

TegMeR[®] 812-New Ester for Acrylic and HNBR Elastomers

Acrylic elastomers are used in many demanding applications that require high heat resistance over extended periods of time. These polymers are typically plasticized at minimum levels with efficient, low-temperature esters. Normally, they do not use high levels because these polymers have moderately low modulus values.

The standard esters used are very efficient in lowering hardness and providing low temperature but are very volatile at the high temperature ranges in which they are exposed. This study compares two esters, TegMeR[®] 812 and Paraplex[®] A-8000, which provide improved low-temperature performance after long-term high heat aging. The new esters were compared to the standard esters in two different acrylic elastomers. High- and low-temperature testing was conducted with the emphasis on the retention of physical properties after heat aging.

Acrylic elastomers are used for applications requiring -135°C to -150°C continuous service with intermittent exposure to -190°C. Acrylic elastomers have a saturated backbone with pendant groups attached through a carbonyl of, potentially, ethyl, butyl, ethylene and oxyethyl. In addition, acrylic elastomers are polar and thus many ester plasticizers are compatible. However, because of the high temperature post-cure required by most acrylics and the higher maximum temperature of application, only a few plasticizers show a reasonable degree of utility.

In this study, we focused on two new esters in two types of acrylic elastomers, Vamac G and AR HyTemp AR212HR. Polymeric esters are well known for their permanence in these elastomers but suffer when trying to improve low-temperature performance. TegMeR[®] 812 and Paraplex[®] A-8000 are lower molecular weight esters that have high heat resistance but provide much needed low-temperature properties after high heat aging. In addition, these esters can be used in Hydrogenated Nitrile Butadiene Rubber (HNBR). These are some data showing similar benefits of these esters for low-temperature improvement.

Formulation: **Vamac G** – 100.00, **N-550** – 68.00*, **Naugard 445** – 2.00, **Stearic Acid** – 1.50, **Armeen 18D** – 0.50, **Vanfre VAM** – 1.00, **Plasticizer as noted** – 20.00
 *N-550 Control Compound – 50.00

Mill Addition: **Vulcofac ACT 55** – 1.80, **Diak 1** – 1.50

Recipe Variable	Control	TP-759	Plasthall® DBEEEG	TegMeR® 812	Paraplex® A-8000
Mooney Viscosity at 121°C (250°F)					
Minimum Viscosity <u>Oscillating Disc Rheometer at</u> <u>177°C (350°F)</u>	36.0	22.0	22.0	22.7	25.1
M _L	3.3	6.5	1.6	1.6	1.8
M _H	63.3	46.7	38.7	42.7	35.9
t' _c (90), min	6.0	6.9	6.3	8.4	6.6
1.25*t' _c (90), min	7.5	8.6	7.8	10.5	8.2
Sheets cured for 5 min at 175°C (347°F) Shallows in Compound # 5 sheets Sheets post cured for 4 h at 175°C (347°F)					
Weight Loss after Post Cure, %	-1.4	-3.1	-1.6	-1.4	-1.8
Roll Spew					
Temperature at -18°C, -3°C, 25°C 24 h through 96 h	None	None	None	None	None
Original Physical Properties					
Tensile, Ultimate, psi	2580	2065	2080	2255	2270
Elongation at Break, %	235	205	205	205	230
Hardness Duro A, pts.	75	76	73	76	75
Specific Gravity	1.209	1.232	1.229	1.228	1.232
T _g by DSC, °C	-31	-44	-46	-44	-39
Compression Set, % 22 h at 100°C	22	32	33	29	37
Recipe Variable	Control	TP-759®	Plasthall® DBEEEG	TegMeR® 812	Paraplex® A-8000
Low Temperature Impact - Brittleness					
Brittle Point, as molded, all pass, °C	-32	-42	-42	-42	-36
after Air Oven Aging, 1 week at 175°C (347°F), all pass, °C	-30	-31	-29	-38	-36
Low Temperature Torsion - Gehman As molded, Relative Modulus					

T ₁₀ , °C	-36	-41	-41	-42	-38
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After Air Oven Aging, 1 w at 175°C (347°F), all pass, °C

Relative Modulus

T ₁₀ , °C	-29	-34	-34	-36	-32
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Air Oven Aging, 1 w at 175°C (347°F)

Tensile Change, %	-6	-8	-8	-14	-16
Elongation Change, %	-11	-5	2	-5	-2
Hardness Change, pts.	1	5	8	0	0
Weight Change, %	-1.6	-5.4	-6.2	-2.5	-1.8
T _g by DSC, °C	-30	-38	-37	-41	-38

Fluid Resistance

IRM 901 Oil, 1 w at 150°C (302°F)

Hardness Change, pts.	-7	1	3	1	-1
Volume Change, %	9.0	0.2	-0.8	-0.8	3.1
Weight Change, %	6.2	-1.2	-2.5	-1.9	1.4

IRM 903 Oil, 1 w at 150°C (302°F)

Hardness Change, pts.	-20	-24	-19	-22	-25
Volume Change, %	57	50	47	46	52
Weight Change, %	44	37	35	33	38

ASTM SF 105, 1 w at 150°C (302°F)

Hardness Change, pts.	-14	-11	-7	-10	-11
Volume Change, %	27	17	15	15	19
Weight Change, %	19	11	9.2	9.3	13

Transmission Fluid, 1 w at 150°C (302°F)

Hardness Change, pts.	63	70	70	70	68
Volume Change, %	21	11	9	9	13
Weight Change, %	15	6	5	5	8

Formulation: HyTemp AR212HR – 100.00, N-550 – 50.00, Stearic Acid – 1.00, Vanox CDPA – 2.00, Armeen 18D – 0.50, Vanfre VAM – 0.50, Plasticizer as noted – 10.00

Mill Addition: Rheogran XLA60 – 2.00, HMDC (Diak 1) – 0.60

Recipe Variable	Control	TegMeR® 812	Paraplex® A-8000	Plasthall® DBEEEG	Plasthall®TOTM	Paraplex G-50
Mooney Viscosity at 125°C (257°F)						
Minimum Viscosity	61.7	42.0	45.4	42.4	43.4	44.9
t35, min	8.7	9.3	9.3	9.7	10.2	9.5
Oscillating Disc Rheometer at 190°C (374°F)						
M _L		9.0	6.0	6.5	5.9	6.3
M _H		41.1	31.3	32	30.8	30.9
t'c(90), min		9.7	9.5	9.5	9.3	9.1
1.25*t'c(90), min		12.1	11.8	11.9	11.7	11.4
Sheets cured for 4 min at 190°C (374°F) Sheets post cured for 4 h at 177°C (351°F)						
Weight Loss after Post Cure, %		-1.5	-1.7	-1.9	-1.8	-1.7
Roll Spew						
Temperature at -18°C, -3°C, 25°C						
24 h through 96 h		None	None	None	None	None
Original Physical Properties						
Tensile, Ultimate, psi		1525	1265	1280	1235	1300
Elongation at Break, %		205	235	245	245	240
Hardness Duro A, pts.		62	51	52	53	52
Specific Gravity		1.259	1.243	1.247	1.244	1.239
Tg by DSC, °C		-27.3	-38.7	-34.7	-39.3	-34.1
Compression Set , 504 h at 175°C, %		56	54	71	62	57

Recipe Variable	Control	TegMeR® 812	Paraplex® A-8000	Plasthall® DBEEEG	Plasthall® TOTM	Paraplex® G-50
Low Temperature Impact - Brittleness						
Brittle Point, as molded, all pass, °C	-22	-31	-28	-34	-28	-25
Low Temperature Torsion - Gehman						
As Molded, Relative Modulus						
T10, °C	-30	-41	-37	-42	-37	-33
Air Oven Aging, 7 d at 190°C (374°F)						
Stress at 100% Elongation, psi	615	370	360	430	480	470
Tensile, Ultimate, psi	1225	960	1030	1035	1110	1135
Tensile Change, %	-20	-24	-20	-16	-15	-11
Elongation Change, %	5	2	-6	2	6	-12
Hardness Duro A, pts	68	58	57	68	66	59
Hardness Change, pts.	6	7	5	15	14	7
Weight Change, %	-3.1	-4.8	-3.6	-8.3	-8.6	-3.7
Tg by DSC, °C	-25.9	-31.8	-30.8	-26.6	-26.1	-30.2
Air Oven Aging, 1000 h at 175°C (347°F)						
Stress at 100% Elongation, psi	800	840	640	645	700	795
Tensile, Ultimate, psi	955	940	980	925	965	1050
Tensile Change, %	-37	-26	-23	-25	-26	-17
Elongation Change, %	-44	-51	-37	-39	-38	-49
Hardness Duro A, pts	77	73	67	76	74	71
Hardness Change, pts.	15	22	15	23	22	19
Weight Change, %	-5.4	-8.8	-6.8	-11	-11	-6.9

Formulation: Therban A3907 – 100.0, N-990 – 40.00, Naugard 445 – 1.00, PE-AC-617 – 1.00, Kadox 911C – 3.00, Maglite DE – 3.00, ZMTI – 0.53, TAIC – 1.50, Plasticizer as noted – 10.00

Mill Addition: Trigonox 101-45B-PD – 8.00

Recipe Variable	TegMeR® 812	Plasthall® TOTM	Control
<u>Mooney Viscosity at 125°C (257°F)</u>			
Minimum Viscosity	32.8	32.1	47.5
t35, min	> 60.0	> 60.0	> 60.0
<u>Oscillating Disc Rheometer at 170 °C (338 °F)</u>			
M _L	6.0	6.5	9.8
M _H	36.7	48.1	71.7
t'c(90), min	28.3	34.8	27.9
1.25*t'c(90), min	35.3	43.5	34.8
<u>Roll Spew</u>			
Temperature at -18°C, -3°C, 25°C			
24 h through 96 h	None	None	None
<u>Original Physical Properties</u>			
Tensile, Ultimate, psi	2365	1890	2000
Elongation at Break, %	440	315	265
Hardness Duro A, pts.	58	59	63
Specific Gravity	1.148	1.144	1.157
Tg by DSC, °C	-26	-29	-24
<u>Compression Set</u> , % 70 h at 135°C	37	30	28
<u>Low Temperature Impact - Brittleness</u>			
Brittle Point, as molded, all pass, °C	-46	-49	-43
<u>Low Temperature Torsion - Gehman</u>			
As Molded, Relative Modulus			
T10, °C	-35	-32	-27
Apparent Modulus of Rigidity	112.8	133.8	154.1

Recipe Variable	TegMeR® 812	Plasthall® TOTM	Control
<u>Air Oven Aging, 7 d at 150°C (302°F)</u>			
Tensile Change, %	11	22	21
Elongation Change, %	-11	-8	4
Hardness Duro A, pts	65	67	69
Hardness Change, pts.	7	8	6
Weight Change, %	-3.2	-4.3	-2.7
<u>Air Oven Aging, 14 d at 150°C (302°F)</u>			
Tensile Change, %	4	32	32
Elongation Change, %	-23	-11	-8
Hardness Duro A, pts	66	68	71
Hardness Change, pts.	8	9	8
Weight Change, %	-3.7	-6.0	-2.9
Tg by DSC, °C	-28	-23	-23
<u>Fluid Resistance</u>			
<u>IRM 901 Oil, 168 h at 135°C (275°F)</u>			
Tensile, Ultimate, psi	2255	2150	2330
Tensile Change, %	-5	14	17
Elongation Change, %	-11	-2	4
Hardness Change, pts.	4	4	2
Volume Change, %	-4.9	-7.1	-0.8
Weight Change, %	-4.4	-6.0	-0.7
<u>IRM 903 Oil, 168 h at 135°C (275°F)</u>			
Tensile Change, %	-33	-15	-13
Elongation Change, %	-31	-16	-11
Hardness Duro A, pts	57	58	60
Hardness Change, pts.	-1	-1	-3
Volume Change, %	8.1	6.0	13
Weight Change, %	6.6	5.1	11
<u>ASTM Fuel C Dry Out – 22 h at 70°C</u>			
Hardness Duro A, pts	61	61	64
Hardness Change, pts.	3	2	1
Volume Change, %	-4.8	-6.4	0.4
Weight Change, %	-5.1	-6.2	-0.5