DuPont™ Vamac®
An Alternate Accelerator for Vamac® Terpolymers: Vulcofac® ACT 55

Technical Information — Rev. 2, July 2010

Introduction

DOTG (di-ortho-tolyl guanidine) is an accelerator used in sulfur-cured elastomers like natural rubber, SBR and NBR. It is also used in the curing system of ACM and AEM elastomers containing an acidic cure site and in certain EPDM compounds.

DuPont™ Vulcofac® ACT 55 (tertiary amine complex) is an accelerator developed and commercialized by Safic-Alcan in 2006 as an alternative to guanidine accelerators like DOTG. Please read and understand the MSDS before handling and use of Vulcofac® ACT 55.

This study compares the properties obtained with Vulcofac® ACT 55 in Vamac® ethylene acrylic elastomer terpolymer compounds with compounds cured with a combination of a primary diamine as curing agent and a guanidine as accelerator.

The two guanidine accelerators considered in this study are:

DOTG (=di-ortho-tolyl guanidine) providing good compression set and high modulus

DPG (=diphenyl guanidine) providing good flex fatigue resistance

For handling precautions, processing and safety information, please refer to the “Safe Handling and Processing of Vamac® and Vamac® Compounds” available from www.vamac.dupont.com.

Formulation

A design of experiment was conducted to evaluate the impact of Vulcofac® ACT 55 in a Vamac® G recipe. Eighteen Vamac® compounds using Vulcofac® ACT 55 were tested in this study (see *Table 1*). In parallel, three standard Vamac® G recipes using DOTG and/or DPG were selected as a reference for seals and hoses made from Vamac® compounds. All Vamac® compounds were mixed in the same conditions (full cooling, load factor = 70%, rotor speed = 40 rpm, mixing time = 180 seconds and dumping temperature = 85 °C) using a 1.6 L Francis Shaw Intermeshing internal mixer. All ingredients, except for curatives, were charged using an upside-down mixing technique.

Table 1. DOE (Design of Experiment) with DuPont™ Vulcofac® ACT 55

Control Compounds	1	2	3	Vulcofac® ACT 55 Variations
	2 runs	**1 run**	**1 run**	
DuPont™ Vamac® G	100 phr	100 phr	100 phr	100 phr
Naugard® 445	2	2	2	2
Sterling SO N-550	60	60	60	60
Vanfre® VAM	1	1	1	1
Rhenosin® W759	10	10	10	10
Stearic Acid	1.5	1.5	1.5	0 and 1.5 phr
Armeen® 18 D	0.5	0.5	0.5	0, 0.075 and 1.5 phr
Diak™ No. 1	1.5	1.25	1.25	1, 1.5 and 2 phr
Ekaland DOTG/C 100%	4	2		
Rhenogram DPG 80%		2	4	0, 0.5 and 1 phr Salicylic Acid 0, 2 and 4 ph Vulcofac® ACT 55

Rheological Properties

A. Mooney Viscometer Results

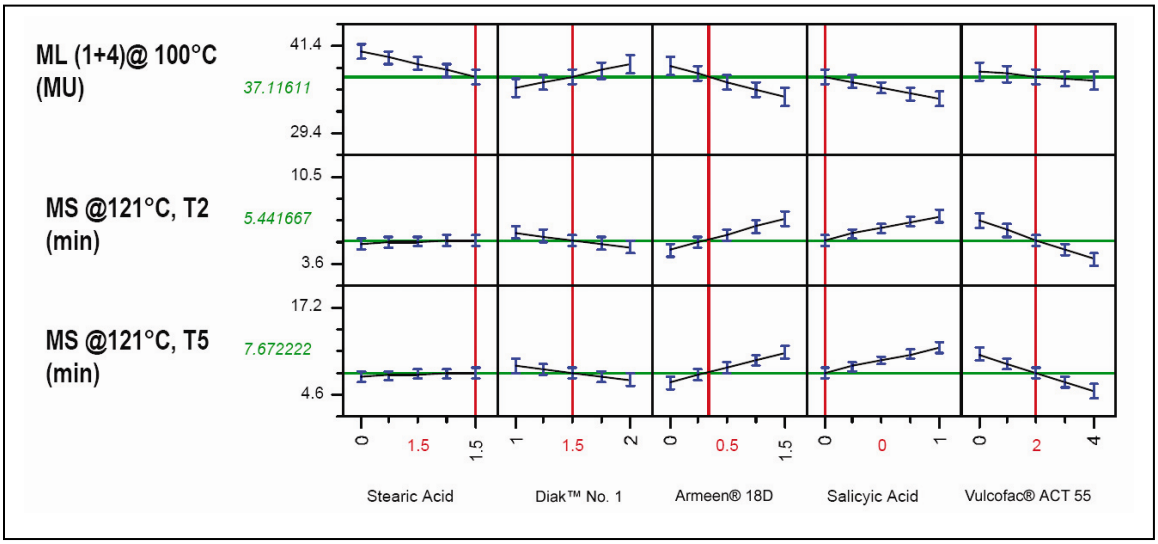
The three “control compounds” have similar viscosity and scorch behavior (see *Table 2*).

Table 2. Control Compounds — Mooney Viscometer Results

	Control 1 (DOTG)	Control 2 (DOTG/DPG)	Control 3 (DPG)	2 phr Vulcofac® ACT 55
ML (1+4) at 100 °C, (MU)	34.1	33.8	33.6	37.1
MS at 121 °C, t2, min	6.3	6.1	6.2	5.4
MS at 121 °C, t5, min	8.9	8.5	8.6	7.7

All Vamac® compounds that contain Vulcofac® ACT 55 are scorchier and therefore higher in Mooney than “control compounds” using DOTG and/or DPG according to the results presented in *Figure 1*. Vamac® recipes using Vulcofac® ACT 55 should be corrected by adding more retarding agent like Armeen® 18D or Salicylic Acid in order to reach a scorch similar to DOTG or DPG.

Figure 1. DOE with Vulcofac® ACT 55 — Mooney Viscosity Results



B. Moving Die Rheometer Results

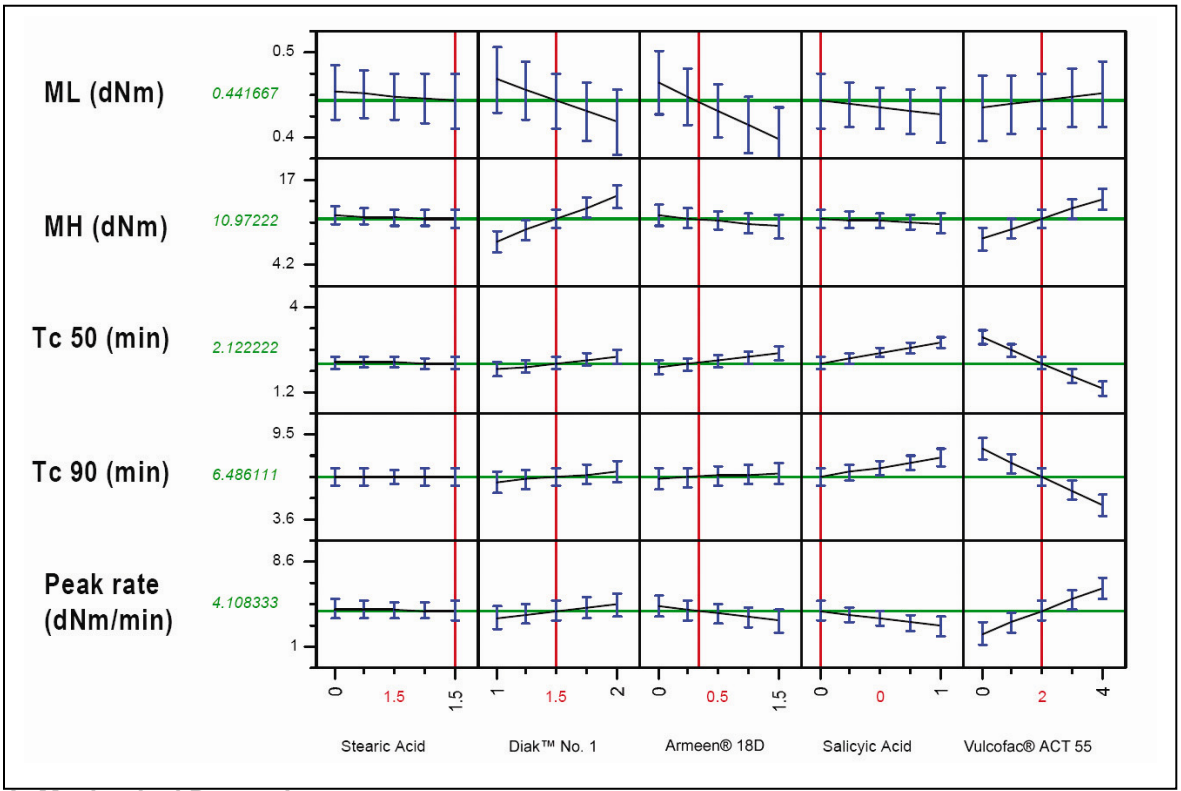
All compounds were tested in the same conditions using MDR equipment (conditions: 0.5°/12 min at 180 °C). “Control 1” compound (1.5 phr Diak™ 1 + 4 phr DOTG), a standard recipe used for seals and gaskets, clearly has a higher crosslink density (higher MH) and higher cure rate (higher peak rate) than the 2 other Vamac® compounds: “Control 2” (= 1.25 phr Diak™ 1 + 2 phr DOTG/2 phr DPG) and “Control 3” (= 1.25 Diak™ 1 + 4 phr DPG) have been considered for hose applications (see *Table 3*).

Table 3. Control Compounds — MDR Results

	Control 1 (DOTG)	Control 2 (DOTG/DPG)	Control 3 (DPG)	2 phr Vulcofac® ACT 55
ML, dNm	0.35	0.37	0.37	0.44
MH, dNm	12.30	9.17	7.66	10.97
Tc50, min	1.96	1.63	1.51	2.12
Tc90, min	5.37	5.06	4.51	6.48
Peak rate, dNm/min	4.3	3	3.4	4.1

Vulcofac® ACT 55 is similar to DOTG in cure kinetics of Vamac® compounds as can be seen in Figure 2. Vulcofac® ACT 55 should cure slightly slower than DOTG.

Figure 2. DOE with Vulcofac® ACT 55 — Moving Die Rheometer results



A. Mechanical Properties

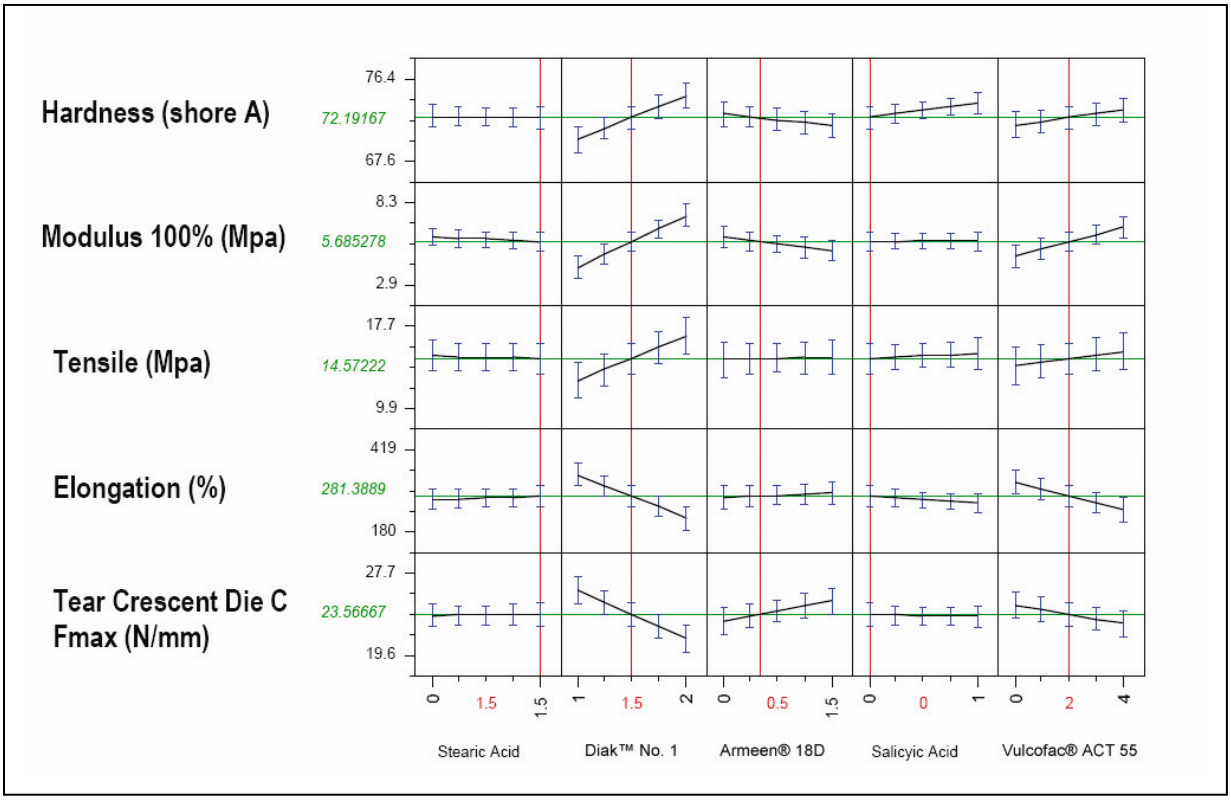
Vamac® slabs were molded by compression during 5 min at 180 °C and post-cured 4 h at 175 °C in an air circulating oven. Test specimens were die-cut from slabs.

Vulcofac® ACT 55 seems to bring about a higher hardness level and lower elongation than DOTG in Vamac® compounds when comparing *Table 4* and *Figure 3*.

Table 4. Control Compounds — Mechanical Properties

	Control 1 (DOTG)	Control 2 (DOTG/DPG)	Control 3 (DPG)	2 phr Vulcofac® ACT 55
Hardness, Shore A, 1 sec	69.8	69.3	68.3	72.2
Modulus 100%, MPa	4.8	4	4.1	5.7
Tensile Strength, MPa	15.6	14.8	13.9	14.6
Elongation, %	325	369	328	281
Tear Crescent Die C. Fmax, dNm/min	26.8	28.2	29.0	23.6

Figure 3. DOE with Vulcofac® ACT 55 — Mechanical properties



B. Compression Set

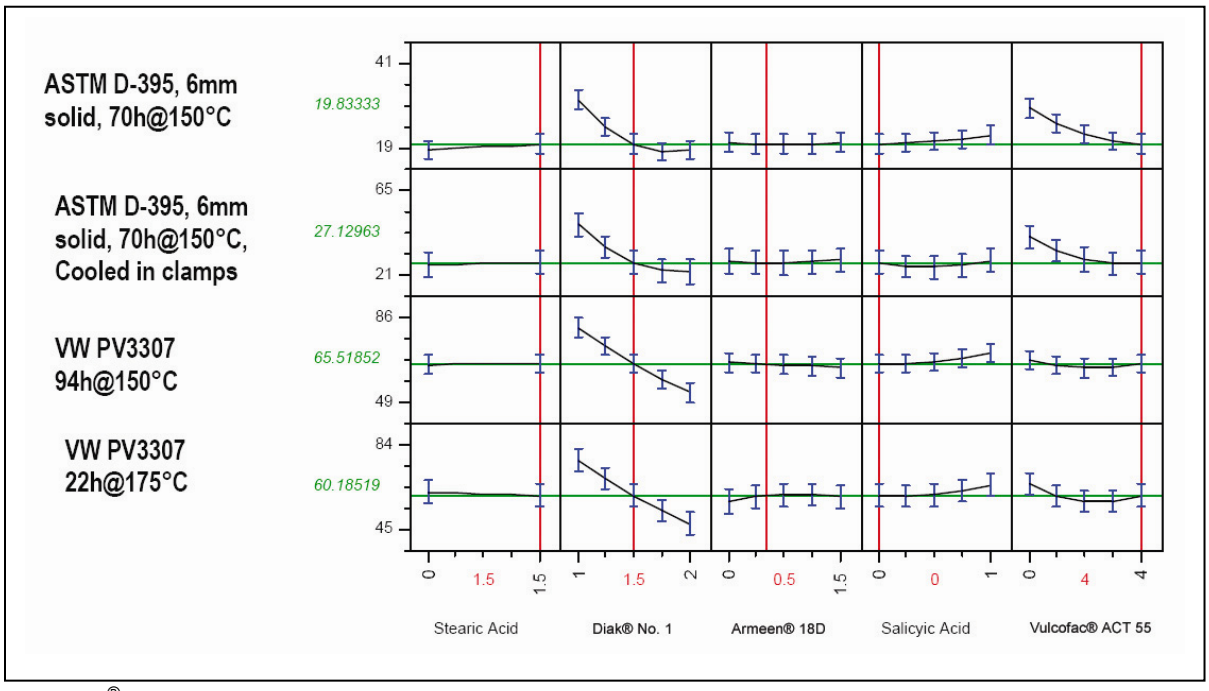
There are several test procedures commonly used to measure compression set. The standard test is ASTM D395 (VDA 675 216A or ISO815) which requires samples to be removed from the clamps immediately after removal from the oven. Alternatively, procedures VDA 675 216B (often applied for Daimler Chrysler parts) and VDA 675 218 (identical to Volkswagen specification PV 3307) ask for measurement of compression set after cooling the test pieces within the clamps. As a result these tests give significantly higher compression set values as shown in *Table 5*.

Table 5. Control Compounds — Compression Set

	Control 1 (DOTG)	Control 2 (DOTG/DPG)	Control 3 (DPG)	2 phr Vulcofac® ACT 55
ASTM D395, 70 h at 150 °C, 6 mm molded pips	19%	23%	28%	24%
ASTM D395, 70 h at 150 °C, VDA 67521B				
2 h cooled in clamps, 6 mm molded pips	27%	34%	42%	30%
VW PV 3307, 94 h at 150 °C, 2 mm disks	58%	58%	77%	61%
VW PV 3307, 22 h at 175 °C, 2 mm disks	59%	62%	77%	58%

Figure 4 shows that Vulcofac® ACT 55 has compression set performance comparable to DOTG (see *Table 5*) so the sealing force should be similar for both DOTG and Vulcofac® ACT 55 compounds.

Figure 4. DOE with Vulcofac® ACT 55 — Compression Set



Vulcofac® ACT 55 can be used as an accelerator in combination with a primary diamine to cure DuPont™ Vamac® terpolymers. Nevertheless, some compound adjustments are necessary to meet properties of existing Vamac® compounds based on DOTG.

Materials Used in Formulations — General Composition and Supplier

Material	Composition	Supplier
Polymer		
Vamac® G	Ethylene Acrylic Elastomer	DuPont
Release Aids		
Armeen® 18D	Octadecyl Amine	Akzo Nobel
Ofalub SEO	Complex Organic phosphate ester	Safic-Alcan
Vanfre® VAM	Complex Organic phosphate ester	R.T. Vanderbilt
Stearic Acid	Stearic Acid	Sigma Aldrich Chemie
Antioxidants		
Naugard® 445	Diphenyl Amine	Uniroyal Chemical
Plasticizers		
Rhenosin® W759	Mixed Ether/Ester Plasticizer	Rhein Chemie
Nycoflex® ADB 30	Mixed Ether/Ester Plasticizer	Safic-Alcan
Fillers		
Spheron® SO N550	Carbon Black	Cabot Corporation
Curatives		
Rubber chem. Diak™ #1	Hexamethylene Diamine Carbamate	DuPont
Vulcofac® HDC MB 75	Hexamethylene Diamine Carbamate	Safic-Alcan
Ekaland DOTG/C	Di-ortho-tolyl Guanidine	MLPC International
Rhenogran DPG 80%	Diphenyl Guanidine	Rhein Chemie
Vulcofac® ACT 55	Animated Derivatives	Safic-Alcan

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