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APPENDICES

Appendix A

The Overall Compositions of Rubber

Table A-1 Natural rubber

Characteristic	Tolerance
Dirt content (max, %wt)	0.04
Ash content (max, %wt)	0.40
Volatile matter (max, %wt)	0.80
Nitrogen (max, %wt)	0.60
Plasticity retention index (min)	60
Mooney viscosity ML (1+ 4) @100 °C	59.2

Table A-2 Properties of ethylene-propylene-diene copolymer (NORDEL™ IP 5565)

Typical polymer	Value
Mooney viscosity ML (1+ 4) @125 °C	65
Polymer composition (%wt)	
Ethylene	50
Propylene	42.5
Ethylidenenobornene (ENB)	7.5
Molecular weight distribution	Medium
Product density (g/cc)	0.86
Ash content (max, %wt)	0.1
Total volatiles (max, %wt)	0.4

A=Peak area except at 5.2 ppm

B=Peak area at 5.2 ppm

C=Peak area of saturated $-\text{CH}_2-$ and $-\text{CH}_3$

$$A = 10C + 7B$$

$$C = \frac{A-7B}{10}$$

Total peak area = Peak area of saturated $-\text{CH}_2-$ and $-\text{CH}_3$ + Peak area at 5.2 ppm

$$C = \frac{A-7B}{10} + B$$

$$C = \frac{A-3B}{10} + B$$

$$\begin{aligned} \% \text{Hydrogenation} &= \left[\frac{(\text{Peak area of sat. } -\text{CH}_2- \text{ and } -\text{CH}_3)}{(\text{Total peak area})} \right] \times 100 \\ &= \frac{\left(\frac{A-7B}{10} \right)}{\left(\frac{A+7B}{10} \right)} \times 100 \\ &= \frac{A-7B}{A+3B} \times 100 \end{aligned}$$

For example: A = 89.11 and B = 1.00

$$\begin{aligned} \% \text{Hydrogenation} &= \frac{89.11-7(1.00)}{89.11+3(1.00)} \times 100 \\ &= 89.14 \% \end{aligned}$$

B.2. Calculation of Thioacetate Content

Composition of HNRTA was determined from ^1H NMR spectra using the integrated peak ratio of proton of a double bond (a, 5.1 ppm) to $-\text{CH-S}-$ (b, 3.6 ppm). The percentage of thioacetate in HNR was calculated using the formula:

$$\% \text{Thioacetate in HNR} = \left(\frac{b}{a + b} \right) \times 100$$

a = peak area at 5.1 ppm.

b = peak area at 3.6 ppm.

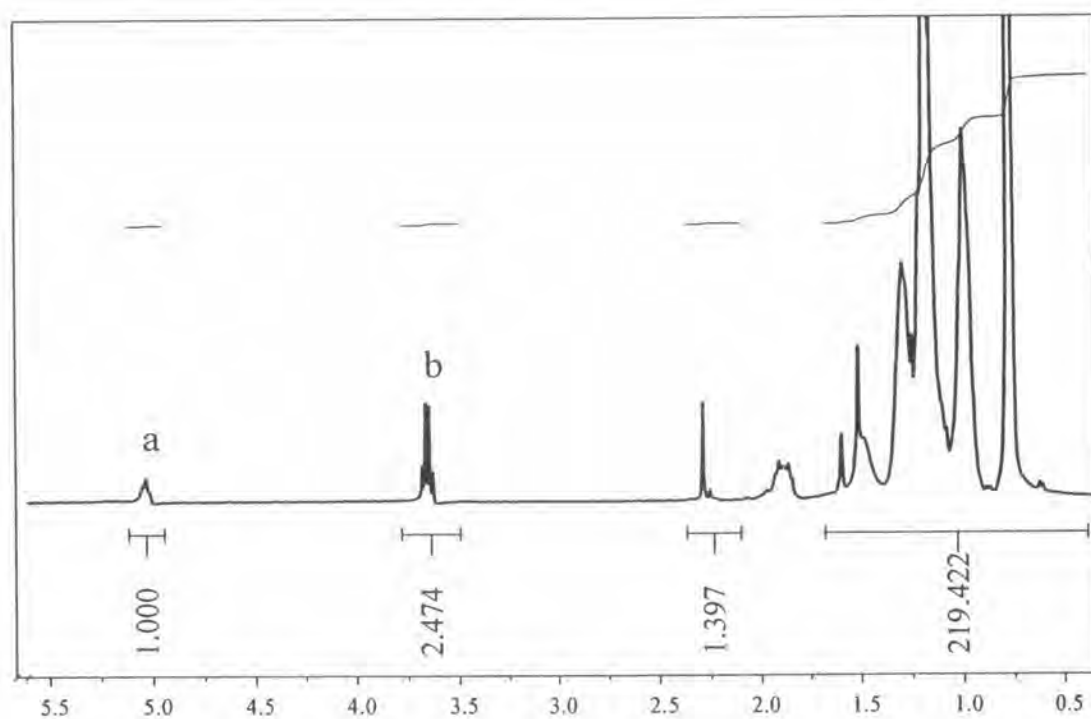


Figure B-2 ^1H -NMR spectra of HNRTA

For example: a = 1.00 and b = 2.47

$$\begin{aligned} \% \text{Thioacetate in HNR} &= \left(\frac{2.47}{1.00 + 2.47} \right) \times 100 \\ &= 71.18 \% \end{aligned}$$

1 g of NR contained $1/68 = 14.7$ mmol of double bond in structure.

1 g of HNR with 90 %hydrogenation contained 1.47 mmol of double bond in structure.

$$\text{Thioacetate content in HNR} = \frac{\% \text{Thioacetate in HNR}}{100} \times \text{double bond}$$

$$= \frac{71.18}{100} \times 1.47$$

$$= 1.04 \text{ mmol/g}$$

B.3. Calculation of %Methanolysis

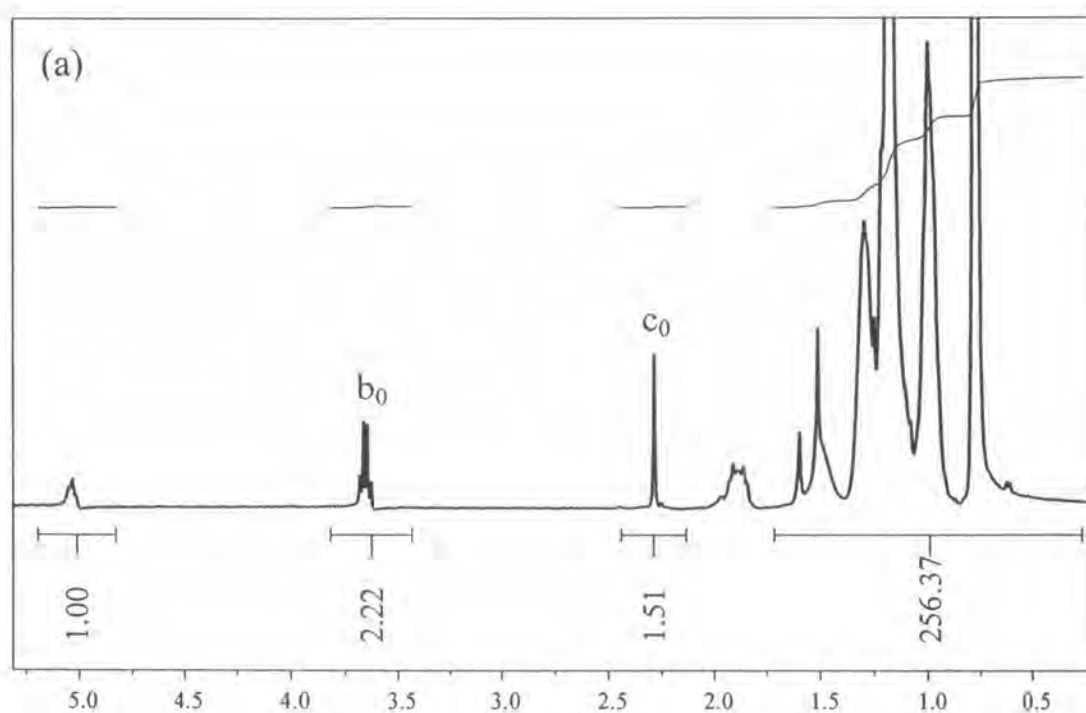
The degree of methanolysis was calculated from the ratio of $-\text{CH-S-}$ (b, 3.6 ppm) to methyl group of thioacetate (c, 2.3 ppm) before (0, HNRTA) and after (t, HNRSH) reaction by using the following equations:

Before methanolysis, the ratio of peak area of CH_3 -thioacetate/ $-\text{CH-S-}$ = $\frac{c_0}{b_0}$

After methanolysis, the ratio of peak area of CH_3 -thioacetate/ $-\text{CH-S-}$ = $\frac{c_t}{b_t}$

Reacted CH_3 -thioacetate in methanolysis = $\frac{c_0}{b_0} - \frac{c_t}{b_t}$

$$\begin{aligned} \therefore \% \text{Methanolysis} &= \frac{\frac{c_0}{b_0} - \frac{c_t}{b_t}}{\frac{c_0}{b_0}} \times 100 \\ &= \left\{ 1 - \left[\frac{c_t}{b_t} \times \frac{b_0}{c_0} \right] \right\} \times 100 \end{aligned}$$



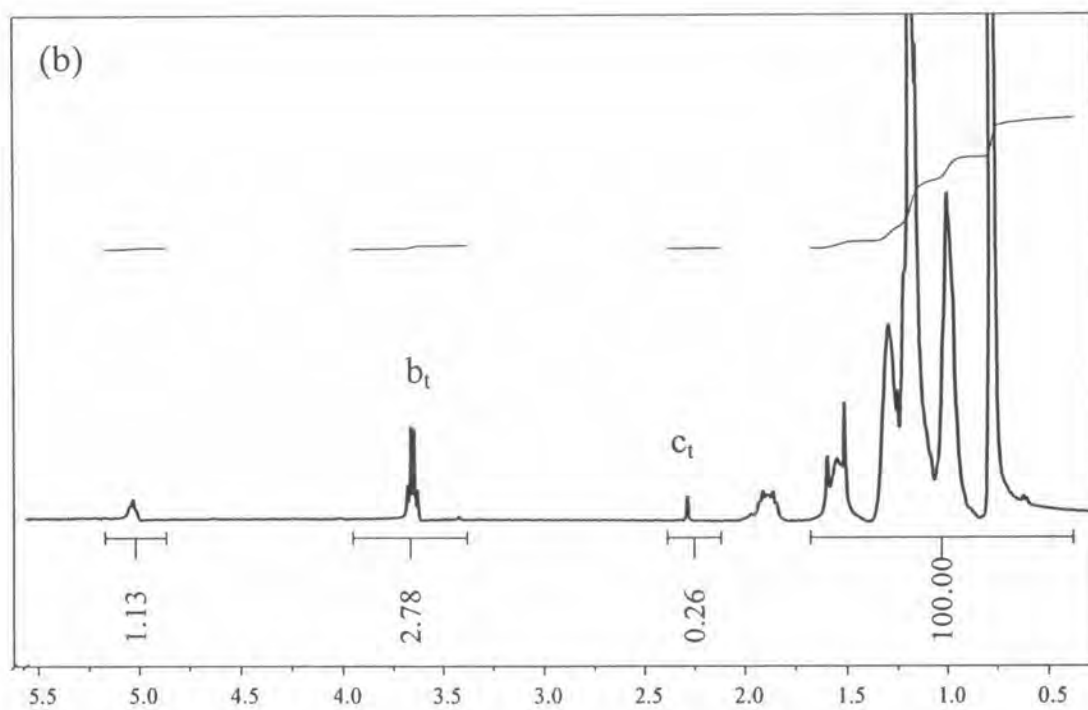


Figure B-3 ^1H -NMR spectra of (a) HNRTA and (b) HNRSH.

For example: $b_0 = 2.22$, $c_0 = 1.51$, $b_t = 2.78$ and $c_t = 0.26$

$$\% \text{Methanolysis} = \left(1 - \frac{2.22 \times 0.26}{1.51 \times 2.78} \right) \times 100$$

$$= 86.2\%$$

Appendix C

HNRTA Content

Table C-1 Effect of reaction time on HNR at 80% and 90% hydrogenation: the mole ratio of [C=C]/[TAA]/[AIBN] = 1/1/0.1 at 80°C under N₂ atmosphere.

Time (h)	90% hydrogenation of HNR		80% hydrogenation of HNR	
	Thioacetate content (mmol/g)	Conversion (%)	Thioacetate content (mmol/g)	Conversion (%)
8	0.175	11.93	0.744	25.32
16	0.432	29.41	1.016	34.55
24	0.584	39.72	1.172	39.86
32	0.749	50.97	1.342	45.63
40	0.818	55.68	1.499	50.97
48	0.966	65.70	1.653	56.21
56	0.988	67.21	1.723	58.60
64	0.993	67.54	1.875	63.77
72	1.040	70.72	1.895	64.45

Table C-2 Effect of thioacetic acid concentration on HNR at 80% and 90% hydrogenation: the mole ratio of $[C=C]/[AIBN] = 1/0.1$ at $80^{\circ}C$ for 48 h under N_2 atmosphere.

[TAA]/[C=C]	90% hydrogenation of HNR		80% hydrogenation of HNR	
	Thioacetate content (mmol/g)	Conversion (%)	Thioacetate content (mmol/g)	Conversion (%)
0.50	0.658	44.79	1.260	42.86
0.75	0.818	55.68	1.440	48.98
1.00	0.966	65.70	1.653	56.21
2.00	1.029	69.97	1.782	60.60
3.00	1.076	73.20	1.932	65.70

Table C-3 Effect of initiator concentration on HNR at 80% and 90% hydrogenation: the mole ratio of $[TAA]/[C=C] = 1/0.1$ at $80^{\circ}C$ for 48 h under N_2 atmosphere.

[Initiator]/[C=C]	90% hydrogenation of HNR		80% hydrogenation of HNR	
	Thioacetate content (mmol/g)	Conversion (%)	Thioacetate content (mmol/g)	Conversion (%)
0.025	0.688	46.79	1.326	45.11
0.050	0.847	57.66	1.451	49.36
0.100	0.966	65.70	1.653	56.21
0.300	1.000	68.01	1.653	56.22
0.500	1.058	71.96	1.670	56.78

Appendix D

Properties of Rubber Blends after Vulcanization

Table D-1 Tensile strength and elongation at break of NR vulcanizates obtained from EPV system.

Properties	Tensile Strength (MPa)					Elongation at break (%)				
	1	2	3	Ave.	S.D.	1	2	3	Ave.	S.D.
Before ageing	14.8	15.1	15.8	15.2	0.53	692	761	697	712	39
After ageing	9.64	9.57	9.61	9.67	0.05	645	735	687	690	45

Table D-2 Tensile strength and elongation at break of HNR/NR vulcanizates obtained from CV system with 5 phr of HNRTA and HNRSH.

Compatibilizer	Tensile Strength (MPa)					Elongation at break (%)				
	1	2	3	Ave.	S.D.	1	2	3	Ave.	S.D.
Before ageing										
None	4.22	3.00	4.36	3.86	0.75	612	573	594	593	20
HNRTA	4.35	3.72	3.71	3.93	0.37	492	409	453	451	42
HNRSH	5.31	3.37	4.78	4.49	1.00	395	362	405	387	23
After ageing										
None	4.22	3.00	4.36	3.86	0.75	177	138	304	206	86
HNRTA	3.45	3.72	5.61	4.26	1.17	357	304	386	348	42
HNRSH	5.31	3.37	4.78	4.49	1.00	203	292	251	248	44

Table D-3 Tensile strength and elongation at break of HNR/NR vulcanizates obtained from EPV system as functions of HNR/TA and HNR/SH content.

Compatibilizer (phr)	Tensile strength (MPa)						Elongation at break (%)					
	0	2	3	5	10	15	0	2	3	5	10	15
Before ageing												
HNR/TA												
1	6.93	8.69	8.07	9.30	8.54	9.51	863	795	727	746	745	727
2	6.70	8.26	8.82	9.16	9.94	9.40	817	820	777	680	725	696
3	7.31	8.44	9.81	9.36	10.2	9.14	806	778	751	731	638	678
Ave.	6.98	8.46	8.90	9.27	9.57	9.35	829	798	752	719	703	700
S.D.	0.31	0.21	0.87	0.10	0.90	0.19	31	21	25	34	57	24
HNR/SH												
1	6.93	9.33	9.05	9.21	9.53	8.63	863	779	725	637	651	636
2	6.70	7.03	9.26	9.36	10.7	10.5	817	763	685	668	622	660
3	7.31	9.12	9.01	9.68	9.53	10.3	806	748	722	688	659	621
Ave.	6.98	8.49	9.11	9.42	9.95	9.83	829	763	711	664	644	639
S.D.	0.31	1.27	0.13	0.24	0.71	1.04	31	15	23	26	19	20
After ageing												
HNR/TA												
1	6.43	7.65	7.98	9.69	9.53	10.3	764	723	695	510	502	496
2	6.32	8.16	8.52	8.96	9.37	9.41	770	677	696	594	491	381
3	5.94	7.99	8.70	7.95	9.89	9.24	683	750	571	650	583	384
Ave.	6.23	7.94	8.40	8.86	9.60	9.65	739	716	654	585	526	420
S.D.	0.25	0.26	0.38	0.87	0.27	0.57	49	37	71	70	50	66
HNR/SH												
1	6.43	5.91	8.45	9.62	10.5	10.4	764	701	584	661	488	451
2	6.32	7.79	8.92	8.20	9.06	9.83	770	727	678	577	608	589
3	5.94	9.23	7.61	7.72	8.99	8.39	683	606	602	503	584	502
Ave.	6.23	7.65	8.33	8.51	9.55	9.57	739	678	621	580	560	514
S.D.	0.25	1.66	0.67	0.99	0.90	1.08	49	64	50	79	63	70

Table D-4 Tensile strength and elongation at break of EPDM/NR vulcanizates obtained from EPV system as functions of HNRTA and HNRSH content.

Compaibilizer (phr)	Tensile strength (MPa)				Elongation at break (%)			
	0	3	5	10	0	3	5	10
Before ageing								
HNRTA								
1	5.75	5.78	5.86	5.23	832	764	709	713
2	5.16	5.31	5.24	5.30	795	746	758	629
3	5.36	5.20	5.80	7.77	773	792	798	752
Ave.	5.42	5.43	5.63	6.10	800	767	755	698
S.D.	0.30	0.31	0.34	1.45	30	23	45	63
HNRSH								
1	5.75	4.86	6.12	6.85	832	757	741	686
2	5.16	5.73	5.86	6.80	795	787	727	741
3	5.36	6.03	5.72	6.85	773	752	785	632
Ave.	5.42	5.54	5.90	6.83	800	766	751	686
S.D.	0.30	0.60	0.20	0.03	30	19	30	54
After ageing								
HNRTA								
1	4.01	5.48	6.45	7.87	763	703	716	646
2	5.13	4.22	5.89	6.57	781	748	686	631
3	3.90	3.97	4.19	6.66	766	709	658	625
Ave.	4.35	4.56	5.51	7.03	770	720	687	634
S.D.	0.68	0.81	1.18	0.73	10	25	29	11
HNRSH								
1	4.01	5.75	6.18	7.74	763	729	701	683
2	5.13	4.04	6.87	6.03	781	767	790	713
3	3.90	3.80	3.43	7.11	766	690	733	652
Ave.	4.35	4.53	5.49	6.96	770	729	741	683
S.D.	0.68	1.06	1.82	0.86	10	39	45	31

Table D-5 Crosslink density of HNR/NR vulcanizates obtained from EPV system as functions of HNRTA and HNRSH content.

Compatibilizer Content (phr)	No.	Swollen weight (g)	Deswollen weight (g)	Density (g/cm ³)	Volume fraction	Crosslink density×10 ⁴ (mol/cm ³)	Ave.	S.D.
None	1	5.321	0.691	0.90	0.126	0.52		
	2	5.234	0.683	0.90	0.126	0.52	0.52	0.00
	3	5.534	0.721	0.90	0.126	0.52		
HNRTA								
2	1	5.084	0.714	0.91	0.133	0.58		
	2	5.187	0.753	0.91	0.136	0.61	0.60	0.01
	3	5.177	0.710	0.91	0.134	0.59		
3	1	4.984	0.702	0.91	0.135	0.61		
	2	5.177	0.732	0.91	0.136	0.61	0.61	0.01
	3	5.077	0.720	0.91	0.137	0.62		
5	1	4.512	0.660	0.91	0.140	0.66		
	2	4.391	0.632	0.91	0.138	0.63	0.64	0.03
	3	4.270	0.610	0.91	0.137	0.62		
10	1	4.986	0.722	0.90	0.140	0.66		
	2	5.074	0.756	0.90	0.144	0.70	0.67	0.02
	3	4.922	0.712	0.90	0.140	0.66		
15	1	4.974	0.775	0.87	0.155	0.82		
	2	4.967	0.753	0.87	0.151	0.77	0.81	0.02
	3	4.891	0.760	0.87	0.155	0.81		
HNRSH								
2	1	4.645	0.707	0.91	0.146	0.72		
	2	4.598	0.666	0.91	0.139	0.64	0.65	0.06
	3	4.783	0.662	0.91	0.133	0.58		

Table D-5 (Cont.)

Compatibilizer Content (phr)	No.	Swollen weight (g)	Deswollen weight (g)	Density (g/cm ³)	Volume fraction	Crosslink density×10 ⁴ (mol/cm ³)	Ave.	S.D.
3	1	4.988	0.785	0.90	0.153	0.79	0.77	0.02
	2	4.735	0.726	0.90	0.149	0.74		
	3	4.578	0.722	0.90	0.153	0.79		
5	1	4.387	0.750	0.90	0.166	0.95	0.97	0.04
	2	4.142	0.734	0.90	0.172	1.03		
	3	4.039	0.693	0.90	0.166	0.95		
10	1	3.548	0.601	0.90	0.164	0.93	1.03	0.13
	2	3.551	0.615	0.90	0.168	0.97		
	3	3.589	0.675	0.90	0.183	1.18		
15	1	3.870	0.723	0.90	0.181	1.16	1.18	0.04
	2	3.792	0.708	0.90	0.181	1.16		
	3	3.587	0.689	0.90	0.186	1.23		

Table D-6 Crosslink density of HNR/NR vulcanizates obtained from CV system with 5 phr of HNRTA and HNRSH.

Compatibilizer	No.	Swollen weight (g)	Deswollen weight (g)	Density (g/cm ³)	Volume fraction	Crosslink density×10 ⁴ (mol/cm ³)	Ave.	S.D.
None	1	4.025	0.823	0.87	0.205	1.52	1.98	0.50
	2	3.338	0.754	0.87	0.226	1.92		
	3	3.607	0.911	0.87	0.253	2.51		
HNRTA	1	3.040	0.712	0.87	0.234	2.09	1.98	0.35
	2	2.976	0.720	0.87	0.242	2.26		
	3	3.372	0.702	0.87	0.208	1.58		
HNRSH	1	2.712	0.690	0.911	0.246	2.35	2.17	0.30
	2	2.491	0.632	0.911	0.245	2.33		
	3	2.670	0.610	0.911	0.221	1.81		

Table D-7 Crosslink density of EPDM/NR vulcanizates obtained from EPV system as functions of HNRTA and HNRSH content.

Compatibilizer Content (phr)	No.	Swollen weight (g)	Deswollen weight (g)	Density (g/cm ³)	Volume fraction	Crosslink density × 10 ⁴ (mol/cm ³)	Ave.	S.D.
Non	1	3.714	0.362	0.90	0.090	0.26		
	2	3.893	0.404	0.90	0.098	0.31	0.32	0.06
	3	3.998	0.414	0.90	0.108	0.38		
HNRTA								
3	1	3.719	0.362	0.90	0.094	0.28		
	2	3.736	0.404	0.90	0.102	0.34	0.34	0.05
	3	3.829	0.414	0.90	0.108	0.38		
5	1	4.428	0.530	0.90	0.117	0.45		
	2	4.387	0.525	0.90	0.118	0.45	0.44	0.02
	3	4.316	0.519	0.90	0.113	0.42		
10	1	4.197	0.753	0.90	0.172	1.03		
	2	4.248	0.748	0.90	0.168	9.78	1.07	0.12
	3	4.318	0.798	0.90	0.184	1.21		
HNRSH								
3	1	4.324	0.515	0.90	0.115	0.43		
	2	4.400	0.526	0.90	0.116	0.44	0.44	0.03
	3	4.593	0.546	0.90	0.115	0.43		
5	1	5.481	0.678	0.90	0.120	0.47		
	2	5.476	0.675	0.90	0.119	0.47	0.47	0.02
	3	5.478	0.676	0.90	0.119	0.47		
10	1	3.855	0.784	0.90	0.196	1.39		
	2	3.882	0.783	0.90	0.196	1.38	1.36	0.00
	3	3.879	0.761	0.90	0.192	1.31		

Table D-8 Hardness of HNR/NR and EPDM/NR vulcanizates as functions of HNRTA and HNRSH content.

Rubber blends	Compatibilizer Content (phr)		Hardness			Ave.	S.D.			
			1	2	3					
Conventional vulcanization										
HNR/NR	None	0	49.0	50.0	49.5	49.5	0.50			
	HNRTA	5	50.0	50.0	50.5	50.2	0.29			
	HNRSH	5	50.0	51.0	50.5	50.5	0.50			
Efficient-peroxide vulcanization										
HNR/NR	None	0	40.0	41.0	40.0	40.3	0.58			
		HNRTA	2	41.5	41.0	42.0	41.5	0.50		
			3	42.0	41.5	41.0	41.5	0.50		
			5	41.5	41.5	42.0	41.7	0.29		
			10	43.0	42.0	42.0	42.3	0.58		
			15	42.5	42.8	42.5	42.6	0.17		
	HNRSH	2	42.0	41.5	41.0	41.5	0.50			
		3	41.5	42.0	41.8	41.8	0.25			
		5	42.0	42.0	42.5	42.2	0.29			
		10	43.0	42.0	42.5	42.5	0.50			
		15	43.5	43.0	43.0	43.2	0.29			
		EPDM/NR	None	0	34.5	34.0	33.0	33.8	0.76	
				HNRTA	3	34.0	34.0	34.5	34.2	0.29
					5	35.0	34.5	34.5	34.7	0.29
			10		36.0	34.0	35.5	35.2	1.04	
HNRSH	3		34.0	34.0	34.5	34.2	0.29			
	5		35.0	35.0	35.5	35.2	0.29			
	10		35.0	36.0	36.5	35.8	0.76			

VITA

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