Producers of Specialty Chemicals

Struktol Company of America

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# Struktol Rubber Lab Project

## The Effects of STRUKTOL® JV 46F
## and
## STRUKTOL® SCA 985 Silane
## in a
## Natural Rubber Off-The-Road
## Tire Formulation

April 2007

Project 07019-pad-n

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CERTIFICATE NUMBER 94.109 CERTIFIED ISO 9001 STRUKTOL COMPANY of AMERICA

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# Formulae

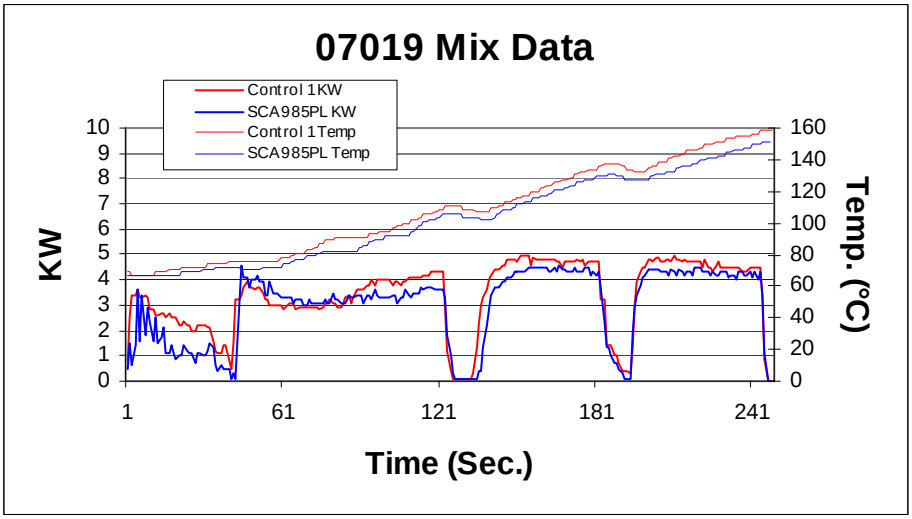
| Ingredient | Control | SCA 985 PL | JV 46F 2.5 | JV 46F 2.5 SCA 985PL 2.0 | JV 46F 5 | JV 46F, SCA 985PL 2 |
|---|---|---|---|---|---|---|
| SMR 5 | 100 | 100 | 100 | 100 | 100 | 100 |
| STRUKTOL® A 86 | .25 | .25 | .25 | .25 | .25 | .25 |
| N220 | 40 | 40 | 40 | 40 | 40 | 40 |
| SILICA VN 3 | 20 | 20 | 20 | 20 | 20 | 20 |
| ST ACID | 2.5 | 2.5 | 2.5 | 2.5 | 2.5 | 2.5 |
| ZnO | 4 | 4 | 4 | 4 | 4 | 4 |
| 6PPD | 2.5 | 2.5 | 2.5 | 2.5 | 2.5 | 2.5 |
| TMQ | 1 | 1 | 1 | 1 | 1 | 1 |
| STRUKTOL® 40 MS | 6 | 6 | 6 | 6 | 6 | 6 |
| STRUKTOL® SCA 985PL | 0 | 2 | 0 | 2 | 0 | 2 |
| STRUKTOL® JV 46F | 0 | 0 | 2.5 | 2.5 | 5 | 5 |
| **TOTAL** | **176.25** | **178.25** | **178.75** | **180.75** | **181.25** | **183.25** |
| 2ND PASS | | | | | | |
| TBBS | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 |
| SULFUR | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 |
| **TOTAL** | 179.25 | 181.25 | 181.75 | 183.75 | 184.25 | 186.25 |

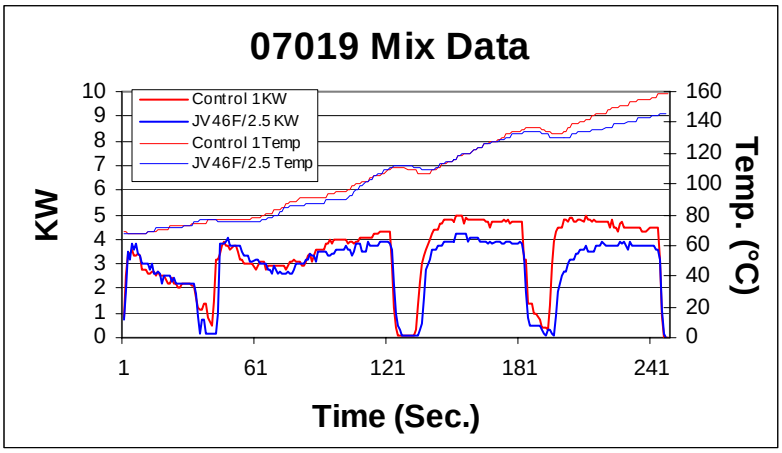
1st PASS MIX – ROTOR SPEED – 77 – RAM PRESSURE 30 – FILL FACTOR 70%
0 SECONDS ADD RUBBER AND PEPTIZER
30 SECONDS ADD BLACK, SILICA, ZINC OXIDE, 6PPD, TMQ AND ADDITIVES
120 SECONDS BRUSH AND SWEEP
180 SECONDS BRUSH AND SWEEP
240 SECONDS DISCHARGE

2nd PASS MIX SPEC – ROTOR SPEED 77 – RAM PRESSURE 30
0 SECONDS LOAD ½ MB, ADD CURES, REST MB
30 SECONDS BRUSH AND SWEEP
120 SECONDS OR 212F DISCHARGE

# Mixing Observations

| Compound | Temp (°C) | Probe Temp(°C) | Energy (WH) | COMMENTS 1ST PASS | COMMENTS 2ND PASS |
|---|---|---|---|---|---|
| CONTROL 1 | 157 | 178.2 | 232 | Dusty/rough/lacy/ Batch ok | Very stiff nearly stalled the banbury/hard to cut |
| SCA 985PL | 150 | 172.5 | 203 | Dusty/rough/lacy/ Batch ok | Door would not open/got to 250F/stayed in banbury longer |
| JV 46F 2.5 | 145 | 165.2 | 195 | Dusty/rough/lacy/ Batch ok | Batch ok/very hard to cut |
| JV 46F 2.5 SCA 985PL 2.0 | 146 | 162.6 | 194 | Not as dusty/not as rough/ Batch ok | Batch ok/very hard to cut |
| JV 46F 5 | 147 | 158.3 | 190 | Not as dusty/smoother than 4/ Batch ok | Batch ok/very hard to cut |
| JV 46F 5 SCA 985PL 2 | 143 | 156.8 | 184 | Not as dusty/same as 5/ Batch ok | Batch ok/very hard to cut |

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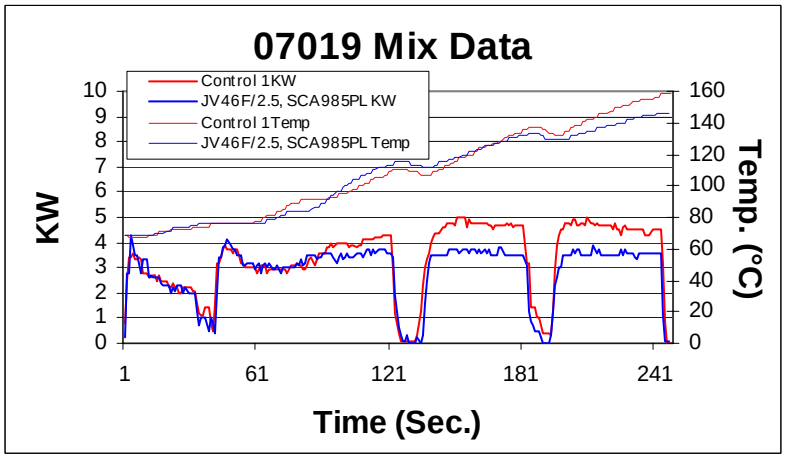
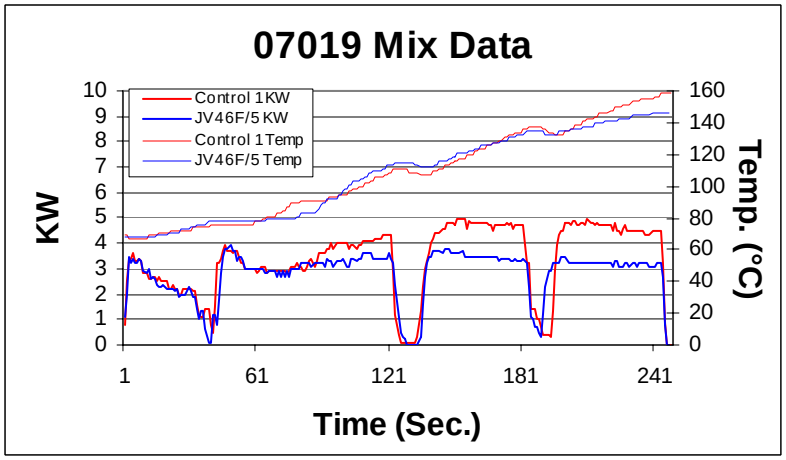
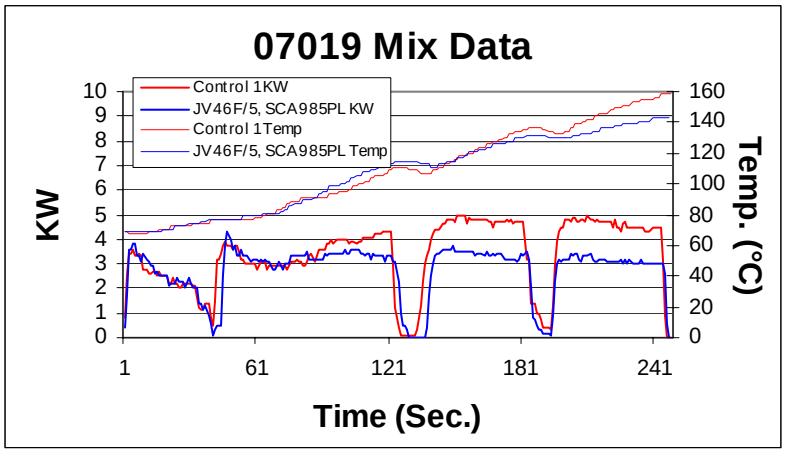
Control 1KW
JV46F/2.5 KW
Control 1Temp
JV46F/2.5 Temp
KW
Temp. (°C)
Time (Sec.)
0
1
2
3
4
5
6
7
8
9
10
0
20
40
60
80
100
120
140
160
1
61
121
181
241


07019 Mix Data
Control 1KW
JV46F/2.5, SCA985PL KW
Control 1Temp
JV46F/2.5, SCA985PL Temp
KW
Temp. (°C)
Time (Sec.)
0
1
2
3
4
5
6
7
8
9
10
0
20
40
60
80
100
120
140
160
1
61
121
181
241

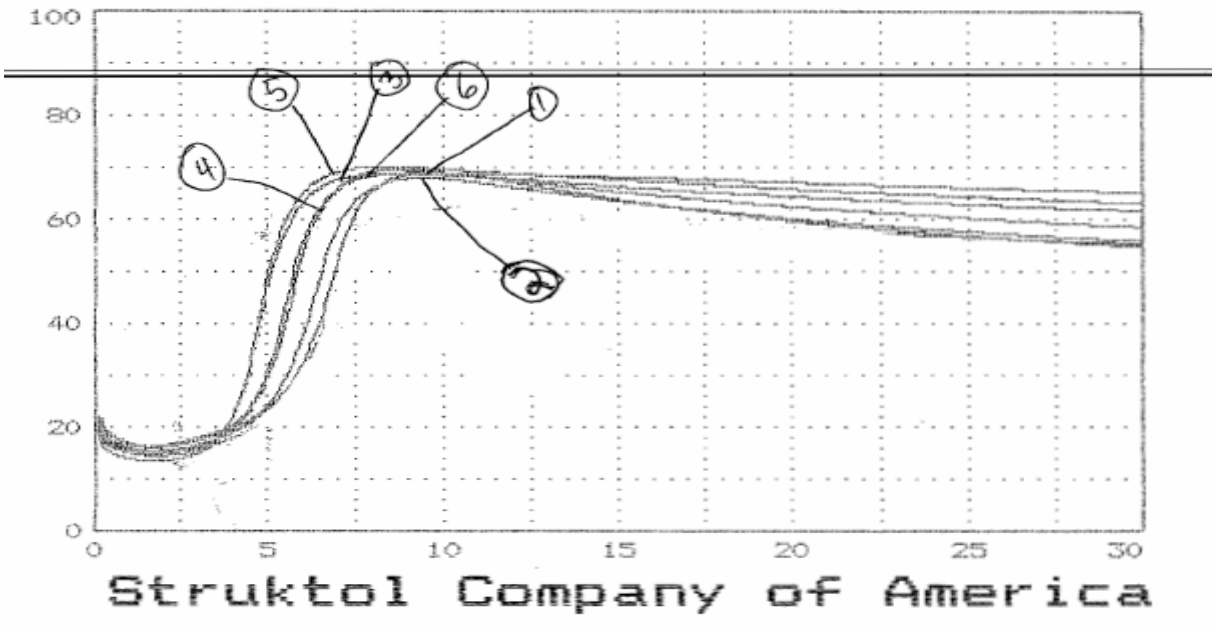

07019 Mix Data
Control 1KW
JV46F/5 KW
Control 1Temp
JV46F/5 Temp
KW
Temp. (°C)
Time (Sec.)
0
1
2
3
4
5
6
7
8
9
10
0
20
40
60
80
100
120
140
160
1
61
121
181
241




# Rheometer Data

160°C; 3° arc; 100 range; 30 minutes – Tech Pro

| Compound | Min Torque | Max Torque | $Ts_2$ | $T_{50}$ | $T_{90}$ | T-2 Reversion |
|---|---|---|---|---|---|---|
| CONTROL 1 | 16.16 | 69.05 | 3.17 | 6.63 | 7.79 | 12.13 |
| SCA 985PL | 14.83 | 67.96 | 3.21 | 6.21 | 7.42 | 12.25 |
| JV 46F 2.5 | 16.10 | 69.68 | 3.38 | 5.67 | 6.79 | 11.83 |
| JV 46F 2.5, SCA 985PL 2.0 | 15.35 | 68.88 | 3.42 | 5.50 | 6.67 | 13.83 |
| JV 46F 5 | 13.51 | 70.03 | 3.21 | 4.79 | 5.96 | 14.08 |
| JV 46F 5, SCA 985PL 2 | 14.49 | 69.05 | 3.29 | 4.83 | 6.08 | 20.25 |



# Mooney Viscosity

ML (1+4) @ 100°C

| Compound | Initial Viscosity | ML (1+4) | Initial Viscosity 2 weeks age | ML (1+4) | Initial Viscosity 4 weeks age | ML (1+4) |
|---|---|---|---|---|---|---|
| CONTROL 1 | 104.3 | 78.4 | 119.8 | 80.5 | 141.9 | 79.9 |
| SCA 985PL | 93.5 | 69.2 | 102.7 | 70.7 | 121.4 | 72.6 |
| JV 46F 2.5 | 112.4 | 72.0 | 121.8 | 73.5 | 123.7 | 72.8 |
| JV 46F 2.5, SCA 985PL 2.0 | 99.4 | 68.6 | 109.2 | 69.5 | 130.7 | 71.1 |
| JV 46F 5 | 95.0 | 62.5 | 98.4 | 62.9 | 112.5 | 61.3 |
| JV 46F 5, SCA 985PL 2 | 94.6 | 63.1 | 102.1 | 62.8 | 119.2 | 64.5 |

# Mooney Scorch

ML @ 125°C

| Compound | Min Torque | $T_5$ | $T_{35}$ | Cure Index |
|---|---|---|---|---|
| CONTROL 1 | 62.8 | 21.48 | 39.03 | 17.55 |
| SCA 985PL | 59.2 | 20.99 | 35.60 | 14.61 |
| JV 46F 2.5 | 63.0 | 22.02 | 28.87 | 6.85 |
| JV 46F 2.5, SCA 985PL 2.0 | 63.1 | 22.19 | 28.09 | 5.90 |
| JV 46F 5 | 55.0 | 18.70 | 23.01 | 4.31 |
| JV 46F 5, SCA 985PL 2 | 58.1 | 18.86 | 22.58 | 3.72 |

# Tensile Data

Unaged

| Compound | Cure time @ 160°C | Shore A Duro | Tensile (MPa) | Elongation (%) | 100% Mod. (MPa) | 200% Mod. (MPa) | 300% Mod. (MPa) |
|---|---|---|---|---|---|---|---|
| CONTROL 1 | 7.5 | 64 | 25.1 | 559 | 2.3 | 6.3 | 11.8 |
| SCA 985PL | 7.5 | 64 | 25.3 | 563 | 2.3 | 6.3 | 11.7 |
| JV 46F 2.5 | 7.5 | 65 | 26.5 | 570 | 2.5 | 6.3 | 11.7 |
| JV 46F 2.5, SCA 985PL 2.0 | 7.5 | 67 | 26.7 | 560 | 2.6 | 6.7 | 12.2 |
| JV 46F 5 | 7.5 | 67 | 25.2 | 539 | 2.5 | 6.2 | 11.5 |
| JV 46F 5, SCA 985PL 2 | 7.5 | 67 | 25.5 | 546 | 2.7 | 6.6 | 11.9 |

# Tear Strength

ASTM D – 624 Die C  N/MM

| Compound | 23ºC |
|---|---|
| CONTROL 1 | 138.3 |
| SCA 985PL | 123.1 |
| JV 46F 2.5 | 124.4 |
| JV 46F 2.5, SCA 985PL 2.0 | 122.8 |
| JV 46F 5 | 127.3 |
| JV 46F 5, SCA 985PL 2 | 136.3 |

# Rebound

Cured at 160ºC

| Compound | Cure | 0ºC | 23ºC | 100ºC |
|---|---|---|---|---|
| CONTROL 1 | 15 | 21 | 32 | 52 |
| SCA 985PL | 15 | 21 | 34 | 54 |
| JV 46F 2.5 | 15 | 21 | 35 | 57 |
| JV 46F 2.5, SCA 985PL 2.0 | 15 | 19 | 33 | 56 |
| JV 46F 5 | 15 | 21 | 34 | 58 |
| JV 46F 5, SCA 985PL 2 | 15 | 21 | 34 | 59 |

# Oven Aged

70 Hrs @ 100ºC

| Sample | Shore A | Points Cchange | Tensile (Mpa) | Pct. Change | Elongation % | Pct. Change | 100% Mod. (Mpa) | Pct. Change | 200% Mod. Mpa | Pct. Change | 300% Mod. (Mpa) | Pct. Change |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| CONTROL 1 | 73 | 9 | 23.4 | -6.8 | 440 | -21.3 | 5.4 | 134.8 | 12.1 | 92.1 | 18.3 | 55.1 |
| SCA 985PL | 74 | 10 | 23.9 | -5.5 | 434 | -22.9 | 5.6 | 143.5 | 12.4 | 96.8 | 18.6 | 59.0 |
| JV 46F 2.5 | 74 | 9 | 21.8 | -17.7 | 419 | -26.5 | 5.1 | 104.0 | 11.0 | 74.6 | 17.0 | 45.3 |
| JV 46F 2.5 SCA 985PL 2.0 | 74 | 7 | 23.3 | -12.7 | 418 | -25.4 | 5.6 | 115.4 | 12.0 | 79.1 | 18.2 | 49.2 |
| JV 46F 5 | 74 | 7 | 22.2 | -11.9 | 409 | -24.1 | 5.6 | 124.0 | 11.3 | 82.3 | 17.3 | 50.4 |
| JV 46F 5 SCA 985PL 2 | 74 | 7 | 22.6 | -11.4 | 418 | -23.4 | 5.5 | 103.7 | 11.5 | 74.2 | 17.3 | 45.4 |

# Heat Build Up

250 lb. Weight; 0.325” Throw; 45 minute test

| Compound | Time |
|---|---|
| CONTROL 1 | 137.2 |
| SCA 985PL | 137.0 |
| JV 46F 2.5 | 127.9 |
| JV 46F 2.5, SCA 985PL 2.0 | 127.8 |
| JV 46F 5 | 122.5 |
| JV 46F 5, SCA 985PL 2 | 117.9 |

# Spiral Mold Flow

Cured at 160ºC

| Compound | Cure | Weight 1 | Weight 2 | Weight 3 | Average Weight |
|---|---|---|---|---|---|
| CONTROL 1 | 7.5 | 1.547 | 1.464 | 1.447 | 1.486 |
| SCA 985PL | 7.5 | 1.597 | 1.570 | 1.620 | 1.596 |
| JV 46F 2.5 | 7.5 | 1.578 | 1.564 | 1.487 | 1.543 |
| JV 46F 2.5<br>SCA 985PL 2.0 | 7.5 | 1.587 | 1.515 | 1.521 | 1.541 |
| JV 46F 5 | 7.5 | 1.689 | 1.581 | 1.618 | 1.629 |
| JV 46F 5<br>SCA 985PL 2 | 7.5 | 1.535 | 1.582 | 1.549 | 1.555 |

# MER

Tension/Compression/Cylindrical / 1 HZ

| Compound | Tan delta average of 3 / 23c | Tan delta average of 3 / 100c |
|---|---|---|
| CONTROL 1 | .152 | .125 |
| SCA 985PL | .157 | .118 |
| JV 46F 2.5 | .129 | .115 |
| JV 46F 2.5, SCA 985PL 2.0 | .129 | .104 |
| JV46F 5 | .120 | .100 |
| JV 46F 5, SCA 985PL 2 | .117 | .090 |

# Capillary Rheometer Data

100 C, 180 sec preheat
Die L/D ratio: 15:1: 90 entrance angle: 1.5 mm orifice

| | Apparent Stress (Pa) | Apparent Viscosity (Pa-s) | Apparent Stress (Pa) | Apparent Viscosity (Pa-s) |
|---|---|---|---|---|
| **Shear** | **500/s** | **500/s** | **1000/s** | **1000/s** |
| CONTROL 1 | 242,430 | 484.89 | 293,110 | 293.10 |
| SCA 985PL | 218,610 | 437.25 | 256,470 | 256.46 |
| JV 46F 2.5 | 205,180 | 410.38 | 244,870 | 244.86 |
| JV 46F 2.5, SCA 985PL 2.0 | 199,680 | 399.39 | 233,270 | 233.26 |
| JV 46F 5 | 183,810 | 367.63 | 218,000 | 217.99 |
| JV 46F 5, SCA 985PL 2 | 178,310 | 356.64 | 215,560 | 215.55 |

## Conclusions:

**Processing**

The results given in the Mixing Observations table and also the Mixing Curves for these batches show that the presence of JV46F in the recipe causes a dramatic reduction in Mixing Energy and also a considerable reduction in Dump Temperature.

It was also noted that the batches that contained 5 phr of JV 46F gave a smoother, less dusty sheet on the batch off mill.

The Mooney Viscosity figures show that the batches that contained 5 phr of JV 46F gave lower Initial Viscosity and lower ML(1+4) reading than the Control compound. Also the climb in Initial viscosity on storage was lower for those batches that contained JV46F.

**Physical properties**

JV 46F did not have any effect on the Ts2 reading on the OD Rheometer but did appear to reduce T90 slightly.

Physical properties such as tensile, modulus and tear were unaffected by the silane or the JV 46F.

On the Rebound test the compounds that contained JV 46F had slightly higher rebound than the Control compound at both 23ºC and at 100ºC.

On the MER tester the tan δ values at 23ºC of the compounds that contained JV 46F were slightly lower than the Control; whereas at 100ºC the tan δ values of the compounds that contained JV 46F (especially at the 5phr level) were significantly lower than the Control

The results on the Capillary Rheometer followed the same trends as were observed on Mooney Viscosity testing.

On the Spiral Flow test all of the additive combinations increased flow by a small amount.