DuPont™ Vamac® Ultra IP

Technical Information — Rev. 2, July 2010

Introduction

The latest manufacturing technology allows polymerization of AEM grades with optimized structure and chemical composition, which provides significant improvements over the existing standard DuPont™ Vamac® elastomers. These grades, designated and sold as Vamac® Ultra, provide true step-change improvement in processability, performance and customer value for targeted applications.

Vamac® Ultra IP (developmental name VMX-3040) is the first of the Ultra family products to be commercialized. During the development phase, a goal was to identify a product having improved performance in injection molding processes compared to Vamac® G to reduce the frequency of mold cleaning. The high viscosity of Vamac® Ultra IP compared to standard AEM grades results in better mixing as well as increased green strength of compounds, and helps to avoid collapse during extrusion processes. The optimized polymer structure ensures gains in physical properties resulting in improved performance of rubber parts such as seals, dampers and extruded hoses. Low abrasion values may allow extended use of Vamac® Ultra IP in dynamic seals.

Major Performance Properties and Applications

The best physical properties of Vamac® Ultra IP are obtained in rubber parts having a hardness range between 50 and 90 Shore A. Extensions of this hardness range may be more easily achieved with Vamac® Ultra IP than standard AEM using appropriate compounding.

Vamac® Ultra IP combines dry heat resistance of 175 °C over a period of 1000 h (six weeks) with very good resistance to automotive lubricants. Exposure of peak temperatures of 200 °C are possible for up to four days. At the same time, the Tg of –31 °C provides very good low temperature flexibility.

Good compression set and compressive stress relaxation properties make Vamac® Ultra IP an excellent choice for sealing applications. Good resistance to Blow-By (hot air, acids, oil and petrol fumes), present in automotive crankcase venting systems and air ducts combined with increased dynamic resistance are additional attributes of Vamac® Ultra IP.

Like every other grade of Vamac®, Vamac® Ultra IP is halogen-free. Typical properties of Ultra IP are shown in *Table 1*.

Handling Precautions

Because Vamac® Ultra IP contains small amounts of residual methyl acrylate monomer, adequate ventilation should be provided during storage and processing to prevent worker exposure to methyl acrylate vapor. Additional information may be found in DuPont Material Safety Data Sheet (MSDS), and bulletin, *Safe Handling and Processing of Vamac®* (VME-A10628), available from DuPont.

(Previously VMX-3040)

Table 1. DuPont™ Vamac® Ultra IP — Typical Product Properties

Property	Limit	Method
Form	Bale size is nominally: 560 mm by 370 mm by 165 mm (22 in. by 15 in. by 7 in.)	Visual Inspection
Color	Clear	Visual Inspection
Total Volatile Matter	< 0.6%	DuPont MP 726-1
Polymer Viscosity ML (1+4), 100 °C (MU)	29 ± 4	ASTM D 1646

Mixing

Vamac® Ultra IP has a higher viscosity than Vamac® G which permits better and faster dispersion of fillers and other compounding ingredients, especially in low hardness compounds or in formulations with high plasticizer levels. Black Incorporation Time (B.I.T., time between addition of the carbon black to the Banbury and peak of energy consumption) of the compounds shown in *Table 2* was reduced by 17% when replacing Vamac® G with Vamac® Ultra IP.

Table 2. Mixing Compounds of Vamac® Ultra IP and Vamac® G

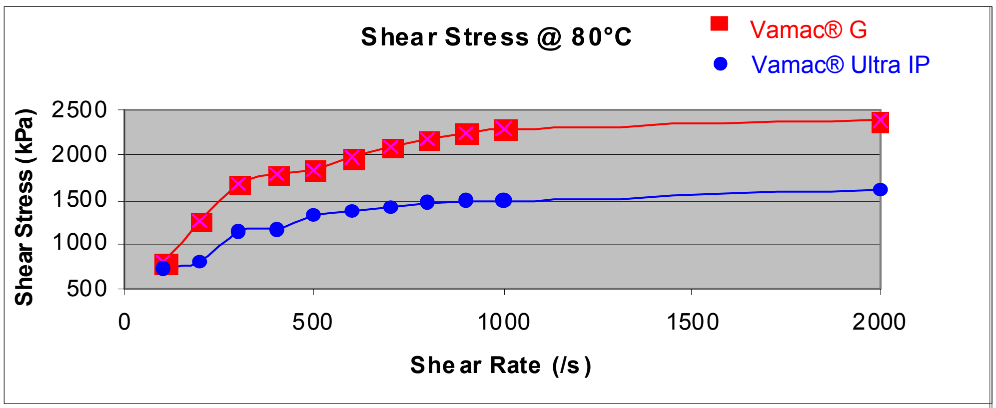
Compound No.	Vamac® G	Vamac® Ultra IP
Vamac® G	100	
Vamac® Ultra IP		100
Naugard® 445	2	2
Armeen® 18D	0.5	0.5
Stearic acid	1.5	1.5
Vanfre® VAM	1	1
Spheron® SO N550	65	65
Rhenosin W 759	10	10
Diak™ No.1	1.5	1.5
Vulcofac® ACT 55	2	2
Francis Shaw 1.7 litre Banbury mixing		
Rotor Speed (rpm)	40	40
B.I.T. (sec)	48	40
Temperature at B.I.T. (°C)	77	75
Discharge : Time (sec) / Temperature (°C)	180 / 95	180 / 96
Load factor (%)	66	66
Polymer Mooney ML (1+4), 100 °C (MU)	16.8	30.1
Compound Mooney Viscosity, ML (1+4), 100 °C (MU)	41.8	67.4

Processing

Standard AEM grades have Mooney viscosities ML (1+4) at 100 °C of below 20 MU. This provides good compound flow and adhesion to other substrates for bonded parts. In extrusion, DuPont™ Vamac® Ultra provides the compound viscosity level or green strength needed to avoid collapse of extruded, uncured compounds. Stickiness of the extruded compound strips for feeding to injection molding machines can also be reduced.

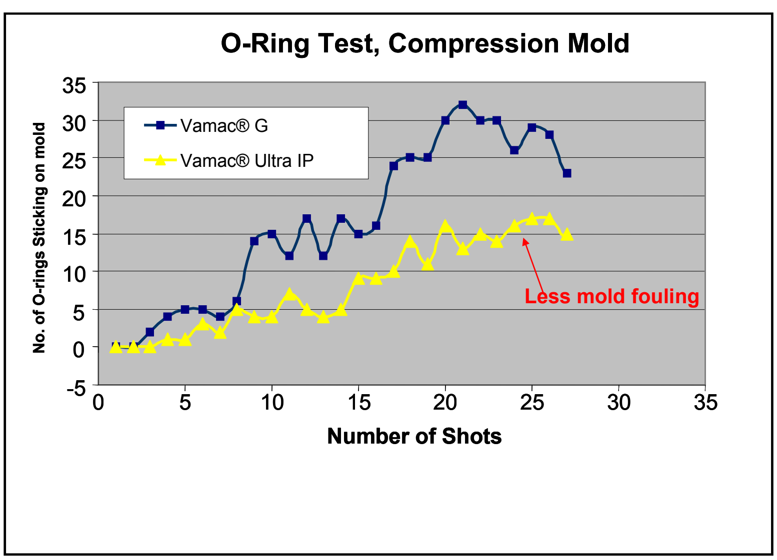
Vamac® Ultra IP offers significant improvements in molding processes. Higher viscosity helps reduce blistering problems in compression molding. In addition to having significantly higher compound Mooney viscosity than Vamac® G, Vamac® Ultra IP also offers better compound flow in injection molding operations as shown in attached Capillary Rheometer plot for 80 Shore A compounds in Chart 1.

Chart 1 – Capillary Rheometer Results



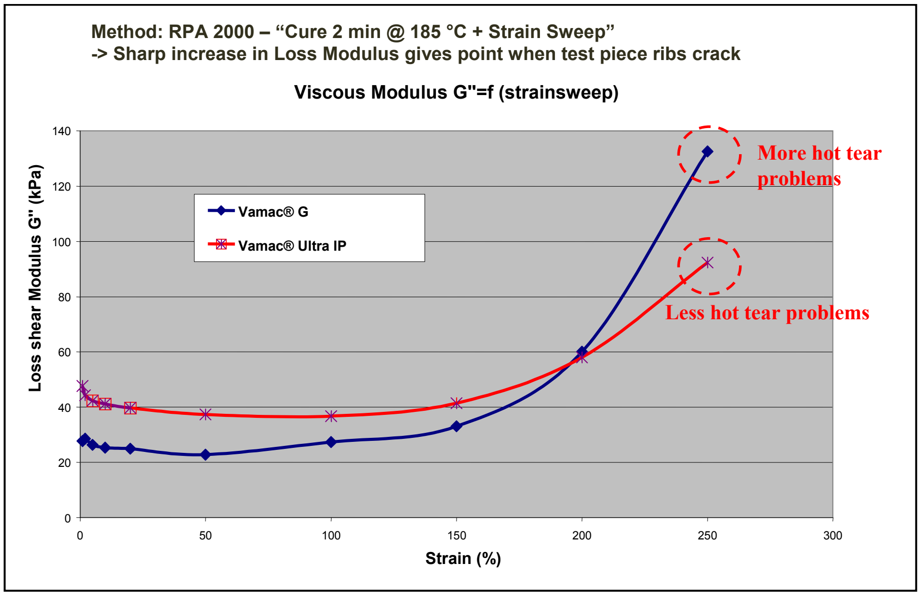
Vamc® Ultra IP has lower mold fouling. An internal test is used to compare the mold-sticking characteristics of small O-rings that are compression molded. The test employs a mold with two sets of 60 cavities. After each vulcanization cycle, the O-rings are blown out by pressurized air of defined pressure under conditions of defined distance between air valve and mold. The number of O-rings sticking to the mold after each cycle is reported. Chart 2 shows a comparison between Vamac® Ultra IP and Vamac® G with identical compound formulations using this test procedure. The number of O-rings sticking to the mold is clearly decreased for Vamac® Ultra IP.

Chart 2 – O-Ring Test Results



Another benefit of using DuPont™ Vamac® Ultra IP in molding processes is improved hot tear strength and higher elongation-at-break at demolding temperatures. Higher elongation-at-break and hot tear properties may be demonstrated by use of RPA 2000 equipment using a cure time of 2 minutes at 185 °C, followed by a strain-sweep, as shown in Chart 3. The point at which the loss modulus shows a sharp increase is the point when ribs on the test specimen crack. This loss modulus rise occurs at lower stress levels and higher elongation for Vamac® Ultra IP compared to Vamac® G indicating better demolding characteristics. This benefit can be further improved by using a lower curative level. Higher hot tear also results in easier tear trim.

Chart 3 – RPA Test



Compounding and Physical Properties

Vamac® Ultra IP is formulated in a manner similar to that used for other Vamac® terpolymers. The difference in polymer composition allows use of lower levels of diamine curative. This helps to reduce compound cost. The reduction in curative level does not adversely affect MH or compression set. MH values are equivalent to or slightly higher than those observed for Vamac® G. In addition, lower curative levels help reduce cure time and to increase maximum cure speed.

The higher viscosity of Vamac® Ultra IP also allows addition of higher levels of plasticizer and result in better low temperature flexibility while maintaining good processing behavior.

Table 3 shows physical property data for two compounds of Vamac® Ultra IP and a standard 78 Shore A hardness compound of Vamac® G. One compound of Vamac® Ultra IP includes 1.5 phr of the curative Diak™ No. 1 whereas the second compound uses a lower curative level of 1.2 phr of Diak™ No. 1. The compound Mooney Viscosity and MH of are higher for Vamac® Ultra IP compared to Vamac® G.

Table 3. Formulations and Rheology

Compound No.	Vamac® G	Vamac® Ultra IP	Vamac® Ultra IP Low Diak™ No. 1
Vamac® G	100		
Vamac® Ultra IP		100	100
Naugard® 445	2	2	2
Armeen® 18D	0.5	0.5	0.5
Stearic acid	1.5	1.5	1.5
Vanfre® VAM	1	1	1
Spheron® SO N550	65	65	65
Rhenosin® W 759	10	10	10
Diak™ No.1	1.5	1.5	1.2
Vulcofac® ACT 55	2	2	2
Mooney ML(1+4) 100 °C (MU)	41.8	67.4	66.4
Mooney Scorch 121 °C T5 (min)	7.5	9.2	8.6
MDR 180 °C / 0.5deg / 12 min			
ML (dNm)	0.51	0.82	0.84
MH (dNm)	13.2	16.7	14.6
Ts2 (min)	0.88	0.81	0.81
tc10 (min)	0.7	0.73	0.68
tc50 (min)	2.2	2.4	2.1
tc90 (min)	6.8	7.1	6.4
Peak rate (dNm/min)	4.1	4.7	4.7

Table 4 reports some original physical properties and compressions sets of three compounds, as well as low temperature flexibility (as indicated by T_g) and abrasion test results.

Physical properties such as tensile and tear strength or elongation-at-break are significantly better for Vamac® Ultra IP in comparison to Vamac® G. Standard compression set also shows advantages for Vamac® Ultra IP even at a lower curative level. However, if compression set according to Volkswagen specification PV3307 is to be optimized, higher curative levels should be considered.

Low temperature properties are substantially identical for Vamac® G and Vamac® Ultra IP. Sandpaper abrasion and resilience values indicate significant differences between the two polymers. Good abrasion results may be of interest for shaft seal or ball bearing seal applications.

Table 4. Physical Properties

Compound No.	Vamac® G	Vamac® Ultra IP	Vamac® Ultra IP Low Diak™ No. 1
Cure 5 min at 180 °C / Post-Cure (4 h at 175 °C)			
Hardness (Sh. A)	78	79	78
M 100% (MPa)	8.5	7.1	6.3
Tensile Strength (MPa)	16.3	18.1	17.6
Elongation (%)	195	261	290
Delft Tear Fmax (N/mm)	21.8	23.8	26.7
Compression Set (70 h / 150 °C), 12 mm molded disks (%)	21	15	12
Compression Set (168 h / 150 °C), 12 mm molded disks (%)	23	17	19
Compression Set (70 h / 175 °C), 12 mm molded disks (%)	29	25	25
Compression Set (1008h / 175 °C), 12 mm molded disks (%)	60	54	57
Compression Set (70 h / 190 °C), 12 mm molded disks (%)	34	30	31
VW CSet PV3307 (94 h / 23 °C) (%)	29	30	42
VW CSet PV3307 (94 h / 150 °C) (%)	46	49	65
Tg by DSC (°C)	–36	–37	–37
Sandpaper Abrasion (mm^3)	164	132	133
Resiliency Test (%)	38	44.4	43.6

Heat Ageing

For the following heat and oil ageing tests, compounds that do not include plasticizer were used to eliminate any effect evaporation of volatile ingredients at high temperatures. These compounds are usually not representative of Vamac® applications in the range of 150 °C. Ether-ester plasticizers or polymeric esters are used in amounts of between 10 to 20 phr for sealing applications for a good balance of low temperature flexibility and oil swell. However, for extremely high temperature requirements, such as for air ducts used in turbo charged engines, very low plasticizer levels are recommended.

Data for three Shore A 55 to 60 compounds are shown in *Table 5*. Two of the formulations are based on Vamac® Ultra IP at two different curative levels and the third is based on Vamac® G.

Vamac® Ultra IP compounds exhibit higher initial tensile strength and elongation values compared to Vamac® G compounds. After ageing in dry heat (air), higher retention of physical properties are observed and a lower percentage change in properties is obtained for Vamac® Ultra IP as shown in *Table 5*. Thus, longer functionality of parts made from Vamac® Ultra IP compounds can be expected.

Table 5 – Results on Heat Ageing of Three 60 Shore A Compounds

Compound No.	**Vamac® G**	**Vamac® Ultra IP**	**Vamac® Ultra IP Low Diak™ No. 1**
Vamac® G	100		
Vamac® Ultra IP		100	100
Naugard® 445	2	2	2
Armeen® 18D	0.5	0.5	0.5
Stearic acid	1.5	1.5	1.5
Vanfre® VAM	1	1.5	1.5
Spheron® SO N550	30	30	30
Diak™ No.1	1.5	1.5	1.17
Ekaland DOTG	4	4	4
Original properties			
Hardness (Sh. A, 1 sec.), 6 mm plied	55	59	57
M 100% (MPa)	2.6	2.6	2.3
Tensile Strength (MPa)	13.2	15.5	16.8
Elongation (%)	326	377	472
Tear ISO-34-1, Method A (N/mm)	5.7	6.3	7.4
Compression set (168 h / 150 °C), 6mm plied disks	27	26	28
Compression set (70 h / 175 °C), 12mm molded disks	17	17	17
Heat ageing 504 h at 175 °C			
Hardness (Sh. A, 1 sec.), 6 mm plied	60	59	57
Hardness change (pts)	5	0	0
M 100% (MPa)	3.4	3.0	2.4
M 100% Change (%)	32	13	5
Tensile Strength (MPa)	6.5	12.2	10.1
Tensile Strength Change (%)	-51	-21	-40
Elongation (%)	151	302	309
Elongation Change (%)	-54	-20	-35
Heat ageing 168 h at 190 °C			
Hardness (Sh. A, 1 sec.), 6 mm plied	59	58	54
Hardness change (pts)	4	–1	–3
M 100% (MPa)	3.3	2.8	2.5
M 100% Change (%)	29	6	11
Tensile Strength (MPa)	9.2	12.2	11.1
Tensile Strength Change (%)	–30	–21	–34
Elongation (%)	195	286	309
Elongation Change (%)	–40	–24	–35

Ageing in Automotive Fluids

The compounds shown in *Table 5* were tested for fluid ageing in Lubrizol® OS206304 and Dexron® VI, two fluids widely used in the automotive industry as reference fluids for engine oils and automatic transmissions. Results are shown in *Table 6*. All compounds exhibited similar volume swell. Overall properties of DuPont™ Vamac® Ultra IP are superior to those of Vamac® G after ageing and in particular, the compound at low curative level exhibits the best properties. In applications where Vamac® is fully immersed in lubricants, such as oil seal or oil hose applications, addition of plasticizers at levels in the range of between 10 and 20 phr is recommended. Thus, a reasonable volume swell of 5 to 10% can easily be achieved. If needed, Vamac® Ultra IP can be blended at any ratio with Vamac® GLS, a standard AEM grade that is designed for lower volume swell.

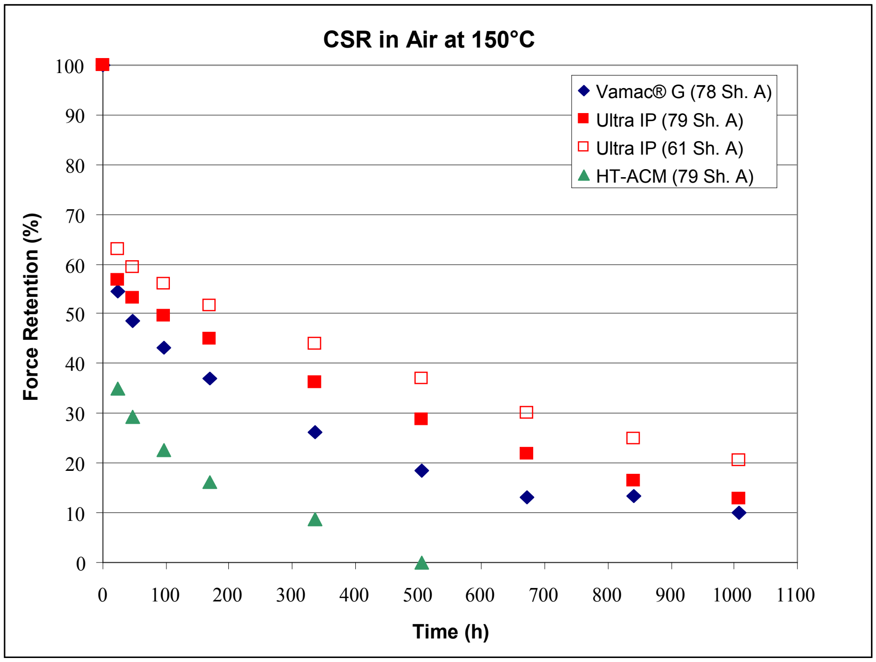
Table 6. Results after ageing in Automotive Lubricants

	Vamac® G	Vamac® Ultra IP	Vamac® Ultra IP Low Diak™ No. 1
Ageing in Lubrizol® OS 206304, 5W40 (504 h at 160 °C)			
Hardness (Sh. A, 1 sec.), 6 mm plied	49	51	49
Hardness Change (pts)	–6	–8	–8
M 100% (MPa)	2.5	2.6	2.1
Tensile Strength (MPa)	8.9	11.1	11.6
Tensile Strength Change (%)	–33	–34	–26
Elongation (%)	242	280	338
Elongation Change (%)	–26	–30	–24
Volume Change (%)	23	23	24
Weight Change (%)	17	17	18
Ageing in Petro Canada Dexron® VI (168 h at 150 °C)			
Hardness (Sh. A, 1 sec.), 6 mm plied	46	48	45
Hardness Change (pts)	–5	–6	–9
M 100% (MPa)	2.2	2.2	1.6
Tensile Strength (MPa)	11.9	12.2	14.4
Tensile Strength Change (%)	–7	–25	–13
Elongation (%)	265	293	391
Elongation Change (%)	–39	-43	–29
Volume Change (%)	21	22	22
Weight Change (%)	15	16	16

Compressive Stress Relaxation (CSR)

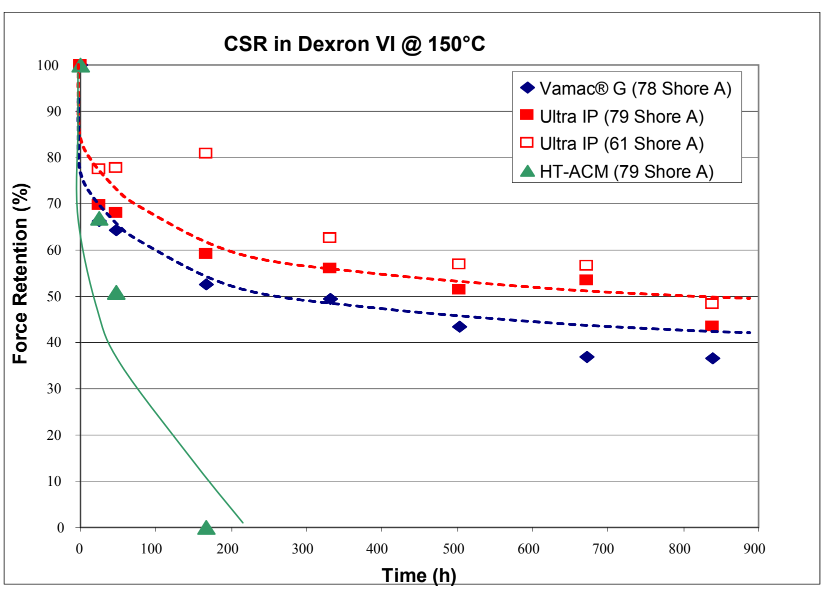
The improved ageing in dry heat and oil of Vamac® Ultra IP also results in better force retention in CSR tests in air at elevated temperature. Chart 4 shows a comparison between Vamac® and HT-ACM compounds, all diamine cured, in the range of 60 to 80 Shore A. Tests were conducted according to ASTM D6147, using a Shawbury-Wallace equipment combined with Dyneon fixtures and with cylindrical specimen (6 mm high, 13 mm diameter) at 150 °C. Vamac® clearly shows higher retention of sealing force.

Chart 4 – Compressive Stress Relaxation in Air



Force retention of Vamac® Ultra IP and HT-ACM based compounds in automatic transmission fluid Dexron® VI, is shown in Chart 5. Both Vamac® G and Ultra IP compounds out-perform HT-ACM compounds. Shawbury Wallace equipment & Shawbury Wallace fixtures were used according to ISO 3384 Method B. In the same test, a compound based on HT-ACM lost all of its sealing force after only about 200 hours at 150 °C.

Chart 5 – Compressive Stress Relaxation in ATF Dexron VI



Starting Point Formulations

Table 7 provides several starting point formulations based on DuPont™ Vamac® Ultra IP for a hardness range between 60 and 85 Shore A.

Table 7. DuPont™ Vamac® Ultra IP Compounds Starting Formulation

Compound No.	1	2	3	4	5	6
Vamac® Ultra IP	100	100	100	100	100	100
Naugard® 445	2	2	2	2	2	2
Armeen® 18D	0.5	0.5	0.5	0.5	0.5	0.5
Stearic acid	1.5	1.5	1.5	1.5	1.5	1.5
Vanfre® VAM	1	1	1	1	1	1
Spheron® SO N550	40	52	72	65	80	
Spheron® 4000						50
Rhenosin W 759	10	10	15	10	10	5
Diak™ No.1	1.2	1.2	1.2	1.2	1.2	1.2
Vulcofac® ACT 55	2	2	2	2	2	2
Press-Cure 5 min. at 180 °C, Post-Cure (4 h at 175 °C)						
Properties at Room temperature						
Hardness (Sh. A)	61	70	79	78	84	61
M 50 % (MPa)	1.3	2.0	3.3	3.0	4.6	1.3
M 100% (MPa)	2.8	4.5	6.8	6.3	9.7	2.5
Tensile Strength (Mpa)	17.7	17.9	16.5	17.6	17.2	19.9
Elongation (%)	409	345	276	290	218	401
Delft Tear Fmax (N/mm)	18.8	24.3	23.9	26.7	29.2	18.7
Comp. Set (70 h / 150 °C), 12 mm molded slabs (%)	12	13	14	15	18	14
Comp. Set (70 h / 190 °C), 12 mm molded slabs (%)	28	29	33	31	32	27
Tg by DSC (°C)	-37	-37	-40	-37	-38	35
Sandpaper Abrasion (mm³)	147	132	135	133	144	167

List of Compound Ingredients

Material	Chemical Composition	Supplier
Polymer		
Vamac® G	Ethylene Acrylic Elastomer	DuPont
Vamac® Ultra IP	Ethylene Acrylic Elastomer	
Release Aids		
Armeen® 18D	Octadecyl Amine	Akzo Nobel
Vanfre® VAM	Complex Organic Phosphate Ester	R.T. Vanderbilt
Stearic Acid		
Anti-Oxidant		
Naugard® 445	Diphenyl Amine	Chemtura
Plasticizer		
Rhenosin® W 759	Mixed Ether/Ester Plasticizer	Rhein Chemie
Filler		
Spheron® SO N550	Carbon Black	Cabot
Spheron® 4000	Carbon Black	Cabot
Curatives		
Diak™ No. 1	Hexamethylene Diamine Carbamate	DuPont
Accelerators		
Vulcofac® ACT 55	Proprietary Compound	Safic-Alcan
Ekaland® DOTG	Di-ortho-tolyl Guanidine	MLPC International
Test Fluids		
Dextron® VI	Transmission Fluid	Petro Canada
Lubrizol® OS206304 5W40	5W40 Reference Engine Oil	Lubrizol

Test Methods

Test	Method
Rheology	
Mooney Viscosity	D1646
Mooney Scorch	D1646
MDR	D5289
Physical Properties	
Hardness	D2240
Tensile Strength, Elongation, Modulus	D412
Compression Set	D395
Compression Set	Volkswagen PV3307
Compressive Stress Relaxation (CSR)	D6147 and ISO 3384
Aging in Air Oven	D573
Fluid Aging	D471
Tg by DSC	D7426
Resiliency Test	ISO 4662
Sandpaper Abrasion Test	ISO 4649
Delft Tear	ISO 34-2

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