

Vamac® Ultra LT

Ethylene Acrylic Elastomer - Technical Data

Description

Vamac[®] Ultra LT provides improved low temperature properties for AEM compounds particularly when used in <u>combination</u> with higher viscosity Ultra grades, like Vamac[®] Ultra IP, and a low volatility plasticizer. Compounds using these three ingredients exhibit good heat resistance, good dynamic properties, and good processability, while having good low temperature properties both before and <u>after</u> heat and fluid ageing.

Key low temperature properties for extended compounds:

- Compound Tg (glass transition temperature) as low as -50 °C
- TR10 (Temperature of retraction 10%) values as low as -43 °C

Vamac[®] Ultra LT - Product Properties

Property	Target Values	Method
Mooney Viscosity ML1+4 at 100 °C	12	ASTM D1646
Volatiles	≤0.6 wt %	Internal DuPont Test
Form (25kg nominal bale size)	51.6 x 34.4 x 13.6 cm	Visual Inspection
Color	Clear to light yellow translucent	Visual Inspection

Handling Precautions

Because Vamac[®] ethylene-acrylic elastomers contain small amounts of residual methyl acrylate and n-butyl acrylate monomers, adequate ventilation should be provided during storage and processing to prevent worker exposure to acrylic monomer vapor. Additional information may be found in the Vamac[®] product Safety Data Sheet (SDS), and DuPont[™] bulletin, *Safe Handling and Processing of Vamac*[®].

'Standard' Low Temperature Vamac® Compounds

The traditional way to improve the low temperature properties of Vamac® compounds has been to start with Vamac® G and add high levels of plasticizers. These compounds have good initial low temperature properties and they can meet the end use requirements.

However, the compounds are difficult to mix and to mold because they are relatively low in viscosity and they are also "tacky". Also, as these compounds undergo heat ageing in air at elevated temperatures they lose most of their plasticizer due to vitalization. This can be shown by measuring the weight loss during ageing and by measuring the increase in the Tg after heat ageing. The weight loss for compounds with the standard plasticizer is relatively high and the heat aged Tg approaches the Tg of a control compound with no plasticizer.

The combination of processing problems and poor low temperature properties after heat ageing has made it difficult to use compounds made based on Vamac[®] G in applications that need outstanding low temperature properties.



Vamac[®] Ultra LT

Vamac[®] Ultra LT (formerly VMX4017) is an ethylene acrylate copolymer from DuPont that uses an acidic cure site monomer, so compounds can be cured with diamines. The key attribute of Ultra LT is the low Tg of -42°C which is about 10°C lower than Vamac[®] G. This translates into a Vamac[®] Ultra LT compound that has a Tg that is about 10°C lower than a comparable Vamac[®] G compound. Compounds made with Vamac[®] Ultra LT have similar heat resistance, compression set and CSR (compressive stress relaxation) properties when compared to a compound made from Vamac[®] G.

There are always trade-offs in elastomer compounds. The compounds using the Vamac[®] Ultra LT have constraints around fluid resistance and processing.

- The fluid resistance is not as good as with Vamac[®] G compounds. For example, a 70 Shore A compound with Vamac[®] Ultra LT, and no plasticizer will have a VI (volume increase) of around 27% in a synthetic engine oil like Mobil 1 5W30 and around 80% in IRM 903 (after ageing for one week at 150C).
- Vamac[®] Ultra LT compounds are not easy to mix and mold because they are low in viscosity and they
 are relatively "tacky".

Low Volatility Plasticizers

Several plasticizer suppliers have introduced polyether/ester plasticizers that have good low temperature properties along with lower volatility compared to the standard plasticizers used in compounds based on Vamac®. Compounds made with these low volatility plasticizers have good initial low temperature properties and they maintain these low temperature properties after ageing in air for one week at 175 °C or for six weeks at 150 °C.

When used in combination with Vamac[®] Ultra LT they improve the low temperature performance of the compounds by up to 8°C. The Tg of a Vamac[®] Ultra LT compound with 15 phr of the plasticizer can be as low as -50°C and the TR10 can be as low as -43 °C.

An added benefit of the plasticizers is that they can improve the fluid resistance. A 70 Shore A Vamac[®] Ultra LT compound with 15 phr plasticizer will have about 19% VI in Mobil 5W30 and about 68% VI in IRM 903.

The low volatility plasticizer improves the low temperature properties and the fluid resistance, but it does not improve the processing problems of the Vamac[®] Ultra LT compounds. It actually makes the processing more difficult because it lowers the compound viscosity and it increases the "tackiness".

Vamac® Ultra IP

Vamac® Ultra IP is an improved processing (IP) version of Vamac® G with a Mooney Viscosity (ML 1+4 at 100°C) of 29 MU compared to 16 MU for Vamac® G. Vamac® Ultra LT Mooney Viscosity is 11 MU.

A combination of Vamac® Ultra IP and Vamac® Ultra LT can be used to improve the processability of a compound based only on Vamac® Ultra LT. The compound viscosity will increase, and the compound will become less "tacky".

The addition of Ultra IP will also improve the fluid resistance. A 70 Shore A compound that uses 50 phr Ultra IP, 50 phr Vamac[®] Ultra LT and 15 phr plasticizer will have about 13% VI in Mobil 1 5W30 and about 58% VI in IRM 903.



The Ultra IP polymer has a Tg of about -32°C so it will increase the Tg of a compound based solely on Vamac[®] Ultra LT. The compound mentioned above – a 50/50 blend of Vamac[®] Ultra LT and Ultra IP along with 15 phr of plasticizer – has a Tg of about -47°C. This is still a very good low temperature compound.

Low Temp after Fluid and Heat Ageing

The low temperature properties of compounds based on Vamac[®] Ultra LT and Vamac[®] Ultra IP were measured initially and also after ageing in air and ageing in fluids. These compounds have very good low temperature properties after testing both ageing conditions. They are significantly better than Vamac[®] G compounds.

An interesting observation was seen with compounds that had plasticizer levels that varied from 0 to 30 phr. As expected there was a large difference in the initial Tg values. After fluid ageing all of the compounds had Tg values that were very close – and were similar to a compound with about 15 phr plasticizer. The compounds with no plasticizer had a significant <u>decrease</u> in Tg after fluid ageing while the compounds with 30 phr plasticizer had a significant <u>increase</u> in Tg after fluid ageing. The compounds with 15 phr plasticizer had little or no change in Tg after fluid ageing.

Most of the compounds discussed in this bulletin use either 0 or 15 phr plasticizer.

Major Performance Properties and Applications

Cured compounds made from Vamac[®] Ultra IP, Ultra LT and a low volatility plasticizer have a good combination of properties including a wide operating window for end use temperatures that ranges from -50 °C up to 175°C. The compounds can withstand short term temperature spikes up to 200 °C.

The cured compounds are typically rated as class E for heat resistance using the ASTM D2000 system and this means that they will pass a heat rating test of 70 hours at 175 °C (347 °F).

The fluid resistance of a cured compound depends on the formulation used. Some key factors are the level of each polymer, the plasticizer and carbon black levels and the cure package. Typical values for volume swell in IRM 903 fluid after ageing for 168 hours at 150 °C (302°F) ranges from 60 to 80%. This means that the compounds are rated as Type E using ASTM D2000. Some of the compounds are between 40 and 60% swell in IRM 903 and they are rated as Type F.

IRM 903 is a standard reference fluid with a relatively high aromatic content. It is much more aggressive to Vamac[®] compounds compared to transmission fluids, engine oils and high temperature greases. This means that the VI in IRM 903 may not be a good predictor of how a compound performs in the actual end use fluid. For comparison – the volume swell for these new compounds ranges from about 13 to 27% in Mobil 1 5W30 (after one week/ 150 °C) and from about 30 to 45% in SF105.

The compression set values for compounds based on Vamac[®] Ultra IP, Ultra LT and the low volatility plasticizer were measured after 70 hours at 150 °C using the ASTM plied method as well as the ISO molded buttons. The comp set values ranged from 15 to 30% and the results followed the usual trends. The comp set improved as the curative level increased and when the plasticizer levels decreased.



CSR tests run on Vamac[®] Ultra LT compounds with plasticizer and the compounds show very good properties for six weeks at 150 °C in engine oils. The percent retained sealing force is a relatively high value and this is probably due to the relatively high volume-swell. The Vamac[®] Ultra LT compounds have higher values compared to the Vamac[®] G compounds which in turn have higher retained sealing forces compared to Vamac[®] GLS compounds.

It is not easy to measure dynamic properties of elastomeric compounds with a simple test that predicts good end use performance. The end use requirements vary considerably from end use to end use – factors such as temperature (both high and low), frequency and strain rate all play a part in end use performance.

The DeMattia flex test was used to study the dynamic properties of the compounds based on Vamac[®] Ultra LT. The test was run on non-pierced samples at 150°C (ASTM D430, ISO 132) and on pierced samples at 23°C (ASTM D813, ISO 133). The trends seen with these compounds were comparable to other studies with Vamac[®] compounds. The compounds had better flex properties when the hardness was lowered and when the compression set was higher (milder cure package).

The two trends that favor better dynamic properties – lower hardness and a milder cure package – also lead to higher volume swell in fluids. One needs to balance the compound properties to meet all of the end use requirements.

The properties of compounds based on Vamac[®] Ultra LT, Vamac[®] Ultra IP and a low volatility plasticizer make them useful for a wide range of automotive applications. Some of these are molded boots, powertrain seals and gaskets, rocker cover seals, transmission oil coolant hoses, power steering hoses, turbocharger hoses, crankcase ventilating tubes, coverings for fuel and coolant hoses, O-rings, grommets and crankshaft dampers.

These compounds are halogen free and thus can be used for flame retardant, low-smoke, non-halogen wire and cable jackets and in non-halogen, low smoke flooring.

Vamac[®] Ultra LT compounds are well suited for injection, transfer or compression molding. They also can be extruded.

Compound and Vulcanizate Properties

The following table shows the formulation for six different compounds based on Vamac[®] Ultra LT, Vamac[®] Ultra IP and low volatility plasticizer with predicted properties. The first two compounds are control compounds based on Vamac[®] G. The first compound has no plasticizer and the second one has 15 phr of a "standard" plasticizer. The next two compounds are based on Vamac[®] Ultra LT where one compound has no plasticizer and the other has 15 phr of a low volatility plasticizer. The last two compounds are based on 50/50 blends of Vamac[®] Ultra LT and Vamac[®] Ultra IP and they have 0 or 15 phr of a low volatility plasticizer. The compound formulations also include 1.5 phr Stearic acid, 1.0 phr Vanfre[®] VAM, 0.5 phr Armeen[®] 18D and 2 phr Naugard[®] 445.

Extensive DOE's (Design of Experiment) have been run on these compounds and models have been developed to predict the properties. The black level in the models was adjusted to give a hardness of about 70 Shore A so that the compounds could be compared at equal hardness values. The cure conditions used in the modeling work included a 10-minute press cure at 180°C followed by a four-hour post cure at 175°C. Predicted values for Mooney viscosity, and initial cured physicals are shown in Table 1 with compounds of Vamac[®] Ultra LT having the best low temperature properties.



Table 1 – Comparison of Predicted Low Temperature Compound Properties

					50/50	50/50
	Vamac [®] G	G control	Vamac [®]	Ultra LT	Ultra LT/	Ultra LT/
	control	with 15	Ultra LT	with 15 phr	Ultra IP	Ultra IP with
	with no	phr std	with no	low vol	with no	15 phr low
	plasticizer	plasticizer	plasticizer	plasticizer	plasticizer	vol plasticizer
Vamac [®] Ultra LT			100	100	50	50
Vamac [®] Ultra IP					50	50
Vamac [®] G	100	100				
"standard" plasticizer	0	15				
Low Volatility plasticizer			0	15	0	15
N550 carbon black	48	68	52	63	49	60
Diak™ #1	1.5	1.5	1.5	1.5	1.5	1.5
Vulcofac® ACT 55	2	2	2	2	2	2
Mooney Viscosity						
ML (1+4) at 100 °C	57	41	47	32	58	39
Hardness, Shore A, pts	70	70	70	70	70	70
Tensile Strength, MPa	19	14	16	13	19	16
Elongation at Break, %	251	243	215	192	249	222
100% Modulus, MPa	6.0	5.6	6.2	6.1	6.2	6.1
Tg by DSC, Initial, °C	-29	-39	-44	-50	-40	-47
TR 10, °C	-28	-32	-37	-43	-30	-36

The compounds based on Vamac[®] Ultra LT have lower viscosities than the Vamac[®] G controls and lower tensile and elongation values. The 50/50 blends of Vamac[®] Ultra LT and Vamac[®] Ultra IP have similar viscosities and similar physicals to the Vamac[®] G controls. The low temperature properties improve as the plasticizer level is increased and also as more Vamac[®] Ultra LT is used. The best low temperatures are seen in compound 4 which has all Vamac[®] Ultra LT and 15 phr of plasticizer.

Compression Set

The predicted compression values of the compounds are shown below. All of the compounds have good compression set values. The G control compound with the standard plasticizer has the highest compression set.

Table 1 (continued) – Comparison of Predicted Low Temperature Compound Properties

					50/50	50/50
	Vamac [®] G	G control	Vamac [®]	Ultra LT	Ultra LT/	Ultra LT/
	control	with 15	Ultra LT	with 15 phr	Ultra IP	Ultra IP with
Compression Set	with no	phr std	with no	low vol	with no	15 phr low
(70 hour / 150 °C), %	plasticizer	plasticizer	plasticizer	plasticizer	plasticizer	vol plast
ISO molded buttons	15	27	17	21	16	20
ASTM plied buttons	13	24	14	17	13	15



Fluid Ageing

The compounds were fluid aged for one week at 150 °C in three different fluids. The predicted VI results for each fluid are shown below. As expected, the Vamac® Ultra LT compound with no plasticizer has the highest VI values. The 50/50 blend of Ultra LT /Ultra IP with 15 phr plasticizer has relatively good fluid resistance. The VI results in IRM 903 are much higher than the engine oils. It is important to run the fluid ageing tests in the actual fluid used in the end use application.

Table 1 (continued) – Comparison of Predicted Low Temperature Compound Properties

					50/50	50/50
	Vamac [®] G	G control	Vamac [®]	Ultra LT	Ultra LT/	Ultra LT/
Fluid Resistance	control	with 15	Ultra LT	with 15	Ultra IP	Ultra IP with
(168 hrs at 150 °C),	with no	phr std	with no	phr low vol	with no	15 phr low
Volume Increase (VI) %	plasticizer	plasticizer	plasticizer	plasticizer	plasticizer	vol plast
Mobil 1 5W30	19	12	27	19	21	13
SF 105	27	21	46	37	36	29
IRM 903	55	48	80	68	68	58

Low Temperature Properties after air ageing and after fluid ageing

Low temperature properties are typically measured only on the initial cured compounds. For the DOE model used in this study the Tg by DSC was also measured after ageing in air for one week at 175 °C as well as after ageing in IRM 903 for 168 hours at 150 °C.

The Vamac® G compound with the standard plasticizer had the largest predicted increase in Tg after air ageing as the Tg increased by 9 °C. This is mainly due to the use of the "standard" plasticizer. This compound had the highest weight loss during ageing which reflects the loss of plasticizer. The compounds with no plasticizer or with the low volatility plasticizer had an increase in Tg of only one to three °C after heat ageing.

The compounds based on Vamac[®] Ultra LT or Ultra LT/Ultra IP had very good low temperature properties after fluid ageing. The Tg values were about -50 °C. Both Vamac[®] G controls had Tg's of about -40 °C after fluid ageing.

To ensure good end use performance a suggestion is to measure the low temperature properties of the compounds after heat ageing in air and fluid ageing in the end use fluid.

Table 1 (continued) – Comparison of Predicted Low Temperature Compound Properties

					50/50	50/50
	Vamac [®] G	G control	Vamac [®]	Ultra LT	Ultra LT/	Ultra LT/
Low Temperature	control	with 15	Ultra LT	with 15 phr	Ultra IP	Ultra IP with
Properties After Ageing	with no	phr std	with no	low vol	with no	15 phr low
Tg (by DSC), °C	plasticizer	plasticizer	plasticizer	plasticizer	plasticizer	vol plast
Initial	-29	-39	-44	-50	-40	-47
Air Ageing, 168hr at 175°C	-26	20	40	-49	-38	-45
7 til 7 tgenig, 100m at 175 C	-20	-30	-42	-49	-30	-45



Dynamic Properties

The typical way to improve the dynamic properties of a Vamac[®] compound is to go to a softer compound (lower hardness and/or modulus) and to a lower state of cure (higher compression set). These two methods were modeled for compounds based on blends of Vamac[®] Ultra LT and Ultra IP. There was a significant improvement in dynamic properties as measured by DeMattia flex. However, the drawback with these two changes is that both increase the VI values for fluid ageing and both changes make it more difficult to process the compounds. One needs to balance the dynamic properties versus the fluid ageing requirements and the processability requirements.

Table 2 shows three compounds that differ significantly in predicted DeMattia flex properties. The first column has a 70 Shore A hardness compound with a high state of cure (low comp set) while the last column has a 60 Shore A compound with a low state of cure (relatively high comp set). The compound in the last column has the best dynamic properties – but it also has the lowest viscosity and the highest VI numbers. The dynamic properties of the 60 Shore A compound are between 15x and 100x better than the 70 Shore A compound.

Table 2 – Comparison of Predicted Dynamic Properties

	70 Shore A	65 Shore A	60 Shore A
	high	intermediate	low
	state of cure	state of cure	state of cure
Vamac [®] Ultra LT	50	50	50
Vamac [®] Ultra IP	50	50	50
Low Volatility plasticizer	15	15	15
N550 carbon black	60	54	48
Diak™ #1	1.5	1.3	1.1
Vulcofac® ACT 55	2	1.5	1
Mooney Viscosity, ML (1+4) at 100°C, MU	39	33	29
Hardness, Shore A, pts	70	65	60
Tensile Strength, MPa	16	15	15
Elongation at Break %	220	270	330
100% Modulus, MPa	6.1	4.4	3.2
Tg (by DSC), Initial, °C	-47	-46	-46
TR 10, °C	-36	-36	-36
Compression Set (70 hour / 150°C)			
ISO molded buttons	20	23	27
ASTM plied buttons	15	16	18
Volume Increase/ VI (168h / 150°C), %			
Mobil 1 5W30	13	15	17
SF 105	29	32	35
IRM 903	58	64	72
DeMattia (ASTM D813, pierced, 23°C)			
Relative performance cycles to 8.5 mm cut length DeMattia (ASTM D430, 150°C)	1	4	15
Relative performance average cycles to failure	1	11	120



Compounds based on Vamac[®] Ultra LT, Ultra IP and a low volatility plasticizer can be designed to meet compounds with outstanding low temperature properties. As with all elastomer compounds there will be tradeoffs between end use performance requirements as well as processability.

The ASTM test methods used in the work are shown below along with the comparable ISO method.

	ASTM	Comparable ISO
	method	method
Rheology		
Mooney Viscosity	D 1646	289-1
Mooney Scorch	D 1646	289-2
MDR	D 5289	6502
Physicals		
Hardness	D 2240	7619-1
Tensile, Elongation, Modulus	D 412	37
Tear, Die C	D 624	34
Fluid Ageing	D 471	1817
Compression Set, method B	D 395	815-1
Tg by DSC	D 3418	22768
Ageing in Air	D 573	188
Temperature of Retraction	D 1329	2921
DeMattia – not pierced	D 430	132
DeMattia – pierced	D 813	133



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