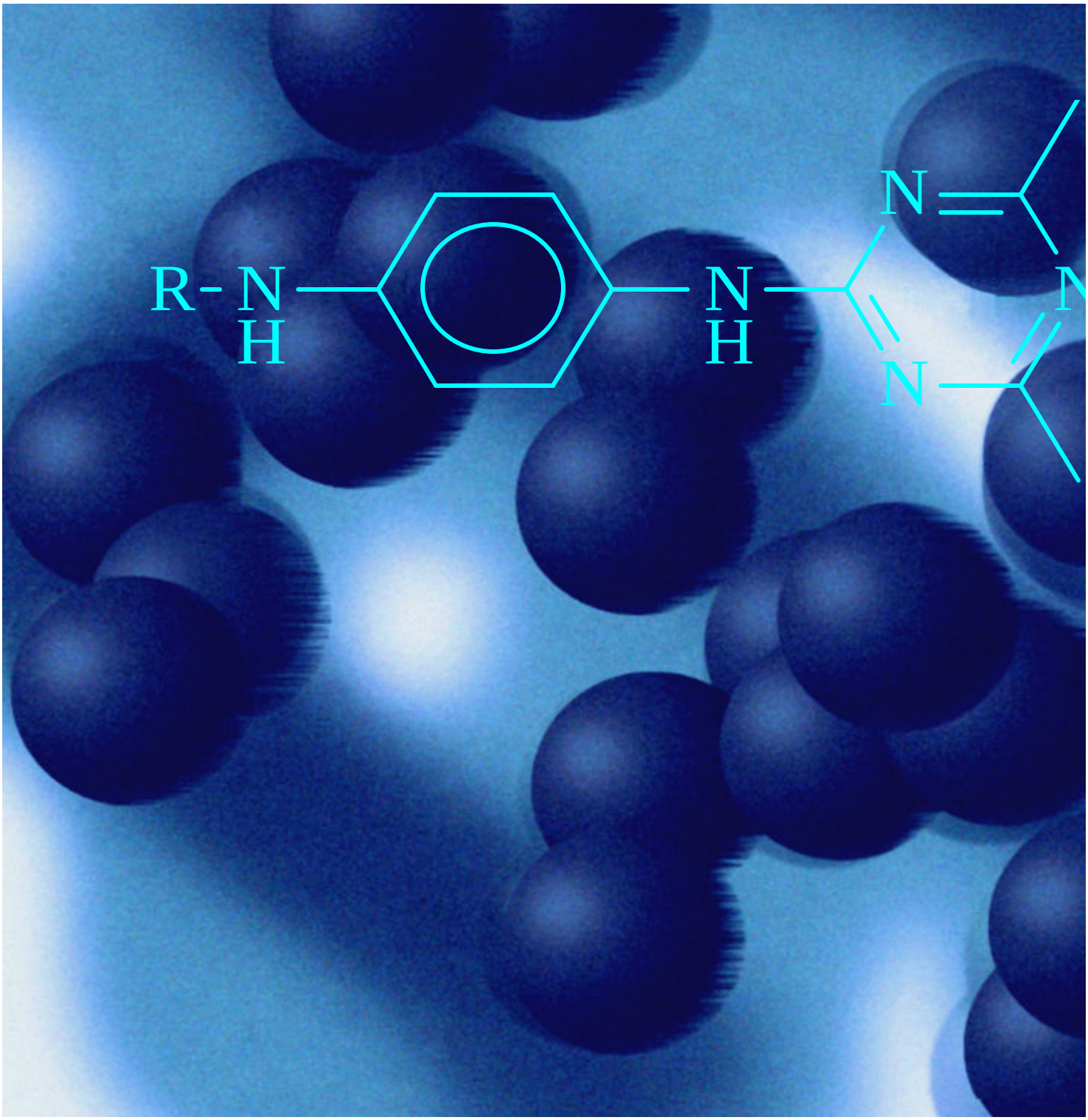
DURAZONE® 37

A Non-Staining Antiozonant

Crompton Uniroyal Chemical

Finding better solutions.



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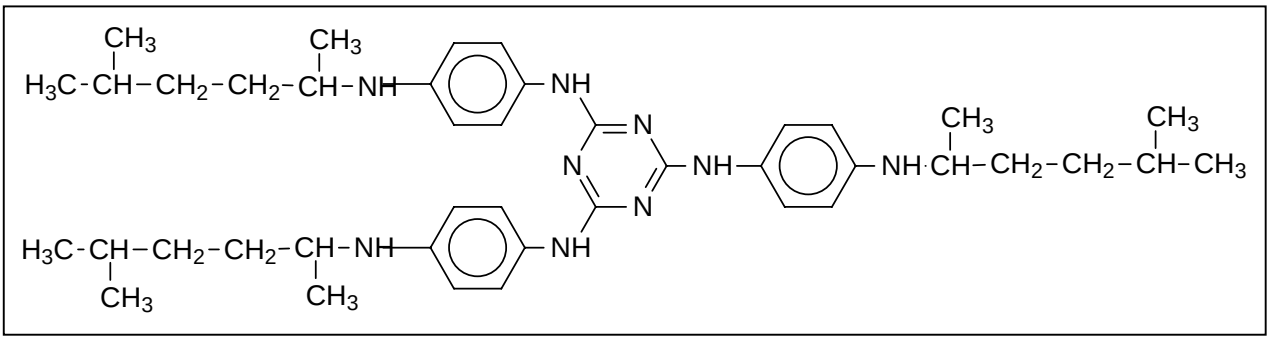
INTRODUCTION



2,4,6-Tris-(N-1,4-dimethylpentyl-p-phenylenediamino)-1,3,5-triazine

Durazone 37 is a revolutionary rubber chemical developed by Uniroyal Chemical as a protective agent for natural and synthetic rubbers. Durazone 37 is unique because it offers excellent antiozonant protection WITHOUT STAINING. Durazone 37 provides excellent static ozone protection, improved flex life and excellent antioxidant protection.

Durazone 37 exhibits excellent solubility in natural rubber, and good solubility in polychloroprene, chlorobutyl and bromobutyl rubbers, EPDM, synthetic polyisoprene and nitrile rubbers. Solubility in styrene-butadiene and polybutadiene rubbers is fair. Durazone 37 demonstrates antiozonant protection in SBR and BR, but will result in significant bloom in applications where dynamic ozone protection is required. Generally speaking, the recommendations as to the use of Durazone 37 depend mostly upon the type of polymer, as well as the compounding ingredients, curing package, compounding conditions, processing conditions and degree of protection required.

Actual commercial testing of Durazone 37 in an improved NR/EPDM sidewall tire shows that it not only protects the tire from the degrading effects of ozone, heat, oxygen and flex fatigue, but it does so without the staining so often seen with p-phenylenediamines. Since it is nonstaining, Durazone 37 can be used in the rubber compounds that are adjacent to white or light colored compounds. Testing has shown that a black sidewall tire containing Durazone 37 will not cause migratory staining of the white sidewall.

P-phenylenediamine type antiozonants must be used in combination with wax as a migratory aid for maximum antiozonant protection. In some instances, this application is unacceptable because high levels of wax can lead to a white bloom. Durazone 37 demonstrates outstanding static ozone protection without the use of wax.

Using Durazone 37 and a p-phenylenediamine type antiozonant together in a compound in which staining is not a issue provides even greater protection against ozone than could have been obtained by using either one alone. Because of the

synergy that takes place between these chemicals, greater long term dynamic and static ozone protection can be obtained.

Durazone 37 is an excellent antiozonant and antioxidant for applications where maximum static ozone protection and nonstaining characteristics are necessary. It is useful in tires, hose, footwear, mechanical goods, roofing, wire and cable and, in fact, is recommended in almost any rubber product where nonstaining static and dynamic ozone and aging protection are required. It is especially effective in natural rubber and in blends of NR with olefin based rubbers such as Uniroyal Chemical's Royalene® EPDM polymers and Trilene® Liquid EPDM polymers.

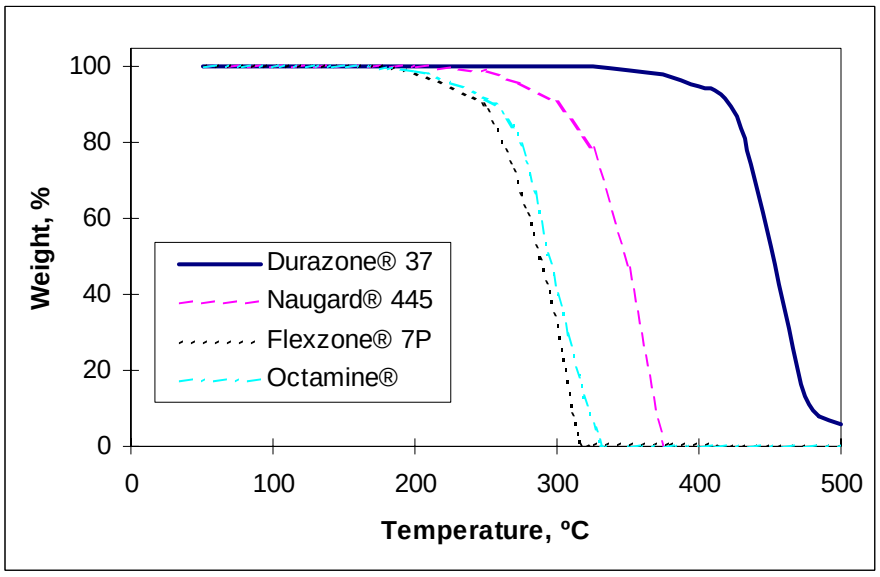
Durazone 37 permits the manufacturer of rubber products to achieve a higher level of quality in their products. This bulletin contains basic recommendations for the use of Durazone 37 in natural and synthetic rubber.

Amounts to use (per 100 parts of RHC): Durazone 37 is available as an easy to disperse flake that is relatively nonvolatile and chemically stable under severe service conditions. The amount of Durazone 37 necessary for compounding will vary depending upon the application. Most applications require between 2 and 4 phr.

Effect on rate of cure: Little to no effect has been observed in most polymers. Durazone 37 gives superior bin scorch safety compared to most p-phenylenediamines in NR, EPDM, NBR, BR, SBR, CR and IIR.

Processing: Durazone 37 provides optimum performance when introduced into the rubber compound at or above its melting point of 65-75 °C (149-167 °F).

Volatility: Durazone 37 is less volatile at elevated temperatures than most all other antiozonants and antioxidants. See the following Thermogravimetric Analysis (TGA) curves that were generated at a heating rate of 10 °C per minute (18 °F per minute).



IN A NATURAL RUBBER COMPOUND

In a natural rubber compound, Durazone 37 offers the added advantage of being a nonstaining antiozonant, while offering excellent protection from ozone attack and flex cracking resistance. It also demonstrates excellent heat aging resistance.

In the recipe to the right, the processing properties are not significantly affected by either Durazone 37 or Flexzone® 7P, as indicated by the compound Mooney scorch and rheometer data. Further, similar physical properties and dynamic properties are achieved with either Durazone 37 or Flexzone 7P.

RECIPE	PHR
SMR-5CV	100.0
N326 Carbon Black	50.0
Zinc Oxide	3.0
Naphthenic Oil	15.0
Stearic Acid	1.0
Antiozonant	as shown
Delac® S	0.7
Sulfur, 80% Oiled	2.2

The dynamic properties are illustrated in fatigue to failure and ozone belt testing data. Although Durazone 37 and Flexzone 7P both provide excellent dynamic properties and similar processing characteristics, p-phenylenediamines such as Flexzone 7P are know to stain the surface. Durazone 37, on the other hand, has the added advantage of being nonstaining. Other physical properties are shown on the following page.

	Control	Durazone 37	Flexzone 7P
Antiozonant, phr	--	4.0	4.0
Mooney at 100ºC (212ºF)			
Viscosity, ML1+4	34	34	36
Mooney at 132ºC (270ºF)			
Scorch time, t3, minutes	19	18	16
Rheometer at 150ºC (302ºF)			
Scorch time, ts1, minutes	1.7	1.6	1.3
Cure time, t'c90, minutes	3.8	3.3	3.4
ML, dN-m	4.5	4.5	4.8
(in-lb)	(4.0)	(4.0)	(4.3)
MH, dN-m	25.0	24.2	24.3
(in-lb)	(22.1)	(21.4)	(21.5)
Extension Cycling Fatigue Test – ASTM D4482			
Aged 70 h at 100ºC (212ºF), kc	55.5	122.0	132.4
Ozone Belt Test – ASTM D3395 50 pphm Ozone @ 40ºC (104ºF)			
after 24 hours, rating*	c	ok	ok
after 72 hours, rating*	c	vs	vvs

*** Appearance rating:** ok = no cracks (0) vvs = very, very slight (1) vs = very slight (2) s = slight (2.5) c = cracked (3)

	Control	Durazone 37	Flexzone 7P
Antiozonant, phr	--	4.0	4.0
Physical Properties, cured 10 min. at 177ºC (350ºF)			
Tensile Strength, MPa	13.4	15.1	11.8
(psi)	(1940)	(2190)	(1720)
Elongation at Break, %	660	750	660
300% Modulus, MPa	3.4	2.8	3.1
(psi)	(490)	(400)	(450)
Hardness, Shore A	47	44	44
Die C Tear Strength, kN/m	33.3	38.6	35.0
(pli)	(190)	(220)	(200)
Aged 2 weeks at 70ºC (158ºF)			
Tensile Strength, % Retained	96	121	130
Elongation, % Retained	74	81	83
300% Modulus, % Retained	167	220	108
Hardness, Points change	+6	+10	+8
Die C Tear Strength, % Retained	126	132	140

MIGRATION STAINING RESULTS – Two Week Outdoor Aging

In Figure 1, the black natural rubber compound containing Flexzone 7P migration stains the white compound adjacent to it.

In Figure 2, the black natural rubber compound containing the nonstaining antiozonant Durazone 37 does not stain the white compound.

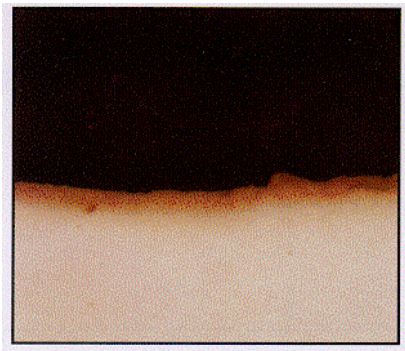


Figure 1 - Black NR compound contains FLEXZONE 7P.

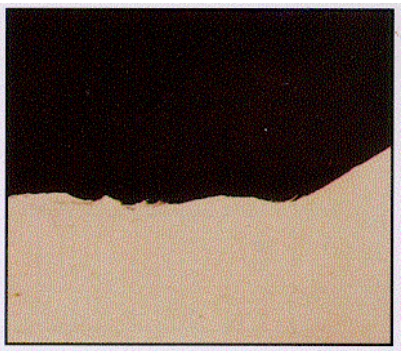


Figure 2 - Black NR compound contains the nonstaining antiozonant DURAZONE 37.

STATIC OZONE RESISTANCE

The following recipe and testing data illustrate the persistent ozone protection offered by Durazone 37 to a NR/BR compound. In outdoor static bent loop agings performed at Naugatuck, Connecticut, Durazone 37 exhibited outstanding static ozone resistance. Samples have been exposed for more than 900 days to the degrading effects of ozone, heat and moisture and have demonstrated no ozone cracking!

RECIPE	PHR
SMR-5CV	50.0
Cis 4 BR	50.0
N326 Carbon Black	50.0
Zinc Oxide	3.0
Stearic Acid	1.0
Aromatic Oil	5.0
Delac® NS	1.0
Sulfur	2.0

	Control	Flexzone 7P	Durazone 37
Antiozonant, phr	--	2.0	2.0
Sunproof® Improved wax, phr	1.5	1.5	1.5
Outdoor Static Bent Loop - ASTM D518 Unaged			
Time to ok rating, days	--	--	1,295
Time to vvs rating, days	--	214	--
Time to vs rating, days	--	247	--
Time to s rating, days	--	--	--
Time to c rating, days	145	549	--
Outdoor Static Bent Loop - ASTM D518 Aged 12 months at room temperature			
Time to ok rating, days	--	--	909
Time to vvs rating, days	66	136	--
Time to vs rating, days	--	--	--
Time to s rating, days	76	164	--
Time to c rating, days	164	212	--

Appearance rating: ok = no cracks (0) vvs = very, very slight (1) vs = very slight (2) s = slight (2.5) c = cracked (3)

In another evaluation, Durazone 37 was evaluated without wax in the formulation. Not only did Durazone 37 exhibit outstanding static ozone protection, but the dynamic ozone resistance was also improved.

	Control	Flexzone 7P	Durazone 37	Durazone 37 with wax
Antiozonant, phr	--	4.0	4.0	4.0
Sunproof® Improved wax, phr	--	--	--	1.5
Outdoor Static Bent Loop - ASTM D518 Unaged				
Time to ok rating, days	--	--	224	224
Time to vvs rating, days	--	110	--	--
Time to vs rating, days	14	148	--	--
Time to s rating, days	--	161	--	--
Time to c rating, days	32	224	--	--
Outdoor Dynamic flexing Unaged				
Time to ok rating, kc	--	29,425	--	--
Time to vvs rating, kc	3,000	--	5,750	5,750
Time to vs rating, kc	4,000	--	17,532	6,173
Time to s rating, kc	6,658	-	23,629	9,513
Time to c rating, kc	8,158	--	--	13,977
Outdoor Dynamic flexing Aged 6 months at room temperature				
Time to ok rating, kc	--	12,351	12,351	--
Time to vvs rating, kc	1,191	--	--	3,415
Time to vs rating, kc	3,416	--	--	8,841
Time to s rating, kc	4,191	-	--	10,616
Time to c rating, kc	6,055	--	--	--

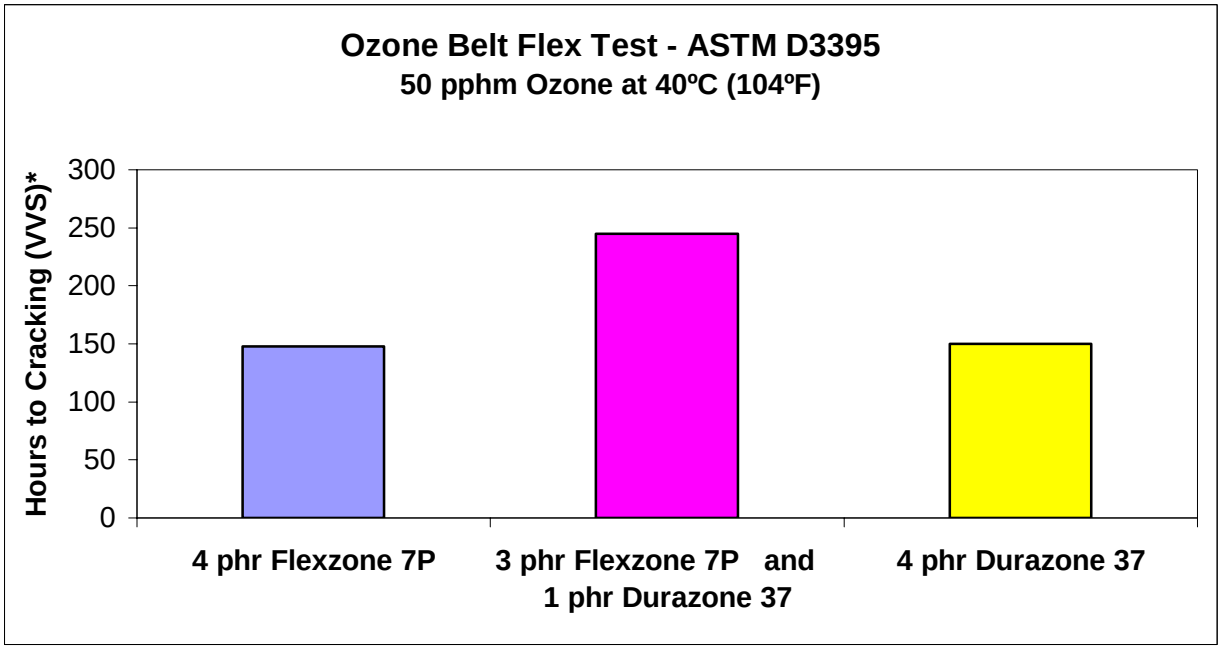
Appearance rating: ok = no cracks (0) vvs = very, very slight (1) vs = very slight (2) s = slight (2.5) c = cracked (3)

IN COMBINATION WITH P-PHENYLENEDIAMINE FOR LONG TERM DYNAMIC OZONE PROTECTION

Durazone 37 behaves synergistically with a p-phenylenediamine (Flexzone® 7P) in the following NR/BR recipe. You can note from the bar graph below that the same level (4 phr) of the blended antiozonants offers far greater resistance to cracking that does the 4 phr of either Durazone 37 or Flexzone 7P in ozone belt flex testing.

RECIPE	PHR
SMR-5CV	55.0
BR 1203	45.0
N351 Carbon Black	25.0
N660 Carbon Black	25.0
Sunproof® Regular	1.5
Zinc Oxide	3.0
Stearic Acid	1.0
Oil	7.0
Delac® NS	1.0
Sulfur	2.0
Antiozonant	as shown

DURAZONE 37 with FLEXZONE 7P in a NR/BR BLACK SIDEWALL COMPOUND



***** vvs = very, very slight (1)

IN BLACK NATURAL RUBBER
WINDSHIELD WIPER COMPOUND

The black NR compound to the right can be used as a beginning formulation for applications such as windshield wiper blades, coated fabric, cable jacket, molded goods, etc. Durazone 37 (Compound B) imparts superior ozone resistance to the compound while enhancing physical properties, and virtually no staining when compared to compounds containing Flexzone® 6H and Flexzone 3C (Compounds A and C respectively).

RECIPE	PHR
SMR-5CV	100.0
Zinc Oxide	5.0
HiSil 233	5.0
N774 Carbon Black	25.0
Stearic Acid	2.0
Delac® S	1.0
DPG	0.3
Sulfur, 80% Oiled	2.5
Antidegradants	as shown

	A	B	C
Naugard® Q, phr	1.5	1.5	--
Durazone 37, phr	--	2.5	--
Flexzone 6H, phr	2.5	--	--
Flexzone 3C, phr	--	--	0.5
Novazone® AS, phr	--	--	1.0
Sunproof® Improved wax, phr	2.0	2.0	1.5
Mooney at 100ºC (212ºF)			
Viscosity, ML1+4	28	24	26
Rheometer at 155ºC (311ºF)			
Scorch time, ts1, minutes	3.3	3.7	3.0
Cure time, t'c90, minutes	6.1	6.2	5.5
ML, dN-m	3.2	2.7	2.8
(in-lb)	(2.8)	(2.4)	(2.5)
MH, dN-m	34.2	33.8	34.2
(in-lb)	(30.3)	(29.9)	(30.3)
Physical Properties, cured 8 min. at 160ºC (320ºF)			
Tensile Strength, MPa	22.8	25.4	25.6
(psi)	(3350)	(3740)	(3770)
Elongation at Break, %	540	630	540
200% Modulus, MPa	3.4	3.1	4.2
(psi)	(500)	(460)	(620)
Hardness, Shore A	57	56	56
Static Ozone Bent Loop 50 pphm Ozone at 40ºC (104ºF)			
Time, hours	1,344	1,344	1,344
Rating*	ok	ok	vvs
Staining (Visual)	severe	negligible	severe

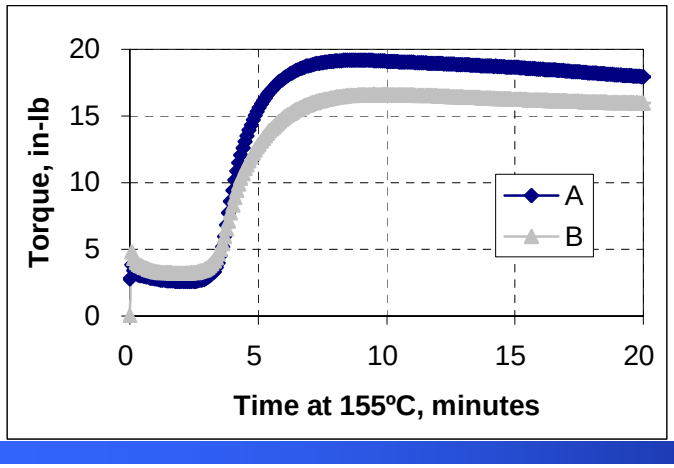
*** Appearance rating:** ok = no cracks (0) vvs = very, very slight (1) vs = very slight (2) s = slight (2.5) c = cracked (3)

IN NATURAL RUBBER, NON-BLACK COMPOUNDS (FOOTWEAR)

NR/EPDM blends have long been used to render ozone resistance to NR compounds without staining. A comparison is shown on the following page between a NR/EPDM compound (B) and a NR compound (A), containing Durazone 37. Both compounds were prepared from non-black typical footwear recipes shown at the right. The use of Durazone 37 in the all NR compound (A) allows one to maintain the higher physical properties typical of natural rubber compounds and at the same time, superior resistance to ozone without staining.

RECIPE	MB-1	MB-2
SMR-5CV	100.0	84.0
Royalene® 535	--	16.0
Zinc Oxide	5.0	5.0
Dixie Clay	50.0	50.0
Royalwhite Light	50.0	50.0
Titanium Dioxide	20.0	20.0
Light Process Oil	5.0	5.0
Stearic Acid	1.0	1.0
Naugex® MBTS	1.0	1.0
Tuex®	0.1	0.1
Tire Sulfur	2.75	2.75
Antidegradants	as shown	

	A	B
MB-1 (NR = 100 phr)	236.85	--
MB-2 (NR/EPDM = 84/16 phr)	--	236.85
Naugawhite® Liquid, phr	2.0	2.0
Naugard® Q, phr	1.0	--
Durazone 37, phr	2.5	--
Sunproof® Regular, phr	2.5	--
Mooney at 100ºC (212ºF)		
Viscosity, ML1+4	36	48
Mooney at 132ºC (270ºF)		
Scorch, t3, minutes	19.0	18.2
Rheometer at 155ºC (311ºF)		
Scorch time, ts2, minutes	2.9	2.8
Cure time, t'c90, minutes	5.8	6.3
ML, dN-m	2.9	3.6
(in-lb)	(2.6)	(3.2)
MH, dN-m	21.7	18.8
(in-lb)	(19.2)	(16.6)



IN NATURAL RUBBER, NON-BLACK COMPOUNDS (FOOTWEAR) … CONTINUED

	A	B
MB-1 (NR = 100 phr)	236.85	--
MB-2 (NR/EPDM = 84/16 phr)	--	236.85
Naugawhite® Liquid, phr	2.0	2.0
Naugard® Q, phr	1.0	--
Durazone 37, phr	2.5	--
Sunproof® Regular, phr	2.5	--
Properties, steam cured 23 min. at 434 kPa (63 psi) 155ºC (311ºF)		
Tensile Strength, MPa	14.2	7.0
(psi)	(2060)	(1010)
Elongation at Break, %	620	450
200% Modulus, MPa	3.1	2.5
(psi)	(450)	(360)
Hardness, Shore A	54	52
Die C Tear Strength, kN/m	39	30
(pli)	(220)	(170)
Unaged Bent Loop Ozone testing at 50 pphm Ozone at 40ºC (104ºF)		
Time to cracked, hours	1,128	6
Rating*	ok	c
Bent Loop Aged 100 Hours in Xenon Arc Weatherometer Before Ozone testing at 50 pphm Ozone at 40ºC (104ºF)		
Time to cracked, hours	8	6
White Lacquer Migration Stain Test		
Unaged, Hunter L Scale reading**	84.4	84.4
Aged 12 hours UV, Hunter L Scale reading	82.5	85.3
Aged 24 hours UV, Hunter L Scale reading	81.2	84.9

*** Appearance rating:** ok = no cracks (0) vvs = very, very slight (1) vs = very slight (2) s = slight (2.5) c = cracked (3)

**** L Scale rating:** 100 = pure white 0 = pure black

IN NR/EPDM BLACK SIDEWALL COMPOUND

In a NR/EPDM blend compound, Durazone 37 offers the added advantage of being a nonstaining antiozonant as well as providing protection equivalent to Flexzone 7P with respect to ozone and flex resistance. It also has excellent heat resistance properties.

While in the following recipe, EPDM improves the ozone cracking resistance of natural rubber in ozone belt testing, the addition of Durazone 37 to the NR/EPDM blend significantly improves the dynamic cracking resistance by a factor of two. Durazone 37 and Flexzone® 7P both provide excellent dynamic properties with similar processing characteristics. However, all p-phenylenediamines, such as Flexzone 7P, are known to stain a compound when they bloom to the surface. This staining will not occur with Durazone 37, resulting in a black sidewall compound that will have a longer lasting, more attractive appearance.

RECIPE	PHR
SMR-5CV	as shown
Royalene® EPDM	as shown
N660 Carbon Black	45.0
Zinc Oxide	3.0
Stearic Acid	1.0
Antiozonant	as shown
Delac® NS	0.85
Dicumyl peroxide, 60%	1.0
Sulfur, 80% Oiled	1.5

	A	B	C	D	E
SMR-5CV, phr	60	60	60	60	60
Royalene® 2914 (75 phr oil)	70	70	70	--	--
Royalene 3114 (50 phr oil)	--	--	--	60	60
Flexzone® 7P, phr	--	2.4	--	--	--
Durazone 37, phr	--	--	2.4	--	2.4
Mooney at 100ºC (212ºF)					
Viscosity, ML1+4	36	30	34	34	37
Mooney at 132ºC (270ºF)					
Scorch, t3, minutes	17.0	17.5	17.3	17.4	16.4
Rheometer at 177ºC (350ºF)					
Scorch time, ts2, minutes	1.5	1.7	1.5	1.5	1.5
Cure time, t'c90, minutes	3.4	4.4	3.6	3.8	3.5
ML, dN-m	4.4	4.7	4.5	4.1	4.2
(in-lb)	3.9	4.2	4.0	3.6	3.7
MH, dN-m	25.4	21.5	24.7	26.2	25.0
(in-lb)	22.5	19.0	21.9	23.2	22.1
Cured 10 min. at 177ºC (350ºF)					
Physical Properties					
Tensile Strength, MPa	17.5	15.5	17.0	18.1	17.7
(psi)	2540	2250	2460	2620	2570
Elongation at Break, %	710	760	750	690	730
300% Modulus, MPa	4.4	3.4	3.6	4.2	4.1
(psi)	640	490	520	610	600
Hardness, Shore A	44	42	46	47	48

	A	B	C	D	E
SMR-5CV, phr	60	60	60	60	60
Royalene® 2914 (75 phr oil)	70	70	70	--	--
Royalene 3114 (50 phr oil)	--	--	--	60	60
Flexzone® 7P, phr	--	2.4	--	--	--
Durazone 37, phr	--	--	2.4	--	2.4
Die B Tear Strength, kN/m	37	35	25	26	28
(pli)	210	200	140	150	160
Extension cycling fatigue - ASTM D4482					
Aged 70 hrs at 100ºC (212ºF)					
Cycles to cracking, kc	273	368	378	194	264
Ozone Belt Test - ASTM D3395					
50 pphm O_3 at 40ºC (104ºF)					
Rating after 300 hours	ok	ok	ok	ok	ok
Aged 3 days at 100ºC (212ºF)					
Tensile Strength, % Retained	52	88	93	52	87
Elongation, % Retained	84	79	86	85	87
300% Modulus, % Retained	71	155	136	80	125
Hardness, points change	-1	+8	+4	-4	+2
Tear Strength, % Retained	52	85	92	66	87

CONTACT STAINING RESULTS

In Figure 3, the black NR/EPDM compound containing Flexzone 7P contact stains the white compound adjacent to it. In Figure 4, the black NR/EPDM compound containing Durazone 37 does not stain the adjacent white compound.

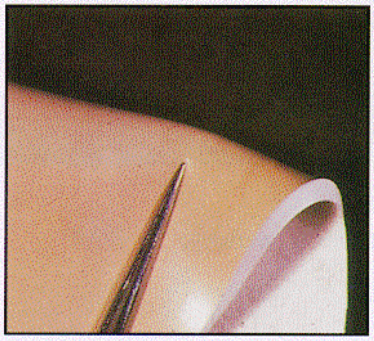


Figure 3 - Black NR/EPDM compound contains FLEXZONE 7P.

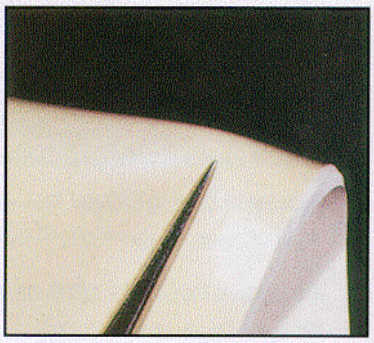


Figure 4 - Black NR/EPDM compound contains the nonstaining antiozonant DURAZONE 37.

IN NR/EPDM BLACK SIDEWALL COMPOUND … CONT'D

Tire Testing results for a 60/40 NR/EPDM black sidewall tire.

OZONE WHEEL TESTING CONDITIONS
Run continuously for 72 hours @ 40ºC (104ºF)

Inflation	138 kPa (20 psi)
Speed	48 k/h (30 mph)
Load	100%
Ozone	50 pphm

RESULTS

	Flexzone® 7P	Durazone 37
Crack Rating*	vvs	ok
Surface Appearance, visual	yellow	black

*** Appearance rating:** ok = no cracks (0) vvs = very, very slight (1) vs = very slight (2) s = slight (2.5) c = cracked (3)

Durazone 37 provides excellent ozone cracking resistance and maintains the clean black appearance of the sidewall compared to the yellow surface appearance of the tire sidewall containing Flexzone 7P. The slight yellowing appearance in the tread portion of the tire sidewall containing Durazone 37 (upper right of Figure 6) is due to the Flexzone 7P added to the tread compound.

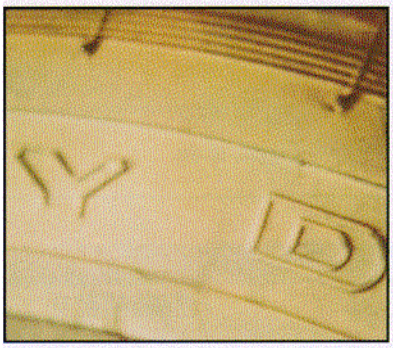


Figure 5 - Sidewall contains FLEXZONE 7P.

Figure 6 - Sidewall contains the nonstaining antiozonant DURAZONE 37.

IN NR/EPDM LIQUID EPDM COMPOUND

Blends of EPDM with NR improve ozone resistance. However, high levels of EPDM have to be used and the vulcanizates of these blends exhibit some undesirable properties such as reduced building tack, tensile strength and modulus. The use of Trilene® liquid EPDM and the nonstaining Durazone 37 together improve the static ozone resistance while retaining the tensile strength and compression set which are good intrinsic properties of natural rubber. This approach is illustrated in the recipe at the right. It utilizes the synergistic effect of small amounts of Trilene liquid EPDM, Royalene EPDM and Durazone 37 combined with natural rubber to obtain excellent static ozone protection and physical properties without staining.

RECIPE	PHR
SMR-5CV	80.0
Antiozonant	as shown
Zinc Oxide	5.0
Royalene® 535 EPDM	8.0
York Whiting	50.0
Stearic Acid	1.0
Trilene® 67	6.0
Sulfur	1.8
Naugex® MBTS	1.0
DPG	0.3
N-(cyclohexylthio)phthalimide	0.4

Since good dispersion is essential for better properties, the natural rubber and Durazone 37 should be added first to the mixer, followed by the high molecular weight Royalene EPDM. After mixing for about one minute, a small portion of the filler is added, followed by all of the Trilene liquid EPDM. The remaining ingredients are then mixed. Curatives can be added to the batch if the temperature is not excessive, or they can be added on the mill after dropping the batch.

	Naugard® Q	AO-B	Durazone 37
Antidegradant, phr	3.0	3.0	3.0
Mooney			
Viscosity at 100ºC (212ºF), ML1+4	34	42	27
Scorch at 125ºC (257ºF), t3, minutes	12.5	8.3	10.4
Rheometer at 150ºC (302ºF)			
Scorch time, ts2, minutes	3.0	2.2	2.6
Cure time, t'c90, minutes	6.8	6.0	6.3
ML, dN-m (in-lb)	4.0 (3.5)	5.7 (5.1)	2.8 (2.5)
MH, dN-m (in-lb)	30.2 (26.7)	29.5 (26.1)	28.6 (25.3)
Physical Properties, cured 12 min. at 160ºC (320ºF)			
Tensile Strength, MPa (psi)	15.8 (1975)	13.7 (2285)	14.7 (2140)
Elongation at Break, %	700	735	700
300% Modulus, MPa (psi)	1.8 (255)	1.7 (240)	1.8 (255)
Hardness, Shore A	48	48	49
Static Ozone Agings 50 pphm Ozone at 40ºC (104ºF)			
ASTM D518 Bent Loop, 818 hrs.	ok	ok	ok
ASTM D1149 20% Elong., 818 hrs	ok	ok	ok

AO-B = 6-ethoxy-2,2,4-trimethyl-1,2-dihydroquinoline

*** Appearance rating:** ok = no cracks (0) vvs = very, very slight (1) vs = very slight (2) s = slight (2.5) c = cracked (3)

IN SBR TIRE TREAD COMPOUND

In a SBR compound, Durazone 37 offers the added advantage of nonstaining as well as providing the equivalent protection of ozone resistance and flex cracking resistance as Flexzone® 7P

In this recipe, the use of Durazone 37 in the SBR tread compound provides a long lasting tire tread that will not migration stain the sidewall adjacent to it. Note in the data below that the dynamic modulus and tangent delta results, which relate to road handling and rolling resistance, respectively, are equal to those provided by the use of Flexzone 7P.

RECIPE	PHR
Linear SBR S-1210	50.00
Oil Extended SBR S-750	68.75
N234 Carbon Black	55.00
Zinc Oxide	3.00
Aromatic Oil	1.25
Stearic Acid	1.50
Sunproof® Regular wax	0.50
Antiozonant	as shown
Thiocarbamyl Sulfenamide	0.50
Delac® MOR	0.50
Sulfur	1.80

	Flexzone 7P	Durazone 37
Antiozonant, phr	1.0	1.0
Dynamic Properties, Cured 10 min. @ 177ºC (350ºF)		
Extension Cycling Fatigue Test - ASTM D4482		
Aged 70 hrs @ 100ºC (212ºF), kc	82.8	107.4
Dynamic Properties		
Tangent Delta at 4ºC (39ºF)	0.184	0.179
Tangent Delta at 28ºC (82ºF)	0.166	0.155
Tangent Delta at 53ºC (127ºF)	0.143	0.131
Tangent Delta at 77ºC (171ºF)	0.133	0.121
Tangent Delta at 102ºC (216ºF)	0.120	0.120
Dynamic Modulus (G*) at 4ºC (39ºF), MPa	8.53	7.75
Dynamic Modulus (G*) at 28ºC (82ºF), MPa	6.16	5.53
Dynamic Modulus (G*) at 53ºC (127ºF), MPa	4.62	4.34
Dynamic Modulus (G*) at 77ºC (171ºF), MPa	4.00	3.73
Dynamic Modulus (G*) at 102ºC (216ºF), MPa	3.58	3.39
Mooney		
Viscosity at 100ºC (212ºF), ML1+4	78	80
Scorch at 132ºC (270ºF), t3, minutes	38	40
Rheometer at 177ºC (350ºF)		
Scorch time, ts1, minutes	2.6	2.5
Cure time, t'c90, minutes	5.7	6.4
ML, dN-m	9.9	10.6
(in-lb)	(8.8)	(9.4)
MH, dN-m	40.7	40.2
(in-lb)	(36.0)	(35.6)

IN SBR TIRE TREAD COMPOUND … CONT'D

	Flexzone 7P	Durazone 37
Antiozonant, phr	1.0	1.0
Unaged Physical Properties		
Cured 10 minutes @ 177ºC (350ºF)		
Tensile Strength, MPa	23.3	22.5
(psi)	(3390)	(3270)
Elongation at Break, %	550	530
300% Modulus, MPa	9.9	9.6
(psi)	(1430)	(1390)
Hardness, Shore A	64	64
Die C Tear Strength, kN/m	68	72
(pli)	(390)	(410)
Aged 2 weeks at 70ºC (158ºF)		
Tensile Strength, % Retained	95	87
Elongation, % Retained	76	72
300% Modulus, % Retained	145	143
Hardness, points change	+5	+6
Die C Tear Strength, % Retained	100	107

MIGRATION STAINING RESULTS

In Figure 7, the black tread compound containing Flexzone 7P migration stains the white compound adjacent to it. In Figure 8, the black tread compound containing the nonstaining antiozonant Durazone 37 does not migration stain the white compound.

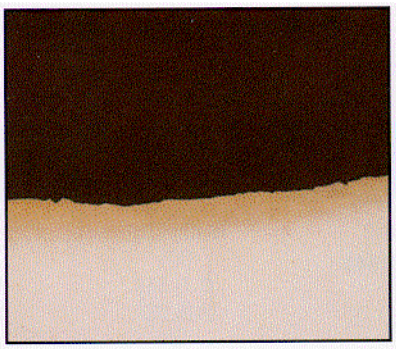


Figure 7 - Black tread compound contains FLEXZONE 7P.

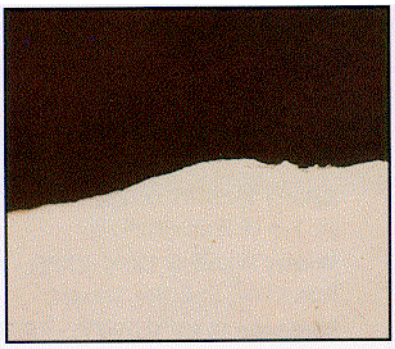


Figure 8 - Black tread compound contains the nonstaining antiozonant DURAZONE 37.

IN BLACK SBR COMPOUND
(EXTRUDED WINDSHIELD GASKET)

Sometimes, blends of SBR with EPDM are utilized to obtain an ozone resistant compound that is extremely cost competitive. However, at times this procedure leads to a reduction in physical properties and/or insufficient ozone resistance. The SBR compound, containing Durazone 37 (Compound C) in combination with a strong antioxidant/weak antiozonant (Naugard® Q) and wax (Sunproof® Extra) offers an acceptable combination of good physical properties and excellent ozone resistance.

Note: Royalene 637P contains 75 phr oil.

RECIPE	A	B	C
Royalene® 637P	105.0	--	--
Royalene 512	40.0	--	--
Royalene 535	--	15.0	--
SBR 1778	--	85.0	100.0
N550 Black	55.0	55.0	55.0
Suprex Clay	68.0	68.0	68.0
Cumar P-25 Resin	21.0	21.0	21.0
Zinc Oxide	4.1	4.1	4.1
Stearic Acid	1.4	1.4	1.4
Naugard® Q	--	3.0	4.0
Flexzone® 6H	--	0.5	--
Durazone 37	--	2.5	2.5
Sunproof® Extra	--	2.0	2.0
Monex®	0.4	0.4	0.4
Sulfur	1.4	1.4	1.4
Naugex® MBT	2.4	2.4	2.4

	A	B	C
Mooney			
Viscosity at 100ºC (212ºF), ML1+4	95	60	60
Scorch at 132ºC (270ºF), t3, minutes	--	14.0	14.0
Rheometer at 160ºC (320ºF)			
Scorch time, ts1, minutes	1.8	1.6	1.7
Cure time, t'c90, minutes	8.6	4.6	5.0
ML, dN-m (in-lb)	7.9 (7.0)	6.4 (5.7)	6.1 (5.4)
MH, dN-m (in-lb)	32.5 (28.8)	18.5 (16.4)	17.2 (15.2)
Physical Properties, cured 12 min. at 177ºC (350ºF)			
Tensile Strength, MPa (psi)	11.2 (1620)	8.3 (1200)	9.5 (1380)
Elongation at Break, %	740	600	710
300% Modulus, MPa (psi)	3.5 (510)	4.1 (590)	3.6 (520)
Hardness, Shore A	67	59	56
Static Ozone Aged 72 hours at 50 pphm Ozone at 40ºC (104ºF)			
Triangular Pieces around 2" Mandrels			
Rating* after 296 hours	ok	ok	ok
White Lacquer Migration Stain Test			
Unaged, Hunter L Scale reading**	94.1	84.0	83.3
Aged 12 hours Sunlamp, Hunter L	85.6	76.3	83.3

*** Appearance rating:** ok = no cracks (0) vvs = very, very slight (1) vs = very slight (2) s = slight (2.5) c = cracked (3)

**** L Scale rating:** 100 = pure white 0 = pure black

ANTIOXIDANT PROTECTION IN A SBR COMPOUND

Durazone 37 has been shown to impart good resistance to heat aging and oxidation in a SBR compound.

RECIPE	PHR
SBR	100.0
N339 Carbon Black	70.0
Aromatic Oil	35.0
Zinc Oxide	2.5
Stearic Acid	1.0
Sunproof® Improved wax	0.5
Antidegradant	2.5
Delac® NS	1.0
DPG	0.3
Sulfur	1.75

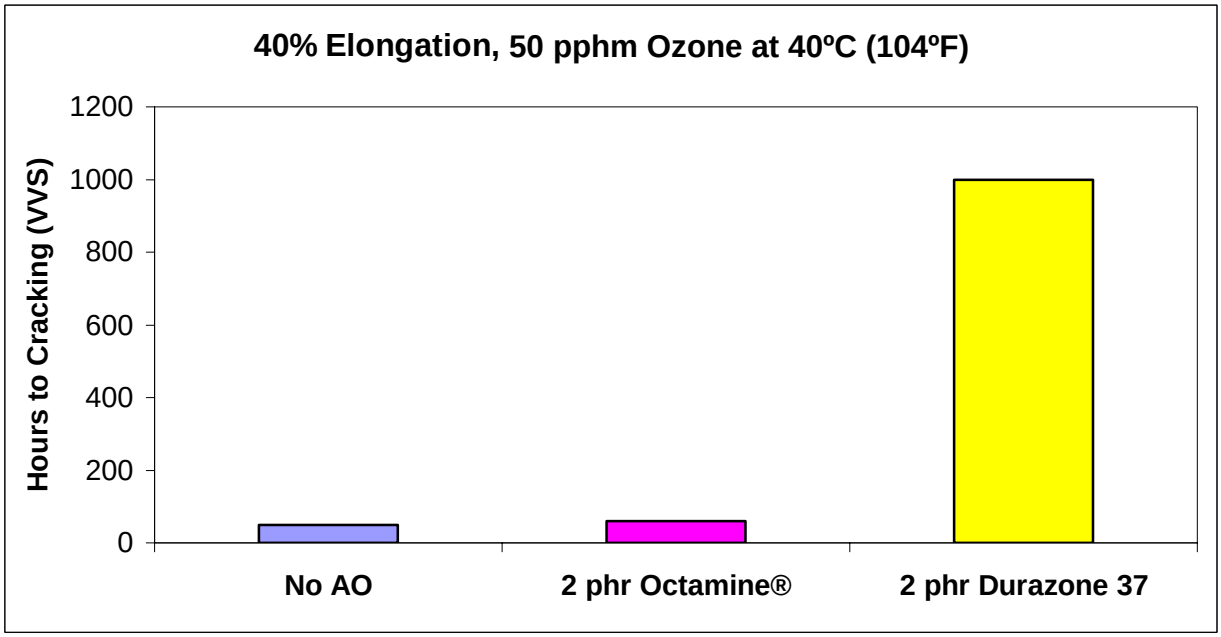
	Flexzone 7P/ Naugard Q	Durazone 37/ Vanox MTI*	Flexzone 7P/ Vanox MTI	Flexzone 7P/ Durazone 37/ Vanox MTI
Antidegradant, phr	2.5/2.5	2.5/2.5	2.5/2.5	1.25/1.25/2.5
Physical Properties, cured 10 min. at 176ºC (349ºF)				
Tensile Strength, MPa	20.7	20.5	22.4	19.7
(psi)	(3040)	(3010)	(3290)	(2900)
Elongation at Break, %	690	800	750	800
300% Modulus, MPa	7.0	5.6	6.7	5.2
(psi)	(1030)	(830)	(990)	(760)
Hardness, Shore A	60	61	61	62
Tear Strength, kN/m	65	67	61	65
(pli)	(370)	(380)	(350)	(370)
Aged 2 weeks at 70ºC (158ºF)				
Tensile Str., % Retained	28	48	29	49
Elongation, % Retained	5	20	70	21
Hardness, points change	+28	+25	+26	+25
Tear Str., % Retained	37	50	42	48

* Vanox MTI = 2-mercaptotoluimidazole

IN POLYCHLOROPRENE RUBBER

Durazone 37 has been evaluated in polychloroprene rubber and it does not adversely affect the scorchiness of the compound. Normally, amine antiozonants are not utilized in polychloroprene compounds, and only amine antioxidants, which function as weak antiozonants are utilized so as to reduce the effect of decreased scorch time in these compounds. The graph below illustrates the superior performance that can be obtained from Durazone 37 when compared to a currently used antidegradant.

NEOPRENE W COMPOUND
STRESSED STATIC OZONE TESTING



Durazone 37 was evaluated in the Neoprene GW compound recipe in the box to the right, which is a typical recipe for use as a conveyor belt, cable jacket, coated fabric, molded goods, hose cover, etc. The physical properties appear on the next page.

RECIPE	PHR
Neoprene GW	100.0
Reogen	5.0
Magnesium Oxide	4.0
Vanfre AP-2	3.0
N990 Carbon Black	50.0
Crown Clay	50.0
Zinc Oxide	5.0
Sulfads	1.5
DOTG	0.5
END-75 (ETU)	0.75
Antidegradants	as shown

IN POLYCHLOROPRENE RUBBER … CONT'D

It has been shown that the utilization of Durazone 37 does not affect either rheological or physical properties. Compare Compound C to Compounds A and B. One of the major advantages in using Durazone 37 is the ability to obtain excellent ozone resistance while maintaining a shiny black surface.

	A	B	C
Antidegradant, phr	Octamine®	Novazone® AS	Durazone 37
Mooney			
Viscosity at 100ºC (212ºF), ML1+4	62	68	65
Scorch at 121ºC (250ºF), t3, minutes	6.5	5.9	6.0
Rheometer at 160ºC (320ºF)			
Scorch time, ts1, minutes	1.2	1.1	1.2
Cure time, t'c90, minutes	7.9	7.4	6.3
ML, dN-m (in-lb)	7.6 (6.7)	8.7 (7.7)	8.4 (7.4)
MH, dN-m (in-lb)	52.9 (46.8)	54.2 (48.0)	55.4 (49.0)
Physical Properties, cured 20 min. at 160ºC (320ºF)			
Tensile Strength, MPa (psi)	21.6 (3140)	21.5 (3120)	21.0 (3050)
Elongation at Break, %	400	390	370
300% Modulus, MPa (psi)	18.6 (2700)	18.2 (2640)	18.4 (2670)
Hardness, Shore A	70	74	72
Ozone Agings 50 pphm Ozone at 40ºC (104ºF)			
Static Bent Loop, hours	120	192	192
Rating*	c	ok	ok
Surface Color	black	brown	black
Dynamic Flexing, 25% Extension			
hours	96	264	192
Rating*	c	c	c
Static 40% Elongation			
hours	120	192	192
Rating*	c	ok	ok
Aged 5 days at 135ºC (275ºF)			
Tensile Strength, % Retained	62	67	55
Elongation, % Retained	20	23	16
Hardness, points change	+21	+12	+16

*** Appearance rating:** ok = no cracks (0) vvs = very, very slight (1) vs = very slight (2) s = slight (2.5) c = cracked (3)

IN NON-BLACK NITRILE COMPOUND (OIL WELL CABLE JACKET)

The formulation to the right can be utilized as an oil well cable jacket, outsole, oil seal, coated fabric, conveyor belt, molded goods, etc. Non-black NBR compounds maintain a high level of their rheological and physical properties when compounded with Durazone 37 in combination with Naugard® Q. An added effect by utilizing Durazone 37 in combination with antioxidants and a wax is increased ozone protection without staining.

RECIPE	PHR
Paracril BJLT-M40	100.0
Zinc Oxide	5.0
Stearic Acid	1.0
Paraplex G-33	20.0
HiSil 233	40.0
Suprex Clay	35.0
Naugex® MBTS	2.5
Ethyl Tuex®	1.5
Tuex®	1.5
Tire Sulfur	0.2
Antidegradants	as shown

	A	B	C	D
Vanox MTI, phr	2.0	--	--	--
Aminox®, phr	1.0	--	--	--
Naugard Q, phr	--	--	--	1.0
Durazone 37, phr	--	2.0	2.5	2.5
Sunproof® Junior, phr	--	2.0	2.0	2.0
Moooney				
Viscosity at 100ºC (212ºF), ML1+4	56	44	50	48
Scorch at 121ºC (250ºF), t3, min.	33.5	30.0	30.7	31.7
Rheometer at 150ºC (302ºF)				
Scorch time, ts1, minutes	5.8	5.5	5.4	5.3
Cure time, t'c90, minutes	10.8	9.7	9.6	9.9
ML, dN-m (in-lb)	4.7 (4.2)	4.3 (3.8)	5.0 (4.4)	4.6 (4.1)
MH, dN-m (in-lb)	33.0 (29.2)	31.1 (27.5)	33.3 (29.5)	31.8 (28.1)
Properties, steam cured 40 min. at 345 kPa (50 psi) 148ºC (298ºF)				
Tensile Strength, MPa (psi)	13.1 (1930)	11.4 (1670)	12.4 (1820)	11.6 (1710)
Elongation at Break, %	760	830	790	790
200% Modulus, MPa (psi)	2.2 (320)	1.8 (270)	1.9 (280)	2.2 (330)
Hardness, Shore A	69	64	65	66
Die C Tear Strength, kN/m (pli)	32 (180)	30 (170)	28 (160)	32 (180)
Static Ozone Agings 50 pphm Ozone at 40ºC (104ºF)				
Bent Loop, hrs to cracked rating	48	144	312	360
Aged 166 hours at 125ºC (257ºF)				
Tensile Strength, % Retained	89	94	81	95
Elongation, % Retained	98	78	82	80
Hardness, points change	+11	+18	+16	+17

IN NON-BLACK NITRILE/PVC COMPOUND (HOSE COVER COMPOUND)

Although blends of NBR/PVC provide good ozone resistance, they tend to degrade when exposed to UV prior to exposure to ozone, and the ozone resistance is reduced. By utilizing Durazone 37, the deficiency can be overcome in both press cured and steam cured compounds.

RECIPE	PHR
Paracril OZO	100.0
Zinc Oxide	5.0
HiSil EP	50.0
Stearic Acid	1.0
Titanium Dioxide	5.0
DOP	15.0
Spider Sulfur	2.00
Naugex® MBTS	1.25
Tuex®	0.20
Antidegradants	as shown

Durazone 37 can be used in the recipe in the box to the right to improve ozone resistance, after UV exposure, in NBR/PVC compounds for use in applications such as hose covers, outsoles, cable jackets, molded goods, conveyor belts, etc. Compounds, shown below, and containing Durazone 37 and blends of Durazone 37 with Naugard® NBC exhibit slightly increased cure rate while maintaining good scorch safety, and also exhibit equally good physical properties and better ozone resistance than those compounds without Durazone 37 or Durazone 37/Naugard NBC blend.

	A	B	C	D
Octamine®, phr	1.0	1.0	1.0	1.0
Naugard NBC, phr	--	--	0.7	0.7
Sunproof® Junior, phr	2.0	2.0	2.0	2.0
Durazone 37, phr	--	3.0	--	3.0
Moooney				
Viscosity at 100ºC (212ºF), ML1+4	54	51	54	50
Scorch at 121ºC (250ºF), t3, min.	45+	45+	45+	45+
Rheometer at 150ºC (302ºF)				
Scorch time, ts1, minutes	14.1	10.5	11.5	8.7
Cure time, t'c90, minutes	21.0	18.2	17.7	14.6
ML, dN-m (in-lb)	4.1 (3.6)	3.7 (3.3)	3.9 (3.5)	3.3 (3.7)
MH, dN-m (in-lb)	33.1 (29.3)	31.8 (28.1)	36.5 (32.3)	33.1 (29.3)
Press cured 40 min. at 150ºC (302ºF)				
Tensile Strength, MPa (psi)	12.8 (1860)	14.6 (2120)	13.9 (2020)	14.9 (2160)
Elongation at Break, %	530	620	560	560
200% Modulus, MPa (psi)	5.7 (830)	5.0 (720)	5.5 (800)	5.5 (800)
Hardness, Shore A	72	70	72	71
Die C Tear Strength, kN/m (pli)	51 (290)	47 (270)	49 (280)	49 (280)
Properties, steam cured 40 min. at 380 kPa (55 psi) 150ºC (302ºF)				
Tensile Strength, MPa (psi)	9.5 (1380)	9.2 (1340)	10.1 (1470)	8.1 (1170)
Elongation at Break, %	470	510	500	450
200% Modulus, MPa (psi)	4.8 (690)	3.9 (570)	4.6 (670)	4.3 (630)
Hardness, Shore A	69	70	68	60
Die C Tear Strength, kN/m (pli)	44 (250)	44 (250)	44 (250)	39 (220)

IN NON-BLACK NITRILE/PVC COMPOUND (HOSE COVER COMPOUND) … CONT'D

	A	B	C	D
Octamine®, phr	1.0	1.0	1.0	1.0
Naugard NBC, phr	--	--	0.7	0.7
Sunproof® Junior, phr	2.0	2.0	2.0	2.0
Durazone 37, phr	--	3.0	--	3.0
Static Ozone Agings 50 pphm Ozone at 40ºC (104ºF)				
Press Cured				
Bent Loop, hours	36	1,011	1,011	1,011
Rating*	c	ok	ok	ok
Steam Cured				
Bent Loop, hours	267	1,011	603	1,011
Rating*	c	ok	c	ok
Aged 48 hrs in Xenon Weatherometer prior to Ozone Testing				
Press Cured				
Bent Loop, hours	699	1,011	1,011	1,011
Rating*	c	vvs	vvs	vvs
Steam Cured				
Bent Loop, hours	8	201	78	267
Rating*	c	c	c	c
Aged 240 hrs in Xenon Weatherometer prior to Ozone Testing				
Press Cured				
Bent Loop, hours	78	1,011	771	1,011
Rating*	c	vs	c	ok
Steam Cured				
Bent Loop, hours	8	8	8	8
Rating*	c	c	c	c

*** Appearance rating:** ok = no cracks (0) vvs = very, very slight (1) vs = very slight (2) s = slight (2.5) c = cracked (3)

COMPOSITION: 2,4,6-Tris-(N-1,4-dimethylpentyl-p-phenylenediamino)-1,3,5-triazine

FORM: An easily dispersed dark purple flake.

TYPICAL PROPERTIES:

Melting Range	65-75 °C (149-167 °F)
Molecular Weight	694
Specific Gravity	1.05
Volatility at 21 °C (70 °F)	Low

SOLUBILITY: Soluble in most organic solvents. Insoluble in water.

STORAGE STABILITY: In excess of two (2) years when stored under normal conditions in a cool, dry place away from any direct sources of heat and light. Keep containers closed when not in use.

HANDLING PRECAUTIONS: Avoid all personal contact. Observe good personal hygiene. Impervious gloves, protective clothing and eye protection should be worn when handling. In the absence of adequate ventilation, NIOSH-certified respiratory protection should be used as necessary.

FEATURES: Offers excellent static and dynamic ozone protection and excellent antioxidant protection.

DISCOLORATION: Durazone 37 is a nonstaining antiozonant offering superior static ozone protection, excellent dynamic protection and excellent antioxidant protection. Durazone 37 will not migrate stain from a black compound to a white compound adjacent to it. Therefore, migration staining of the white compound will not occur.

RECOMMENDED USES: As a compounding ingredient, Durazone 37 is added into rubber above its melting point of 75 ºC (167 ºF) in quantities of 2 to 4 parts per 100 parts RHC.