Plasthall® P-7092 and Plasthall® 8-10 TM-E Evaluated in Peroxide-Cured HNBR Compounds Based Upon Therban 1707, Tornac® and Zetpol 2000

Many of the manufacturers of Highly Saturated Nitrile elastomers consider trimellitate type plasticizers to provide a good balance of performance properties for HNBR compounds servicing 150°C applications. In this preliminary study, Plasthall® P-7092 (a polyester glutarate) and Plasthall® 8-10 TM-E (a linear trimellitate) are evaluated at 20.0 PPHR in peroxide-cured compounds based upon Therban 1707, Tornac® A and Zetpol 2000. The recipe used was provided to us by one of the HNBR elastomer manufacturers and unplasticized compounds were used as controls. Plasthall® P-7092 and Plasthall® 8-10 TM-E were found to provide comparable performance with regard to Mooney Viscosity reduction, compatibility and air oven volatility.

- Plasthall® P-7092 provided the following compound performance benefits:
  - Excellent permanence following ASTM Fuel C dry out
  - Excellent permanence following ASTM Oil #1 immersion
  - Excellent permanence following ASTM Oil #3 immersion
  - Slight improvement in Gehman low-temperature flexibility
- Benefits provided by Plasthall® 8-10 TM-E:
  - Excellent low-temperature impact and Gehman flexibility improvement
  - Good permanence following distilled water immersion dry out

Plasthall® P-7092 was selected for in-depth evaluation based on results obtained from several screening studies. Additional test work is planned to further examine P-7092 and other plasticizers in HNBR formulations. Results will be published as they become available.

Recipes for Banbury Mixing and Physical Testing

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Recipes for Banbury Mixing and Physical Testing

Recipe EN06-160	**1**	**3**	**4**	**5**	**7**	**8**	**9**	**11**	**12**
Therban 1707	100.0	100.0	100.0	-	-	-	-	-	-
Tornac® A	-	-	-	100.0	100.0	100.0	-	-	-
Zetpol 2000	-	-	-	-	-	-	100.0	100.0	100.0
N-550	65.0	→	→	→	→	→	→	→	→
Maglite® D	2.0	→	→	→	→	→	→	→	→
Kadox 930	2.0	→	→	→	→	→	→	→	→
Vulkanox ZMB-2	0.4	→	→	→	→	→	→	→	→
Vulkanox DDA	1.0	→	→	→	→	→	→	→	→
Diak #7	1.5	→	→	→	→	→	→	→	→
Plasthall P-7092	20.0	-		20.0	-		20.0	-	
PlasthallL 8-10TM-E	-	20.0	-	-	20.0	-	-	20.0	-
Sub Total	191.9	191.9	171.9	191.9	191.9	171.9	191.9	191.9	171.9
Mill Addition:									
Retilox F-40KEP	7.0	→	→	→	→	→	→	→	→
TOTAL	198.9	198.9	178.9	198.9	198.9	178.9	198.9	198.9	178.9
Major Variable	**Therban P-7092**	**→ 810TME**	**→ UNPLAS**	**Tornac® P-7092**	**→ 810TME**	**→ UNPLAS**	**Zetpol P-7092**	**→ 810TME**	**→ UNPLAS**
Recipe EN 06-160	**1**	**3**	**4**	**5**	**7**	**8**	**9**	**11**	**12**
Recipe variable	**Therban P-7092**	**→ 810TME**	**→ UNPLAS**	**Tornac® P-7092**	**→ 810TME**	**→ UNPLAS**	**Zetpol P-7092**	**→ 810TME**	**→ UNPLAS**

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Viscosity and Curing Properties

Mooney Viscosity: at 150°C (302°F)

Minimum Viscosity	37.5	35.5	73.0	48.5	39.5	83.0	49.5	42.0	87.0
t5, minutes	4.8	5.8	4.3	5.3	6.5	4.5	4.8	5.5	4.3
t35, minutes	9.3	11.0	7.8	10.3	12.3	8.3	9.0	10.5	7.5

Oscillating Disc Rheometer: at 180°C (356°F)

ML, lbf/in.	20.5	31.5	53.0	28.0	31.0	57.0	27.0	36.5	62.0
MH, lbf/in.	106.0	93.5	138.0	96.0	84.0	134.0	112.0	97.0	152.0
ts2, min.	2.5	2.2	1.8	2.3	2.2	1.8	2.0	2.0	1.5
t'c(90), min.	6.8	7.8	6.8	6.6	8.0	7.1	6.5	7.1	6.0
Cure Rate Index	23.3	17.9	20.0	23.3	17.2	18.9	22.2	19.7	22.2
1.25 x t'c(90), min.	8.5	9.8	8.5	8.3	10.0	8.9	8.1	8.9	7.5

Original Physical Properties

Stress at 100% Elong., MPa	7.8	6.6	12.2	6.7	5.9	12.4	8.3	5.9	12.8
psi	1125	950	1775	975	850	1800	1200	850	1850
Stress at 200% Elong., MPa	20.7	17.8	-	18.3	15.5	26.9	22.4	16.0	
Tensile, Ultimate, MPa	21.7	21.2	20.7	22.2	21.2	26.9	24.3	20.3	27.2
psi	3150	3075	3000	3225	3075	3900	3525	2950	3950
Elongation at Break, %	200	240	170	250	280	200	220	280	190
Hardness, Duro A, pts.	73	68	80	73	69	79	75	71	81
Specific Gravity	1.184	1.169	1.194	1.197	1.178	1.204	1.193	1.175	1.201

Roll Spew Compatibility:									
Temperature at: 23°C, 0°C, -21°C									
Exudation, 24h thru 96h	None	→	→	→	→	→	→	→	→
Recipe EN06-160	**1**	**3**	**4**	**5**	**7**	**8**	**9**	**11**	**12**
Recipe Variable	**Therban P-7092**	**→ 810TME**	**→ UNPLAS**	**Tornac® P-7092**	**→ 810TME**	**→ UNPLAS**	**Zetpol P-7092**	**→ 810TME**	**→ UNPLAS**
Low-Temperature Impact:									
As Molded									
All Pass, °C	-47	-53	-46	-41	-53	-38	-41	-50	-45
Low-Temperature Torsion:									
Gehman (As Molded)									
T_2, °C	-16	-16	-14	-16	-18	-16	-19	-21	-19
T^5, °C	-23	-27	-22	-24	-28	-22	-25	-29	-24
T_{10}, °C	-25	-30	-25	-25	-31	-24	-26	-31	-26
T_{100}, °C	-31	-36	-32	-30	-36	-30	-33	-38	-33
Apparent Mod. of Rigid., psi	167	133	295	133	111	269	181	139	334
Temperature at:									
500 psi, °C	-20	-24	-12	-23	-27	-16	-24	-28	-13
5,000 psi, °C	-29	-34	-26	-28	-34	-26	-28	-35	-26
10,000 psi, °C	-29	-37	-20	-29	-35	-27	-31	-37	-27
25,000 psi, °C	-33	-39	-32	-33	-39	-30	-34	-40	-31
50,000 psi, °C	-37	-43	-34	-37	-44	-32	-38	-45	-35

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Air Oven Aging, 70 h at 150°C									
Tensile, Ultimate, MPa	26.0	19.5	26.4	23.4	22.1	27.2	27.2	24.5	29.6
psi	3775	2825	3825	3400	3200	3950	3950	3550	4300
Tensile Change, %	+20	-8	+28	+5	+4	+1	+12	+20	+9
Elongation at Break, %	200	180	150	200	220	140	200	260	140-
Elongation Change, %	0	-25	-12	-20	-21	-30	-9	-7	-26
Hardness, Duro A, pts.	81	77	86	83	78	86	81	78	87
Hardness Change, pts.	+8	+9	+6	+10	+9	+7	+6	+7	+6
Weight Change, %	-2.4	-2.5	-2.3	-2.4	-2.6	-2.4	-1.9	-2.1	-2.0
180° Bend, All Pass	Yes	→	→	→	→	→	→	→	→
Recipe EN06-160	**1**	**3**	**4**	**5**	**7**	**8**	**9**	**11**	**12**
Recipe Variable	**Therban**	**→**	**→**	**Tornac®**	**→**	**→**	**Zetpol**	**→**	**→**
	P-7092	**810TME**	**UNPLAS**	**P-7092**	**810TME**	**UNPLAS**	**P-7092**	**810TME**	**UNPLAS**
Distilled Water Immersion, 70 h at 100°C									
Tensile, Ultimate, MPa	23.4	18.9	25.2	21.4	21.0	26.0	21.4	21.9	27.9
psi	3400	2750	3650	3100	3050	3775	3100	3175	4050
Tensile Change, %	+8	-11	+22	-4	-1	-3	-12	+8	+3
Elongation at Break,	220	230	180	250	270	220	220	260	190
Elongation Change., %	+10	-4	+6	0	-4	+10	0	-7	0
Hardness, Duro A, pts.	72	71	79	72	70	79	73	72	82
Hardness Change, pts.	-1	+3	-1	-1	+1	0	-2	+1	+1
Volume Change, %	+8.5	+3.4	+2.8	+9.9	+3.6	+2.8	+7.9	+3.1	+2.5
Weight Change, %	+7.8	+3.4	+2.9	+8.8	+3.6	+3.0	+7.2	+3.1	+2.7
180° Bend, All Pass	Yes	→	→	→	→	→	→	→	→
Distilled Water Dry Out, 22 h at 85°C									
Hardness, Duro A, pts.	78	75	84	79	72	84	79	75	83
Hardness Change, pts.	+5	+7	+4	+4	+3	+5	+4	+4	+2
Volume Change, %	2.3	-1.7	1.5	-2.2	-1.7	-1.9	-2.3	-1.6	-1.6
Weight Change, %	-1.6	-.96	-.86	-1.5	-.95	-1.0	-1.4	-.93	-.85

ASTM Fuel C Immersion, 70 h at 40°C		.							
Tensile, Ultimate, MPa	9.0	9.3	9.8	10.9	10.7	11.2	10.9	10.7	12.6
psi	1300	1350	1425	1575	1550	1625	1575	1550	1825
Tensile Change, %	-59	-56	-53	-51	-50	-58	-55	-47	-54
Elongation at Break, %	90	120	90	130	140	90	120	120	80
Elongation Change, %	-55	-50	-47	-48	-50	-55	-45	-57	-58
Hardness, Duro A, pts.	56	53	60	52	51	59	56	55	62
Hardness Change, pts.	-17	-15	-20	-21	-18	-20	-19	-16	-19
Volume Change, %	+63	+51	+65	+56	+42	+56	+57	+44	+57
Weight Change, %	+43	+34	+45	+38	+28	+38	+39	+30	+39
180° Bend, All Pass	Yes	→	→	→	→	→	→	→	→

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Recipe EN06-160 Recipe Variable	1 Therban P-7092	3 → 810TME	4 → UNPLAS	5 Tornac® P-7092	7 → 810TME	8 → UNPLAS	9 Zetpol P-7092	11 → 810TME	12 → UNPLAS
ASTM Fuel C Dry Out, 22 h at 70°C									
Hardness, Duro At pts.	76	80	81	77	81	80	78	80	82
Hardness Change, pts.	+3	+12	+1	+4	+12	+1	+3	+9	+1
Volume Change, %	-5.5	-13	-.67	-4.9	-12	-1.3	-4.7	-12	-.37
Weight Change, %	-5.1	-11	-.93	-4.8	-11	-1.5	-4.2	-10	-.77
ASTM Oil #1 Immersion, 70 h at 150°C									
Tensile, Ultimate, MPa	24.5	22.1	23.8	24.5	23.6	23.3	26.0	24.1	30.0
psi	3550	3200	3450	3550	3425	3375	3775	3500	4350
Tensile Change, %	+13	+4	+15	+10	+11	-13	+7	+19	+10
Elongation at Break, %	210	210	190	240	270	160	240	230	170
Elongation Change, %	+5	+13	+12	-4	-4	-20	+9	-18	-11
Hardness, Duro A, pts.	75	78	79	76	80	80	78	81	81
Hardness Change, pts.	+2	+10	-1	+3	+11	+1	+3	+10	0
Volume Change, %	0	-9.8	+1.4	-1.2	-11	-.41	-.78	-10	+.16
Weight Change, %	-.52	-8.5	+.82	-1.5	-9.6	-.52	-.90	-8.8	+.07
180° Bend, All Pass	Yes	→	→	→	→	→	→	→	→

ASTM Oil #3 Immersion, 70 h at 150°C									
Tensile, Ultimate, MPa	22.2	19.7	24.8	21.0	21.4	25.5	22.9	22.1	27.2
psi	3225	2850	3600	3050	3100	3700	3325	3200	3850
Tensile Change, %	+2	-7	+20	-5	+1	-5	-6	+8	0
Elongation at Break, %	210	220	190	250	320	200	230	260	180
Elongation Change, %	+5	-8	+12	0	+14	0	+5	-7	-5
Hardness, Duro A, pts.	67	66	69	69	70	72	71	70	73
Hardness Change, pts.	-6	-2	-11	-4	-1	-7	-4	-1	-8
Volume Change, %	+21	+12	+25	+15	+6.3	+18	+16	+7.3	+19
Weight Change, %	+17	+9.6	+20	+11	+4.9	+14	+12	+5.6	+15
180° Bend, All Pass	Yes	→	→	→	→	→	→	→	→

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Test Methods

Compounds for performance testing were mixed in a BR Banbury except for curatives which were added on a two-roll, 6 x 12 inch mill. Test specimens for compound performance properties were molded as follows: Press temperature - 180°C (356°F), Press time - 1.25 x t'c(90) minutes and at 833 psi on the sheet surface. Specimens for original properties, low temperature, immersions, air agings and roll spew were die cut from molded sheets .075 ± .005 inch thick.

Mooney Viscometer	ASTM D1646-81, Monsanto Viscometer, large rotor, 1 minute preheat.
Oscillating Disc Rheometer	ASTM D2084-81, Monsanto Rheometer, Square Die, 30 arc, 900 cpm, 0-100 range, 30 min. motor speed, 20 sec. preheat. MH at central point of torque rise, rate - one lb. in./5 min.
Original Properties	
Tensile, Elongation, Modulus	ASTM D412-83, Method A Die C
Hardness	ASTM D2240-84, ls reading
Specific Gravity	ASTM D792-66 (1979), para. 11.1
Compatibility	
Roll Spew, 96 h at 23°C, O°C, -21°C	Hallstar Method 01-79
Plasticizer Analysis	
Acid Value	AOCS Cd 3a-63
Saponification Value	AOCS Cd 3-25
Viscosity, cps	ASTM D1824-83
Specific Gravity	ASTM D2111
Water, %	Karl Fischer
Low-Temperature	
Impact (Brittleness)	ASTM D2137-83, Method A
Gehman	ASTM D1053
Air Oven Aging	ASTM D573-81
Fluid Immersion	ASTM D471-79

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Materials List

Material	Description	Supplier
Therban 1707	Hydrogenated Acrylonitrile - Butadiene	Mobay Corp.
Tornac® A	Hydrogenated Acrylonitrile - Butadiene	Polysar
N-550	FEF Carbon Black	Ashland
Maglite® D	Magnesium Oxide	Hallstar
Kadox 930	Zinc oxide - French Process	Hallstar
Vulkanox 2MB-2	Benzimidazol	Mobay Corp.
Vulkanox DDA	Styrenated Diphenylamine	Mobay Corp.
Diak #7	Triallyl Isocyanurate	DuPont
Plasthall® P-7092	Polyester Glutarate	Hallstar
Plasthall® 810TME	Linear Trimellitate	Hallstar
Retilox F-40 KEP	Bis (t-butylperoxy-isopropyl)	Akron Chem. benzene