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Evaluation of the Use of Processing Aids in Silicone Elastomers and Cellular Silicones

W. E. Cady, E. S. Jessop
and B. M. McKinley

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May 23, 1982

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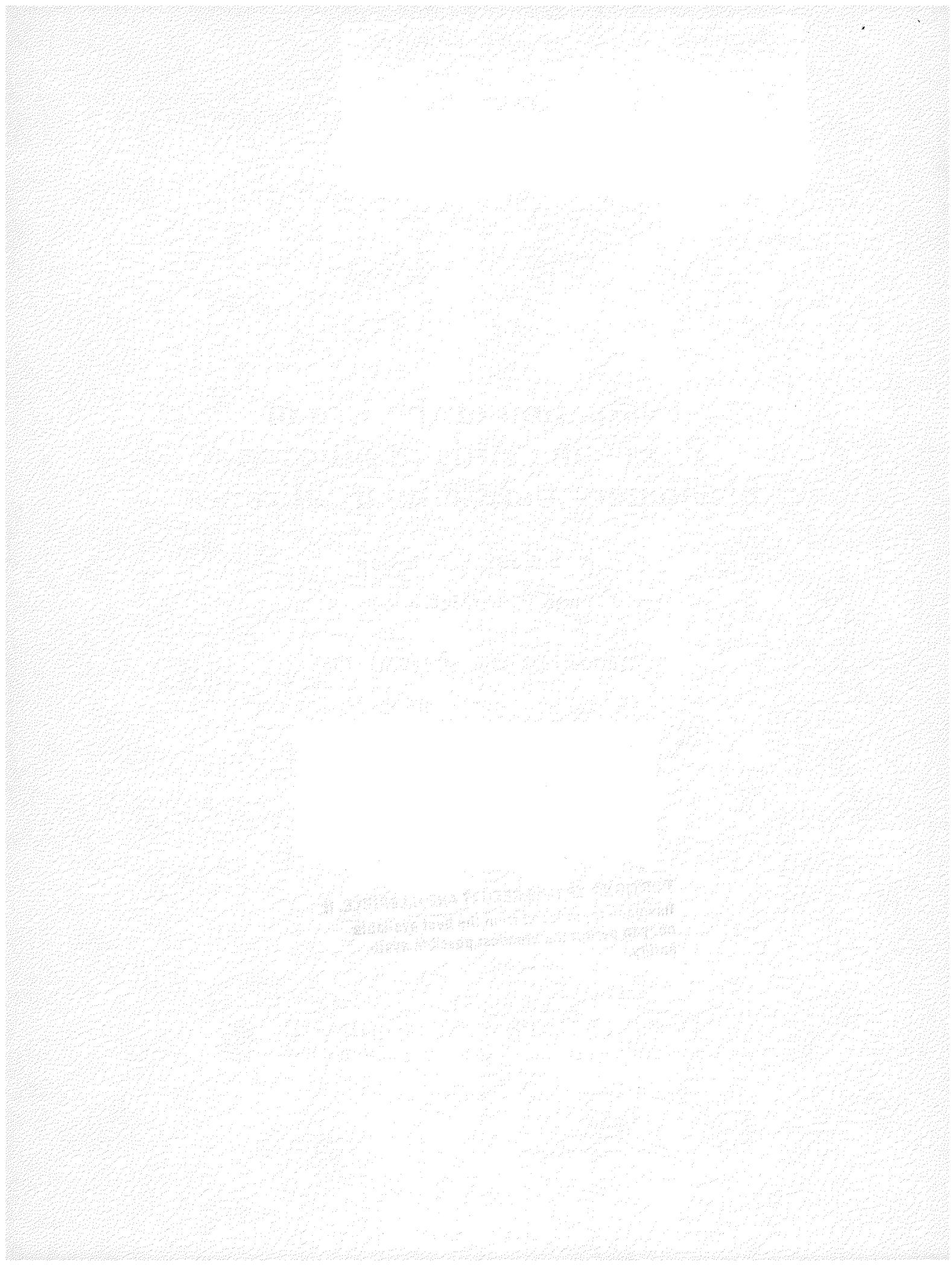
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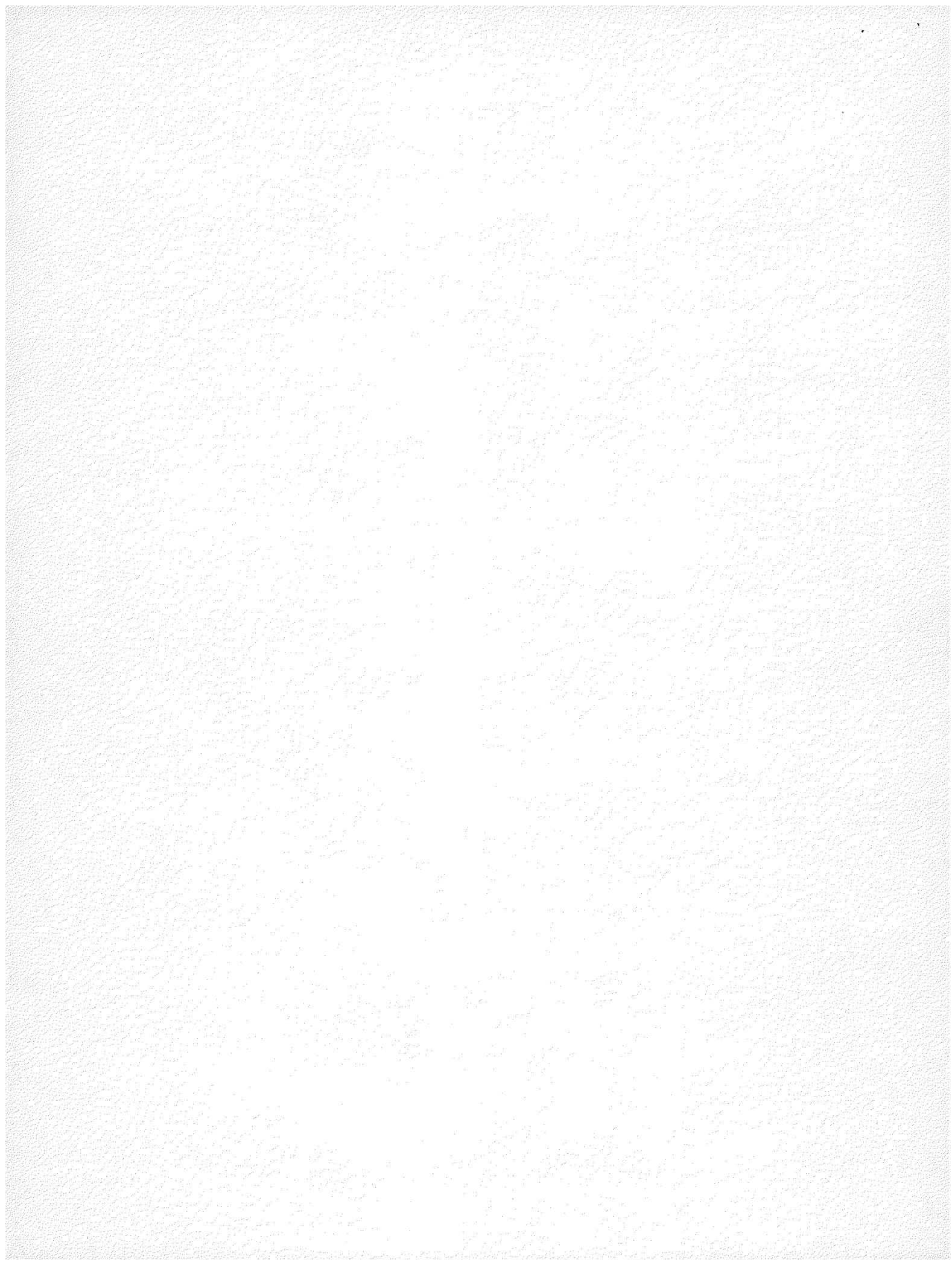
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Glossary

Banbury intensive mixer	Mixer produced by Farrel-Birmingham Co., Inc., Ansonia-Derby, CT.
BKC	The Bendix Corporation, Kansas City Division, Kansas City, MO.
Cabosil Grade MS7	A grade of silicon dioxide made by the Cabot Corporation, Boston, MA.
Cadox BS	Fifty percent benzoyl peroxide in a dimethylsilicone oil. A curing agent produced by the Chemical Department, McKesson and Robbins, Inc., New York, NY.
Cadox TS50	Fifty percent paste of 2,4-dichlorobenzoyl peroxide in dimethylsilicone oil. A curing agent produced by the Chemical Department, McKesson and Robbins, Inc., New York, NY.
Δ area	The hysteresis loop between the third cycle load and unload curves of load-deflection.
Δt	Difference in starting thickness between the first and third cycles of the load-deflection curves as measured by the Instron test machine.
DMS	Dimethylsiloxane repeating unit in our silicone polymers.
DPS	Diphenylsiloxane repeating unit in our silicone polymers.
DuPont	E. I. DuPont de Nemours and Co., Wilmington, DE.
EEMCO rubber mill	Two-roll mill produced by the Erie Engine and Manufacturing Co., Erie, PA.
Esperox 497XL	Tertiary butyl per-2-methylbenzoate at 40% concentration on calcium carbonate. A peroxide curing agent produced by Witco Chemical Corporation, U.S. Peroxygen Division, Richmond, CA.
Fisher	Fisher Scientific Co., Pittsburgh, PA.
GE	General Electric Co., Silicone Products Department, Waterford, NY.
GPC	Gel permeation chromatography.
Haake intensive mixer	One-pound mixer, System 80, produced by Haake, Inc., Saddle Brook, NJ.
Hi Sil 233	A grade of silicon dioxide produced by Pittsburgh Plate Glass Industries, Inc., Pittsburgh, PA.
Instron Test Machine	Universal test machine produced by the Instron Corporation, Canton, MA.
L60VB	Vinyl-end-blocked, block-polymerized silicone gum produced by LLNL.
L97KVB	Vinyl-end-blocked, equilibrium-polymerized silicone gum produced by LLNL.
L1668	Intermediate reinforced silicone gum produced by LLNL.
L3219	Heat-striped L1668 produced by LLNL.
L3223	Molding compound of L3219, temporary filler and curing agent or cellular silicone cushion produced from the molding compound by LLNL. Nominal cushion density of 0.63-0.65 Mg/m ³ .
L3260	Like L3223 but with a nominal density of 0.52-0.55 Mg/m ³ .
LLNL	Lawrence Livermore National Laboratory, Livermore, CA.
Luperco 101XL	2,5-dimethyl-2,5 bis (t-butyl peroxyl) hexane at 50% concentration on an inert filler. A peroxide curing agent produced by Lucidol Division, Pennwalt Corporation, Buffalo, NY.
M_n	Number-average molecular weight from GPC measurements based on polystyrene equivalents.
MN60VB	Commercial equivalent of L60VB silicone polymer produced by McGhan-NuSil Corporation, Carpenteria, CA.
MW	Molecular weight.
M_w	Weight-average molecular weight from GPC measurements based on polystyrene equivalents.
MWD	Molecular weight distribution, or the ratio of M_w/M_n .

MVS	Methylvinylsiloxane repeating unit in our silicone polymers.
n	Number of samples tested.
NMR	Nuclear magnetic resonance.
Pasadena Hydraulics press	Molding press made by Pasadena Hydraulics, Inc., El Monte, CA.
% C of V	Coefficient of variation; (S/av)100.
Rex O	Durometer Hardness Tester, Type O, Model 1600, made by Rex Gage Co., Glenview, IL.
RT	Room temperature.
S	Standard deviation.
SE33	A grade of silicone polymer produced by GE and used as a carrier for the other components of GE's SE4210 processing aid.
SE54	A grade of silicone polymer produced by General Electric's Silicone Products Department, Waterford, NY.
SE4210	Processing aid for use in compounding silicone gum produced by General Electric's Silicone Products Department, Waterford, NY.
Sherritt-Gordon Mines	Sherritt-Gordon Mines, Ltd., Fort Saskatchewan, Alberta, Canada.
Shore A	Durometer Hardness Tester, Type A, made by Shore Instrument and Manufacturing Co., Jamaica, NY.
TGA	Thermal gravimetric analysis.
UCC	Union Carbide Corporation, Silicone Rubber, New York, NY.
U/L ratio	Ratio of 3rd cycle unload to load values at the same deflection.
V/V ₀	Equilibrium swell ratio or ratio of swollen polymer volume to original polymer volume. A measure of degree of crosslinking.
Y1587	Ethoxy-end-blocked, dimethylsiloxane fluid produced by UCC.

Evaluation of the Use of Processing Aids in Silicone Elastomers and Cellular Silicones

Abstract

We evaluated two processing aids, UCC's Y1587 and GE's SE4210, in GE's SE54, type 97, and type 60 silicone gums. We tested uncured L1668 reinforced gums, cured L3219 elastomers, and L3223 and L3260 cellular silicones. In the new type 97KVB and type 60VB silicone gums, Y1587 produces a softer cushion than SE4210. The type of processing aid has no significant effect on uncured L1668 and has no effect on the compression set of the higher density L3223. For lower density L3260 cushions, Y1587 produces a significantly lower compression set than SE4210. If compression set is not a big consideration, or if cushion density is high, the choice of a processing aid can be used to control cushion stiffness along with the amount of reinforcing SiO_2 , vinyl content, and porosity. Y1587 is preferred for all silicone gums, and it is important that it be used in the 97KVB and 60KVB gums.

Introduction

Lawrence Livermore National Laboratory has used cellular silicones as support materials and space fillers in weapons for two decades. Our sole commercial source of these materials has ceased production of both the silicone gum and filled molding compounds. Therefore, we have developed a silicone technology to both synthesize gums and compound them to cellular materials. We are in the process of developing replacement gums and compounding them to equivalent or better cellular silicone than we have had in the past.

The gums we are evaluating include GE's SE54 silicone gum; our L97KVB gum¹ which we hope is an improved version of the commercial gum we have been using; and our L60VB² which is an improved alternating block copolymer.

In the first of the series of compounding steps, silicone gum is mixed with two grades of SiO_2 and a processing aid to create a reinforced gum, L1668. The L1668 is bin aged at RT for about one month and then heat stripped to the base

gum, L3219. The L3219 is filled with a temporary filler and activated with a peroxide curing agent to create a molding compound, e.g. L3223 or L3260. The molding compound is compression-die molded and cured, water washed to remove temporary filler, dried, and post cured to create our product.

With our original commercial gum (UCC's W97), UCC's Y1587, an ethoxy-end-blocked dimethylsiloxane fluid, was used as the processing aid. We have also used Y1587 in all our replacement gums. General Electric recommends that their SE4210 processing aid be used with their SE54 gum. We have also used SE4210 in all our replacement gums in order to compare the two processing aids. We made one comparison of the use of Y1587 with no use of a processing aid.

This report evaluates the use of Y1587, SE4210 or no processing aid on the properties of uncured and cured L1668, cured L3219 elastomer, and cellular silicones, L3223 and L3260.

Experimental

Materials

We used two lots of SE54 gum (161 and 167); one lot of L97KVB, one lot of MN97KVB, one lot of L60VB, and several experimental lots of

MN60VB in our comparison of processing aids. The SE54 gums were obtained from General Electric Co. Our L97KVB was synthesized in-house to be a random equilibrium terpolymer of dimethyl-, diphenyl-, methylvinylsiloxane containing 0.7

Table 1. Properties of uncured silicone gums by gum and identification number.

	SE 54	L97KVB	MN97KVB	L60VB	MN60VB			
	Lot 161 ^a	Lot 167	TJ150	TJ164	TJ154	DR115	DR114	RD001VR26
Molecular weight^b								
M_w	514,000	745,000	590,000	514,150	478,000	671,350	418,700	320,750
M_n	146,000	333,500	250,000	192,400	182,500	204,000	151,300	127,750
MWD	3.52	2.24	2.38	2.67	2.62	3.30	2.77	2.51
Wt% MVS ^c	0.29 ^d	0.27	0.7	0.69	0.80	0.67	0.69	0.71
Wt% DPS ^c	14.3 ^e	14.3	9.6	8.52	9.64	9.70	10.1	9.45

^a Bendix data, Reference 3.

^b By GPC, based on polystyrene equivalents.

^c By NMR except as noted.

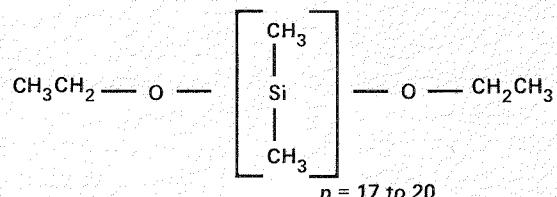
^d By iodine monochloride titration.

^e By pyrolysis gas chromatography.

wt% methylvinylsiloxane and 9 wt% diphenylsiloxane. The MN97KVB gum was synthesized in a 200-lb batch by McGhan-NuSil under our guidance to duplicate L97KVB. Both L97 and MN97 were vinyl-end-blocked. The L60VB was synthesized in-house to be an alternating block condensation terpolymer of DMS, DPS, and MVS with the same MVS and DPS contents as L97KVB. The MN60VB's were small scale experimental attempts by McGhan-NuSil to produce our L60VB. All condensation polymers were also vinyl-end-blocked. SE54 is an equilibrium terpolymer of much lower MVS and higher DPS contents than L97 and L60. Data for the polymer lots are listed in Table 1.

To prepare L1668 we normally add 32 parts of Cabosil MS7 SiO_2 and 6 parts of HiSil 233 SiO_2 plus a processing aid to the silicone gum. We have varied MS7 over the range of 25 to 38 parts and HiSil 233 at 6 parts or none at all. We used only one commercial lot each of MS7 and HiSil 233 in our experiments. The other ingredient in L1668 is the processing aid. Because we do not understand the interaction among silicone gum, SiO_2 , and the processing aid we had no way of knowing the optimum amount of either processing aid to use, and we had no way of judging equivalent amounts of the two processing aids (Y1587 and SE4210) to be used for comparison. For our standard formulations we used 10 or 11 parts of Y1587 per hundred parts of gum, by weight as was done by UCC; for SE4210 we used 5 or 6 parts as presumably is done by GE with SE54 gum. For amounts of MS7 above 32 parts we increased the amount of SE4210 and for amounts of MS7 below 32 parts we decreased the amount of Y1587 (see Table 5). We did not adjust the amount of either processing aid for the absence of HiSil 233.

Only one lot of UCC's Y1587 (355022178) has been used to date. For SE4210, lot HF230 was used in L1668 TJ82 and thereafter lot JE240 was used. BKC analytical data on SE4210, Y1587, and SE33, the major component of SE4210, are shown in Table 2. Thermal gravimetric analyses of both processing aids are shown in Table 3; these were run at a heating rate of 10°C/min in a nitrogen atmosphere. Y1587 is much more volatile than SE4210 and the excess is probably much more effectively stripped during the conversion of L1668 to L3219 and during the long post cure than it is for SE4210. Table 4 shows the component composition of SE4210 as determined by BKC. SE33, the major component of SE4210, is a GE commercial gum consisting primarily of dimethylsiloxane and a small amount of methylvinylsiloxane, but containing no phenylmethylsiloxane or diphenylsiloxane. Y1587 is probably an ethoxy-end-blocked dimethylsiloxane fluid with a structure as follows⁶:



Y1587 apparently contains small amounts of octamethylcyclotetrasiloxane and oligomers of the above structure where $n = 4, 8, 12$, and 16.

To cure L3219, we activated L3219 with one of the following peroxide curing agents:

Cadox TS50

Cadox BS

Luperco 101XL

Esperox 497XL

Table 2. Analyses of the two processing aids and the major component of SE4210, SE33.^{4,5,6}

	Y1587	SE4210	SE33
SiO ₂ , wt%	—	13.0	—
Hydroxyl content, wt%	0.12	0.50	—
Vinyl content, wt% as MVS	—	0.13 ^a	0.23 ^a
	—	0.10 ^b	0.20 ^b
MW by GPC			
M _w	~2000	594,000	533,000
M _n	1500-1700	300,000	270,000
MWD	—	1.98	1.98

^a By the iodine monochloride method.

^b By gas chromatography.

The curing agent used in a L3219 was the same that we used for the following molding compound but the concentration was not always the same. All curing agents were purchased from commercial sources.

To prepare the molding compound we filled L3219 either with urea and activated it with TS50, BS or 497XL or with NH₄Cl and activated it with 101XL. We used two ureas; one was supplied to us by BKC and was purchased by them from Sherritt-Gordon Mines; the other was our own supply which we had obtained from DuPont. The NH₄Cl was supplied by Fisher and handground by us. All fillers were screened to -25 to +40 mesh, U.S. Sieve Nos.

Compounding

For preparing L1668's we have established a standard addition sequence for the gum, MS7, HiSil 233, and the processing aid. L1668's TJ81, TJ82, TJ113, and TJ114 were made prior to the

Table 3. TGA analyses of the two processing aids. Losses are expressed as wt%.

	Y1587 ^a	SE4210 ^b
Onset of weight loss, °C	75	88
Cumulative loss, wt%		
at 100°C	1.1	1.5
at 200°C	20.4	10.8
at 300°C	66.1	20.0
at 400°C	76.1	25.4
at 500°C	80.0	50.8
at 600°C	—	87.0

^a LLNL analysis.

^b BKC analysis, Reference 4.

Table 4. Component composition of SE4210.⁷

Component	Wt%	Measurement Method
SiO ₂	15	TGA
SE-33	50	GPC
Silanol (MW ~ 1500)	23	By difference
D ₄ and D ₅ cyclics	12	GPC

adoption of this standard technique. Later formulations in which we varied the amounts of ingredients followed the standard procedure as closely as possible. In all cases, comparative pairs of formulations were made by the same procedure. Most mixings were made in our 1-lb size Banbury intensive mixer but some of the later mixings were made in our new Haake intensive mixer. Comparative formulations mixed in different mixers will be so noted. Mixing times in the Banbury totaled 5 min and in the Haake they totaled 7 min. Final blending and sheeting of all L1668's were done on our EEMCO 2-roll rubber mill. The 6 mm thick sheets (approx) of L1668 were bin aged at RT for at least 28 days and then were heat stripped in an air-circulating oven at 171° to 177°C for 18 h to produce L3219 base gums. Curing agents were added to L1668's and L3219's on the rubber mill. Activated L1668's and L3219's were molded into about 2 mm thick sheets in a flat-slab steel mold at an appropriate time and temperature in a Pasadena Hydraulics press; the sheets were then post cured at an appropriate time and temperature.

L3223 and L3260 molding compounds were prepared by our standard technique. Curing agent was added to L3219 on the 2-roll mill. Temporary filler was added to the activated L3219 either in the Banbury mixer or in the Haake mixer. Comparative formulations made in different mixers will be noted. Final mixing and sheeting were done on the 2-roll mill.

Molding compounds were converted to cellular silicones in a series of steps. Flat sheets were molded in a flat-slab steel mold at an appropriate time and temperature in a Pasadena Hydraulics press to a nominal 2.6 mm thickness except for earlier formulations (TJ108-111, TJ113A, and TJ114) which were 2.0 mm thick as noted. All comparative formulations are of the same nominal thickness. The press-cured sheets of molding compound were washed in about 90°C water in a washing machine using our standard washing sequence. The washed cushions were dried at RT for 16 h and then oven dried at 149°C for 4 h. Dried cushions were post cured for 24 h at 249°C.

Testing

All cured L3219 elastomers were tested for tensile properties and all L3223 and L3260 cushions were tested for load-deflection properties on an Instron test machine with a cross head speed of 1.25 mm/min at RT. Disks of L3219, L3223, and L3260 were weighed, measured, and their densities calculated. The compression sets on L3223's

and L3260's were measured by our confined-die test. A 31.7 cm² disk of material was compressed to the appropriate percentage of original free height in a closed die and placed in an air oven at 149°C for 24 h. After cooling to RT under compression, the sample was removed from the die and its free height measured as a function of time at RT. Compression set was calculated from height at 1 h recovery.

Results

SE54 Silicone Gum

L1668's

The compositions of all L1668's made for this study are listed in Table 5. The data for the two pairs of uncured L1668's made from the two lots of SE54 are listed in Table 6. Y1587 produces a softer reinforced gum for both lots of SE54. Using Y1587, both lots of SE54 lose more weight on bin

aging and have more extractables than with SE4210. Properties of cured L1668's from SE54 lot 167 are shown in Table 7. Y1587 produces a softer reinforced elastomer with higher extractables and higher swell ratio (V/V₀) which indicates less crosslinking with Y1587. The ultimate tensile strength with Y1587 seems to be slightly higher and the ultimate elongation is much greater than with SE4210.

Table 5. Compositions of L1668's, parts by weight.

Sample Identification	Gum Identification	Parts	SiO ₂		Processing Aid	
			MS7	HiSil 233	Y1587	SE4210
TJ81	SE54 lot 161	100	38	6	10	—
TJ82	SE54 lot 161	100	38	6	—	7
TJ113	SE54 lot 167	100	38	6	10	—
TJ114	SE54 lot 167	100	38	6	—	8
TJ115	SE54 lot 167	100	32	6	11	—
TJ116	SE54 lot 167	100	32	6	11	—
TJ117	SE54 lot 167	100	32	6	—	—
TJ150	L97KVB-TJ150-Blend	100	32	6	10	—
TJ150-2	L97KVB-TJ150-Blend	100	32	6	10	—
TJ150SE	L97KVB-TJ150-Blend	100	32	6	—	6
TJ172	L97KVB-TJ150-Blend	100	25	0	8	—
TJ168	L97KVB-TJ150-Blend	100	25	0	—	5
TJ164	MN97KVB-RD004-067	100	32	6	10	—
TJ175	MN97KVB-RD004-067	100	32	6	10	—
TJ176	MN97KVB-RD004-067	100	32	6	—	6
TJ154	L60VB-III-49-Blend	100	32	6	10	—
TJ177	L60VB-III-49-Blend	100	32	6	—	6
TJ173	L60VB-III-49-Blend	100	25	0	8	—
TJ169	L60VB-III-49-Blend	100	25	0	—	5
TJ159I	MN60VB-DR115	100	32	6	10	—
TJ159II	MN60VB-DR115	100	32	6	—	5
TJ160I	MN60VB-DR114	100	32	6	10	—
TJ160II	MN60VB-DR114	100	32	6	—	5
TJ161I	MN60VB-RD001VR26	100	32	6	10	—
TJ161II	MN60VB-RD001VR26	100	32	6	—	5

Table 6. Properties of uncured L1668's made from SE54 silicone gums and Y1587 or SE4210 processing aid.

Property	SE54 lot 161		SE54 lot 167	
	Y1587 TJ 81	SE4210 TJ 82	Y1587 TJ113	SE4210 TJ114
Hardness, Rex O, after aging				
0 day	34	36	22	27
1 day	42	50	29	35
6 days	43	53	—	—
7 days	—	—	31	44
14 days	47	58	37	48
21 days	—	—	40	49
23 days	50	60	—	—
28 days	—	—	41	49
80 days	53	62	—	—
Aging loss, wt%, after aging				
23 days	1.22	0.95	—	—
28 days	—	—	0.46	0.23
CHCl₃ extractables, wt%, after aging				
1 day	47.2	37.6	32.4	44.6
30 days	—	—	33.7	19.9
37 days	37.9	15.5	—	—
83 days	38.6	10.3	—	—
Swell ratio (V/V₀) in chlorobenzene after aging				
1 day	—	—	4.54	4.36
30 days	—	—	4.11	5.66

Comparative properties of SE54 lot 167 with and without Y1587 are shown in Table 8. These L1668's do not compare to T113 of Table 6 because of the difference in the MS7 contents and the switch to the now standard addition sequence. TJ115 and TJ116 are duplicates and they agree well in properties. Omission of Y1587 from SE54 produced a much harder reinforced gum; there is very little weight loss on bin aging and extractables are greatly reduced.

L3219's

The compositions and cure cycles of all L3219's made for this evaluation are shown in Table 9. The properties of the two pairs of cured L3219's made from lot 161 of SE54 and the pair made from lot 167 are listed in Table 10. The two comparative pairs from lot 161 show higher weight loss on post cure, higher V/V₀, slightly greater ultimate elongation and perhaps greater ultimate tensile strength for Y1587. For lot 167, the Y1587 formulation shows higher weight loss on post cure, higher extractables, higher swell ratio and both higher elongation and tensile strength. Y1587 produces a slightly softer elastomer with greater tensile strength and elongation and less crosslinking.

Properties of the two L3219's made from SE54 lot 167 gum and cured with 101XL peroxide with and without Y1587 are shown in Table 11. Omission of Y1587 results in a harder elastomer with reduced tensile strength and greatly reduced elongation. Without Y1587, crosslinking is slightly greater and extractables are reduced.

L3223's

The composition and cure cycles of all L3223's relative to the comparison of Y1587 and SE4210 are listed in Table 12. The load-deflection properties of the cushions made from SE54 lot 161 are shown in Table 13. No comparison can be made because both formulations utilizing SE4210 could not be processed into L3223 cushions. With lot 167 of SE54 we were able to produce cushions so the load-deflection data are tabulated in Table 14. The confined compression-set results for both lots of SE54 gums are shown in Table 15. Use of Y1587 produces a much softer cushion with greater resiliency than does use of SE4210. The type of processing aid used has no effect on compression set although final recovery is better with Y1587.

Load-deflection and set properties of the L3223 cushions made from SE54 lot 167 with and

Table 7. Properties of cured L1668's made from SE54 silicone gum (lot 167) and Y1587 or SE4210 processing aid.

	Y1587 TJ113	SE4210 TJ114
Composition, parts by weight		
L1668	100	100
Cadox TS50	1.5	1.5
Cure,		
Temperature, °C	116	116
Time, min	15	15
Post cure		
Temperature, °C	249	249
Time, hr	16	16
Properties		
Hardness, Shore A		
Cured	35	49
Post cured	38	64
Loss on post cure, wt%	6.21	4.7
CHCl ₃ extractables, wt%	3.53	2.34
V/V ₀ in chlorobenzene	3.62	2.73
Density, Mg/m ³	1.192	1.200
Ultimate tensile strength		
n	5	5
Av, kPa	6233	5440
S, kPa	683	683
Ultimate elongation		
Av, %	468	266
S, %	43	27

without Y1587 are shown in Tables 16 and 17 respectively. TJ115 and 115 CBA are duplicates except for a slight difference in the amount of NH₄Cl used in the molding compounds. The duplicates are not statistically alike in load-deflection but they are similar. The difference may be due to the density difference as the denser cushion is stiffer. For set, the duplicates are very different and we have no explanation for the difference. Without Y1587 the L3223 cushion is stiffer than either of the duplicates; it is stiffer than TJ115 CBA which it matches more closely in density even though it should not be, based on the amount of NH₄Cl used in the molding compounds. The confined compression set and ultimate recovery without Y1587 match that of TJ115 CBA with Y1587. Elimination of Y1587 produces a stiffer cushion but has no effect on compression set.

Type 97 Silicone Gums

L1668's

A. **L97KVB gum.** Properties of all uncured L1668's from our L97KVB silicone gum are listed

Table 8. Properties of uncured L1668's made from SE54 lot 167 silicone gum and Y1587 processing aid or no processing aid.

Property	Y1587 TJ115	None TJ116	None TJ117
Hardness, Rex O, after aging			
0 day	12	20	22
1 day	19	—	—
4 days	25	—	—
5 days	—	27	34
7 days	—	—	36
11 days	27	—	—
14 days	25	30	47
21 days	26	30	50
28 days	26	30	50
Aging loss, wt%, after			
28 days	0.59	0.68	0.06
CHCl ₃ extractables, wt%, after aging			
1 day	61.0	52.5	32.3
28 days	—	43.4	20.4
32 days	44.7	—	—
V/V ₀ after aging			
1 day	3.12	3.09	3.95
32 days	3.45	—	—

in Table 18. Formulations TJ150, TJ150-2, and TJ150SE were compounded with our standard amounts of 32 parts of MS7 SiO₂ and 6 parts of HiSil 233 SiO₂. TJ150 and TJ150-2 are duplicates. Formulations TJ168 and TJ172 were attempts to produce an improved cushion by reducing the MS7 to 25 parts to increase the resiliency of the elastomer and by eliminating HiSil 233, which appears to be a useless ingredient. The processing aids in these two formulations were adjusted proportionally to the MS7 contents but not to the HiSil 233 contents.

The duplication with Y1587 is good. Results with SE4210 are very much like those with Y1587. Type of processing aid has no effect on the properties of standard L1668's.

For the modified formulations, processing aids do have an effect; Y1587 produces a harder uncured L1668.

B. MN97KVB gum. Properties of standard uncured L1668's from MN97KVB, the commercial equivalent of our L97KVB gum, are shown in Table 19. TJ164 and TJ175 are excellent equivalents. The type of processing aid has no effect on the uncured L1668's.

L3219's

A. **L97KVB.** Properties of all relevant cured L3219's from L97KVB gum are listed in Table 20.

Table 9. Compositions and cure cycles of L3219's.

Sample Ident.	Gum Ident.	L1668 Ident.	Curing Agent		Cure Cycle		Post-Cure Cycle	
			Ident.	Parts ^a	min	°C	h	°C
TJ81a	SE54 lot 161	TJ81	TS50	1.5	15	113	24	249
TJ81b	SE54 lot 161	TJ81	BS	1.5	15	113	24	249
TJ82a	SE54 lot 161	TJ82	TS50	1.5	15	113	24	249
TJ82b	SE54 lot 161	TJ82	BS	1.5	15	113	24	249
TJ113	SE54 lot 167	TJ113	TS50	1.5	15	116	16	249
TJ114A	SE54 lot 167	TJ114	TS50	1.5	15	116	16	249
TJ115B	SE54 lot 167	TJ115	101XL	0.5	10	171	16	249
TJ117C	SE54 lot 167	TJ117	101XL	0.5	10	171	16	249
TJ150E	L97KVB-TJ150	TJ150	497XL	2.1	60	125	16	249
TJ150-2E	L97KVB-TJ150	TJ150-2	497XL	2.0	60	125	16	249
TJ150SEE	L97KVB-TJ150	TJ150SE	497XL	2.0	60	125	16	249
TJ172E	L97KVB-TJ150	TJ172	497XL	2.0	60	125	16	249
TJ168E	L97KVB-TJ150	TJ168	497XL	2.0	60	125	16	249
TJ164E	MN97KVB-RD004-067	TJ164	497XL	2.0	100	125	16	249
TJ175E	MN97KVB-RD004-067	TJ175	497XL	2.0	60	125	16	249
TJ176E	MN97KVB-RD004-067	TJ176	497XL	2.0	100	125	16	249
TJ154E	L60VB-III49	TJ154	497XL	2.0	60	125	16	249
TJ177E	L60VB-III49	TJ177	497XL	2.0	100	125	16	249
TJ173E	L60VB-III49	TJ173	497XL	2.0	60	125	16	249
TJ169E	L60VB-III49	TJ169	497XL	2.0	60	125	16	249
TJ159-II	MN60VB-DR115	TJ159-II	497XL	2.0	60	125	16	249
TJ160-I	MN60VB-DR114	TJ160-I	497XL	2.0	60	125	16	249
TJ160-II	MN60VB-DR114	TJ160-II	497XL	2.0	60	125	16	249
TJ161-II	MN60VB-RD001VR 26	TJ161-II	497XL	2.0	60	125	16	249

^a Parts of curing agent per hundred parts of L3219, by weight.

Table 10. Properties of cured L3219's made from SE54 silicone gums and Y1587 or SE4210 processing aid.

Property	SE54 lot 161				SE54 lot 167	
	TS50		BS		TS50	SE4210
	Y1587	SE4210	Y1587	SE4210	Y1587	SE4210
TJ81a	TJ82a	TJ81b	TJ82b	TJ113	TJ114A	
Hardness, Shore A						
Cured	45	45	55	54	42	52
Post cured	40	45	51	53	39	57
Loss on post cure, wt%	2.5	1.4	2.0	1.7	3.21	2.72
Density, Mg/m ³	1.198	1.190	1.190	1.184	1.193	1.199
CHCl ₃ extractables, wt%	3.7	3.4	2.2	2.2	3.05	2.27
V/V ₀ in chlorobenzene	3.42	3.06	2.85	2.66	3.19	2.60
Tensile properties						
n	5	5	5	5	5	5
Ultimate tensile strength, kPa						
Av	5888	4137	5599	5123	6364	5668
S	586	186	745	358	462	221
% C of V	9.95	4.50	13.3	7.00	7.26	3.89
Ultimate elongation, %						
Av	376	278	212	178	360	226
S	31	13	27	15	16	9
% C of V	8.24	4.68	12.7	8.43	4.44	3.98

Table 11. Properties cured L3219's made from SE54 lot 167 silicone gum and Y1587 processing aid or no processing aid.

Property	Y1587 TJ115B	None TJ117C
Hardness, Shore A		
Cured	42	55
Post cured	43	57
Loss on post cure, wt%	—	2.1
Density, Mg/m ³	1.172	1.184
CHCl ₃ extractables, wt%	2.8	1.97
V/V ₀ in chlorobenzene	3.23	2.86
Tensile properties		
n	5	3
Ultimate tensile strength, kPa		
Av	6736	5688
S	228	103
% C of V	3.38	1.81
Ultimate elongation, %		
Av	416	213
S	11	6
% C of V	2.64	2.82

TJ150E and TJ150-2E are considered duplicate standard formulations even though TJ150 contained 5% more curing agent and was made from a L1668 that was heat stripped after one week of bin aging instead of after four weeks of bin aging. TJ150SEE is also a standard formulation. TJ168E and TJ172E are modified formulations with reduced MS7 and no HiSil 233. These formulations contain less equivalent weights of 497XL curing agent than do the TJ150's because they contain more L97KVB gum by weight in the L1668's. All formulations were cured and post cured identically.

In spite of the differences between TJ150E and TJ150-2E, the duplication is good. L3219 with SE4210 is similar to the duplicate L3219's with Y1587; tensile strengths are the same but elongation, swell ratio and CHCl₃ extractables are slightly less with SE4210. These differences are probably not significant. For the modified formulations the type of processing aid has no effect on properties of the cured L3219's.

Table 12. Composition and cure cycles of L3223 molding compounds and cushions.

Sample Ident.	Gum Ident.	L3219 Ident.	Type	Filler Source	Curing Agent		Cure Cycle		Post-cure Cycle		
					Part ^a	Type	Parts ^a	min	°C	h	°C
TJ108	SE54 lot 161	TJ81	Urea	LLNL	100	TS50	4.5	15	113	24	249
TJ109	SE54 lot 161	TJ81	NH ₄ Cl	LLNL	105	BS	3.0	15	113	24	249
TJ110	SE54 lot 161	TJ82	Urea	LLNL	100	TS50	4.5	—	—	—	—
TJ111	SE54 lot 161	TJ82	NH ₄ Cl	LLNL	105	BS	3.0	—	—	—	—
TJ113A	SE54 lot 167	TJ113	Urea	LLNL	100	TS50	4.5	15	113	24	249
TJ114	SE54 lot 167	TJ114	Urea	LLNL	100	TS50	4.5	15	113	24	249
TJ115C	SE54 lot 167	TJ115	NH ₄ Cl	BKC	105.5	101XL	0.5	10	171	24	249
TJ115CBA	SE54 lot 167	TJ115	NH ₄ Cl	BKC	110	101XL	0.5	10	171	24	249
TJ117CBA	SE54 lot 167	TJ117	NH ₄ Cl	BKC	105.5	101XL	0.5	10	171	24	249
TJ150EBU	L97KVB-TJ150	TJ150	Urea	BKC	100	497XL	2.0	60	125	24	249
TJ150-2EBU	L97KVB-TJ150	TJ150-2	Urea	BKC	100	497XL	2.0	60	125	24	249
TJ150SEEBU	L97KVB-TJ150	TJ150SE	Urea	BKC	100	487XL	2.0	60	125	24	249
TJ172EBU	L97KVB-TJ150	TJ172	Urea	BKC	100	497XL	2.0	60	125	24	249
TJ168EBU	L97KVB-TJ150	TJ168	Urea	BKC	100	497XL	2.0	60	125	24	249
TJ164ELU	MN97KVB-RD004-067	TJ164	Urea	LLNL	100	497XL	2.0	100	125	24	249
TJ176EBU	MN97KVB-RD004-067	TJ176	Urea	BKC	100	497XL	2.0	100	125	24	249
TJ154EBU	L60VB-III49	TJ154	Urea	BKC	100	497XL	2.0	100	125	24	249
TJ177EBU	L60VB-III49	TJ177	Urea	BKC	100	497XL	2.0	100	125	24	249
TJ173EBU	L60VB-III49	TJ173	Urea	BKC	100	497XL	2.0	60	125	24	249
TJ169EBU	L60VB-III49	TJ169	Urea	BKC	100	497XL	2.0	60	125	24	249
TJ159-IIEBU	MN60VB-DR115	TJ159-II	Urea	BKC	100	497XL	2.0	—	—	—	—
TJ160IIEBU	MN60VB-DR114	TJ160-I	Urea	BKC	100	497XL	2.0	100	125	24	249
TJ160IIIEBU	MN60VB-DR114	TJ160-II	Urea	BKC	100	497XL	2.0	—	—	—	—
TJ161IIIEBU	MN60VB-RD001VR26	TJ161-II	Urea	BKC	100	497XL	2.0	—	—	—	—

^a Parts per hundred parts of L3219, by weight.

Table 13. Load-deflection properties of L3223 cushions from SE54, lot 161, silicone gum and Y1587 processing aid. Formulations using SE4210 could not be processed into L3223 cushions, so no comparison can be made with L3223 using Y1587.

Property	TS50 and Urea						BS and NH ₄ Cl					
	Y1587			SE4210			Y1587			SE4210		
	n	Av	S	n	Av	S	n	Av	S	n	Av	S
3rd cycle load, kPa, at deflections of												
5%	5	34.1	2.36	—	—	—	5	28.0	0.66	—	—	—
10%	5	64.7	2.47	—	—	—	5	57.7	0.97	—	—	—
20%	5	121.3	2.88	—	—	—	5	103.1	3.15	—	—	—
30%	5	220.1	5.51	—	—	—	5	167.3	7.70	—	—	—
40%	5	602.6	18.3	—	—	—	5	391.2	21.4	—	—	—
3rd cycle unload, kPa, at deflections of												
5%	5	18.3	1.95	—	—	—	5	16.4	0.64	—	—	—
10%	5	40.8	1.74	—	—	—	5	39.6	0.31	—	—	—
20%	5	79.1	1.58	—	—	—	5	73.1	1.07	—	—	—
30%	5	151.8	5.07	—	—	—	5	119.6	6.03	—	—	—
40%	5	494.1	24.5	—	—	—	5	318.3	21.2	—	—	—
3rd cycle U/L at deflections of												
10%	5	0.632	0.0098	—	—	—	5	0.686	0.0097	—	—	—
20%	5	0.652	0.0077	—	—	—	5	0.710	0.0132	—	—	—
30%	5	0.689	0.0088	—	—	—	5	0.715	0.0056	—	—	—
3rd cycle hysteresis loop (Δ area)	5	248.6	30.5	—	—	—	5	171.3	27.6	—	—	—
Thickness, mm												
1st cycle	5	2.11	0.028	—	—	—	5	2.03	0.008	—	—	—
3rd cycle	5	2.05	0.026	—	—	—	5	1.99	0.007	—	—	—
Δt	5	0.073	0.0060	—	—	—	5	0.039	0.0015	—	—	—
Density, Mg/m ³	7	0.6738	0.0027	—	—	—	6	0.6353	0.0037	—	—	—

^a Could not be processed.

B. MN97KVB. Properties of the cured L3219's from MN97KVB gum are shown in Table 21. All three are standard formulations and TJ164E and TJ175E are duplicates but TJ175E was press cured only 60 min instead of 100 min. The duplicates are very much alike; they have equal V/V's indicating equal crosslinking in spite of the difference in time of press cure. Results with SE4210 are like the duplicates with Y1587. Again type of the processing aid has no effect on the properties of cured L3219's.

L3223's

A. L97KVB. The load-deflection properties of standard-formulation L3223's made from L97KVB gum are listed in Table 22 and the modified formulations are shown in Table 23. Corresponding confined-compression-set data are shown in

Tables 24 and 25. All five formulations contain the same parts by weight of BKC urea and 497XL curing agent and were cured and post cured alike. The modified formulations contain less urea and less curing agent per gram of L97KVB gum than do the standard formulations because of the reduction of SiO₂ fillers.

The standard duplicate L3223's have similar load-deflection properties. The cushion with SE4210 is much stiffer and less resilient than the duplicates with Y1587. The duplicate cushions with Y1587 have the same compression set and ultimate recovery. The L3223 with SE4210 has greater set than duplicate L3223's with Y1587, but the same ultimate recovery. For the modified formulations SE4210 produces a slightly stiffer cushion with slightly greater resilience than does Y1587 but does not effect compression set or 7-day recovery. We found a 7-day recovery to be

Table 14. Load-deflection properties of L3223 cushions from SE54, lot 167, silicone gum and Y1587 or SE4210 processing aid.

Property	TS50 and Urea					
	Y1587			SE4210		
	n	Av	S	n	Av	S
3rd cycle load, kPa, at deflections of						
5%	5	39.8	1.68	4	68.4	1.87
10%	5	74.6	2.94	4	123.6	2.36
20%	5	126.8	5.57	4	208.0	4.95
30%	5	200.0	6.39	4	327.2	9.26
40%	5	468.0	19.5	4	762.4	45.1
3rd cycle unload, kPa, at deflection of						
5%	5	31.9	1.54	4	45.0	0.96
10%	5	60.8	2.62	4	87.7	1.68
20%	5	98.8	4.55	4	147.3	3.19
30%	5	153.4	3.76	4	234.8	7.37
40%	5	400.6	12.5	4	626.7	43.4
3rd cycle U/L at deflections of						
10%	5	0.816	0.0038	4	0.709	0.0063
20%	5	0.779	0.0026	4	0.708	0.0042
30%	5	0.767	0.0087	4	0.718	0.0026
3rd cycle hysteresis loop (Δ area)	5	170.5	46.1	4	367.0	12.7
Thickness, mm						
1st cycle	5	2.44	0.042	4	2.43	0.047
3rd cycle	5	2.14	0.040	4	2.35	0.047
Δt	5	0.047	0.0040	4	0.072	0.0096
Density, Mg/m ³	5	0.6541	0.0038	5	0.6477	0.0048

Table 15. Confined-compression-set properties at 35% compression of L3223 cushions made from SE54 silicone gums and Y1587 or SE4210 processing aid.

Property	Lot 161						Lot 167					
	Y1587			TS50 and Urea			Y1587			TS50 and Urea		
	BS and NH ₄ Cl			TJ109			TJ113A			SE4210		
	n	Av	S	n	Av	S	n	Av	S	n	Av	S
Original thickness, mm	4	2.00	0.028	4	2.03	0.046	4	2.17	0.037	4	2.34	0.053
Density, Mg/m ³	4	0.6320	0.0058	4	0.6708	0.0019	4	0.6460	0.0027	4	0.6405	0.0011
Compression, %	4	34.97	0.100	4	34.95	0.081	—	—	—	—	—	—
Set, %	4	23.17	2.17	4	34.26	1.45	4	10.85	1.54	4	10.55	0.49
Recovery, % of original thickness after												
2 min	4	88.45	2.37	4	81.88	1.15	4	93.05	1.53	4	95.33	0.36
1 hr	4	91.85	0.77	4	88.03	0.49	4	96.20	0.54	4	96.30	0.16
24 hrs	4	93.55	0.30	4	91.08	0.43	4	97.30	0.37	4	96.78	0.21
7 days	4	94.30	0.42	4	93.10	0.27	4	98.03	0.51	4	97.23	0.30
28 days	4	95.40	0.36	4	94.18	0.54	4	98.73	0.41	4	97.88	0.47

Table 16. Load-deflection properties of L3223 cushions made from SE54, lot 167, silicone gum with Y1587 processing aid and with no processing aid.

Property	Y1587			TJ115CBA			None		
	n	Av	S	n	Av	S	n	Av	S
3rd cycle load, kPa, at deflections of									
5%	5	41.0	0.58	3	37.2	1.04	3	45.3	0.62
10%	5	71.2	1.38	3	62.8	1.65	3	81.8	1.01
20%	5	119.4	2.13	3	105.7	4.10	3	142.4	1.57
30%	5	198.4	2.98	3	180.1	12.9	3	239.1	2.23
40%	5	519.1	7.33	1	498.2	—	3	637.8	10.2
3rd cycle unload, kPa, at deflections of									
5%	5	26.8	0.26	3	22.6	0.45	3	29.3	0.71
10%	5	49.5	0.73	3	41.7	0.89	3	57.8	0.89
20%	5	84.0	0.95	3	72.1	0.52	3	103.8	1.32
30%	5	141.8	1.86	3	127.2	5.82	3	178.7	1.63
40%	5	449.5	9.14	1	442.5	—	3	587.8	17.5
3rd cycle U/L at deflections of									
10%	5	0.696	0.0063	3	0.664	0.0205	3	0.707	0.0046
20%	5	0.703	0.0060	3	0.683	0.0219	3	0.729	0.0026
30%	5	0.715	0.0025	3	0.707	0.0196	3	0.748	0.0021
3rd cycle hysteresis loop (Δ area)	5	221.0	10.7	3	201.5	26.2	3	232.9	12.8
Thickness, mm									
1st cycle	5	2.76	0.035	3	2.62	0.016	3	2.67	0.026
3rd cycle	5	2.70	0.035	3	2.52	0.015	3	2.58	0.023
Δt	5	0.067	0.0034	3	0.101	0.0104	3	0.088	0.0032
Density, Mg/m ³	5	0.6687	0.0015	4	0.660	0.0111	4	0.6625	0.0037

Table 17. Confined-compression-set properties at 35% compression of L3223 cushions made from SE54, lot 167, silicone gum with Y1587 processing aid and with no processing aid.

Property	Y1587			TJ115CBA			None		
	n	Av	S	n	Av	S	n	Av	S
Original thickness, mm	4	2.75	0.038	3	2.62	0.018	3	2.73	0.046
Density, Mg/m ³	4	0.6671	0.0047	3	0.6392	0.0073	4	0.6435	0.0029
Compression, %	4	35.02	0.014	—	—	—	—	—	—
Set, %	3	10.38	0.58	3	4.83	2.04	4	4.99	0.65
Recovery, % of original thickness after									
2 min	3	88.87	2.24	3	97.87	0.60	4	97.71	0.64
1 hr	3	96.36	0.21	3	98.29	0.73	4	98.25	0.22
24 hrs	2	97.45	0.071	3	98.68	0.65	4	98.56	0.062
7 days	4	97.85	0.55	3	98.62	0.62	4	98.73	0.16
28 days	4	98.10	0.48	3	98.87	0.65	4	98.98	0.29

Table 18. Properties of uncured L1668's made from L97KVB silicone gum and Y1587 or SE4210 processing aid.

Property	Y1587		SE4210
	TJ150-2	TJ150	TJ150SE
Hardness, Rex O after aging			
0 days	44	46	40
4 weeks	60	60	61
After aging 4 weeks			
Loss, wt%	4.47	5.1	5.50
CHCl ₃ extractables, wt%	20.67	17.10	—
V/V _o in chlorobenzene	6.09	6.09	—
Stripping loss, wt%	7.13	8.69	7.95
		TJ172	TJ168
Hardness, Rex O, after aging			
0 days	34	22	
28 days	50	—	
33 days	—	42	
Loss, wt%, after aging			
28 days	6.78	—	
33 days	—	6.54	
Stripping loss, wt%	11.3	9.42	

as useful a measurement as a 28-day recovery so we ceased measuring 28-day recoveries.

For L97KVB, SE4210 produces a stiffer cushion than Y1587 but has no significant effect on compression set. Its effect on resiliency is variable and depends upon the amount of SiO₂ present.

B. MN97KVB. TJ164ELU and TJ176EBU are standard formulations comparable to the L3223's from L97KVB but they were both press cured for 100 min instead of 60 min. Also TJ164ELU was filled with LLNL urea instead of BKC urea; the urea source is not considered a significant variable.

The molding compound for the L3223 with Y1587 was compounded in the Banbury mixer and that with SE4210 was compounded in the Haake mixer. We do not consider the mixer to be a significant variable but we are still evaluating this

difference. Load-deflection results for the standard L3223's from MN97KVB are listed in Table 26 and their set properties are shown in Table 27. Again, as with the standard formulations from L97KVB, SE4210 produces a much stiffer and less resilient cushion but does not effect compression set on 7-day recovery.

L3260's

All of our early work was done with L3223 which is defined by a nominal cushion density of 0.65 Mg/m³ corresponding to ~45% porosity because that was the material of weapon interest. Later the interest shifted to a softer, lower density material such as L3260 which is defined by a nominal density of 0.50 Mg/m³ (~57% porosity) so we began to build up a body of knowledge on L3260. The compositions and cure cycles of all L3260's relevant to the processing aid study are shown in Table 28.

A. L97KVB. We attempted to prepare L3260 from our modified formulations with Y1587 (TJ172) and SE4210 (TJ168) but could not mill either formulation into a useful molding compound.

B. MN97KVB. TJ175ELU and TJ176EBU are standard formulations but there are differences. TJ175 used LLNL urea and was compounded in the Banbury mixer and TJ176 used BKC urea and was compounded in the Haake mixer. These differences are not considered to be significant.

Table 19. Properties of uncured L1668's made from MN97KVB silicone gum and Y1587 or SE4210 processing aid.

Property	Y1587		SE4210
	TJ164	TJ175	TJ176
Hardness, Rex O, after aging			
0 days	55	51	50
28 days	73	70	76
Loss, wt%, after aging 28 days	6.39	6.26	5.81
Stripping loss, wt%	10.6	10.7	8.7

Table 20. Properties of cured L3219's made from L97KVB silicone gum and Y1587 or SE4210 processing aid.

Property	32 parts MS7			25 parts MS7	
	6 parts HiSil 233		SE4210	No parts HiSil 233	
	Y1587	TJ150-2E		TJ150E ^a	TJ168E
Hardness, Shore A					
Cured	65	69	69	62	65
Post cured	68	71	75	62	63
Loss on post cure, wt%	3.14	1.83	2.31	1.89	1.33
Density, Mg/m ³	1.193	1.193	1.198	1.141	1.143
CHCl ₃ extractables, wt%	1.48	1.16	1.04	1.07	0.91
V/V ₀ in chlorobenzene	2.34	2.26	1.94	2.51	2.47
Tensile properties					
n	5	5	5	5	5
Ultimate tensile strength, kPa					
Av	3516	3179	3509	1717	1641
S	359	207	221	193	117
% C of V	10.2	6.51	6.30	11.3	7.13
Ultimate elongation, %					
Av	90	74	60	70	66
S	10	2	7	10	5
% C of V	11.1	2.70	11.7	14.3	7.58

^a Heat stripped L668 after 1 week bin aging.

Table 21. Properties of cured L3219's made from MN97KVB silicone gum and Y1587 or SE4210 processing aid.

Property	Y1587		SE4210
	TJ164	TJ175	TJ176
Hardness, Shore A			
Cured	68	70	69
Post cured	68	70	71
Loss on post cure, wt%	2.41	3.77	2.13
Density, Mg/m ³	1.187	1.191	1.200
CHCl ₃ extractables, wt%	—	1.09	0.97
V/V ₀ in chlorobenzene	2.30	2.29	2.21
Tensile properties			
n	5	5	5
Ultimate tensile strength, kPa			
Av	3082	2903	3206
S	200	186	379
% C of V	6.49	6.41	11.8
Ultimate elongation, %			
Av	80	76	70
S	10	5	7
% C of V	12.5	6.58	10.1

Table 22. Load-deflection properties of L3223 cushions made from L97KVB silicone gum and Y1587 or SE4210 processing aid.

Property	Y1587			TJ150EBU			SE4210		
	TJ150-2EBU			TJ150EBU			TJ150SEEBU		
	n	Av	S	n	Av	S	n	Av	S
3rd cycle load, kPa, at deflections of									
5%	4	78.5	1.92	5	72.8	0.99	4	100.2	1.95
10%	4	145.1	2.58	5	134.3	1.59	4	182.8	4.03
20%	4	258.2	3.73	5	239.0	2.92	4	336.3	9.57
30%	4	417.6	5.82	5	378.0	5.06	4	590.2	24.3
40%	4	969.2	23.4	5	831.1	10.4	—	—	—
3rd cycle unload, kPa, at deflections of									
5%	4	61.0	1.88	5	55.6	2.01	4	65.2	0.51
10%	4	115.8	2.46	5	106.3	2.52	4	130.6	1.66
20%	4	202.7	3.57	5	186.4	2.97	4	250.4	6.42
30%	4	333.7	6.16	5	300.6	0.89	4	460.9	20.6
40%	4	890.7	34.4	5	763.1	13.9	—	—	—
3rd cycle U/L at deflections of									
10%	4	0.798	0.0031	5	0.791	0.0119	4	0.715	0.0079
20%	4	0.785	0.0026	5	0.780	0.0054	4	0.745	0.0040
30%	4	0.799	0.0035	5	0.795	0.0044	4	0.781	0.0032
3rd cycle hysteresis loop (Δ area)	4	314.1	4.33	5	285.2	15.6	4	424.7	16.3
Thickness, mm									
1st cycle	4	2.77	0.037	5	2.73	0.033	4	2.84	0.034
3rd cycle	4	2.72	0.029	5	2.67	0.030	4	2.77	0.028
Δt	4	0.047	0.0086	5	0.058	0.0168	4	0.069	0.0066
Density, Mg/m ³	5	0.6559	0.0006	5	0.651	0.005	5	0.6573	0.0021

Load-deflection results for these standard L3260's from MN97KVB are listed in Table 29. For L3223, we load to a minimum of 40% deflection but for lower density L3260 we load to a minimum of 45% deflection. Compression-set properties of the two cushions made from MN97KVB are shown in Table 30. Again, because of the density difference we compress L3223 to a nominal 35% of original thickness and L3260 to a nominal 40%. The cushion with SE4210 is slightly stiffer but also has a higher density which could account for the increase in stiffness at higher compressions. The density difference may be due to the use of different mixers, a weighing error, or to the use of different processing aids. SE4210 produces a considerably less resilient cushion than does Y1587 and this is probably not related to the density difference. SE4210 increases set significantly.

Type 60VB Silicone Gums

L1668's

A. L60VB. The properties of the three standard uncured L1668's and two modified L1668's made from our L60VB silicone gum are listed in Table 31. Duplicate batches of TJ154 with Y1587 were prepared and tested and then combined before stripping to produce one large batch of L3219. The duplication of the two batches of TJ154 is very good. SE4210 produces a harder uncured gum than Y1587, but otherwise the two L1668's are alike. For the modified formulations SE4210 produces a softer uncured gum and has less weight loss on stripping than Y1587.

B. MN60VB experimental gums. Data for the McGhan-NuSil 60VB formulations are included to

Table 23. Load-deflection properties of L3223 cushions made from L97KVB silicone gum and Y1587 or SE4210 processing aid, and containing 25 parts of MS7 and no HiSil 233.

Property	Y1587 TJ172EBU			SE4210 TJ168EBU		
	n	Av	S	n	Av	S
3rd cycle load, kPa, at deflections of						
5%	3	56.3	1.57	4	62.4	1.76
10%	3	111.3	3.57	4	122.9	2.72
20%	3	192.4	7.52	4	214.4	3.95
30%	3	292.8	11.6	4	327.9	6.45
40%	3	605.3	31.6	4	695.8	25.3
3rd cycle unload, kPa, at deflections of						
5%	3	44.6	0.89	4	52.7	1.82
10%	3	94.1	2.61	4	106.8	2.98
20%	3	162.3	5.85	4	181.5	3.79
30%	3	244.4	7.26	4	275.6	5.47
40%	3	541.2	9.67	4	622.7	26.2
3rd cycle U/L at deflections of						
10%	3	0.846	0.0037	4	0.868	0.0055
20%	3	0.843	0.0027	4	0.847	0.0029
30%	3	0.835	0.0084	4	0.841	0.0010
3rd cycle hysteresis loop (Δ area)	3	200.0	31.0	4	201.1	9.82
Thickness, mm						
1st cycle	3	2.81	0.023	4	2.80	0.039
3rd cycle	3	2.77	0.024	4	2.77	0.033
Δt	3	0.028	0.026	4	0.028	0.021
Density, Mg/m³	5	0.6424	0.0068	5	0.6449	0.0033

Table 24. Confined-compression-set properties at 35% compression of L3223 cushions made from L97KVB silicone gum and Y1587 or SE4210 processing aid.

Property	Y1587			SE4210		
	TJ150-2EBU	n	Av	TJ150EBU	n	Av
Original thickness, mm	3	2.74	0.038	4	2.69	0.0094
Density, Mg/m ³	3	0.6456	0.0012	4	0.6434	0.0024
Compression, %	3	34.98	0.023	3	35.04	0.12
Set, %	3	5.66	0.38	3	5.53	0.44
Recovery, % of original thickness after						
2 min	3	97.83	0.14	3	97.84	0.25
1 hr	3	98.02	0.13	3	98.06	0.16
24 hrs	3	98.15	0.14	3	98.29	0.04
7 days	3	98.42	0.14	3	98.54	0.16
28 days	3	98.63	0.09	3	98.53	0.22

Table 25. Confined-compression-set properties at 35% compression of L3223 cushions made from L97KVB silicone gum and Y1587 or SE4210 processing aid, and containing 25 parts of MS7 and no HiSil 233.

Property	Y1587 TJ172EBU			SE4210 TJ168EBU		
	n	Av	S	n	Av	S
Original t, mm	3	2.76	0.030	4	2.78	0.029
Density, Mg/m ³	3	0.6360	0.0038	4	0.6445	0.0034
Compression, %	3	35.09	0.14	4	34.97	0.059
Set, %	3	5.85	0.83	3	5.29	0.59
Recovery, % of original thickness after						
1 hr	3	97.95	0.30	3	98.15	0.21
7 days	3	98.19	0.21	3	98.53	0.17

Table 26. Load-deflection properties of L3223 cushions made from MN97KVB silicone gum and Y1587 or SE4210 processing aid.

Property	Y1587 TJ172EBU			SE4210 TJ168EBU		
	n	Av	S	n	Av	S
3rd cycle load, kPa, at deflections of						
5%	5	62.5	0.51	3	82.9	1.41
10%	5	119.9	0.75	3	152.5	2.53
20%	5	217.9	1.47	3	288.6	4.73
30%	5	344.6	1.84	3	520.6	8.53
40%	5	742.1	6.23	1	1503.6	—
3rd cycle unload, kPa, at deflections of						
5%	5	48.3	0.70	3	60.2	1.11
10%	5	96.7	0.61	3	117.9	1.89
20%	5	173.2	1.00	3	226.6	3.69
30%	5	274.8	2.14	3	416.3	7.64
40%	5	647.8	15.3	—	—	—
3rd cycle U/L at deflections of						
10%	5	0.806	0.0034	3	0.773	0.0035
20%	5	0.795	0.0024	3	0.785	0.0021
30%	5	0.797	0.0044	3	0.800	0.0028
3rd cycle hysteresis loop (Δ area)	5	273.4	18.6	3	346.4	5.80
Thickness, mm						
1st cycle	5	2.75	0.013	3	2.76	0.032
3rd cycle	5	2.72	0.015	3	2.70	0.033
Δt	5	0.031	0.015	3	0.053	0.0022
Density, Mg/m ³	5	0.6565	0.0016	5	0.6721	0.0018

Table 27. Confined-compression-set properties at 35% compression of L3223 cushions made from MN97KVB silicone gum and Y1587 or SE4210 processing aid.

Property	Y1587			SE4210		
	n	Av	S	n	Av	S
Original thickness, mm	3	2.72	0.033	5	2.71	0.038
Density, Mg/m ³	3	0.6572	0.0025	5	0.6695	0.0023
Compression, %	3	34.99	0.075	3	34.95	0.12
Set, %	3	5.91	0.70	3	5.61	0.64
Recovery, % of original thickness after						
2 min	3	97.95	0.34	—	—	—
1 hr	3	97.92	0.26	3	98.04	0.22
24 hrs	3	97.96	0.046	—	—	—
7 days	3	98.11	0.093	3	98.38	0.27
28 days	3	98.40	0.093	—	—	—

illustrate the complexity of the interaction of gum, SiO₂ and processing aid and our lack of understanding of it. The only measured property of the MN60VB's that shows any significant variation is molecular weight which ranges over a factor of two, yet the processability of these three gums varied widely. Properties of the three pairs of formulations that we tried to make are shown in Table 32. Both the high and low molecular weight gums could be processed only with SE4210. For DR114 SE4210 produces a harder uncured gum with less weight loss on aging and less weight loss on stripping than does Y1587.

L3219's

A. L60VB. Properties of the two pairs of cured L3219's from the standard and modified formula-

tions of L60VB are listed in Table 33. TJ177E was cured for 100 min instead of 60 min like the rest. For both the standard and modified formulations the type of processing aid has no significant effect on the properties of cured L3219 elastomer.

B. MN60VB experimental gums. The four formulations that were processable into L1668's were cured to L3219 elastomers with the properties shown in Table 34. For the only comparison of Y1587 and SE4210 that can be made (gum DR114), there is no major difference in processing aids. All four L3219's are much alike.

L3223's

A. L60VB. The load-deflection properties of L3223 cushions made from standard formulations of L60VB are shown in Table 35 and their

Table 28. Compositions and cure cycles of L3260 molding compounds and cushions.

Sample Ident.	Gum Ident.	L3219 Ident.	Filler		Curing Agent Type	Cure Cycle Min	Post cure cycle hrs	Post cure cycle C°
			Type	Source				
TJ172EBU	L97KVB-TJ150	TJ172	Urea	BKC	170	497XL	2.0	—
TJ168EBU	L97KVB-TJ150	TJ168	Urea	BKC	170	497XL	2.0	—
TJ175ELU	MN97KVB-RD004067	TJ175	Urea	LLNL	170	497XL	2.0	100 125 24 249
TJ175EBU	MN97KVB-RD004067	TJ176	Urea	BKC	170	497XL	2.0	100 125 24 249
TJ154EBU	L60VB	TJ154	Urea	BKC	170	497XL	2.0	100 125 24 249
TJ177EBU	L60VB	TJ177	Urea	BKC	170	497XL	2.0	100 125 24 249
TJ173EBU	L60VB	TJ173	Urea	BKC	170	497XL	2.0	60 125 24 249
TJ169EBU	L60VB	TJ169	Urea	BKC	170	497XL	2.0	—

^a Parts per hundred parts of L3219, by weight.

Table 29. Load-deflection properties of L3260 cushions made from MN97KVB silicone gum and Y1587 or SE4210 processing aid.

Property	Y1587			SE4210		
	Banbury Mixer LLNL Urea TJ175ELU			Haake Mixer BKC Urea TJ176EBU		
	n	Av	S	n	Av	S
3rd cycle load, kPa, at deflections of						
5%	5	34.8	0.63	4	35.2	1.03
10%	5	63.5	1.23	4	66.3	3.20
20%	5	108.5	2.94	4	123.3	6.89
30%	5	161.1	4.88	4	202.4	11.7
40%	5	266.3	9.77	4	383.2	24.9
3rd cycle unload, kPa, at deflections of						
5%	4	29.5	0.62	4	25.0	0.89
10%	4	54.5	1.14	4	50.4	2.72
20%	4	91.0	2.86	4	96.0	5.63
30%	4	134.3	4.85	4	159.2	1.59
40%	4	229.7	10.6	4	313.9	21.7
3rd cycle U/L at deflections of						
10%	4	0.854	0.0036	4	0.760	0.0058
20%	4	0.837	0.0030	4	0.779	0.0041
30%	4	0.832	0.0022	4	0.787	0.0026
3rd cycle hysteresis loop (Δ area)	4	127.6	9.52	4	220.2	11.0
Thickness, mm						
1st cycle	4	2.72	0.021	4	2.70	0.008
3rd cycle	5	2.68	0.019	4	2.65	0.017
Δt	4	0.033	0.0036	4	0.055	0.017
Density, Mg/m³	5	0.5226	0.0052	5	0.5507	0.0048

Table 30. Confined-compression-set properties at 40% compression of L3260 cushions made from MN97KVB silicone gum and Y1587 or SE4210 processing aid.

Property	Y1587			SE4210		
	Banbury Mixer LLNL Urea TJ175ELU			Haake Mixer BKC Urea TJ176EBU		
	n	Av	S	n	Av	S
Original thickness, mm	3	2.68	0.023	6	2.68	0.047
Density, Mg/m³	3	0.5205	0.0046	6	0.5461	0.0061
Compression, %	3	40.46	0.39	3	39.93	0.079
Set, %	3	4.59	0.68	3	7.29	0.65
Recovery, % of original thickness after						
1 hr	3	98.31	0.41	3	97.09	0.26
7 days	3	98.29	0.18	3	97.84	0.10

Table 31. Properties of uncured L1668's made from L60VB silicone gum and Y1587 or SE4210 processing aid.

Property	Y1587		SE4210 TJ177
	Batch 1 TJ154	Batch 2	
Hardness, Rex O, after aging			
0 days	37	—	38
1 day	—	45	—
28 days	52	53	65
After aging 28 days			
Loss, wt%	1.91	1.90	1.86
CHCl ₃ extractables, wt%	33.0	25.6	—
V/V ₀ in chlorobenzene	4.87	5.06	—
Stripping loss, wt%		4.45 ^a	4.42
		<u>TJ173</u>	<u>TJ169</u>
Hardness, Rex O, after aging			
0 days	27	15	—
28 days	41	—	—
33 days	—	33	—
Loss, wt%, after aging			
28 days	2.49	—	—
33 days	—	2.29	—
Stripping loss, wt%		6.86	4.67

^a Combined batches.

Table 32. Properties of uncured L1668's made from experimental MN60VB silicone gums and Y1587 or SE4210 processing aid.

Property	DR-115		DR-114		RD001VR26	
	Y1587 TJ159-I ^a	SE4216 TJ159-II	Y1587 TJ160-I	SE4210 TJ160-II	Y1587 TJ161-I ^a	SE4210 TJ161-II
Hardness, Rex O, after aging						
0 days	—	43	33	32	—	32
28 days	—	54	49	59	—	53
Loss, wt%, after aging 28 days	—	0.84	0.81	0.57	—	1.56
Stripping loss, wt%	—	3.59	4.70	2.71	—	4.07

^a Cannot be processed.

Table 33. Properties of cured L3219's made from L60VB silicone gum and Y1587 or SE4210 processing aid.

Property	Standard Formulation ^a		Modified Formulation ^b	
	Y1587 TJ154E	SE4210 TJ177E	Y1587 TJ173E	SE4210 TJ169E
Hardness, Shore A				
Cured	63	65	63	60
Post cured	63	68	63	59
Loss on post cure, wt%	2.67	1.97	1.73	1.53
Density, Mg/m ³	1.178	1.180	1.123	1.123
CHCl ₃ extractables, wt%	1.18	0.96	0.85	0.78
V/V ₀ in chlorobenzene	2.28	2.25	2.50	2.55
Tensile properties				
n	5	5	5	5
Ultimate tensile strength, kPa				
Av	3144	2834	1434	1413
S	496	317	110	90
% C of V	15.8	11.2	7.67	6.37
Ultimate elongation, %				
Av	52	74	58	68
S	11	11	5	4
% C of V	21.2	14.9	8.62	5.88

^a Thirty-two parts of MS7 and six parts of HiSil 233.

^b Twenty-five parts of MS7 and no HiSil 233.

Table 34. Properties of cured L3219's made from experimental MN60VB silicone gums and Y1587 or SE4210 processing aid.

Property	DR115 ^a	DR114		RDOOIVR26 ^b
	SE4216 TJ159-IIIE	Y1587 TJ160-IE	SE4210 TJ160-IIIE	SE4210 TJ161-IIIE
Hardness, Shore A				
Cured	65	65	65	68
Post cured	67	67	68	69
Loss on post cure, wt%	1.10	1.67	1.39	1.45
Density, Mg/m ³	1.175	1.163	1.180	1.183
CHCl ₃ extractables, wt%	0.90	1.10	0.90	0.62
V/V ₀ in chlorobenzene	2.20	2.31	2.19	2.14
Tensile properties				
n	5	5	5	5
Ultimate tensile strength, kPa				
Av	2130	1986	2110	2199
S	165	97	255	207
% C of V	7.75	4.88	12.1	9.41
Ultimate elongation, %				
Av	48	62	46	42
S	4	4	5	8
% C of V	8.33	6.45	10.9	19.0

^a L1668 with Y1587 from this gum (TJ160-IIIE) could not be processed.

^b L1668 with Y1587 from this gum (TJ161-I) could not be processed.

compression-set properties are listed in Table 36. Corresponding data for modified formulations are listed in Tables 37 and 38. The two standard formulations were made in different mixers. The standard SE4210 cushion has a greater density and also is stiffer and less resilient than the standard Y1587 cushion. SE4210 results in a significant increase in compression set.

For the modified formulations, Y1587 produces a slightly softer cushion than SE4210 but resiliency is the same. The type of processing aid has no effect on compression set or 7-day recovery of the modified formulations.

B. MN60VB experimental gums. Of the four processable L1668's from the three experimental MN60VB's only the one L1668 based on lot DR114 and Y1587 could be processed into L3223. Load-deflection and set properties of this single L3223 (TJ160-IEBU) are listed in Tables 39 and 40 respec-

tively. Although SE4210 allowed all three gums to be processed through the cured L3219 stage compared to one gum for Y1587, the only formulation to be successfully molded into an L3223 cushion was based on Y1587. Y1587 seems to have more versatility in accommodating the vagaries of silicone gums than does SE4210.

L3260's

A. L60VB. The load-deflection properties of L3260 cushions made from standard formulation L60VB are shown in Table 41 and those of L3260 from modified formulations are listed in Table 42. Corresponding compression set data are shown in Table 43. The SE4210 standard cushion is denser and stiffer at higher compressions, is less resilient, and has considerably higher compression set than the Y1587 cushion. The modified formulation could not be processed with SE4210.

Table 35. Load-deflection properties of L3223 cushions made from L60VB silicone gum and Y1587 or SE4210 processing aid.

Property	Y1587			SE4210				
	Banbury Mixer TJ174EBU	n	Av	S	Haake Mixer TJ177EBU	n	Av	S
3rd cycle load, kPa, at deflections of								
5%	5	60.5	2.85		3	67.9		1.07
10%	5	116.4	5.57		3	132.7		0.67
20%	5	209.4	10.8		3	264.5		1.68
30%	5	329.8	17.7		3	485.6		6.14
40%	5	703.1	43.4		3	1451.7		35.3
3rd cycle unload, kPa, at deflections of								
5%	5	50.9	2.47		3	53.3		0.58
10%	5	100.7	5.26		3	109.3		0.27
20%	5	176.2	9.39		3	219.1		2.03
30%	5	275.5	15.1		3	404.2		7.03
40%	5	639.8	40.9		3	1429.0		74.6
3rd cycle U/L at deflections of								
10%	5	0.864	0.0044		3	0.824		0.0039
20%	5	0.842	0.0022		3	0.828		0.0029
30%	5	0.835	0.0026		3	0.832		0.0041
3rd cycle hysteresis loop (Δ area)	5	198.1	13.8		3	243.1		24.2
Thickness, mm								
1st cycle	5	2.77	0.027		3	2.70		0.030
3rd cycle	5	2.73	0.022		3	2.66		0.029
Δt	5	0.041	0.0061		3	0.044		0.0109
Density, Mg/m³	5	0.6466	0.0020		5	0.6812		0.0038

Table 36. Confined-compression-set properties at 35% compression of L3223 cushions made from L60VB silicone gum and Y1587 or SE4210 processing aid.

Property	Y1587			SE4210				
	Banbury Mixer TJ175ELU	n	Av	S	Haake Mixer TJ176EBU	n	Av	S
Original thickness, mm	3	2.71		0.036	6	2.70		0.060
Density, Mg/m ³	3	0.6442		0.0040	6	0.6771		0.0064
Compression, %	3	35.03		0.13	4	35.04		0.20
Set, %	3	3.84		0.29	4	6.97		0.33
Recovery, % of original thickness after								
2 min	3	98.22		0.021	—	—	—	—
1 hr	3	98.65		0.10	4	97.56		0.11
24 hrs	3	98.78		0.18	—	—	—	—
7 days	3	98.93		0.24	4	98.08		0.16
28 days	3	99.29		0.23	—	—	—	—

Table 37. Load-deflection properties of L3223 cushions made from L60VB silicone gum and Y1587 or SE4210 processing aid, and containing 25 parts of MS7 and no HiSil 233.

Property	Y1587			SE4210				
	TJ173EBU	n	Av	S	TJ169EBU	n	Av	S
3rd cycle load, kPa, at deflections of								
5%	4	55.6		0.69	5	60.0		2.27
10%	4	110.5		1.22	5	117.9		4.18
20%	4	190.4		3.69	5	205.1		7.50
30%	4	282.2		6.83	5	311.6		12.4
40%	4	575.2		25.6	5	639.8		41.7
3rd cycle unload, kPa, at deflections of								
5%	4	51.5		0.41	5	55.4		2.23
10%	4	113.1		1.12	5	110.3		3.94
20%	4	172.0		3.17	5	186.6		3.62
30%	4	251.6		5.33	5	279.2		3.76
40%	4	537.7		19.1	5	589.2		35.2
3rd cycle U/L at deflections of								
10%	4	0.933		0.0047	5	0.935		0.0028
20%	4	0.903		0.0015	5	0.910		0.0032
30%	4	0.891		0.0031	5	0.896		0.0039
3rd cycle hysteresis loop (Δ area)	4	112.0		9.43	5	119.2		12.9
Thickness, mm								
1st cycle	4	2.78		0.0371	5	2.84		0.0310
3rd cycle	4	2.73		0.0248	5	2.81		0.0274
Δt	4	0.049		0.0183	5	0.030		0.0056
Density, Mg/m ³	5	0.6233		0.0030	5	0.6334		0.0045

Table 38. Confined-compression-set properties at 35% compression of L3223 cushions made from L60VB silicone gum and Y1587 or SE4210 processing aid, and containing 25 parts of MS7 and no HiSil 233.

Property	Y1587 TJ175ELU			SE4210 TJ176EBU		
	n	Av	S	n	Av	S
Original thickness, mm	3	2.74	0.031	5	2.80	0.040
Density, Mg/m ³	3	0.6200	0.0013	5	0.6302	0.0024
Compression, %	3	35.17	0.26	5	35.04	0.12
Set, %	3	3.69	0.43	5	3.21	0.10
Recovery, % of original thickness after						
1 hr	3	98.70	0.15	5	98.88	0.032
7 days	3	98.74	0.21	5	99.21	0.24

Table 39. Load-deflection properties of L3223 cushions made from experimental MN60VB silicone gum, DR114, and Y1587 processing aid.

Property	TJ160-IEBU		
	n	Av	S
3rd cycle load, kPa, at deflections of			
5%	4	61.5	4.79
10%	4	123.2	7.78
20%	4	222.5	15.9
30%	4	362.9	33.8
40%	4	813.8	128.4
3rd cycle unload, kPa, at deflections of			
5%	4	53.3	3.86
10%	4	109.5	6.13
20%	4	192.5	12.4
30%	4	312.1	26.5
40%	4	739.5	108.7
3rd cycle U/L at deflections of			
10%	4	0.885	0.0098
20%	4	0.866	0.0080
30%	4	0.861	0.0091
3rd cycle hysteresis loop (Δ area)	4	184.1	18.7
Thickness, mm			
1st cycle	4	2.76	0.0244
3rd cycle	4	2.73	0.0279
Δt	4	0.0236	0.0043
Density, Mg/m ³	5	0.6461	0.0157

Table 40. Confined-compression-set properties at 35% compression of L3223 cushions made from experimental MN60VB silicone gum, DR114, and Y1587 processing aid.

Property	TJ160-IEBU		
	n	Av	S
Original thickness, mm	3	2.73	0.025
Density, Mg/m ³	3	0.6521	0.0194
Compression, %	3	34.97	0.075
Set, %	3	3.82	0.54
Recovery, % of original thickness after			
2 min	3	98.55	0.20
1 hr	3	98.66	0.19
24 hrs	3	98.91	0.22
7 days	3	98.64	0.04
28 days	3	98.69	0.17

Table 41. Load-deflection properties of L3260 cushions made from L60VB cellular silicone gum and Y1587 or SE4210 processing aid.

Property	Y1587			SE4210		
	Banbury Mixer TJ154EBU	n	Av	S	n	Av
3rd cycle load, kPa, at deflections of						
5%	5	33.5	0.68	3	30.7	0.18
10%	5	60.8	1.60	3	56.3	0.76
20%	5	101.5	3.03	3	104.1	2.61
30%	5	145.5	4.77	3	171.1	6.81
40%	5	229.1	9.49	3	339.6	8.10
3rd cycle unload, kPa, at deflections of						
5%	5	28.7	0.40	3	25.0	0.28
10%	5	52.9	0.89	3	47.2	0.58
20%	5	86.5	1.86	3	87.7	2.26
30%	5	123.2	3.22	3	145.4	6.82
40%	5	198.7	7.47	3	294.6	35.7
3rd cycle U/L at deflections of						
10%	5	0.869	0.0076	3	0.839	0.0049
20%	5	0.852	0.0062	3	0.843	0.0057
30%	5	0.846	0.0083	3	0.849	0.0129
3rd cycle hysteresis loop (Δ area)	5	112.8	12.8	2	127.9	13.3
Thickness, mm						
1st cycle	5	2.69	0.014	3	2.66	0.008
3rd cycle	5	2.66	0.010	3	2.61	0.003
Δt	5	0.031	0.0094	3	0.047	0.011
Density, Mg/m³	5	0.5027	0.0026	5	0.5391	0.0059

Table 42. Load-deflection properties of L3260 cushions made from L60VB silicone gum, Y1587 processing aid, and containing 25 parts of MS7 and no HiSil 233. The modified formulation with SE4210 could not be processed.

Property	Y1587 TJ173EBU			SE4210 TJ169EBU ^a		
	n	Av	S	n	Av	S
3rd cycle load, kPa, at deflections of						
5%	5	30.4	0.67	—	—	—
10%	5	56.6	1.15	—	—	—
20%	5	87.8	2.17	—	—	—
30%	5	119.0	2.78	—	—	—
40%	5	179.1	3.95	—	—	—
3rd cycle unload, kPa, at deflections of						
5%	5	28.3	0.68	—	—	—
10%	5	52.9	1.19	—	—	—
20%	5	79.9	2.01	—	—	—
30%	5	106.9	2.55	—	—	—
40%	5	162.3	3.83	—	—	—
3rd cycle U/L at deflections of						
10%	5	0.936	0.0021	—	—	—
20%	5	0.910	0.0015	—	—	—
30%	5	0.898	0.0015	—	—	—
3rd cycle hysteresis loop (Δ area)	5	60.1	1.87	—	—	—
Thickness, mm						
1st cycle	5	2.79	0.039	—	—	—
3rd cycle	5	2.76	0.027	—	—	—
Δt	5	0.038	0.0170	—	—	—
Density, Mg/m ³	5	0.4807	0.0037	—	—	—

^a Could not be processed.

Table 43. Confined-compression-set properties at 40% compression of L3260 cushions made from L60VB and Y1587 or SE4210 processing aid.

Property	32 parts MS7 6 parts HiSil 233						25 parts MS7 no HiSil 233		
	SE4210 Haake Mixer TJ177EBU			Y1587 Banbury Mixer TJ154EBU			Y1587 Banbury Mixer TJ173EBU		
	n	Av	S	n	Av	S	n	Av	S
Original thickness, mm	4	2.61	0.014	3	2.63	0.020	3	2.73	0.051
Density, Mg/m ³	4	0.5356	0.0050	3	0.5129	0.0182	3	0.4798	0.0006
Compression, %	3	40.09	0.17	3	39.99	0.12	3	40.23	0.41
Set, %	3	7.02	0.65	3	3.30	0.63	3	2.61	0.55
Recovery, % of original thickness after									
2 min	—	—	—	3	98.33	0.35	—	—	—
1 hr	3	97.18	0.27	3	98.68	0.25	3	98.95	0.23
24 hrs	—	—	—	2	98.85	—	—	—	—
7 days	3	97.89	0.24	3	98.90	0.05	3	99.12	0.13
28 days	—	—	—	3	99.00	0.06	—	—	—

Conclusions

L1668's

The interpretation of results is aided by a tabulation of results for a specific property such as uncured hardness of L1668 after bin aging in Table 44.

For SE54 gum with 38 parts of MS7 and 6 parts of HiSil 233, Y1587 produces a softer uncured reinforced gum with higher extractables and greater weight loss on bin aging than does SE4210. Elimination of the processing aid from SE54 with 32 parts of MS7 and 6 parts of HiSil 233 greatly increases the uncured hardness and significantly reduces the weight loss on bin aging and the extractables. Since there is no excess processing aid or the by-product ethanol to be eliminated by aging or extraction, the results on weight loss and extractables are to be expected.

For type 97 gums with 32 parts MS7 and 6 parts HiSil 233, the type of processing aid has no effect on the properties of uncured L1668.

For type 60 gums with 32 parts MS7 and 6 parts HiSil 233, Y1587 produces a softer uncured gum.

For modified L97KVB and L60VB with 25 parts of MS7 and no HiSil 233, Y1587 produces a softer uncured L1668.

The type of processing aid has no profound effect on the properties of uncured reinforced gum (L1668) and the effects it does have are probably of no consequence.

L3219's

Comparative data for extractables and the swell ratio which relates to the extent of crosslinking are shown in Table 45. Strength properties for which we have statistics are listed in Tables 46 and 47.

For SE54 gum Y1587 produces higher ultimate tensile strength and elongation than SE4210 or no processing aid. Y1587 also causes a slightly higher density and probably less hardness in the post-cured elastomer. Y1587 results in higher extractables, a higher V/V_0 which indicates a lesser degree of crosslinking, and a greater weight loss on post cure. In SE54 gum Y1587 produces a lower density and post-cured hardness, higher extractables and higher V/V_0 than no processing aid at all.

In all other gums the type of processing aid has no effect on ultimate tensile strength or post-cure hardness of both standard and modified formulations and has a variable, but probably not significant, effect on elongation. Y1587 produces a lower density in the standard formulation of all gums except SE54 and does not effect density in the modified formulations of L97KVB and L60. Y1587 causes higher extractables and higher weight loss on post cure for all gums and all formulations. Y1587 yields a higher V/V_0 for standard formulations of all gums, but does not effect the V/V_0 in L97KVB and L60 modified formulations.

Table 44. Comparisons of the hardness, Rex O, of uncured L1668 after one month bin aging.

Gum	Parts by weight		Rex O Y1587	Comparison	Rex O SE4210
	MS7	HiSil 233			
SE54 lot 161	38	6	50	<	60
SE54 lot 167	38	6	41	<	49
L97KVB	32	6	60	=	61
L97KVB	32	6	60	=	
L97KVB	25	0	50	>	42
MN97KVB	32	6	73	=	76
MN97KVB	32	6	70	=	
L60VB	32	6	52	<	65
L60VB	32	6	53	<	
L60VB	25	0	41	>	31
MN60VB lot DR114	32	6	49	<	59
			Y1587		None
SE54 lot 167	32	6	26	<	
SE54 lot 167	32	6	30	<	50

Table 45. Comparison of extractables and swell ratio of cured L3219 elastomers.

Gum	Parts by weight		CHCl ₃ Extractables, wt%			V/V ₀		
	MS7	HiSil 233	Y1587	Comparison	SE4210	Y1587	Comparison	SE4210
SE54 lot 161	38	6	3.7	≥	3.4	3.42	>	3.06
SE54 lot 161	38	6	2.2	=	2.2	2.85	>	2.66
SE54 lot 167	38	6	3.05	>	2.27	3.19	>	2.60
L97KVB	32	6	1.16	≥	1.04	2.26	>	1.94
L97KVB	32	6	1.48	>	1.04	2.34	>	2.21
L97KVB	25	0	1.07	>	0.91	2.51	=	2.47
MN97KVB	32	6	—	—	0.97	2.30	>	2.21
MN97KVB	32	6	1.09	>	0.97	2.29	>	2.21
L60VB	32	6	1.18	>	0.96	2.28	≥	2.25
L60VB	25	0	0.85	≥	0.78	2.50	≤	2.55
MN60VB lot DR114	32	6	1.10	>	0.90	2.31	>	2.19
			Y1587		None	Y1587		None
SE54 lot 167	32	6	2.8	>	1.97	3.23	>	2.86

Table 46. Comparisons of the ultimate tensile strength of cured L3219 elastomers, kPa.

Gum	Parts by weight		Y1587			Comparison ^a	SE4210		
	MS7	HiSil 233	n	Av	S		n	Av	S
SE54 lot 161	38	6	5	5888	586	>	5	4137	186
SE54 lot 161	38	6	5	5599	745	=	5	5123	358
SE54 lot 167	38	6	5	6346	402	>	5	5668	221
L97KVB	32	6	5	3179	207	=	5	3509	221
L97KVB	32	6	5	3516	359	=	5	3206	379
L97KVB	25	0	5	1717	193	=	5	1641	117
MN97KVB	32	6	5	3082	200	=	5	2834	317
MN97KVB	32	6	5	2903	186	=	5	1413	90
L60VB	32	6	5	3144	496	=	5	2110	255
L60VB	25	0	5	1434	110	=	5	5688	103
MN60VB	32	6	5	1987	97	=			
			Y1587			None			
SE54 lot 167	32	6	5	6736	228	>	3		

^a By Student's t test.

Table 47. Comparisons of the ultimate elongation of cured L3219 elastomers, %.

Gum	Parts by weight		Y1587			Comparison ^a	SE4210		
	MS7	HiSil 233	n	Av	S		n	Av	S
SE54 lot 161	38	6	5	376	31	>	5	278	13
SE54 lot 161	38	6	5	212	27	=	5	178	15
SE54 lot 167	38	6	5	360	16	>	5	226	9
L97KVB	32	6	5	90	10	>	5	60	7
L97KVB	32	6	5	74	2	>	5	70	7
L97KVB	25	0	5	70	10	=	5	66	5
MN97KVB	32	6	5	80	10	=	5	74	4
MN97KVB	32	6	5	76	5	=	5	68	4
L60VB	32	6	5	52	11	<	5	46	5
L60VB	25	0	5	58	5	<	5	213	6
MN60VB	32	6	5	62	4	>			
			Y1587			None			
SE54 lot 167	32	6	5	416	11	>	3		

^a By Student's t test.

Except for the effect on tensile strength and elongation of cured L3219's from SE54, the type of processing aid has no significant effect on the properties of cured L3219's from a variety of silicone gums.

L3223's

For cushion use, the most significant property of the material is its load-bearing capacity as indicated by its load-deflection curve. Retention of load with time is also of great importance but we cannot test for this conveniently. We do test for short-term, elevated-temperature compression set and use the percentage of set as a measure of the worth of cushion materials (i.e., lower set is better). We also measure density and thickness of cushions because load is a function of density and at lower densities it is also a function of thickness. Comparative data for density are shown in Table 48. Compression set data are listed in Table 49. Various data on load-deflection are shown in Tables 50, 51, 52, 53, and 54.

Y1587 may produce a lower density in type 97 and type 60 gums but the difference is small and not significant.

Type of processing aid has no effect on the compression set of L3223's from any silicone gum. Elimination of the processing aid reduces set in SE54 gum.

The major definition of L3223's is the load and unload-deflection properties. These values at 30% deflections are compared in Tables 50 and 51. For all gums, Y1587 produces a distinctly softer cushion. The effect is reduced for the modified formulations. Y1587 also produced a softer cushion than no processing aid at all.

Table 48. Comparisons of the density of the load-deflection disks of L3223 cellular silicone, Mg/m³.

Gum	Parts by weight		Y1587			Comparison ^a	SE4210		
	MS7	HiSil 233	n	Av	S		n	Av	S
SE54 lot 167	38	6	5	0.6541	0.0038	>	5	0.6477	0.0048
L97KVB	32	6	5	0.6559	0.0006	=	5	0.6573	0.0021
L97KVB	32	6	5	0.651	0.005	<	5	0.6449	0.0033
L97KVB	25	0	5	0.6424	0.0068	=	5	0.6721	0.0018
MN97KVB	32	6	5	0.6565	0.0016	<	5	0.6812	0.0038
L60VB	32	6	5	0.6466	0.0020	<	5	0.6334	0.0045
L60VB	25	0	5	0.6233	0.0030	<	4	0.6625	0.0037
Y1587									
SE54 lot 167	32	6	5	0.6687	0.0015	=	4	0.6625	0.0037
SE54 lot 167	32	6	4	0.660	0.011	=			

^a By Student's t test.

Another property of cushion that may be important is the resiliency of the material. We consider the ratio of the unload values to the load values at the same deflection and the hysteresis in the 3rd cycle unload and load curves as measures of resiliency and show the data for these in Tables 52 and 53; we assume that they both measure the same characteristic of the material. For SE54, Y1587 produces a more resilient cushion. For type 97 and type 60 gums, the type of processing aid has little or no effect. Removal of the processing aid increases resiliency. The correlation of the U/L ratio to the hysteresis loop is not at all clear.

The shift in starting thickness of L3223 from the first to third load cycle is being monitored to see if it is a useful indicator of cushion value. The data are shown in Table 54. The type or amount of processing aid has no apparent effect on Δt .

The major effect of the type of processing aid on L3223 cellular silicone is that Y1587 produces a significantly softer cushion than SE4210.

L3260's

Interest had shifted from L3223's to lower density cushions so we began to collect information on L3260's which is shown in Table 55. The data are complicated by a switch from the Banbury mixer to the Haake mixer. This variable is not considered significant except perhaps for its effect on density as shown in the table; the Haake mixer may produce a lower density than the Banbury. Again Y1587 produces a softer cushion than SE4210 but the difference is not so pronounced as it is with L3223. Y1587 does produce considerably less set than SE4210; this effect had not been observed with the higher density L3223.

Table 49. Comparisons of the confined-compression set of L3223 cellular silicones, %.

Gum	Parts by weight		Y1587			Comparison ^a	SE4210		
	MS7	HiSil 233	n	Av	S		n	Av	S
SE54 lot 167	38	6	4	10.85	1.54	=	4	10.55	0.49
L97KVB	32	6	3	5.66	0.38	=	3	6.46	0.22
L97KVB	32	6	3	5.53	0.44	=	3	5.29	0.59
L97KVB	25	0	3	5.85	0.83	=	3	5.61	0.64
MN97KVB	32	6	3	5.91	0.70	=	3	6.97	0.33
L60VB	32	6	3	3.84	0.29	<	3	4.99	0.65
L60VB	25	0	3	3.69	0.43	=	5	3.21	0.10
				Y1587				None	
SE54 lot 167	32	6	3	10.38	0.58	>	4		

^a By Student's t test.

Table 50. Comparisons of the 3rd cycle load at 30% deflection of L3223 cellular silicone, kPa.

Gum	Parts by weight		Y1587			Comparison ^a	SE4210		
	MS7	HiSil 233	n	Av	S		n	Av	S
SE54 lot 167	38	6	5	200.0	6.39	<	4	372.2	9.26
L97KVB	32	6	4	417.6	5.82	<	4	590.2	24.3
L97KVB	32	6	5	378.0	5.06	<	4	327.9	6.45
L97KVB	25	0	3	292.8	11.6	<	3	520.6	8.53
MN97KVB	32	6	5	344.6	1.84	<	3	485.6	35.3
L60VB	32	6	5	329.8	17.7	<	5	311.6	12.4
L60VB	25	0	4	282.2	6.83	<	3	239.1	2.23
				Y1587				None	
SE54 lot 167	32	6	5	198.4	2.98	<	3		
SE54 lot 167	32	6	3	180.1	1.29	<			

^a By Student's t test.

Table 51. Comparisons of the 3rd cycle unload at 30% deflection of L3223 cellular silicone, kPa.

Gum	Parts by weight		Y1587			Comparison ^a	SE4210		
	MS7	HiSil 233	n	Av	S		n	Av	S
SE54 lot 167	38	6	5	153.4	3.76	<	4	234.8	7.37
L97KVB	32	6	4	333.7	6.16	<	4	460.9	20.6
L97KVB	32	6	5	300.6	0.89	<	3	275.6	5.47
L97KVB	25	0	3	244.7	7.26	<	3	416.3	7.64
MN97KVB	32	6	5	274.8	2.14	<	3	219.1	2.03
L60VB	32	6	5	176.2	9.39	<	5	279.2	3.76
L60VB	25	0	4	251.6	5.33	<	3	178.7	1.63
				Y1587				None	
SE54 lot 167	32	6	5	141.8	1.86	<	3		
SE54 lot 167	32	6	3	127.2	5.82	<			

^a By Student's t test.

Table 52. Comparisons of the 3rd cycle unload-to-load ratio at 30% deflection of L3223 cellular silicones.

Gum	Parts by weight		Y1587			Comparison ^a	SE4210		
	MS7	HiSil 233	n	Av	S		n	Av	S
SE54 lot 167	38	6	5	0.767	0.0087	>	4	0.718	0.0026
L97KVB	32	6	4	0.799	0.0035	>	4	0.781	0.0032
L97KVB	32	6	5	0.795	0.0040	>	4	0.841	0.0010
L97KVB	25	0	3	0.835	0.0084	=	3	0.800	0.0028
MN97KVB	32	6	5	0.797	0.0044	=	3	0.832	0.0041
L60VB	32	6	5	0.835	0.0026	=	5	0.896	0.0039
L60VB	25	0	4	0.891	0.0031	=	None		
Y1587									
SE54 lot 167	32	6	5	0.715	0.0025	<	3	0.748	0.0021
SE54 lot 167	32	6	3	0.707	0.0196	<			

^a By Student's t test.

Table 53. Comparisons of the 3rd cycle hysteresis loop of L3223 cellular silicones.

Gum	Parts by weight		Y1587			Comparison ^a	SE4210		
	MS7	HiSil 233	n	Av	S		n	Av	S
SE54 lot 167	38	6	5	170.5	46.1	<	4	367.0	12.7
L97KVB	32	6	4	314.1	43.3	<	4	424.7	16.3
L97KVB	32	6	5	285.2	15.6	<	4	201.1	9.82
L97KVB	25	0	3	200.0	31.0	=	3	346.4	5.80
MN97KVB	32	6	5	273.4	18.6	<	3	243.1	24.2
L60VB	32	6	5	198.1	13.8	<	5	119.2	12.9
L60VB	25	0	4	112.0	9.43	=	None		
Y1587									
SE54 lot 167	32	6	5	221.0	10.7	=	3	232.9	12.8
SE54 lot 167	32	6	3	201.5	26.2	=			

^a By Student's t test.

Table 54. Comparisons of the difference between 1st cycle and 3rd cycle starting thicknesses of L3223 cellular silicones, mm.

Gum	Parts by weight		Y1587			Comparison ^a	SE4210		
	MS7	HiSil 233	n	Av	S		n	Av	S
SE54 lot 167	38	6	5	0.047	0.0040	<	4	0.072	0.0096
L97KVB	32	6	4	0.047	0.0086	<	4	0.069	0.0066
L97KVB	32	6	5	0.058	0.0168	=	4	0.028	0.0021
L97KVB	25	0	3	0.028	0.026	=	3	0.053	0.0022
MN97KVB	32	6	5	0.031	0.015	=	3	0.044	0.0109
L60VB	32	6	5	0.041	0.0061	=	5	0.030	0.0056
L60VB	25	0	4	0.049	0.0183	=	None		
Y1587									
SE54 lot 167	32	6	5	0.067	0.0034	<	3	0.088	0.0032
SE54 lot 167	32	6	3	0.101	0.0104	=			

^a By Student's t test.

Table 55. Comparisons of the properties of L3260 cellular silicones from MN97KVB and L60VB silicone gums.

	MN97KVB									L60VB								
	Y1587 LLNL Urea Banbury Mixer			SE4210 BKC Urea Haake Mixer			Y1587 BKC Urea Banbury Mixer			SE4210 BKC Urea Haake Mixer								
	n	Av	S	n	Av	S	n	Av	S	n	Av	S	n	Av	S	n	Av	S
Density of L/D disks, Mg/m ³	5	0.5226	0.0052	≤	5	0.5507	0.0048	5	0.5027	0.0026	≤	5	0.5391	0.0059				
3rd cycle																		
Load at 30% deflection, kPa	5	161.1	4.88	≤	5	202.4	11.7	5	145.5	4.77	≤	3	171.1	6.81				
Unload at 30% deflection, kPa	4	134.3	4.85	≤	4	159.2	1.59	5	123.2	3.22	≤	3	145.4	6.82				
Unload/load ratio at 30% deflection	4	0.832	0.0022	≥	4	0.787	0.0026	5	0.846	0.0083	≥	3	0.849	0.0129				
Hysteresis loop (Δ area)	4	127.6	9.52	≤	4	220.0	11.0	5	112.8	12.8	≥	2	127.9	13.3				
Δt, mm	4	0.033	0.0036	=	4	0.055	0.017	5	0.031	0.0094	=	3	0.047	0.011				
Set, %	3	4.59	0.68	≤	3	7.29	0.65	3	3.30	0.63	≤	3	7.02	0.65				

^a By Student's t test.

The major effect of the type of processing aid on L3260 cellular silicone is that Y1587 produces a significantly lower compression set than SE4210.

Also Y1587 produces a slightly softer cushion than SE4210.

Summary

We have evaluated two processing aids; UCC's Y1587 and GE's SE4210 in different silicone gums: GE's SE54, type 97 gums (L97KVB and MN97KVB), and type 60 gums (L60VB and MN60VB). We tested uncured L1668 reinforced gums, cured L3219 elastomers, and L3223 and L3260 cellular silicones.

The type of reinforcing aid has no significant effect on uncured L1668. In L3219 from SE54, Y1587 produces higher ultimate tensile strength and elongation than does SE4210. For all the other silicone gums, the type of processing aid has no effect on the properties of cured L3219 elastomer.

For L3223 cellular silicone, Y1587 produces a significantly softer cushion for all gums. Type of

processing aid has no effect on the compression set of L3223. For L3260 cellular silicone, Y1587 produced a slightly softer cushion for all gums and it results in significantly lower compression set in type 97 and type 60 gums.

If compression set is not a primary concern or if cushion density is high, the choice of a processing aid can be used to control cushion stiffness along with vinyl content of the gum, amount of reinforcing SiO_2 in the L1668 gum, and the porosity of the cellular silicone.

Y1587 is the preferred processing aid for all silicone gums, and it is important that it be used in the new type 97KVB and 60VB gums.

Recommendations

In the new silicone gums of types 97KVB and 60VB, UCC's Y1587 processing aid produces a softer cushion than does GE's SE4210. This softening effect decreases as cushion density decreases (porosity increases). The type of processing aid has no effect on the compression set of the higher density L3223. For lower density L3260 cushions, Y1587 produces a significantly lower compression set than does SE4210. Our experience with difficult-to-process type 60 gums indicates that Y1587 is more versatile in accommodat-

ing to unknown gum variables than SE4210. For these new gums, the Y1587 processing aid should be used.

Although data for GE's SE54 silicone gum is less complete and less conclusive, we recommend that Y1587 be used with SE54 also.

If compression set is not a big consideration, or if cushion density is high, type of processing aid can be used as a tool to manipulate load properties of a cushion along with the amount of reinforcing SiO_2 , vinyl content and porosity.

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