Struktol Company of America
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Producers of Specialty Chemicals

High Performance Silica Tread

with

Next Generation

Struktol Process Additives

Struktol Rubber Lab Project 08044

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CERTIFICATE NUMBER 94.109 CERTIFIED ISO 9001 STRUKTOL COMPANY of AMERICA

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JV46F

•Blend of fatty acid derivatives
•DP 85C
•Zn ~5%

KK 49

•Blend of fatty acid derivatives
•DP 105C
•Zn 10%

CY 48

•Blend of fatty acid derivatives
•DP 97C
•Zn ~8%

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Outline

- Formulations
- Mix Spec
- Rheometer Data
- Physical Property Data
- Processability/Extrusion Data
- Conclusions
- High Surface Area Silica

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Formulations

First Pass	Control		Batch 1	Batch 2	Batch 3
INGREDIENT	MASS		MASS	MASS	MASS
DURADENE 751	103.5		103.5	103.5	103.5
TAKETENE 220	25		25	25	25
N220	10		10	10	10
SCA 985	5		5	5	5
ZnO	2.5		2.5	2.5	2.5
ST. ACID	2		2	2	2
6PPD	2		2	2	2
SUNDEX 790	5		5	5	5
ZS 1165MP	75		75	75	75
JV46F	0		4	0	0
KK 49	0		0	4	0
CY 48	0		0	0	4
Total	**230**		**234**	**234**	**234**

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Formulations

Final Pass	Control	Batch 1	Batch 2	Batch 3
MB control	230			
MB 1		234		
MB 2			234	
MB 3				234
DPG	1.4	1.4	1.4	1.4
CBS	1.7	1.7	1.7	1.7
SULFUR	1.2	1.2	1.2	1.2
Total	**234.3**	**238.3**	**238.3**	**238.3**

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Mix Spec

1st PASS MIX – ROTOR SPEED 120 FILL FACTOR 70% RAM PRESSURE 30 PSI

0 SECONDS ADD RUBBER
30 SECONDS ADD ½ SILICA AND SILANE
75 SECONDS ADD ½ SILICA AND OIL
120 SECONDS BRUSH AND SWEEP
135 SECONDS ADD BLACK, ZnO STEARIC ACID, ADDITIVES AND 6PPD
180 SECONDS BRUSH AND SWEEP
338 F AUTO RAM LIFT ROTOR SPEED CHANGE TO 58 RPM
700 SECONDS DISCHARGE

2ND PASS MIX SPEC ROTOR SPEED 77, FILL FACTOR 65%, RAM PRESSURE 30 PSI

0 SECONDS ADD ½ MB, CURES AND ½ MB
30 BRUSH AND SWEEP
120 SECONDS OR 212F DISCHARGE

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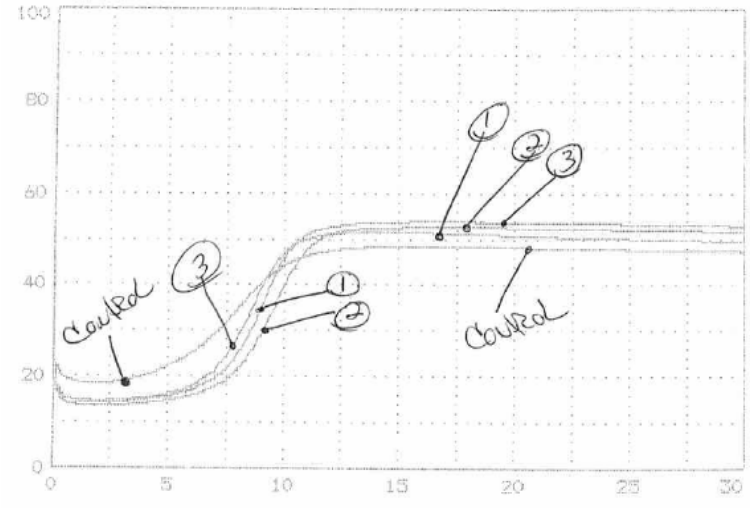
Rheometer Curves



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Rheometer Data

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

Compound	Min Torque	Max Torque	Ts_2	T_{50}	T_{90}
Control	18.34	48.44	4.38	7.92	10.67
Batch 1	14.55	51.58	5.50	8.58	10.38
Batch 2	13.45	52.69	5.96	9.50	11.42
Batch 3	14.26	53.91	5.71	9.04	10.83

Higher cure state with all additives
Lower minimum torque with all additives
Increased scorch safety with all additives
T90 approximately equal

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Mooney Viscosity

ML (1+4) @ 100°C

Compound	Initial Vis	ML (1+4)	3 DAY AGE	ML (1+4)	7 DAY AGE	ML(1+4)	14 DAY AGE	ML(1+4)	28 DAY AGE	ML(1+4)
Control	107.4	78.5	122.0	82.5	132.5	86.4	146.4	88.5	162.4	95.7
Batch 1	93.8	67.8	105.0	72.5	110.3	72.9	124.3	77.0	139.5	84.3
Batch 2	89.6	68.0	97.7	69.2	105.5	70.2	113.7	72.1	127.9	78.0
Batch 3	98.2	70.8	104.2	73.2	112.2	74.7	119.6	76.2	134.5	82.5

Decreased viscosity for better processability for all additives
Better viscosity control with time providing more stable stock storage

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Tensile Data

Unaged

Compound	Cure time @ 160°C	ShoreA Duro	Tensile (MPa)	Elongation (%)	100% Mod. (MPa)	200% Mod. (MPa)	300% Mod. (MPa)
Control	12	62	17.3	607	1.3	3.0	6.0
Batch 1	12	62	18.8	677	1.2	2.5	5.0
Batch 2	12	62	20.3	671	1.4	3.2	6.2
Batch 3	12	63	21.2	634	1.6	3.6	6.9

Equal to better tensile, elongation and modulus with KK 49 and CY 48
Better tensile and elongation with JV46F with slightly lower modulus

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Tear Strength ASTM D – 624 Die C N/MM

Compound	23ºC	100ºC
Control	39.8	40.5
Batch 1	49.6	55.3
Batch 2	53.0	54.9
Batch 3	52.6	49.8

Increased tear at room temperature and 100C with all additives

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MER

Tension/Compression/Cylindrical / 1 Hz

Compound	Tan delta/ average of 3 / 23c	Tan delta/ average of 3 / 100c
Control	.198	.140
Batch 1	.212	.145
Batch 2	.186	.142
Batch 3	.183	.140

Equivalent rolling resistance

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Firestone Flexometer

Heat Build-up

250 lb. Weight; 0.325" Throw; 45 min. test

Compound	Cure Time @ 160°C	Duro	Temp. °C
Control	48	59	159.3
Batch 1	48	59	153.3
Batch 2	48	62	144.5
Batch 3	48	62	148.7

Lower heat build-up for all additives

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Spiral Mold

Compound	Cure Time @ 320°F	1	2	3	Avg.
Control	12	1.658	1.633	1.597	1.629
Batch 1	12	1.830	1.881	1.863	1.858
Batch 2	12	1.952	1.906	1.907	1.922
Batch 3	12	1.806	1.860	1.725	1.797

Better flow characteristics with all additives

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Capillary Rheometer Data

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

Compound	Apparent Stress (Pa)	Apparent Viscosity (Pa-s)	Apparent Stress (Pa)	Apparent Viscosity (Pa-s)
	500/s	500/s	1000/s	1000/s
Control	271,130	542.29	305,940	305.92
Batch 1	243,650	487.33	279,070	279.05
Batch 2	235,710	471.45	269,910	269.89
Batch 3	244,870	489.77	280,290	280.27

Reduced apparent viscosity with all additives
Better extrudability
Less scrap/workaway

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Conclusions

- All additives provide increased cure state and increased scorch safety.
- Reduced viscosity for better processability.
- Better viscosity control with time.
- Superior tensile, elongation and modulus.
- Equivalent rolling resistance.
- Lower heat build-up.
- Flow and extrusion characteristics improved.

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High Surface Area Silica

First Pass	**Control**	**Batch 1**	**Batch 2**	**Batch 3**
INGREDIENT	**MASS**	**MASS**	**MASS**	**MASS**
DURADENE 751	103.5	103.5	103.5	103.5
TAKTENE 220	25	25	25	25
N220	10	10	10	10
SCA 985	5	5	5	5
ZnO	2.5	2.5	2.5	2.5
St ACID	2	2	2	2
6PPD	2	2	2	2
SUNDEX 790	5	5	5	5
ZS P200 MP	75	75	75	75
JV46F	0	4	0	0
KK 49	0	0	4	0
CY 48	0	0	0	4
Total	**230**	**234**	**234**	**234**

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High Surface Area Silica

Final Pass	Control	Batch 1	Batch 2	Batch 3
MB Control	230			
MB 1		234		
MB 2			234	
MB 3				234
DPG	1.4	1.4	1.4	1.4
CBS	1.7	1.7	1.7	1.7
SULFUR	1.2	1.2	1.2	1.2
Total	**234.3**	**238.3**	**238.3**	**238.3**

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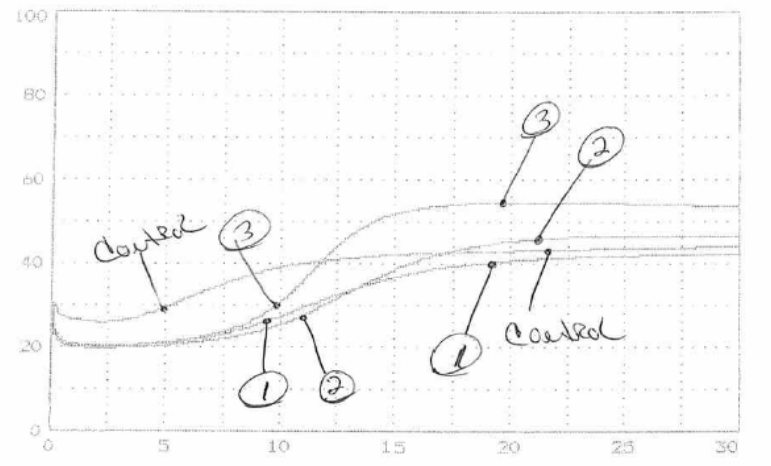
Rheometer Curves



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Rheometer Data

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

Compound	Min Torque	Max Torque	Ts_2	T_{50}	T_{90}
Control	25.73	43.95	4.38	7.58	15.58
Batch 1	20.14	41.97	6.96	12.00	18.50
Batch 2	19.74	46.57	7.38	13.21	18.46
Batch 3	20.20	54.55	6.63	11.42	14.79

(Compared to ZS 1165 MP)
Significantly reduced cure amount
Longer cure times
High surface area silica adsorbing more of the curatives/additives

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Mooney Viscosity

ML (1+4) @ 100°C

Compound	Initial Vis	ML (1+4)	3 DAY AGE	ML (1+4)	7 DAY AGE	ML (1+4)	14 DAY AGE	ML (1+4)	28 DAY AGE	ML (1+4)
Control	137.5	102.5	154.3	106.9	156.8	112.2	175.0	117.3	198.4	122.8
Batch 1	116.2	87.1	126.2	89.6	132.2	92.4	141.9	97.7	159.1	105.6
Batch 2	119.3	89.1	130.2	92.1	136.8	93.9	147.0	98.9	164.6	106.1
Batch 3	119.3	89.7	131.8	94.2	145.6	98.2	157.6	105.4	170.9	110.5

Decreased viscosity for better processability for all additives
Better viscosity control with time providing more stable stock storage

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Tensile Data

Unaged

Compound	Cure time @ 160°C	Shore A Duro	Tensile (MPa)	Elongation (%)	100% Mod. (MPa)	200% Mod. (MPa)	300% Mod. (MPa)
Control	16	61	6.4	406	1.3	2.7	4.5
Batch 1	19	62	12.4	644	1.1	2.2	3.8
Batch 2	19	62	13.3	630	1.3	2.7	4.6
Batch 3	16	62	18.0	579	1.8	3.9	6.9

(Compared to ZS 1165 MP)
Significantly lower tensile and modulus values
Curatives and additives being adsorbed on silica
Poor dispersion?

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Tear Strength ASTM D – 624 Die C N/MM

Compound	23ºC	100ºC
Control	22.1	19.7
Batch 1	35.7	42.0
Batch 2	37.3	41.6
Batch 3	44.9	49.8

Increased tear at room temperature and 100C with all additives

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MER

Tension/Compression/Cylindrical / 1 HZ

Compound	Tan delta/ Average of 3 / 23c	Tan delta/ Average of 3 /100c
Control	.208	.166
Batch 1	.206	.161
Batch 2	.202	.163
Batch 3	.202	.168

Equivalent rolling resistance

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Firestone Flexometer

Heat Build-up

250 lb. Weight; 0.325" Throw; 45 min. test

Compound	Cure Time @ 160°C	Duro	Temp. °C
Control	64	64	183.7
Batch 1	76	60	180.7
Batch 2	76	62	171.9
Batch 3	64	61	208.3

Lower heat build-up for all additives except Batch 3 (CY 48). This compound had very high modulus and tensile values as well as the highest cure amount.

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Spiral Mold

Compound	Cure Time @ 320°F	1	2	3	Avg.
Control	16	1.491	1.559	1.547	1.532
Batch 1	19	1.748	1.742	1.648	1.713
Batch 2	19	1.657	1.661	1.644	1.654
Batch 3	16	1.649	1.604	1.591	1.615

Better flow characteristics with all additives

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Capillary Rheometer Data

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

Compound	Apparent Stress (Pa)	Apparent Viscosity (Pa-s)	Apparent Stress (Pa)	Apparent Viscosity (Pa-s)
	500/s	**500/s**	**1000/s**	**1000/s**
Control	291,280	582.60	320,590	320.57
Batch 1	265,630	531.30	298,610	298.59
Batch 2	286,390	572.83	315,710	315.69
Batch 3	285,780	571.60	313,870	313.86

Slightly reduced apparent viscosity with all additives
Slightly better extrudability

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Three Pass Mix for High Surface Area Silica

- Low cure amount with 2 pass mix
- Longer cure times
- Lower modulus
- Possible poor dispersion

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Mix Spec

1st pass mix spec (65 rpm, fill factor 70%)
0 SECONDS ADD RUBBER
30 SECONDS ADD 1/3 SILICA
60 SECONDS ADD 1/3 SILICA
90 SECONDS ADD SILANE, OIL, CARBON BLACK, ADDITIVES AND REST OF SILICA
180 SECONDS OR 266F RA LIFT
300 SECONDS OR 302F BRUSH AND SWEEP
420 SECONDS OR 320FDISCHARGE
ROTOR SPEED 65 RPM AT DISCHARGE 30 PSI

2nd pass mix spec for batch rotor speed started at 110 rpm, at 320F changed rotor speed to 60 rpm fill factor 70%)
0 SECONDS ADD all MB
320F AUTO RAM LIFT, CHANGE ROTOR SPEED TO 60 RPM
DISCHARGE 7 MINUTES AFTER ROTOR SPEED

Mix spec final pass for batch (65 % fill factor)
0 SECONDS ADD ALL RUBBER
15 SECONDS ADD CURES
60 SECONDS BRUSH AND SWEEP
150 SECONDS OR 226F DISCHARGE
ROTOR SPEED 77 RAM PRESSURE 30 PSI

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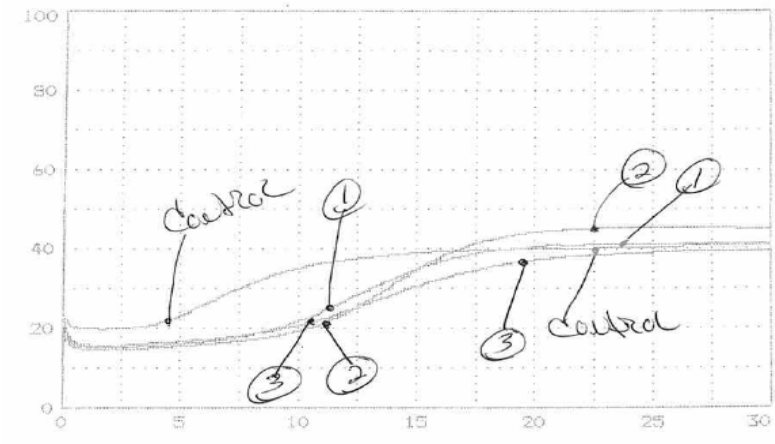
Rheometer Curves



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Rheometer Data

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

Compound	Min Torque	Max Torque	Ts_2	T_{50}	T_{90}
Control	19.79	40.87	4.58	7.83	15.17
Batch 1	15.19	41.33	6.96	12.67	17.75
Batch 2	14.61	45.18	7.38	13.92	18.25
Batch 3	15.72	39.41	7.17	13.79	20.25

Higher cure amount than 2 pass mix except for Batch 3 (CY 48)
T90s approximately equal to 2 pass mix except for Batch 3 (CY 48) significantly longer
Reduced minimum torque with all additives

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Mooney Viscosity

ML (1+4) @ 100°C

Compound	Initial Vis	ML (1+4)	3 DAY AGE	ML (1+4)	7 DAY AGE	ML (1+4)	14 DAY AGE	ML (1+4)	28 DAY AGE	ML (1+4)
Control	112.4	81.4	120.5	83.6	86.6	86.7	136.6	91.5	150.6	99.7
Batch 1	95.8	72.3	98.8	72.4	74.6	74.6	111.8	78.1	123.3	83.8
Batch 2	95.2	72.2	97.9	73.0	73.6	73.7	114.1	78.2	123.2	82.4
Batch 3	98.9	75.1	104.2	75.3	77.3	77.3	117.7	80.6	127.3	87.6

Decreased viscosity for better processability for all additives
Better viscosity control with time providing more stable stock storage

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Tear Strength ASTM D – 624 Die C N/MM

Compound	23ºC	100ºC
Control	27.1	25.7
Batch 1	44.7	64.9
Batch 2	48.3	81.0
Batch 3	44.2	64.8

Increased tear at room temperature and 100C with all additives

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Spiral Mold

Compound	Cure Time @ 320°F	1	2	3	Avg.
Control	18.5	1.665	1.659	1.687	1.670
Batch 1	18.5	1.839	1.868	1.755	1.821
Batch 2	18.5	1.811	1.812	1.865	1.829
Batch 3	20.5	1.813	1.723	1.791	1.776

Better flow characteristics with all additives

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Capillary Rheometer Data

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

Compound	Apparent Stress (Pa)	Apparent Viscosity (Pa-s)	Apparent Stress (Pa)	Apparent Viscosity (Pa-s)
	500/s	500/s	1000/s	1000/s
Control	286,390	572.83	312,650	312.64
Batch 1	253,420	506.87	285,780	285.77
Batch 2	255,250	510.54	284,560	284.55
Batch 3	258,920	517.86	287,620	287.60

Reduced apparent viscosity with all additives
Better extrudability

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Conclusions

- Lower cure amount with high surface area silica.
- Longer cure times with high surface area silica due to cure and additives adsorbing onto silica.
- Significantly lower modulus values than would be expected for a high surface area silica.
- Lab program to evaluate increased cure/additive package has been initiated.

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