes).

The Rheometer data indicated similar torque properties and Tc50 and Tc90 results. However, the Tc95 showed substantially longer cure times for the thermal black compounds. This would be expected with the higher black 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 thermal black compounds, with comparatively lower tensile strength. But, the elongation was lower and the hardness was higher with the thermal black compounds. Again, this would likely be due to the higher carbon black loading.

Crack growth (ASTM D 813) and tear resistance (ASTM D 624) 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 MFT compound had tear resistance properties equal to that of the control, while the N990 compounds had slightly lower tear resistance. Whether or not there is sufficient fatigue resistance with the thermal black 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 thermal black samples showed better adhesion and smooth peeling. The best result was provided by the compound with 80 phr N990 and 30 phr N660.

Permeability

BFGoodrich does not have permeability testing capability and contracts Akron Polymer Laboratory to perform this test. APL performs this test according to ASTM D 1434, 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 Cancarb 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)

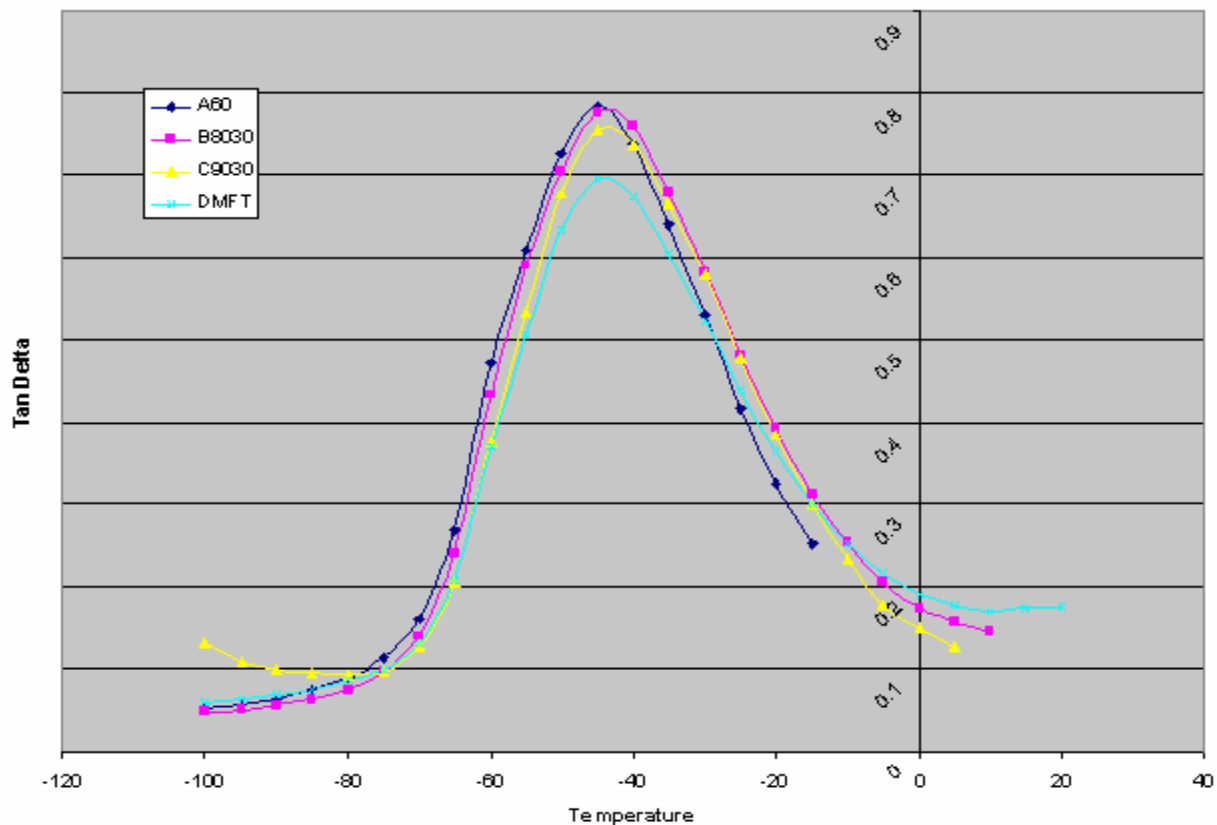
Sample	cm ³ .cm/cm ² .sec.atm	cm ³ .mm/m ² .day.atm
A60	1.67E-08	144.2
B8030	1.21E-08	104.9
C9030	1.47E-08	126.7
DMFT 9030	8.17E-09	70.6

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 D 5279. The second test is the standard method for stiffening at low temperature: flexible polymers and coated fabrics, as per ASTM D 1053 – 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:

Inner Liner: Rheometrics RSA II Low Temperature Dynamic Properties



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 thermal compounds continued until the +10°C level. The lowest tan delta was shown with the MFT compound, and the second lowest with the N990 90/30 compound. Both of these had the highest amounts of thermal black.

The "Gehman Torsion" low temperature stiffness test was performed at Akron Rubber Development Laboratory according to ASTM D 1053 - 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. T₂ refers to twice the room temperature stiffness; T₁₀₀ being 100 x the room temperature stiffness.

Relative Modulus Temperatures, °C

	A60	B8030	C9030	DMFT
T ₂	-18.0	-15.0	-19.0	-10
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 thermal black compounds. The C9030 (N990) compound has a similar relative modulus to the control and the B8030 compound has the highest T₁₀₀ 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 the thermal black does not provide significantly better low temperature properties. But, it is at least of equal value and is not detrimental to the compound. Thus, cost reduction via replacing the more expensive halobutyl and better impermeability remain the primary reasons for using the thermal black in the inner liner.