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NXT* Silane

Coupling Agent for Silica-Reinforced Tire Tread Compounds

MARKETING BULLETIN SILANES - TIRE & RUBBER

NXT silane provides options for enhanced tire performance and overall systems cost-efficiencies for tire manufacturers.

NXT silane, a thiocarboxylate functional silane, has been shown to enable reduced rolling resistance without loss of wet traction, while increasing overall production efficiency for tire manufacturers as compared to standard sulfur silanes.

Silica compounds coupled with NXT silane have exhibited improved silica dispersion, easier mixing, and faster, pliable processing. Compounds containing NXT silane show lower Payne Effect, improved tan δ values, and better resilience properties. In addition, NXT silane typically can be used with high surface area silica and functionalized polymers for easy processing of high performance compounds. Green compounds made with NXT silane have demonstrated stability for a significantly longer time without reflocculation.

Key Features and Typical Benefits

- Fewer non-productive mixing steps
- Reduced compound viscosity
- Faster extrusion
- Improved Payne Effect
- Increased resilience
- Reduced tan δ max
- Excellent dynamic properties at low temperature (-20 °C to +10 °C)

Typical Physical Properties	
Physical Form	Clear liquid
Color	Pale yellow
Specific Gravity at 20 °C, OECD-Guideline No. 102	0.9686
Flash Point, Pensky-Martens Closed Cup, °C (°F)	110 (230)

Typical properties are average data and are not to be used as or to develop specifications.

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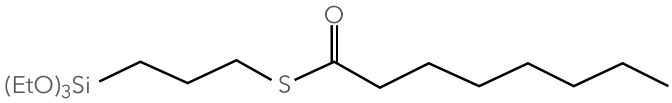
Chemical Structure



3-Octanoylthio-1-propyltriethoxysilane (Mol. Wt. 364)

Table 1: Typical Silica-Reinforced Tire Tread Formulation Used to Evaluate NXT Silane

PHR	Ingredient
103.2	SSBR (oil extended; 55% vinyl, 25% styrene content)
25	BR (butadiene rubber; 96% *cis*)
80	Precipitated silica
10	N-330 carbon black
Variable	TESPD, TESPT or NXT silane (Momentive Performance Materials)
5.0	DAE oil
2.5	ZnO
2.0	Stearic acid
2.0	6PPD (antioxidant/antiozonant)
1.5	Wax
Final Mix Ingredients	
Variable	Sulfur†
2.0	CBS (primary accelerator)
2.0	DPG (secondary accelerator)

† Total theoretical sulfur content for each compound equals 4.93 x 10^{22} atoms

Product formulations are included as illustrative examples only. Momentive makes no representation or warranty of any kind with respect to any such formulations, including, without limitation, concerning the efficacy or safety of any product manufactured using such formulations.

Potential Applications

For improved rolling resistance without loss of wet traction in tires, NXT silane can be considered for use in tire tread compounds to enable high silica loading while typically managing compound viscosity, improving processibility and increasing mixing temperatures, even in functionalized polymer and high surface area silica compounds. NXT silane can be mixed at temperatures as high as 170 °C without scorch and the number and duration of non-productive mixing steps can be minimized to achieve maximum silica dispersion.

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Potential Applications (continued)

Compounding procedures for traditional polysulfide TESPD and TESPT silanes require two or more non-productive mixes. Differences in mixing procedures between NXT silane and these traditional polysulfide silanes are shown in Table 2.

Table 2: Mixing Procedures for Polysulfide and NXT Silanes

Two Step Mix Procedure for S4 (TESPT) Silane		Two Step Mix Procedure for S2 (TESPD) and NXT Silanes	
Size "OOC" mixer – 4.2 L capacity, 70% FF		Size "OOC" mixer – 4.2 L capacity, 70% FF	
First Non-Productive Pass at 55 rpm, 70 °C		**First Non-Productive Pass at 55 rpm, 70 °C**	
$t_{0\ min}$	Add polymers, ram down mix (RDM) 30 seconds	$t_{0\ min}$	Add polymers, ram down mix (RDM) 30 seconds
$t_{0.5\ min}$	Add 50% silica, all silane, RDM 60 seconds	$t_{0.5\ min}$	Add 50% silica, all silane, RDM 60 seconds
$t_{1.5\ min}$	Add 50% silica, chemicals, RDM 60 seconds	$t_{1.5\ min}$	Add 50% silica, chemicals, RDM 60 seconds
$t_{2.5\ min}$	Add CB, oil at 125 °C	$t_{2.5\ min}$	Add CB, oil at 125 °C
$t_{2.5\ min}$	Reduce rotor speed to 35 rpm, RDM 90 seconds	$t_{2.5\ min}$	Reduce rotor speed to 35 rpm, RDM 90 seconds
$t_{4\ min}$	Sweep, RDM 120 seconds	$t_{4\ min}$	Sweep, RDM 120 seconds
$t_{4\ min}$	Hold temperature at 135-140 °C for 2 minutes	$t_{4\ min}$	Hold temperature at 145-150 °C for 2 minutes
$t_{6\ min}$	Discharge at 145 °C	$t_{6\ min}$	Discharge at 155 °C
Mill Blend, sheet off 80-90 °C roll mill, cool to near room temperature		Mill Blend, sheet off 80-90 °C roll mill, cool to near room temperature	
Second Non-Productive Pass at 50 rpm, 70 °C		**Second Non-Productive Pass at 50 rpm, 70 °C**	
$t_{0\ min}$	Add compound from first pass, RDM 60 seconds	$t_{0\ min}$	Add compound from first pass, RDM 60 seconds
$t_{1\ min}$	Add remainder of ingredients, RDM 60 seconds	$t_{1\ min}$	Add remainder of ingredients, RDM 60 seconds
$t_{2\ min}$	Sweep, RDM 120 seconds	$t_{2\ min}$	Sweep, RDM 120 seconds
$t_{2\ min}$	Hold temperature at 135-140 °C for 2 minutes	$t_{2\ min}$	Hold temperature at 145-150 °C for 2 minutes
$t_{4\ min}$	Discharge at 145 °C	$t_{4\ min}$	Discharge at 155 °C
Mill Blend, sheet off 80-90 °C roll mill, cool to near room temperature		Mill Blend, sheet off 80-90 °C roll mill, cool to near room temperature	
Final Productive Pass at 45 rpm, 50 °C		**Final Productive Pass at 45 rpm, 50 °C**	
$t_{0\ min}$	Add compound from second pass, cure package RDM 60 seconds	$t_{0\ min}$	Add compound from second pass, cure package RDM 60 seconds
$t_{1\ min}$	Sweep, RDM 90 seconds	$t_{1\ min}$	Sweep, RDM 90 seconds
$t_{1\ min}$	Hold temperature at 90-95 °C for 1.5 minutes	$t_{1\ min}$	Hold temperature at 90-95 °C for 1.5 minutes
$t_{2.5\ min}$	Discharge at 100-105 °C	$t_{2.5\ min}$	Discharge at 100-105 °C
Mill Blend, sheet off 80-90 °C roll mill, cool to near room temperature		Mill Blend, sheet off 80-90 °C roll mill, cool to near room temperature	

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Potential Applications (continued)

Table 3: Comparison of Rubber Compounds Containing NXT Silane, Standard S2 (TESPD) Silane or Standard S4 (TESPT) Silane

Silane	S2	S4	NXT Silane	NXT Silane
Silane phr	**5.65**	**6.41**	**6.4**	**6.4**
Hardness Adjustment			None	+5 phr silica
Number of Mixing Steps	2	2	2	2
Reaction Temperature	155	145	160	160
Mooney Viscosity at 100 °C				
ML (1+4)	87	87	77	79
Mooney Scorch at 135 °C				
MS1+, t3, minutes	15	12	14	14
Rheometer (160 °C, 30 Mins.)				
ML, dN-m	3.34	3.47	2.56	2.83
MH, dN-m	21.98	21.76	20.60	22.24
Δ Torque, dN-m	18.64	18.29	18.04	19.41
T90, minutes	18.00	18.28	16.19	17.29
Dynamic Properties				
Strain Sweep at 55 °C				
E′ max	12.8	12.8	12.3	13.8
E′ min	5.3	5.1	5.6	5.8
E′ mean	8.8	8.6	8.8	9.4
E′ initial (MPa)	12.83	12.78	12.29	13.77
ΔE′ (MPa)	7.51	7.67	6.72	7.94
E″ max (MPa)	2.05	2.05	1.68	1.98
Tan δ max	0.22	0.22	0.19	0.20
Interaction Parameter (IP)	100	88	115	102
Physical Properties at 25 °C				
Shore A at RT	71	69	68	71
Shore A at 70 °C	67	65	65	67
Elongation (%)	362	384	371	358
Tensile Strength (MPa)	14.5	14.2	15.5	15.5
50% Modulus (MPa)	1.5	1.5	1.6	1.7
100% Modulus (MPa)	2.6	2.4	2.9	3.0
300% Modulus (MPa)	12.7	11.5	13.3	13.9
M_{300} - M_{100} (MPa)	10.1	9.1	10.4	10.9
RI (M_{300}/M_{100})	4.9	4.8	4.6	4.6
Break Energy (J/cm^3)	20	21	23	22
Rebound				
0°C (%)	9	9	7	8
RT (%)	18	19	19	18
70°C (%)	39	41	43	41
Temp Sweep at 0.5% Strain				
Tan δ at Tg	0.837	0.880	0.889	0.891
Tan δ at 0 °C	0.816	0.801	0.860	0.865
Tan δ at 60 °C	0.203	0.208	0.170	0.189
E* at 25 °C	21	20	18	20

Note: Test data. Actual results may vary.

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Potential Applications (continued)

All figures shown below compare rubber compounds containing S2 (TESPD) silane, S4 (TESPT) silane and NXT silane

Figure 1: Comparison of Batch Viscosities

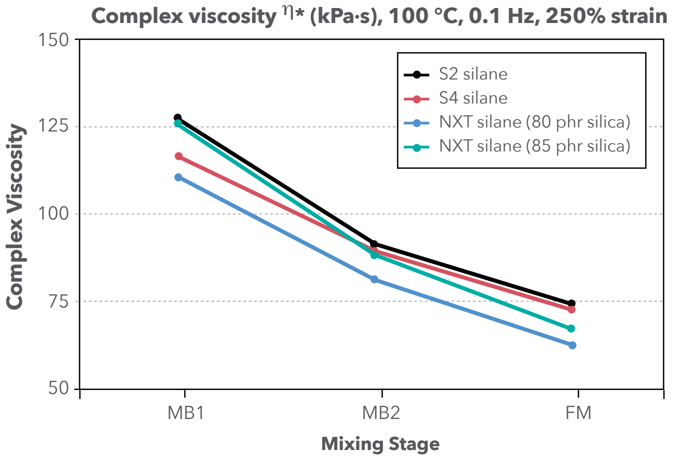


Figure 2: Cure Curve Comparison MDR at 160 °C

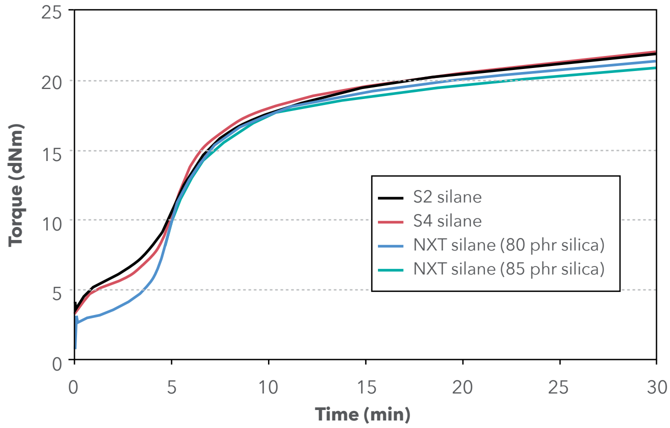
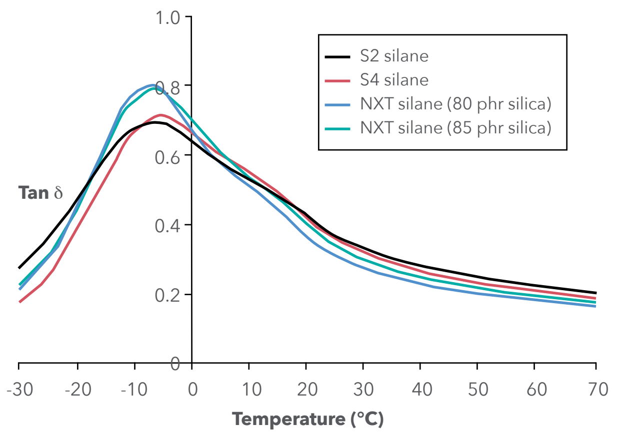


Figure 3: Temperature Sweep, 10.0 Hz, at 0.5% Strain



Note: Test data. Actual results may vary.

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Potential Applications (continued)

Figure 4: Low Viscosity and Improved Storage Stability with NXT Silane

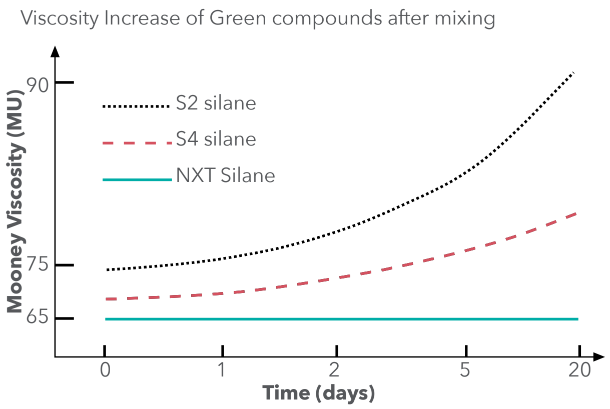


Figure 5: Strain Sweep, 10.0 Hz, at 55 °C

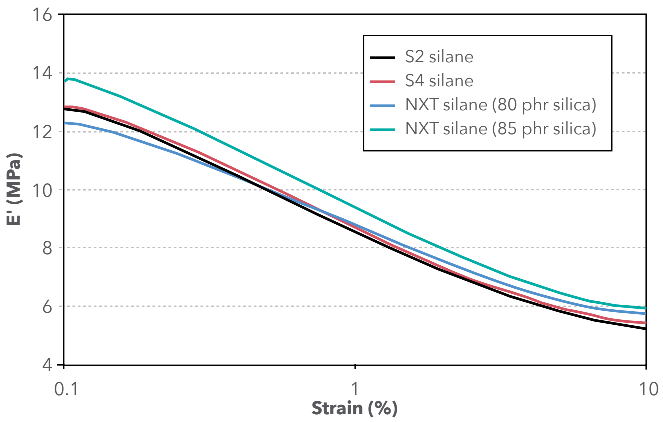


Figure 7: E" Values, 10.0 Hz, at 55 °C

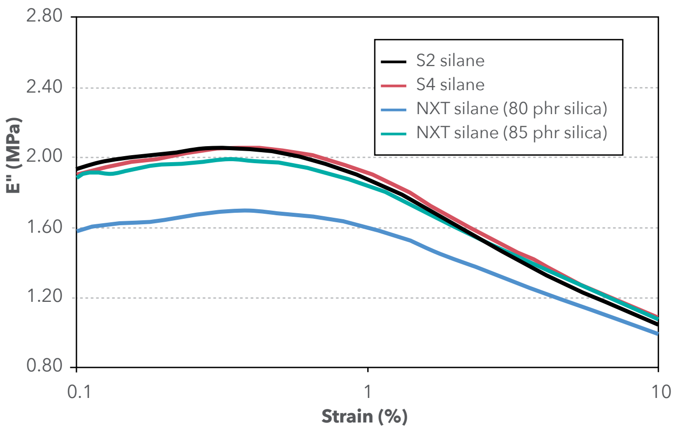
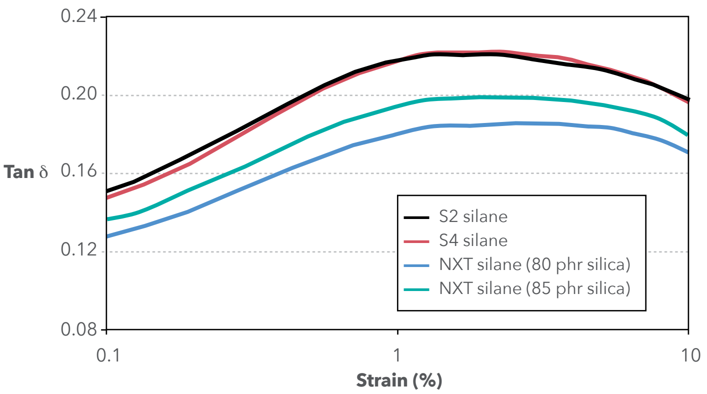


Figure 6: Tan δ Max Values, 10.0 Hz, at 55 °C



Note: Test data. Actual results may vary.

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General Instructions for Use

Please refer to Tables 1 and 2 for details regarding example formulation and mixing procedures using NXT silane.

Dynamic properties with NXT silane compounds are typically superior to standard S2 (TESPD) and S4 (TESPT) silane compounds in the 60-110 phr equivalent silica loading range. At 80 phr silica loading, 6.41 phr of standard S4 (TESPT) silane typically produces 35% more VOCs than 6.4 phr of NXT silane.

Maximum coupling strength can be achieved when there is an 8% loading of NXT silane based on the phr loading of silica. That is equivalent to 6.4-6.8 phr of NXT silane in an 80-85 phr silica formulation, respectively. NXT silane coupling strength was improved with higher mixing temperatures and higher vinyl content of the SBR polymer. In formulations in which the Shore A Hardness is slightly less than the control compound, 1-5 phr of silica can be added to increase hardness without reducing physical or dynamic properties.

NXT silane's reaction mechanism allowed non-conventional solutions for common compound challenges. Modulus 300% may be increased by adding silane, silica, and stearic acid in the first non-productive mix, while adding zinc oxide in the second non-productive mix. The accelerator, DPG (0.5-1.0 phr), can be added late in the second non-productive mix to increase bound rubber content. Compound viscosity can be reduced substantially by adding 0.5-1.0 phr of CBS in the second non-productive mix.

Patent Status

Nothing contained herein shall be construed to imply the nonexistence of any relevant patents or to constitute the permission, inducement or recommendation to practice any invention covered by any patent, without authority from the owner of the patent.

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Customers must evaluate Momentive Performance Materials products and make their own determination as to fitness of use in their particular applications.

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