Plasticizers for CPE Elastomers

Chlorinated polyethylene (CPE) was developed for use in applications involving serviceability where oil resistance, ozone and weathering resistance are important. CPEs can generally be used in applications that encounter service temperatures up to 149°C. This polymer owes its ozone and weathering resistance and relatively good heat aging properties to the fact that it has a saturated backbone. In addition, the presence of chlorine in the elastomer imparts enhanced oil resistance.

CPEs are available on a commercial basis in three standard grades representing chlorine contents ranging from 36–42 percent, along with several variations of polymer viscosity. In this evaluation, we used CM0136, which is described as a general purpose CPE with a chlorine content of 36 percent from Dow.

In our evaluations, two recipes were used. The recipe for Part I employed a TAIC/Trigonox 17/40 cure system and the plasticizers, which were evaluated at 20 PPHR, included DBEA, TOTM, S-75 and G-62. The compound evaluated in Part II was cured with a combination of Triganox 17/40 and Vulcup 40KE. The plasticizers evaluated at 35 PPHR included DBEP, P-7068, P-670, TOTM and G-62.

Part I

Results for this compound indicate that each of the plasticizers evaluated would be suitable for use in CPE depending on the specific properties required. S-75 appears to be well suited for applications requiring good heat resistance up to 150°C. TOTM, S-75 and G-62 could be recommended for use in CPE when a good overall balance of heat and oil resistance are needed up to 150°C.

Plasthall® DBEA was found to provide excellent flex resistance and good brittle point values as-molded, as well as after ASTM's 1 and 3 oil aging and distilled water immersion. Results for DBEA were very impressive for flex crack growth resistance, and this plasticizer would be highly recommended if this property is a major concern.

Part II

Data for this compound again point out the utility of TOTM and G-62 for 150°C air aging applications. Of the compounds tested, TOTM provides the best as-molded, low-temperature brittle point. The adipate-based polyester P-670 provided a number of interesting properties relative to TOTM and G-62. Relative to these two materials, P-670 was found to provide:

- Greater Mooney viscosity reduction
- Lower percent change of elongation, hardness and weight after air aging for 70 h at 150°C
- Lower change of hardness, volume and weight after Fuel C immersion
- Significantly lower elongation change after immersion in ASTM 2 oil for 70 h at 150°C

Plasthall® DBEP was found to impart relatively high elongation values along with good low-temperature impact and Gehman results but has poor permanence. Most notable for DBEP was that it yielded the greatest tear resistance of the compounds tested.

Part I

CPE Compound Data

Recipe	**1**	**2**	**3**	**4**	**5**
CM0136	100.0	100.0	100.0	100.0	100.0
N-330	50.0	50.0	50.0	50.0	50.0
Hi-Sil 233	10.0	10.0	10.0	10.0	10.0
H202-D80	10.0	10.0	10.0	10.0	10.0
Agerite Resin D	0.2	0.2	0.2	0.2	0.2
DAP	10.0	10.0	10.0	10.0	10.0
Plasthall® 200 (DBEP)	35.0	-	-	-	-
Plasthall® P-7068	-	35.0	-	-	-
Plasthall® P-670	-	-	35.0	-	-
Plasthall® TOTM	-	-	-	35.0	-
Paraplex® G-62	-	-	-	-	35.0
Mill Addition					
Trigonox 17/40	2.0	2.0	2.0	2.0	2.0
Vulcup 40KE	6.0	6.0	6.0	6.0	6.0
TOTAL	223.2	223.2	223.2	223.2	223.2

	Plasthall®	**Plasthall®**	**Plasthall®**	**Plasthall®**	**Paraplex®**
Major Variable	**200**	**P-7068**	**P-670**	**TOTM**	**G-62**
Scorch and Cure Properties: at 177°C					
ML, lbf/in	21.7	24.3	15.1	23.2	22.8
MH, lbf/in	44.9	58.8	30.1	60.1	52.3
ts2, min	2.8	2.7	3.7	*2.9*	2.8
1.25 x t'c(90), min	12.5	12.8	13.1	13.1	12.3
Original Physical Properties					
100% Modulus, psi	150	300	200	300	200
Tensile, Ultimate, psi	2200	2500	2100	2500	2350
Elongation at Break, t	540	430	340	440	500
Hardness, Duro A, pts.	74	74	73	75	74
Specific Gravity	1.343	1.336	1.346	1.323	1.327
Tear Resistance, lbf/in	227	189	118	184	200

	Plasthall® 200	Plasthall® P-7068	Plasthall® P-670	Plasthall® TOTM	Paraplex® G-62
Air oven Aging, 70 h at 150°C					
Tensile Change, %	-2	-8	+2	-10	-2
Elongation Change,	-59	-12	-6	-30	-20
Hardness Change, pts.	+18	+11	+5	+7	+9
Weight Change, %	-16	-6.6	-2.6	-3.9	-1.8
Fuel C Immers., 70 h at 23°C					
Tensile Change, %	-80	-76	-76	-74	-77
Elongation Change, %	-48	-58	-56	-61	-54
Hardness Change, pts.	-46	-39	-40	-38	-46
Volume Change, %	+107	+97	+104	+91	+106
Weight Change, %	+60	+55	60	+52	+61
Fuel C Dry Out, 22 h at 70°C					
Hardness Change, pts.	+13	+12	+8	+12	+11
Volume Change, %	-18	-17	-12	-19	-16
Weight change, %	-16	-14	-10	-15	-14
ASTM Oil #2 Immers., 70 h at 150°C					
Tensile Change, %	-34	-20	-12	-22	-30
Elongation Change,	-28	-21	-8.8	-32	-20
Hardness Change, pts.	-37	-32	-30	-29	-35
Volume Change, %	+66	+50	+58	+49	+61
Weight Change, %	+33	+38	+43	+41	+41

	Plasthall® 200	Plasthall® P-7068	Plasthall® P-670	Plasthall® TOTM	Paraplex® G-62
Roll Spew Compatibility					
Temperature at 23°C, -1°C, -21°C through 96 h	NONE	NONE	NONE	NONE	NONE
Low-Temperature Impact					
As Molded:					
All Pass, °C	-40	-33	-34	-42	-35
Low-Temperature TorsionGehman					
T5, °C	-22	-16	-18	-20	-17
T10, °C	-27	-20	-22	-25	-22
Apparent Mod. of Rigid., psi	254	287	225	259	248

Part II

CPE Compound Data Summary

Recipe	**1**	**2**	**3**	**4**
CM0136	100.0	100.0	100.0	100.0
BSWL #202	10.0	10.0	10.0	10.0
Agerite Resin D	0.2	0.2	0.2	0.2
N-660	65.0	65.0	65.0	65.0
DAP	10.0	10.0	10.0	10.0
Elastocal C-75	15.0	15.0	15.0	15.0
Plasthall® 203 (DBEA)	20.0	-		
Plasthall® TOTM	-	20.0		
Monoplex® S-75		-	20.0	
Paraplex® G-62	-		-	20.0
<u>Mill Addition</u>				
TAIC	2.0	2.0	2.0	2.0
Trigonox 17/40	11.0	11.0	11.0	11.0
TOTAL	233.2	233.2	233.2	233.2
Major variable	**Plasthall® 203**	**Plasthall® TOTM**	**Monoplex S-75**	**Paraplex® G-62**
<u>Scorch and Cure Properties</u>				
ML at 121°C	57	63	60	63
t5, 121°C, min	12.0	10.5	11.5	11.0
Cure Time, 160°C, min	10.8	9.5	8.3	12.6
<u>Original Physical Properties</u>				
100% Modulus, psi	550	700	550	750
Ultimate Tensile, psi	1900	2150	1850	2150
Elongation at Break, %	320	280	300	290
Hardness, Duro A, pts.	77	78	77	81
Specific Gravity	1.403	1.410	1.405	1.400
Tear Resistance, lbf/in.	147	143	132	156
Compression Set, % 70 h at 149°C	68	54	93	76
<u>Low-Temperature Properties</u>				
Brittle Point, °C	-19	-18	-11	-12
T F- 45,000 psi, °C	-38	-30	-31	-27
T-135,000 psi, °C	-46	-38	-39	-35

	Plasthall® 203	Plasthall® TOTM	Monoplex S-75	Paraplex® G-62
Flex Resist., initial crack				
length-0.07511, Kilocycles to:				
0.37511 crack length	331	5.2	27.7	6.0
Heat Resistance, 168 h at 150°C				
Elongation Change, %	-75	-64	-37	-52
Hardness Change, pts.	+14	+10	+9	+8
Weight Change, %	-9.4	-4.0	-2.8	-1.8
Brittle Point, °C	+3	-1	-8	-2
ASTM Oil 1, Aged 70 h at 150°C				
Elongation Change, %	-28	-32	-33	-28
Hardness Change, pts.	+6	+6	+6	+3
Volume Change, %	+3.5	+3.4	+4.5	+6.8
180° Bend, all pass	NO	NO	NO	NO
ASTM Oil 3, Aged 70 h at 150°C				
Elongation Change, %	-31	-32	-33	-38
Hardness Change, pts.	-26	-25	-24	-26
Volume Change, %	+56	+52	+56	+55
Brittle Point, °C	-22	-23	-20	-27
Water Aged, 70 h at 100°C				
Elongation Change, %	-34	-14	-20	-7
Hardness Change, pts.	-5	-3	-2	-1
Volume Change, %	+16	+16	+24	+18
Brittle Point, °C	-5	+7	+6	>RT
ASTM Fuel C Aged, 70 h at 23°C				
Elongation Change, %	-50	-54	-53	-57
Hardness Change, pts.	-36	-33	-35	-36
after dry out	+10	+12,	+14	+9
Volume Change,	+96	+91	+95	+95
after dry out	-15	-14	-14	-12
Brittle Pt., after dryout, °C	+4	+7	+5	+8

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Appendix I

Test Methods

Compounds for performance testing were mixed in a BR Banbury, except for curatives, which were added on a two-roll, 6 x 13 inch mill. Test specimens for compound performance properties were molded as follows: press temperature – 170°C (338°F), press time – 1.25 x t'c(90) min and at 833 psi on the sheet surface. Specimens for original properties, low temperature, immersions, air agings and roll spew were die cut from molded sheets .075 ± .005 inch thick.

Mooney Viscometer:	ASTM D1646-87, Monsanto Visco-meter, large rotor, 1 min preheat
Oscillating Disc Rheometer:	ASTM D2084-87, Monsanto Rheometer, Square Die, 3° arc, 900 cpm, 0-100 range, 20 min motor speed, 30 s preheat. MH at central point of torque rise, rate - one lb. in./5 min.
Original Properties:	
Tensile, Elongation, Modulus	ASTM D412-87, Die C
Hardness	ASTM D2240-86, Is reading
Specific Gravity	ASTM D792-86 para. 11.1
Low-Temperature Brittleness	ASTM D2137-83, Method A
Low-Temperature Gehman	ASTM D1053
Air Oven	ASTM D573-88
Fluid Immersions	ASTM D471-79
Roll Spew, 96 h at 23°C, 2°C, -16°C	Hallstar Method 01-79
Tear Resistance	ASTM D624-86

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Part I

Materials List

Material	Description	Supplier
CM0136	Chlorinated Polyethylene Elastomer	Dow Chemical
N-330	Carbon Black	Ashland Chem.
Hi-Sil 233	Precipitated Hydrated Amorphous	PPG
H202-D80	Basic Silicate White Lead	Wyrough & Loser
Agerite Resin D	Polymerized 1, 2-dihydro 2,2, 4 trimethylquinoline	R.T. Vanderbilt
DAP	Diallyl Phthalate	Hallstar
Trigonox 17/40	40% n-butyl 4,4, bis(t-butyl peroxy) valerate on Calcium Carbonate Filler	Akzo Chem.
Vulcup 40KE	40% di(2-tertiary-butyl peroxy-iso-propyl) benzene	Hercules
Plasthall® 220	Dibutoxyethyl Phthalate (DBEP)	Hallstar
Paraplex® G-62	Epoxidized Soybean Oil	Hallstar
Plasthall® P-7068	Low-Molecular Weight Phthalate Polyester	Hallstar
Plasthall® P-670	Polyester Adipate	Hallstar
Plasthall® TOTM	Trioctyl Trimellitate	Hallstar

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Part II

Materials List

Material	Description	Supplier
CM0136	Chlorinated Polyethylene Elastomer	Dow Chemical
BSWL #202	Basic Silicate White Lead-	Eagle Picher
Agerite Resin D	Polymerized 1, 2-dihydro 2,2, 4 trimethylquinoline	R.T. Vanderbilt
DAP	Diallyl Phthalate	Hallstar
Elastocal C-75	Dispersion, 75% CaO in Chlorinated Paraffin	Elast-O-Chem Chem. Co.
N-660	GPF Grade Carbon Black	Cabot Corp.
Trigonox 17/40	40% n-butyl 4,4, bis(t-butyl peroxy) valerate on Calcium Carbonate Filler	Noury Chem.
TAIC	Triallyl isocyanurate	Nippon Kasei Chem.
Plasthall® 203	Dibutoxyethyl Adipate (DBEA)	Hallstar
Monoplex® S-75	Epoxidized Glycol Dioleate	Hallstar
Paraplex® G-62	Epoxidized Soybean Oil	Hallstar
Plasthall® TOTM	Trioctyl Trimellitate	Hallstar