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Safe Processing Curing Systems for Viton Fluoroelastomers

Further details on specific curing agents will be found in the article of the same title on page 831, July 1959 I/EC.

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The combination of vinylidene fluoride and hexafluoropropylene represented in Viton has been carefully chosen to give good processing compounds and high quality vulcanizates. However, the proper utilization of any elastomer is dependent, to a large extent, on the development of safe processing curing agents. The following tables contain data obtained with a number of materials as curing agents for Viton A and Viton A-HV. The influence of these curing agents on processing safety as well as original and aged vulcanizate properties is shown.

Unless otherwise specified in the tables, the press cure is 30 minutes at 300° F., and the oven cure is step cure to 400° F. and 24 hours at 400° F.

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Table I. Triethylenetetramine and Benzoyl Peroxide as Curing Agents for Viton A

Compound	A-1	B-1
Viton A	100	100
Zinc oxide	10	10
Dibasic lead phosphite [a]	10	10
MT carbon black	20	...
Fine silica-silicone oil (100/20) [b]	...	20
Triethylenetetramine	1	...
Benzoyl peroxide	...	2
Mooney scorch at 250° F. (MS)		
Minutes to a 10-point rise	2	3
Stress-strain, original		
Modulus at 100%, p.s.i.	1180	360
Modulus at 200%, p.s.i.	...	580
Tensile strength, p.s.i.	2650	2250
Elongation at break, %	180	720
Hardness, shore A	75	75

[a] Dyphos, National Lead Co., New York 6, N. Y.
[b] HiSil 233, Columbia Southern Chemical Corp., Pittsburgh, Pa. LM-3 oil, Silicone Products Division, Union Carbide Corp.

Table II. Hexamethylenediamine Carbamate as a Curing Agent for Viton An and Viton A-HV

Compound	A-2	B-2	C-2	D-2	E-2
Viton A	100	100	100	...	...
Viton A-HV	...	...	...	100	100
Zinc oxide	10	...	...	...	...
Dibasic lead phosphite	10	...	...	...	...
Magnesium oxide [a]	...	15	15	15	15
MT carbon black	20	20			
MT carbon black	20	20	60	20	60
Hexamethylenediamine carbamate [b]	1	1	1	1	1
Mooney scorch at 250° F. (MS)					
Minutes to a 10-point rise	36	12	9	7	6
Stress-strain, original					
Modulus at 100%, p.s.i.	370	310	950	390	1200
Modulus at 200%, p.s.i.	1040	860	1880	1130	...
Tensile strength, p.s.i.	2450	2325	2200	2500	2400
Elongation at break, %	340	340	200	320	170
Hardness, shore A	67	68	87	67	85

[a] Darlington 601, Darlington Chemical Co., Philadelphia, Pa.
[b] Diak No. 1, E. I. du Pont de Nemours & Co., Inc.

Table III. Ethylenediamine Carbamate as a Curing Agent for Viton A-HV

Compound	A-3	B-3	C-3	D-3
Viton A-HV	100	100	100	100
Magnesium oxide [a]	15	15	15	15
MT carbon black	20	20	20	60
Hexamethylenediamine carbamate	1	...	...	...
Ethylenediamine carbamate [b]	...	0.85	1.0	0.85
Mooney scorch at 250° F. (MS)				
Minutes to a 10-point rise	7	36	25	32
Stress-strain, original				
Modulus at 100%, p.s.i.	390	350	380	1050
Modulus at 200%, p.s.i.	1130	1190	1330	...
Tensile strength, p.s.i.	2500	2875	2875	2600
Elongation at break, %	320	350	320	190
Hardness, shore A	67	69	68	84
Compression set, Method B				
70 hours at 250° F.	35	34	23	32
Stress-strain, after 5 days at 500° F. (oven)				
Tensile strength, % retained	57	59	68	62
Elongation, % retained	97	92	81	105
Hardness, change	+6	+7	+8	+5

[a] Darlington 601.
[b] Diak No. 2, E. I. du Pont de Nemours & Co., Inc.

Table IV. Effect of Remilling on Properties of Stocks

Containing Ethylenediamine Carbamate and Hexamethylenediamine Carbamate

Compound	A-4	B-4
Viton A-HV	100	100
Magnesium oxide [a]	15	15
MT carbon black	20	20
Hexamethylenediamine carbamate	1	...
Ethylenediamine carbamate	...	0.85
Stress-strain, original (no remill)		
Modulus at 100%, p.s.i.	400	410
Modulus at 200%, p.s.i.	1100	1200
Tensile strength, p.s.i.	1675	2400
Elongation at break, %	260	340
Hardness, shore A	69	69
Stress-strain, original (remilled)		
Modulus at 100%	390	350
Modulus at 100%, p.s.i.	390	350
Modulus at 200%, p.s.i.	1130	1190
Tensile strength, p.s.i.	2500	2875
Elongation at break, %	320	350
Hardness, shore A	67	69

[a]Darlington 601.

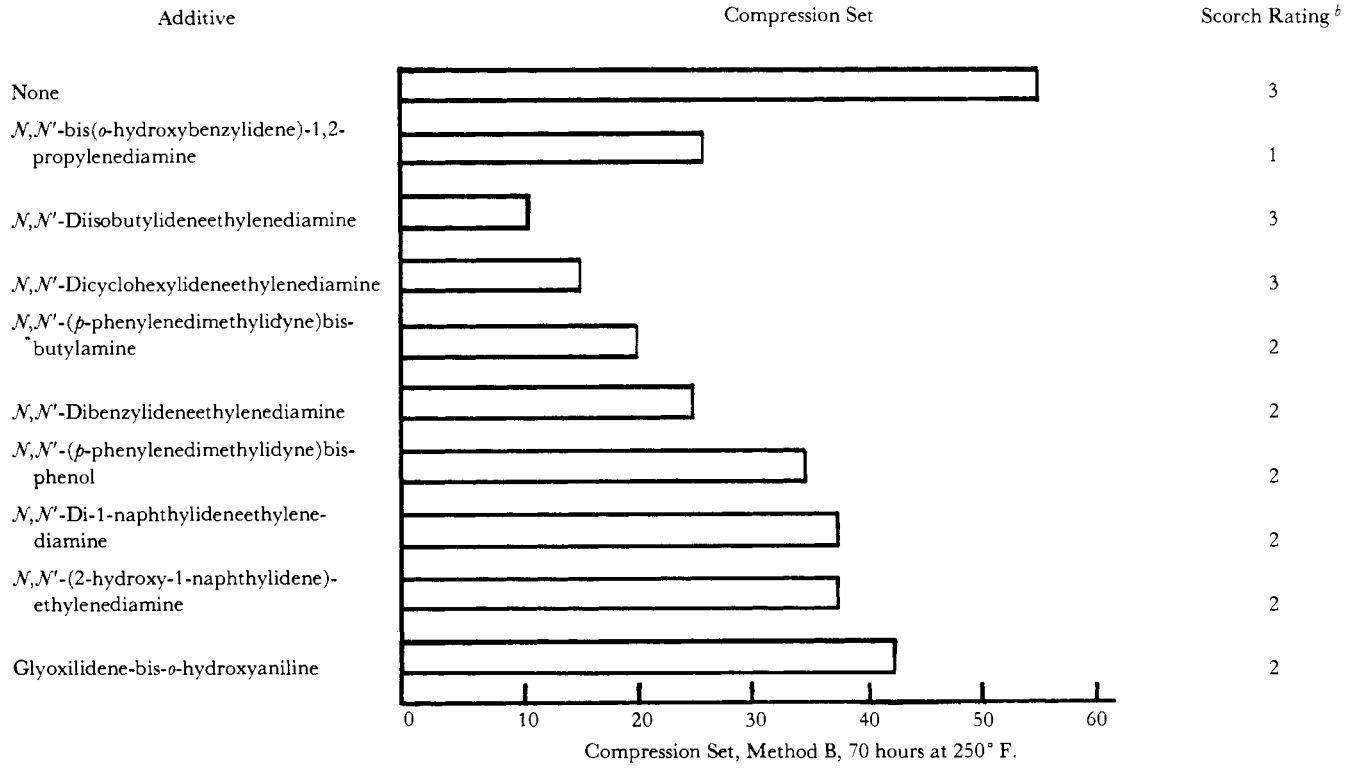
Table V. Curing Viton A-HV with a Combination of *N,N'*-bis(o-hydroxybenzylidene)-1,2-propylenediamine and Hexamethylenediamine Carbamate

Compound	A-5	B-5	C-5	D-5
Viton A-HV	100	100	100	100
Magnesium oxide [a]	15	15	15	15
MT carbon black	20	20	20	20
Hexamethylenediamine carbamate	1	1	0.7	0.7
N,N'-bis(*o*-hydroxybenzylidene)-1,2-propylenediamine	...	1.3	1.0	0.7
Mooney scorch at 250° F. (MS)				
Minutes to a 10-point rise	7	30	> 45	30
Curing conditions				
Press cure, 30 minutes at 300° F.				
Oven cure, step cure to 400° F. and 24 hours at 400° F.				
Stress-strain, original				
Modulus at 100%, p.s.i.	390	580	400	380
Modulus at 200%, p.s.i.	1130	2075	1200	1110
Tensile strength, p.s.i.	2500	2525	2250	2475
Elongation at break, %	320	240	290	300
Hardness, shore A	67	69	65	66
Compression set, Method B				
70 hours at 250° F.	35	16	29	34
Stress-strain, after 5 days at 500° F. (oven)				
Tensile strength, % retained	57	87	76	61
Elongation, % retained	97	46	69	90
Hardness, change	+6	+18	+8	+6

[a]Darlington 601.

Table VI. Effect of Various Diimine Additives on Scorch and Compression Set of Viton A

Viton A	100	Press cure. 30 minutes at 300° F.
Magnesium oxide [a]	15	Oven cure. Step + 24 hours at 400° F.
MT carbon black	60	
Hexamethylenediamine carbamate	1.1	
Additive	1.3	



[a]Maglite D.

[b]1. Same as *N,N'*-bis(*o*-hydroxybenzylidene)-1,2-propylenediamine (40 minutes).[c]
2. Between *N,N'*-bis(*o*-hydroxybenzylidene)-1,2-propylenediamine and hexamethylenediamine carbamate.
3. Same as hexamethylenediamine carbamate (6 minutes).[c]

[c]Mooney scorch at 250° F. (MS), minutes to a 10-point rise.

Table VII. 2,5-Dimethyl Piperazine as a Curing Agent for Viton A-HV

Compound	A-7	B-7	C-7
Viton A-HV	100	100	100
Magnesium oxide [a]	15	15	15
MT carbon black	20	20	20
Hexamethylenediamine carbamate	1	...	...
2,5-Dimethyl piperazine	...	0.8	1.0
Mooney scorch at 250° F. (MS)			
Minutes to a 10-point rise	7	42	31
Stress-strain, original			
Modulus at 100%, p.s.i.	390	330	380
Modulus at 200%, p.s.i.	1130	1050	1350
Tensile strength, p.s.i.	2500	2725	2750
Elongation at break, %	320	380	320
Hardness, shore A	67	65	65
Compression set, Method B			
70 hours at 250° F.	35	32	25
Stress-strain, after 5 days at 500° F. (oven)			
Tensile strength, % retained	57	58	66
Elongation, % retained	97	71	53
Hardness, change	+6	+8	+7

[a] Darlington 601.

Table VIII. Sulfenamide Derivatives of Diamines as Curing Agents for Viton A

Compound	A-8	B-8
Viton A	100	100
Magnesium oxide [a]	15	15
MT carbon black	18	18
Piperazine-bis-thiobenzene	2.7	...
Piperazine-bis-2-thiobenzothiazole	...	2.7
Mooney scorch at 250° F. (MS)		
Minutes to a 10-point rise	> 45	> 45
Curing conditions		
Press cure, 180 minutes at 500° F.		
Oven cure, step cure to 400° F. and 24 hours at 400° F.		
Stress-strain, original		
Modulus at 100%, p.s.i.	470	660
Tensile strength, p.s.i.	2050	2250
Elongation at break, %	300	290
Hardness, shore A	71	74
Compression set, Method B		
70 hours at 250° F.	52	29

[a] Keasby and Mattison XLC, Ambler, Pa.

Table IX. *N,N'*-Bis(o-hydroxybenzylidene)-1,6-hexylenediamine as a Curing Agent for Viton A-HV

Compound	A-9		B-9		C-9	
Viton A-HV	100		100		100	
Magnesium oxide [a]	15		15		15	
MT carbon black	20		20		20	
Hexamethylenediamine carbamate	1		...		...	
N,N'-bis(*o*-hydroxybenzylidene)-1,6-hexylenediamine	...		2		2.5	
Mooney scorch at 250° F. (MS)						
Minutes to a 10-point rise	7		> 45		> 45	
Curing conditions						
Press cure, 30 min.	300° F.	400° F.	300° F.	400° F.	300° F.	400° F.
Oven cure, step cure to 400° F. and 24 hr. at 400° F.						
Stress-strain, original						
Modulus at 100%, p.s.i.	390	410	270	430	270	580
Modulus at 200%, p.s.i.	1130	1200	640	1140	750	1600
Tensile strength, p.s.i.	2500	2400	2800	2975	2700	2925
Elongation at break, %	320	300	470	350	400	330
Hardness, shore A	67	68	68	71	68	73
Compression set, Method B, 70 hours at 250° F.						
No oven cure	65	67	... [b]	94	... [b]	85
Step + 24 hours/400° F.	35	37	... [c]	36	... [c]	27
Stress-strain, after 5 days at 500° F. (oven)						
Tensile strength, % retained	57	61	49	65	59	75
Elongation, % retained	97	100	90	85	100	61
Hardness, change	+6	+8	+4	+11	+6	+11

[a] Darlington 601.
[b] Blowing during test.
[c] Blowing during oven cure.

Table X. Miscellaneous Diimines as Curing Agents for Viton A

Compound	A-10	B-10	C-10	D-10	E-10
Viton A	100	100	100	100	100
Magnesium oxide [a]	15	15	15	15	15
MT carbon black	18	18	18	18	18
Hexamethylenediamine carbamate	1.1	...	...	...	...
N,N'-bis-(*N,N*-dimethyl-*p*-aminobenzylidene)-1,6-hexylenediamine	...	2.1	...	...	...
N,N'-bis-cinnamylidene-1,6-hexylenediamine	...	...	2.1	...	...
N,N'-bis-(*o*-methoxybenzylidene)-1,6-hexylenediamine	...	...	...	2.1	...
N,N'-bis-(*p*-methoxybenzylidene)-1,6-hexylenediamine	...	...	...	...	2.1
Mooney scorch at 250° F. (MS)					
Minutes to a 10-point rise	11	22	29	> 45	> 45
Compression set, Method B					
70 hours at 250° F.	25	25	25	20	23

[a] Keasby & Mattison.