Millathane® 5004

A HIGH PERFORMANCE MILLABLE POLYESTER URETHANE RUBBER

(Formerly known as Vibrathane® 5004)

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TSE INDUSTRIES, INC.

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CLEARWATER, FLORIDA

For the past forty years, TSE Industries has been an industry leader in new, innovative solutions for the rubber industry. Our millable polyurethane gums, sold under the trade name **MILLATHANE**® have grown to be the sales leaders in the world today.

TSE Industries has recently completed its Phase Five Expansion encompassing 300,000 square feet spread over twenty acres in Clearwater, Florida. A large part of this is dedicated to significantly expanding our Research and Development efforts so that we will remain the Technology Leaders into the 22nd Century.

TSE INDUSTRIES, INC.
4370 112th Terrace North, Clearwater, Florida 33762, USA
Tel: (727) 573-7676 (800) 237-7634
Fax: (727) 572-0487
www.TSE-INDUSTRIES.com www.MILLATHANE.com

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MILLATHANE® 5004*

A High Performance Millable Polyester Urethane Rubber

INTRODUCTION

MILLATHANE 5004 is a millable polyurethane elastomer having excellent processing characteristics. **MILLATHANE 5004** stocks can be readily processed on conventional rubber equipment. Vulcanization is carried out by the use of various peroxides including dicumyl peroxide (DCP) or 2,5-dimethyl-2,5-bis (t-butylperoxy) hexane (DBPH).

CURING MILLATHANE 5004

MILLATHANE 5004 compounds cured in the press reach optimum cure in 15 minutes at 320°F (160°C), or 30 to 45 minutes at 305°F (152°C) for thin gauge ASTM slabs 0.075″ (2mm) thick. Rate of curing may be accelerated to 3 to 4 minutes by curing at elevated temperatures of 350 to 400°F (179 to 204°C). The cured stock is a high quality product having good performance at low temperatures and good resistance to dry heat. The cured stock also has excellent abrasion and tear resistant qualities coupled with good stress-strain properties at elevated temperatures and excellent resistance to oxygen, ozone, fuels and oils.

MIXING MILLATHANE 5004

- Add **MILLATHANE 5004** and stearic acid to a Banbury at slow speed and mix for one minute.
- Add the carbon black and continue mixing for 3 minutes.
- The stock temperature will build up to around 230-240°F (110-116°C) during the mixing cycle.
- Discharge and cool the stock to room temperature conditions.
- Add Di-Cup 40C to the stock on a cool sheeting mill (120°F (49°C) maximum roll temperature).
- Avoid contamination by the commonly used rubber chemicals such as sulfur, TUEX, MONEX, MBT, MBTS, cumates, zimates, etc., which have negative effects on the cure.

*Formerly known as Vibrathane® 5004

TYPICAL POLYMER PROPERTIES

Chemical type	Polyester polyurethane
Physical form	Off-white chunks
Specific gravity	1.21
Mooney viscosity (ML 1+4 @ 100° C)	50 to 70
Storage stability	Excellent

TYPICAL PHYSICAL PROPERTIES

(20 pts. HAF Black)

Specific gravity	1.28
Mooney viscosity, (ML 1+4 @ 100° C)	70
Mooney scorch, (MS @ 121° C) t5, minutes	25
Shore A hardness	66
100% modulus, psi (MPa)	510 (3.5)
200% modulus, psi (MPa)	1420 (9.8)
300% modulus, psi (MPa)	2750 (19.0)
Tensile strength, psi (MPa)	4200 (28.9)
Elongation, %	400
Tear strength, ASTM Die C, pli (kN/m)	550 (96.3)
Compression set, Method B, 22 hrs. @ 158°F (70°C), %	20
Torsional hysteresis:	
At room temperature	0.126
At 285°F (141°C)	0.086
Bashore resilience,% rebound	47

LOW TEMPERATURE PROPERTIES

	Green Stocks	20 phr N330 (HAF) Black Stock
Bell Brittle Point °F (°C)	–	-75 (-59)
Temperatures of Retraction (°C)		
TR-10	-38.2	-33.4
TR-30	-35.5	-23.7
TR-50	-31.9	-11.0
TR-70	-24.0	-3.4
Gehman Modulus (°C)		
T_2	-30.7	-25.5
T_5	-33.9	-33.1
T_{10}	-35.7	-35.0
T_{100}	-40.0	-39.5

HEAT AGING PROPERTIES OF MILLATHANE® 5004

	Modulus At 300% psi (MPa)	Tensile psi (MPa)	Elongation (%)	Shore A Hardness
Original	2750 (19.0)	4200 (28.9)	400	66
Aged 70 hrs at 250°F (121°C)	2600 (17.9)	3950 (27.2)	420	62
Aged 70 hrs at 300°F (149°C)	1750 (12.1)	3050 (21.0)	490	62

FUEL RESISTANCE OF MILLATHANE 5004

	Modulus At 300% psi (MPa)	Tensile psi (MPa)	Elongation (%)	Shore A Hardness	Volume Change (%)
Original	2750 (19.0)	4200 (28.9)	400	66	–
Aged 7 days at Room Temp In ASTM Fuel A	2700 (18.6)	3750 (25.9)	370	66	+ 1.3
Aged 7 days at Room Temp In ASTM Fuel B	2400 (16.5)	2400 (16.5)	300	67	+16.0

OIL RESISTANCE OF MILLATHANE 5004

	Modulus At 300% psi (MPa)	Tensile psi (MPa)	Elongation (%)	Shore A Hardness	Volume Change (%)
Original	2750 (19.0)	4200 (28.9)	400	66	–
Aged 70 hours at 250°F (121°C) In ASTM Oil #901	2260 (15.6)	3300 (22.8)	420	67	- 0.67
Aged 70 hours at 250°F (121°C) In ASTM Oil #903	2280 (15.7)	3400 (23.4)	410	66	-1.03

OXYGEN AGING OF MILLATHANE 5004

	Modulus At 300% psi (MPa)	Tensile psi (MPa)	Elongation (%)	Shore A Hardness
Original	2750 (19.0)	4200 (28.9)	400	66
Aged 96 hrs. at 158°F (70°C)	2680 (18.5)	4250 (29.3)	470	66

EFFECT OF CONCENTRATION OF CURING AGENT (Di-Cup) ON PROPERTIES OF MILLATHANE® 5004

Recipe	1	2	3	4	5
MILLATHANE 5004	100	100	100	100	100
Stearic Acid	0.25	0.25	0.25	0.25	0.25
N330 (HAF) Black	20	20	20	20	20
Di-Cup 40C	3	4	5	6	8
Curing Conditions: 45 min. @ 305°F (152°C).					
Physical Properties					
Shore A Hardness	60	66	68	69	70
300% Modulus, psi (MPa)	1730 (11.9)	2750 (19.0)	3500 (24.1)	–	–
Tensile Strength, psi (MPa)	4430 (30.5)	4200 (28.9)	3800 (26.2)	3400 (23.4)	2800 (19.3)
Elongation, (%)	570	400	310	260	210
Tear Strength lbs/inch (ASTM Die C) (kN/m)	630 (110)	610 (107)	660 (116)	600 (105)	420 (74)

MILLATHANE 5004 CARBON BLACK (N330) STUDY

Recipe	1	2	3	4
MILLATHANE 5004	100	100	100	100
Stearic Acid	0.25	0.25	0.25	0.25
N330 (HAF) Black	20	40	60	80
Di-Cup 40C	4.0	4.6	5.3	6.0
Curing Conditions: 30 min. @ 305°F (152°C).				
Physical Properties				
Shore A Hardness	68	75	86	90
100% Modulus, psi (MPa)	600 (4.1)	1320 (9.1)	1440 (9.9)	1660 (11.4)
200% Modulus, psi (MPa)	1500 (10.3)	2840 (19.6)	3000 (20.7)	2310 (15.9)
300% Modulus, psi (MPa)	2800 (19.3)	3910 (26.9)	–	–
Tensile Strength, psi (MPa)	4600 (31.7)	4140 (28.5)	3610 (24.9)	2310 (15.9)
Elongation, (%)	460	340	280	200
Heat Aged 70 hours at 300°F (149°C)				
Shore A Hardness	64	80	89	98
100% Modulus, psi (MPa)	350 (2.4)	880 (6.1)	950 (6.6)	–
200% Modulus, psi (MPa)	950 (6.6)	1990 (13.7)	1900 (13.1)	–
300% Modulus, psi (MPa)	1830 (12.6)	–	–	–
Tensile Strength, psi (MPa)	2950 (20.6)	2760 (19.0)	2100 (14.5)	1450 (10.0)
Elongation, (%)	460	280	220	50
Heat Aged One Week at 300°F (149°C)				
Shore A Hardness	57	79	84	98
100% Modulus, psi (MPa)	210 (1.4)	620 (4.3)	700 (4.8)	–
200% Modulus, psi (MPa)	580 (4.0)	1190 (0.2)	1150 (7.9)	–
300% Modulus, psi (MPa)	1010 (9.0)	1590 (10.9)	–	–
Tensile Strength, psi (MPa)	1610 (11.1)	1600 (11.5)	1500 (10.3)	1420 (9.8)
Elongation, (%)	440	310	230	60

COMPOUNDING MILLATHANE® 5004 WITH HiSIL 233 AND Di-Cup 40C

Recipe	1	2	3
MILLATHANE 5004	100	100	100
Stearic Acid	0.25	0.25	0.25
HiSIL 233	20	20	20
Di-Cup 40C	3	4	6
Curing Conditions: 60 min. @ 305°F (152°C).			
Physical Properties			
Shore A Hardness	60	70	78
300% Modulus, psi (MPa)	850 (5.9)	1190 (8.2)	1850 (12.8)
Tensile Strength, psi (MPa)	3730 (25.7)	3660 (25.2)	3600 (24.8)
Elongation, (%)	650	590	460
Tear Strength lbs/inch (ASTM Die C), (kN/m)	350 (61.2)	400 (70.0)	440 (77.1)

COMPOUNDING MILLATHANE 5004 WITH HiSIL 233 AND VAROX DBPH-50

Recipe	1	2	3
MILLATHANE 5004	100	100	100
Stearic Acid	0.25	0.25	0.25
HiSil 233	20	20	20
Varox DBPH-50	3	4	6
Curing Conditions: 60 min. @ 305°F (152°C).			
Physical Properties			
Shore A Hardness	70	70	73
300% Modulus, psi (MPa)	1160 (8.0)	1460 (10.1)	2040 (14.1)
Tensile Strength, psi (MPa)	4170 (28.8)	4170 (28.8)	3210 (22.1)
Elongation, (%)	570	500	380
Tear Strength lbs/inch (ASTM Die C), (kN/m)	390 (68.3)	400 (70.0)	410 (71.8)

LOW MODULUS VULCANIZATES OF MILLATHANE 5004

Recipe	1	2	3	4
MILLATHANE 5004	100	100	100	100
Stearic Acid	0.25	0.25	0.25	0.25
N330 (HAF) Black	20	20	20	20
Di-Cup 40C	5	5	5	5
Tricresyl Phosphate	–	10	20	25
Curing Conditions: 60 min. @ 305°F (152°C).				
Physical Properties				
Shore A Hardness	68	65	59	56
300% Modulus, psi (MPa)	3500 (24.1)	2070 (14.3)	1260 (8.7)	1260 (8.7)
Tensile Strength, psi (MPa)	3800 (26.2)	3000 (20.7)	3240 (22.3)	3330 (22.9)
Elongation, (%)	310	370	540	530
Tear Strength lbs/inch (ASTM Die C), (kN/m)	660 (115.6)	560 (98.1)	500 (87.6)	480 (84.1)

RAPID CURING OF MILLATHANE® 5004

MILLATHANE 5004, cured for 3 minutes at 350°F (177°C), attains the same high quality properties as stocks cured for 45 minutes at 305°F (152°C). These are typical mechanical properties of rapidly cured millable gums.

TYPICAL MECHANICAL PROPERTIES OF BLACK FILLED MILLATHANE 5004 CURED AT 305°F (152°C), 350°F (177°C), and 400°F (204°C).

Recipe	Control	1	2	3	4
MILLATHANE 5004	100	100	100	100	100
Stearic Acid	0.25	0.25	0.25	0.25	0.25
N330 (HAF) Black	20	20	20	20	20
Di-Cup 40C	4	3	6	3	4
Curing Temperature °F, (°C)	305 (152°C)	350 (177°C)	350 (177°C)	400 (204°C)	400 (204°C)
Curing Time (minutes)	45	3	3	2	2
Mechanical Properties					
Shore A Hardness	68	68	73	65	70
100% Modulus, psi (MPa)	500 (3.4)	390 (2.7)	720 (5.0)	400 (2.8)	520 (3.6)
200% Modulus, psi (MPa)	1470 (10.1)	840 (5.8)	2000 (13.8)	970 (6.7)	1200 (8.3)
300% Modulus, psi (MPa)	2900 (20.0)	1490 (10.3)	3920 (27.0)	1830 (12.6)	2400 (16.5)
Tensile, psi (MPa)	4200 (28.9)	4200 (28.9)	4040 (27.9)	4170 (28.8)	4300 (29.6)
Elongation, (%)	400	660	310	560	450
Tear Strength lbs/inch (ASTM Die C) (kN/m)	550 (96.3)	500 (87.6)	470 (82.3)	570 (99.8)	540 (94.6)

RAPID CURING OF MILLATHANE 5004 ELASTOMERS

Recipe	1	2	3	4	5
MILLATHANE 5004	100	100	100	100	100
Stearic Acid	0.25	0.25	0.25	0.25	0.25
N330 (HAF) Black	20	20	–	–	–
HiSil 233	–	–	40	40	20
Di-Cup 40C	4	5	6	8	5
Curing Temperature °F, (C)	350 (177)	350 (177)	350 (177)	350 (177)	400 (204)
Curing Time (minutes)	3	3	3	4	3
Mechanical Properties					
Shore A Hardness	70	70	76	84	65
300% Modulus, psi (MPa)	2280 (15.7)	3220 (22.2)	950 (6.6)	1850 (12.8)	820 (5.7)
Tensile, psi (MPa)	4140 (28.5)	4030 (27.8)	4050 (27.9)	3650 (25.2)	3830 (26.4)
Elongation, (%)	470	360	580	400	590
Tear Strength lbs/inch (ASTM Die C) (kN/m)	550 (96.3)	510 (89.3)	370 (64.8)	400 (70.0)	320 (56.0)

TYPICAL MECHANICAL PROPERTIES OF NON-BLACK FILLED MILLATHANE® 5004 CURED AT 305°F (152°C), 350°F (177°C), and 400°F (204°C).

Recipe	Control	1	2	3	4	5
MILLATHANE 5004	100	100	100	100	100	100
Stearic Acid	0.25	0.25	0.25	0.25	0.25	0.25
HiSil 233	20	20	40	40	40	40
Di-Cup 40C	4	5	6	7	6	8
Curing Temperature °F, (°C)	305 (152)	350 (177)	350 (177)	350 (177)	400 (204)	400 (204)
Curing Time, minutes	45	3	3	3	2	2
Mechanical Properties						
Shore A Hardness	70	70	83	83	82	83
100% Modulus, psi (MPa)	460 (3.2)	320 (2.2)	480 (3.3)	500 (3.4)	390 (2.7)	400 (2.8)
200% Modulus, psi (MPa)	800 (5.5)	600 (4.1)	850 (5.9)	880 (6.1)	720 (5.0)	730 (5.0)
300% Modulus, psi (MPa)	1190 (8.2)	920 (6.3)	1200 (8.3)	1300 (8.9)	1060 (7.3)	1100 (7.6)
Tensile, psi (MPa)	3660 (25.2)	3790 (26.1)	3640 (25.1)	3520 (24.3)	3000 (20.7)	3200 (22.1)
Elongation, (%)	590	530	500	490	500	540
Tear Strength lbs/inch (ASTM Die C) (kN/m)	400 (70.0)	350 (61.3)	380 (66.5)	380 (66.5)	350 (61.3)	360 (63.0)

FUEL AND OIL RESISTANCE OF BLACK FILLED MILLATHANE 5004 CURED AT 350°F (177°C) FOR 3 MINUTES

	Per Cent Volume Increase	
	Control (45 Min Cure @ 305°F [152°C])	**Rapid Cure (3 Min Cure @ 350°F [177°C])**
ASTM Fuel A, 168 hrs at Room Temperature	+ 1.3	- 0.45
ASTM Fuel B, 168 hrs at Room Temperature	+ 16.0	+15.0
ASTM Oil #901, 70 hrs at 250°F (121°C)	- 0.64	- 3.1
ASTM Oil #903, 70 hrs at 250°F (121°C)	- 1.03	+ 4.5

TSE INDUSTRIES, INC., MILLATHANE DIVISION PRODUCTS LIST:

MILLATHANE® 66 — A polyester-based millable polyurethane which is peroxide curable.

MILLATHANE® 76 — A polyester-based millable polyurethane which is sulfur or peroxide curable.

MILLATHANE® 97 — A transparent polyether-based millable polyurethane which is peroxide curable.

MILLATHANE® 5004 — A polyester polyurethane with excellent processing characteristics and can be easily injection molded and must be vulcanized with peroxide (formerly known as Vibrathane® 5004).

MILLATHANE® CM — A polyether polyurethane rubber exhibiting outstanding low temperature properties and excellent hydrolytic stability (formerly known as Adiprene® CM).

MILLATHANE® E34 — A polyether-based millable polyurethane which is sulfur or peroxide curable.

MILLATHANE® HT — A polyester-based millable polyurethane which is sulfur or peroxide curable and able to withstand elevated temperatures.

CAYTUR® 4 — MBTS/zinc chloride activator for millable polyurethanes.

THANECURE® T9 — Dimeric 2,4-toluene diisocyanate can be used as vulcanization agent for polyurethane rubber; adhesion promoter for rubber to textile and PVC to textile bonding; as a cross linking component in heat activated one compound PUR elastomer systems, one component adhesive systems and one component coatings which include automotive undercoats.

CRYSTAL® 1053 — A semi-permanent mold release agent recommended for applications in rubber, composite, and thermoplastic molding. Excellent mold sealer and inhibits mold build-up.

CRYSTAL® 2000 — A semi-permanent mold release agent used as a mold lubricant and recommended for slab dip, lubricating extruded goods and prevents water spotting in open steam cure.

CRYSTAL® 4100 — A semi-permanent mold release agent used for applications in thermoplastic, epoxy, and urethane molding. Provides a high level of slip to the mold.

CRYSTAL® 7000 — A semi-permanent mold release agent which allows a greater number of releases for urethane integral skin foam.

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TSE INDUSTRIES, INC.
AN ISO 9001 CERTIFIED COMPANY

4370 112th Terrace North, Clearwater, Florida 33762, USA
(727) 573-7676 (800) 237-7634 Fax: (727) 572-0487
www.TSE-INDUSTRIES.com www.MILLATHANE.com

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