

DuPont™ Vamac®

Compounding Processing Guide — Vamac® Dipolymers

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DuPont™ Vamac® ethylene acrylic elastomers are used as the base polymer for a thermoset elastomer compound. Vamac® elastomers or polymers fall into two general categories: (1) terpolymers of ethylene, methyl acrylate and an acidic cure site monomer and (2) dipolymers of ethylene and methyl acrylate. Compounds made from Vamac® terpolymers are typically cured with a diamine and require a post cure to achieve full properties. (See the “Safe Handling and Processing of Vamac® and Vamac® Compounds Guide”.) Optimum Vamac® performance is achieved with post cured terpolymers. Compounds made from Vamac® dipolymers use a peroxide curing system and can develop good properties only after a press cure step. The need for a post cure can be eliminated or significantly reduced with the use of the peroxide cure system used with Vamac® dipolymers. The various Vamac® grades are listed below:

Product Grade	ML(1+4) at 100 °C	Key Feature
Terpolymers		
DuPont™ Vamac® G	16.5	Base grade
DuPont™ Vamac® GLS	18.5	Low oil swell
DuPont™ Vamac® HVG	26.0	High viscosity
DuPont™ Vamac® GXF	18.5	High-temperature performance
Dipolymers		
DuPont™ Vamac® DP	22.0	No post cure

Besides the elimination of the post curing step, other advantages of compounds made from Vamac® dipolymers vs. terpolymers are better bin stability, longer scorch times, less sensitivity to oil additives such as amine stabilizers and an ability to use metal oxides and hydroxides in compound formulations.

Handling Precautions

Vamac® elastomers contain small amounts of residual methyl acrylate monomer and adequate ventilation should be provided during mixing and processing to prevent worker exposure to methyl acrylate monomer. Additional information is available in the Material Safety Data Sheet (MSDS) and in the “Safe Handling and Processing of Vamac® and Vamac® Compounds Guide” available from www.dupontelastomers.com/vamac.

Compounds of Vamac® Dipolymers

Vamac® DP is the only dipolymer grade that is commercially available at this time. Compounds made from Vamac® DP can be used in applications with continuous maximum temperatures of 165 °C (329 °F) and they can survive brief temperatures of around 200 °C. The fluid resistance of a cured compound depends on the carbon black and plasticizer level. Typical values for volume swell in IRM 903 fluid after aging for 168 hours at 150 °C (302 °F) are 50–60% for Vamac® DP. The glass transition temperature, T_g, as measured by DSC will range from –30 to –35 °C for a cured Vamac® dipolymer compound. A cured dipolymer compound can often meet the requirements of an end use bench test at –40 °C.



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The properties of DuPont™ Vamac® dipolymer compounds are well suited for a wide range of automotive applications, such as powertrain seals and gaskets, rocker cover seals, transmission oil coolant hoses, power steering hoses, turbo-charger hoses, crankcase ventilating tubes, coverings for fuel and coolant hoses, O-rings, grommets, spark plug boots and crankshaft dampers.

All Vamac® based compounds do very well in compressive stress relaxation testing (CSR). This indicates that compounds can perform well in sealant applications in various fluids for long periods of time.

Vamac® dipolymers are halogen-free polymers and do not decompose to give off corrosive gases when exposed to flame. They can be used for flame retardant, low-smoke, non-halogen wire and cable jackets and in non-halogen, low smoke flooring.

Compound Formulation Guidelines

The principles of compounding Vamac® dipolymers are similar to conventional technology in that curatives, filters, antidegradants, plasticizers and process aids are all used. As with other high-performance, heat-resistant elastomers, the choice of potential additives tends to be smaller and selection more specific. Equal attention must be given to avoiding ingredients that might give detrimental effects. This is especially true with peroxide cured Vamac® dipolymers.

Grade Selection: The preferred grade for peroxide cured compounds is Vamac® DP. It is possible to use Vamac® GLS in a peroxide cured compound for improved fluid resistance but the tradeoff is that the low temperature properties will be reduced. Also the acidic cure site in Vamac® GLS lowers the effectiveness of the peroxide cure system. It is also possible to use blends of Vamac® DP and Vamac® GLS to achieve a balance of improved fluid resistance while sacrificing some low temperature performance.

Antidegradants: Vamac® dipolymers require the addition of one part semi-staining diphenylamine antioxidant in black formulations. Only one part is used as the antioxidant interferes with the peroxide cure. Peroxides cure by free radical polymerization and anti-oxidants work as free radical traps. If improved heat aging performance is needed, then the level of diphenylamine antioxidant can be increased two parts, with a slight increase in the peroxide level to minimize the effect on cure state and compression set. Two parts nonstaining phenolic antioxidant, such as Santowhite powder, can be used in nonblack compounds.

Contact with zinc (e.g. hose couplings) catalyzes heat aging in Vamac®. Hydrazine antioxidants are effective in preventing the zinc-catalyzed attack. One part of ADK STAB CDA-6 or two parts of inhibitor OABH are suggested. No antiozonants are needed in any Vamac® formulation.

Fillers: Carbon black is the preferred filler for Vamac®. It has no effect on its heat aging properties, and is beneficial to compression set and flex resistance. The majority of Vamac® compounds contain N762 or N774 SRF blacks, or N550 FEF black. All give good reinforcement with or without plasticizer. High-structure blacks such as N-683 or Spheron® 5000 are also suitable. Highly reinforcing blacks such as ISAF tend to respond best without accompanying plasticizer, probably due to the limited shear dispersion available from the polymer during mixing. Low-structure blacks can also be difficult to disperse. MT black may be used as a filler and extender.

Mineral fillers should be selected with care. For high physical properties, tensile and tear, a fumed silica is most effective and has the least effect on heat aging properties. Fumed silica significantly increases compound viscosity and increases compression set, and therefore is normally incorporated at moderate levels of up to 25 parts. Surface-treated talc also is reinforcing, with much less effect on compression set compared with fumed silica.

Calcium carbonate may be used at quite high levels without much effect on heat aging; the best reinforcement is obtained from the precipitated form. Barium sulfate also is suitable as a high-level filler.

Aluminum hydroxide is used frequently as a flame-retarding filler in Vamac®. Zinc-containing fillers are harmful to Vamac® types.

Plasticizers: Use of plasticizers are limited with DuPont™ Vamac® dipolymers as they retard the peroxide cure. The level of plasticizers for Vamac® dipolymers should be limited to 5 to 10 parts. Below are the recommended plasticizers for Vamac® dipolymers and suitable temperature ranges for each.

Linear trimellitate	–45 to 155 °C trioctyl trimellitate
Low volatility alkyl trimellitate	–40 to 170 °C Bisoflex T810T, Plasthall® 810TM
Tri isononyl trimellitate	–40 to 170 °C ADK CizerC-9N
Polyester	–30 to 170 °C Plasthall® 670

Process Aids: Process aids are recommended with Vamac® dipolymers to improve part release and mold fouling. Similar to plasticizers, they retard the peroxide cure and must be used in low levels. For Vamac® dipolymers, the recommended release package is half a part of Armeen® 18D, one part of stearic acid and half to one part of Vanfre® VAM. Higher levels of stearic acid or Vanfre® VAM can be used but impact on final compound properties must be considered. Other release aids such as Technical Processing TE58A or TE88XL can also be used.

Peroxide Cure Systems: Vamac® dipolymer compounds are cured using a peroxide (i.e., Vul-Cup®, Peradox®, Di-Cup®, etc) in combination with a co-agent. Selection of the system depends on desired compound properties and processing conditions. There are two main peroxides used in curing Vamac® dipolymers. A-A-bis(t-butyl peroxy) diisopropylbenzene is the most common peroxide used giving a good balance of scorch safety, tensile properties and compression set. The other peroxide used is dicumyl peroxide, providing faster curing and better compression set but can be scorchy or give poor tear properties. Compound properties will also be impacted by the co-agent selected and the levels of peroxide and co-agent.

There are a variety of co-agents that all provide differences in curing and final properties. The main ones used for Vamac® dipolymers are listed below.

HVA-2	Fast Curing/Optimum Compression Set
TAIC	Slower Cure/Higher Elongation
TAC	Slower Cure/Higher Elongation/Poorer Heat Aging
Ricon® 152	Slower Cure/Higher Elongation
Sartomer® 350	Slower Cure/Higher Elongation/Better Compression Set

Compound and Vulcanizate Properties

Compounds of Vamac® are formulated to meet specific end-use performance requirements. The compounds have to have a balance of good processability and good final cured properties. Several compounds based on Vamac® DP showing the effect of various peroxide/co-agent systems are shown in *Table 1*. The compounds were mixed in a small laboratory internal mixer and sheeted off on a roll mill.

Table 1 — DuPont™ Vamac® Dipolymer Compound Performance

	1	2	3	4
Vamac® DP	100	100	100	100
Naugard® 445	1	1	1	1
Stearic Acid	0.5	0.5	0.5	0.5
Vanfre® VAM	0.5	0.5	0.5	0.5
Armeen® 18D	0.5	0.5	0.5	0.5
Carbon Black FEF N-550	55	55	55	55
VUL-CUP® 40KE	5			
DI-CUP® 40C		5	9	9
HVA-2	2	2		
Ricon® 152			3	
Sartomer® 350				3
TOTAL PHR	164.5	164.5	169.5	169.5
RHEOLOGY PROPERTIES				
Mooney Viscosity, ML (1+4) at 100 °C	43.2	44.0	39.6	36.9
Mooney Scorch at 121 °C				
Minimum, mu	14.2	14.6	13.4	12.5
t(3), m.m.	0	0	0	0
t(10), m.m.	0	0	0	0
MDR at 177 °C, 0.5° arc, 20 mins				
ML, N-m	0.7	0.7	0.7	0.7
MH, N-m	15.7	11.6	17.4	13.9
ts(2), m.m	0.8	0.8	0.6	1.0
t(50), m.m	1.6	1.2	1.5	1.7
tc(90), m.m	5.2	3.1	4.0	3.7
Cure Rate Slope	2.2	2.8	3.3	3.0
CURED PROPERTIES				
Press Cure 10 mins at 177 °C				
Tensile, Tear and Hardness — Original at 23 °C				
Hardness, Durometer A, pts	69	67	69	65
100% Modulus, MPa (psi)	7.9	5.2	6.8	5.6
Tb, MPa (psi)	19.0	16.8	18.1	17.5
Eb, %	206	264	213	246
Tear, Die C, N/mm	34.2	39.4	35.7	33.8
Compression Set, %				
168 hrs at 150 °C	43.8	26.1	22.3	19.2
1008 hrs at 150 °C	54.7	37.6	36.7	35.6
Heat Aging—Air Aging 6 weeks at 150 °C				
Hardness Change in pts	6	8	9	11
Change in Modulus, %	21	12	4	13
Change in Tensile Strength, %	-9	-11	-12	-13
Change in Elongation, %	-13	-4	11	0
Fluid Aging, Aging 6 weeks at 150 °C in SF 105				
Hardness Change in pts	-12	-13	-9	-11
Change in Modulus, %	-10	-7	-4	-7
Change in Tensile Strength, %	-26	-23	-18	-17
Change in Elongation, %	-19	-24	-15	-9
Volume Change, %	31	32	29	31
Fluid Aging, Aging 6 weeks at 150 °C in Dexron® III ATF				
Hardness Change in pts	-10	-12	-11	-9
Change in Modulus, %	-8	-17	-13	-17
Change in Tensile Strength, %	-20	-23	-21	-15
Change in Elongation, %	-19	-22	-10	-5
Volume Change, %	25	27	26	26
Low Temperature Properties				
Tg, DSC, Inflection Point, °C	-33	-33	-34	-34

DuPont™ Vamac® Dipolymer Compound Mixing

Vamac® dipolymer compounds can be mixed on an open roll mill or in an internal mixer. The same mixing rules apply to Vamac® dipolymer compounds as with Vamac® terpolymer compounds. An upside down mixing procedure is recommended. One difference is because of the higher activation temperatures of the peroxide curing agents a slightly higher dump temperature of 5–10 °C can be used. Also, there is no concern with the use of metal stearate anti-stick coatings since no potential ionic crosslinking can occur. A detailed Vamac® compound mixing guide is available.

Vamac® Dipolymer Processing

Vamac® dipolymer compounds can be extruded, molded (injection, transfer, compression) or calendered. Shrinkage during molding is expected and varies with compound formulation and process conditions. Vamac® dipolymer compounds normally have shrinkage values in the range of 2.5–3%.

Extrusion: Extrusion is mainly used for hose and wire and cable applications. Short to moderate L/D extruders are suitable and able to handle a broad range of draw-down ratios. Extruder head temperatures are normally around 70 °C with lower barrel and screw temperatures. Low extruder temperatures are recommended to maximize compound viscosity to prevent flat spotting. Curing is done with steam or air autoclave. Vamac® compounds will “sponge” if inadequate pressure (60–80 psi min.) is applied during curing. Because oxygen inhibits peroxide curing, autoclaves should be purged with nitrogen. Continuous vulcanization can be obtained with high pressure steam systems or salt bath.

Molding: Molds for Vamac® compounds should be hard chrome plated. Molds should be properly vented and have vacuum systems to remove air before filling. Mold temperatures range from 160–190 °C. Injection molding is the preferred molding method for Vamac® compounds due to their low viscosity. Fast cure times of less than a minute can be achieved with injection molding. A detailed guide on injection molding of Vamac® compounds is available.

Calendering: Vamac® compounds can be calendered into high quality, blister free sheets up to 2 mm thick. Thicker sheets can be obtained from plied sheets. Mill rolls should be kept cool (30–50 °C) to maximize melt “green” strength. Curing is with steam or air autoclaves.

Materials Used in Formulations and Test Fluids — General Composition and Supplier

Material	Composition	Supplier
Polymer		
Vamac® DP	Ethylene Acrylic Elastomer	DuPont
Release Aids		
Armeen® 18D	Octadecyl Amine	Akzo Nobel
Vanfre® VAM	Complex Organic phosphate ester	R.T. Vanderbilt
Stearic Acid		
Antioxidant		
Naugard® 445	Diphenyl Amine	Uniroyal Chemical
Fillers		
N550	Carbon Black	
Curatives		
Vul-Cup® 40KE	40% a-a-bist (t-butyl peroxy) diisopropylbenzene	Hercules Incorporated
Di-Cup® 40C	40% Dicumyl Peroxide	GEO Specialty Chemicals
HVA-2	N,N-m-phenylene dimaleimide	DuPont
Ricon® 152	Polybutadiene	Sartomer Technology Company
Sartomer® 350	Trimethylol-propane-trimethacrylate	Sartomer Technology Company
Test Fluids		
Dexron® III	Automatic Transmission Fluid	General Motors Corporation
Service Fluid 105	Service Fluid 105	ASTM Test Monitoring Center

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