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APPLIED SCIENCE RESEARCH LABORATORY UNIVERSITY OF CINCINNATI CINCINNATI 21, OHIO

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PROGRESS REPORT NO. 4 June 1, 1952 - July 31, 1952

ON

DEVELOPMENT OF NEW POLYMERS FOR AIRCRAFT APPLICATION

Navy Bu Aer Contract NOa(s) 52-090 c

Work of a

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Walter Soller

A FILE COPY

Approved:

Date: September 2, 1952

Walter Soller Director

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Summary

A. Chloroprene-Methacrylic Acid Copolymers

1. Curing Studies

Curing studies during this period were confined primarily to the investigation of room temperature cures. The optimum time for several agents has been found to be in the 10-14 day range. Good results, however, have been secured after shorter intervals of time; e.g., 6750 psi tensile strength was obtained with a film containing 5 pts ZnO after 3 days. (See Table I and Figure I)

Further investigation of the heat cure (1 hour at 120°C) versus air-cure for 1 pt MgO is in progress. Three days after pouring the films, the air-cured specimen had a tensile strength of 3500 psi compared with a 5600 value for the heat-cured specimen. After ten days the air-cure reached 6300 in contrast to 6400 for the other. Permanent set and elongation at break for the films were practically identical after ten days. Tensile strength after 17 days will be determined in order to discover whether the aircure eventually surpasses the heat cure. A similar tendency has been noted with ZnO and is being studied further. In any event, it has been shown that, insofar as the properties measured are concerned, the employment of heat with metal oxide cures is unnecessary.

2. Cornell Tests

The ninth series of Cornell rain-erosion test panels has been prepared and submitted for evaluation. Nine of the ten specimens submitted were cured at room temperature; Table II lists this information in detail. Tensile strengths ranged from 2300 to 7900 psi.

3. Effect of Aging on Film Properties

Duplicate film samples of the eighth series of Cornell tests have been checked for tensile strength and elongation after 2 months (Table III). Films cured with 10 parts Zimate and 2 parts DADPM were found to change little in tensile strength, except for specimen 8A, which was originally exceptionally low (2180 psi); in two months this reached a value of 3960 psi comparable to the other specimen cured similarly. In general higher elongation was accompanied by improved permanent set. The ASTM permanent set showed decrease on aging while the 1 minute recovery did not follow a set pattern, except in these cases where very poor initial recovery was realized; here appreciable improvement occurred. With the magnesia cure, there is an improvement in tensile and elastic properties on aging. For specimen 8K, excessive milling was taken to cause the exceptionally low initial tensile strength, but as in the case of 8A upon aging almost one mormal tensile value was noted.

Goodyear's rain erosion resistant material was found to improve upon aging in every respect i.e. tensile changed from 2000 to 2500 psi, elongation from 690 to 900 per cent and ASTM and 1 minute recovery from 126 to 38 and 20 to 12 per cent respectively. Gates' material, on the other hand did not show any "ange in tensile test, but an improvement in elastic properties.

4. Creep Behavior of the C-MA Copolymers

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The creep study has been continued during the past period to the candidate rain erosion coatings. The . bulk of the work has been concentrated on the specimens of the ninth rain erosion test sories. As indicated on Figure II and Table IV these creep specimens show a wide spectrum of compliances. The lower the compliance, i.e. the higher the modulus, the more effective is the cure for a given copolymer. It is indicated on Figure III (Table IV) that the Goodyear's and Gates' rain erosion resistant materials both have lower compliance than any of the C-MA copolymers. Before more significance is placed on the " 1 minute" and "ten minute" compliances, the results of the Cornell rain erosion resistance tests should be available. Other characteristics like smoothness of the film surface and loading will undoubtedly have an effect on the outcome of the rain erosion tests. It is commonly known, as also indicated on Figure IV, that the loading will reduce the compliance i.e. increase the modulus of a given elastomer. For instance, for one C-MA copolymer loaded with 10 parts of hydrophobic silica, the "1 minute" and "10 minute" compliances are respectively 3.8 and 2.6 times those of 20 parts hydrophobic silice. However, no significant difference in the rain erosion due to loading has been observed.

It is observed that with some curing formulations, considerable change in tensile strength results on aging. It has been shown in this connection that for the organis curing system consisting of 10 parts of Zimate and 2 parts of DADPM, a marked decrease in compliance occurs on aging.

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(Figure V and Table IV). Corresponding tensile data is not available, but further runs are planned, for which tensile data will be obtained concurrently with the creep information.

At first, it was believed that creep data on a much smaller time scale could be determined with the help of a moving picture camera. Upon closer study of the problems it has become apparent that this procedure is not feasible. If a load (the tension bob) is applied from rest, the longitudinal vibrations caused by the sudden application of the load do not enable the deformation to be determined before one hundreth of a minute.¹ Consequently the true instantaneous elongation can also not be determined by this method.

B. Chloroprene-Methacrylamide Copolymers

Until recently some difficulty had been experienced in reproducing results with chloroprene emethacrylamide copolymers. It has now been shown that two factors werew responsible for this, and that with their proper control, resonable reproducibility is now being attained. These factors were: (1) variation of time of film conditioning between cure and physical testing, and (2) variable polymer solubility due to insufficient milling. It has since been shown that the first effect arises from the fact that curing is not completed during the oven bake but continues over a period varying from several days to two weeks depending on the curing formulation.

Further progress has been made on curing formulations.

 C.A. Dahlgent, J.O. Hendricks, N.W. Taylor, Ind. Eng. Chem., 43 1404 (1951)

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The during system Ethyl Zimate, sulfur, tagnesia discussed in the previous report has been studied in considerable detail. Best results have been obtained with magnesia at 2 to 8 parts, Ethyl Zimate 3 to 12 parts, and sulfur at 2 parts. A typical film prepared with these formulations exhibited a tensile strength of 4780 psi, an elongation at break of 860% and an ASTM permanent set of 11%. This result was obtained using 6 parts Ethyl Zimate, 2 parts magnesia, and 2 parts sulfur in an 89% chloroprene copolymer at 62% conversion modified with 0.07 parts dodecyl mercaptan. The general properties of the copolymer thus are approaching those of the C-MA copolymers.

With the attainment of an improved cure the modification question can be considered from a different aspect. Although the physical properties of more modified films are lower with an inferior curing system, differences in modifications are of minor importance with the improved curing system discussed above. Complete polymer solubility appears to be a more important consideration.

The fractional precipitation studies mentioned in the previous report have now been completed. These studies indicate that the chloroprene-methacrylamide copolymer is heterogeneous in nature, and contains a considerable portion of what is essentially polychloroprene. The methacrylamide content was largely distributed between two of the five polymer fractions obtained. It is believed that the C-MA copolymers are of a similar heterogeneous nature.

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Program.

A. C-MA Copolymers

Results of the air-cure studies are considered most encourging; this program will be continued. The obtaining of better oxide dispersions (as curing agents) and of antithickening agents for the lacquers also will be investigated. Other possible lines of attack will await the results of the last series of Cornell specimens. The studies to obtain possible correlation between "creep" behavior and rain erosion properties also will be continued.

B. Chloroprene-Methacrylamide Copolymers

Curing studies for the chloropreme-methacrylamide copolymer will be directed toward the possibility of air cures. Several carbon blacks and silica will be evaluated as reinforcing agents. Frimer candidates are also being reexamined for the eventual goal of preparing Cornell test specimens.

Because of the indicated heterogeneity of the copolymer, several grafted copolymers of chleroprene and methacrylamide as well as blended mixtures of polychloroprene and polymethacrylamide are being studied. It is hoped that these investigations will shed further light on the nature of the copolymer.

Experimental

A. C-MA Copolymers

All Cornell specimens were cleaned with toluene after sending with THREE-M-ITE ELEK-TRO GUT CLOTH 320. Bostik primer 1007 was diluted slightly, and four coats applied at 5 minute intervals. After a drying time of about 15-20 minutes, four coats of Bostik tiecoat cement 1024 were brushed on at 10-15 minute intervals. The tie coat cement was charged with Bostik's B component for 1024 just before application. Methyl isobutyl ketone was added as a diluent to the extent of about 1/5 the original volume of tiecoat cement.

The employment of tiecoat was necessitated by the pronounced effect of the mixed ketones now usediin C-MA lacquers (50 by volume of cyclohexanone and methyl isobutyl ketone) upon the Bostik primer. When the tiecoat was not used the primer gradually diffused up through the topcoat layers.

Four coats of C-MA primer were used on panel 9B, 9C, 9F, and 9H. Application (Experiment 2) of coats was at 20-30 minute intervals. After a thorough drying these panels were cured for 4 hours at 120°C.

In the preparation of panel 9G, 8 coats of topcoat lacquer duritaining 5 parts of DuPont's Hydrophobic Silica were first employed followed with a final 4 layers of a non-silica-containing lacquer. This was done in an attempt to secure a smoother outer surface combined with an inner resistance to heat build-up which may possibly have a bearing upon erosion. Although panel 9D had a large flaw along the trailing edge, it was included in the series since the critical leading edge area was of the same quality as the other specimens.

If not kept on ball mill, the slurries of metal oxides tend to form a cake on the bottom of the bottles used. This cake has a tendency to leave lumps in the slurry even when remilled. It has been found that the addition of several drops of an aldehyde-amine accelerator prevented caking completely. The effect, if any, of this additive upon the curing performance remains to be studied. The dispersive power of the addehyde-amines (viacous organic liquids) probably results from the forming of a thin oily film on the oxide particles reducing the strength of adherence between the particles. Theoretically, this should also give a more uniform cure, probided that the small quantity of aldehyde-amine has no deleterious effect on the films.

It is believed that this ability to disperse particles is not limited to the class of compounds mentioned, but that many liquids within a suitable viscosity range should perform in a like manner.

A 560 gram batch of copolymer has been prepared in the event results should warrant a service test in the near future. Polymerization was carried out in the usual manner, and stopped at a conversion of 55% and an acid content of 12.7%.

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B. Chloroprene-Methacrylamide Copolymers

It has been the procedure with respect to physical testing of free films to condition the films a minimum of 48 hours at 21°C and 65% relative humidity prior to testing. Conditioning times have varied from two days to over a week in some instances. It has now been shown that the cure continues after the oven bake and that the elapsed time between baking and testing is significant and must be controlled. The effect of increased conditioning time upon tensile strength is illustrated by the following data.

,q

Table V

Effect of Conditioning Time Upon Tensile Strength of Chloroprene-Methacrylamide Copolymers

Pensile After ba	Strength psi, ke.	4 days	Tensile Strength after bake	ps1, 12 days
	2380	`,	3140	
	1770	, ``	2200	r D
· n (1	1980		3000	<i>"</i>
	2200	, 	2950	и к 1
1 v.	20 30		2770	
. .	2 240		3060	et et an

1. The above films were cured with one part magnesia and baked two hours at 120°C.

It has been established that this effect is not a result of moisture content. The following data also provides information on the length of time required to attain maximum tensile strength.

Table VI

Elapsed Days Since Cure	14 C-1	14 0-2	<u>14 G-1</u>	<u>14 G-2</u>
2	3380	3480	2160	2140
4 .**	5580	3510	250 0	2560
· • • • • • • • • • • • • • • • • • • •	3620	3680	2890	2950
9	3220	3330	2850	2670
11	3620	3490	3 020	3070
14	4040	4020	3090	3030
21	3840	3870	3230	3200
28	3880	.3970	3250	3200

magnesia, 2.5 parts sulfur, and 10 parts Ethyl Zimate; samples 146 G-1 and 14 G-2 were cured with 1 part magnesia. After baking, films 14 C-1 and 14 G-1 were soaked in water for two hours and conditioning then begun; films 14 C-2 and 14 G-2 began the conditioning completely dry. No significant difference can be detected between the results with dry and wet films. Hence it has been concluded that the effect is due to delayed cure and not to moisture content.

The period necessary to attain maximum physical properties is not readily apparent and appears to vary with the nature of the cure. For practical reasons a period of one week has been established as a tentative basis for comparison.

A second factor which has contributed to better reproducibility has been improved solubility. This has been produced by additional milling on the rubber mill. The improvement in tensile strength with milling is illustrated in the following data.

Milling Time, min.	% Soluble	T.S., psi
4	63	3730
6	79	3600
••••••••••••••••••••••••••••••••••••••	84 · · · ·	3890
10	91	4330
12	99	4370

Milling produced no significant change in elongation at break or permanent set; for all of the above films the elongation was approximately 750% and the set 6%. Films were cured with 6 parts Zimate, 2 parts mignesia and 2 parts sulfur and conditioned 1 week before testing. None of the above lacquers were badly gelled; however, the lacquer from the polymer milled 12 minutes was completely gel

Several other variations in procedure have been investigated with the hope of discovering other causes of poor reproducibility; these included: (1) coagulation with sodium sulfate as was practiced in earlier work; (2) elimination of the alkaline wash on the chloroprene (3) recrystallization of the methacrylamide from distilled methyl ethyl ketone; and (4) elimination of the pH control salts. No significant improvement wasnoted in any case except the latter; here the improvement involved only a reduction in the amount of precoagulation during polymerization.

Earlier curing studies had indicated that the cure provided by a combination of Ethyl Zimate, magnesia and sulfur showed considerable promise. Several tensile strengths over 4000 psi were recorded using 10 parts Zimate, 3 parts magnesia, and 2.5 parts sulfur. Occasional blooming was encountered, however; hence a series of films was prepared to establish the optimum quantities of the three components. In all cases the bake was two hours at 120°C.

Table VII

Sffect	of	Variat	ions	of	Zimate,	Magnezia,	and	Sulfur	upon
hysic		ropert	165						

	FALLOD				
Zimate	Magnesia	Sulfur	T.S. psi	Elong.	ASTM <u>5 Set</u>
6.		0	3730	890	10
		ີ້ 2 ປ້	4780	86Ő	" ~ 11 .
		4	4130	780	. 11

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	• 1 • • • •	х - + 1		, - e, a •	ASTM	
Zimate	Magnesia	Sulfur	T.S. psi	Elong. %	Set	
		6	3080	650	10	
6	0	2	4240	1000	6	
	2		4780	860	11	
	4		4770	780	6	
	6		4500	750	6	
	8		4810	760	7	
0	2	2	3410	890	9	
3			4800	930	9	
6			4790	860	11	
9			4620	840	9	
12			4670	740	11	
15 [#]			# 0 70	730	5	

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* Blooming

The amounts of Zimate and magnesia do not appear critical in the range of 3 to 12 parts for the Zimate and 2 to 8 parts for the magnesia. Sulfur shows a sharp maximum in tensile strength at 2 parts. For fut we polymer comparisons a standard cure of 6 parts Zimate, 2 parts magnesia and 2 parts sulfur has been adopted.

The above curing data was obtained on an 89% methacrylamide copolymer at 62% conversion modified with 0.07 parts dodecryl mercaptan. A limited amount of data on an identical copolymer modifier with 0.14 parts dodecyl mercaptan shows a reasonable improvement in physical properties with the Zimate-magnesia-sulfur cure even at the higher mercaptan level. A formulation consisting of 10 parts Zimate, 3 parts magnesia, and 2.5 parts sulfur gave a tensile strength of 3780 psi, an elongation at break of 790% and a permanent set of 9% with only five days of conditioning. It is believed that with the use of the optimum Zimate-magnesiasulfur cure together with proper conditioning these more modified film will be more nearly comparable to those modified with 0.07 parts mercaptan.

A number of other miscellaneous cures have been examined of which the only one that may bear further investigation is one employing Goodyear accelerator 983C. This datais summarized in Table VIII. The bake employed in all cases was 2 hours at 120°C.

Table VIII

Effectiveness of Miscellaneous Curing Agents on Chloroprene-

Cure	T.S. psi	Elong. %	ASTM Set
Goodyear 983c, D.9 cc/gm	4280	910	6
Goodyear 983c, 1.8 cc/gm	3760	970	6
Goodyear 983c, D.9 cc/gm plus 2 parts S	3310	980	6
1 part magnesia	3930	1010	. 9
2.5 parts magnesia	4030	910	8
4 parts magnesim	3460	920	9
6 parts magnesia	3460	660	11
1 part MgO, 5 parts HCHO	2690	750	8
1 part Mg0, 10 parts HCH0	2430	830	9
1 part MgO. 5 m rts HCHO	2280		

2.5 parts S

Further data has been collected illustrating the effect of composition on physical properties.

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-	o on doi j zanzao					<i>.</i> .	"	
		"l par	t magnes:	ia 👘	Zima	te-magne	aia-sul	fur
Ľ	Chloroprene	TS	Elong	Set	TS	Elong	Set	
	90	3860	940	14	4270	810	14	
	87	3530	1000	14	4110	860	6	
•	85	3430	900	14	3960	780	. 7	
	82	3190	870	16	3660	800	··· 8	

Effect of Cure upon Physical Properties of Chloroprene-Methacrylamide Copolymers

All of the above cures were 2 hours at 120°C. The decrease in tensile strength shown is similar to that previously encountered. There still remains a question, however, as to whether the decrease in tensile strength may not be a function of the decreasing solubility.

The fractional precipitation of a chloroprene-methacrylamide copolymer has been completed. An 89% chloroprene copolymer at 51% conversion and modified with 0.14 parts dodecyl mercaptan was dissolved in distilled cyclohexanone at 2% and divided into 5 fractions by fractional precipitation with a 50:50 toluene-methanol mixture. Out of 30 gms of polymer the following fractions were recovered.

Ta	bl.	X
-	N.T.O.	~

Copolymers				Tutut
Fraction No.	Wt.	% Chloroprene	% Boluble	Viscosity
1	5.07	77	31	0.38
2	6.81	95	90	0.55
3	5.81	98	100	0.65
	3.79	98	100	0.39
5	1.17	98	100	0.30

Table IX

The original polymer was 92% soluble in cyclohexanone with an intrinsic viscosity for the soluble portion of 1.09. All viscosities were determined at 30° C.

Free film properties were obtained only on fractions 2 and 3 above; fraction 2 showed a tensile strength of 3030 psi, an elongation of 820% and a set of 13%; the corresponding values for fraction 2 were 2510 psi, 860%, and 10%.

It will be noted that fractions 3, 4, and 5 all have a nominal chloroprene content of 98%; considering the 2% error in the analysis discussed in a previous report these samples can be considered as essentially 100% polychloroprene. Hence at least 35% of a typical chloroprene-methacrylamide copolymer is virtually polychloroprene. The other two fractions both contained methacrylamide. It is believed that these fracti ns contain some actual copolymer or at least grafted polymer. This conclusion cannot be definitely established; however, it is apparent that the methacrylamide content of these fractions cannot be pure polymethacrylamide as this could not have been removed with the insoluble portion from an aqueous emulsion system. The possibility remains that polymethacrylamide insolubility may have been created by the loss of ammonia between amide groups on adjacent polymer chains leading to cross-linking through the formation of imide groups.

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Compilation of Ten	sile Str	engths of	Various Air Cu	res of C-MA	
	Co	polymer A-	<u>-K-20</u> 1		• •
	Tensile	Strength	(psi) after		 9 .
System	3 Days	7 Days	10-11 Days	14 Days	21 Days
5 pts ZnO	6750	7200	7930	7920	7300
1 pt ZnO + 6 pts DADPM	•• ••	4 •	4330	۰۵ می دو. ۱۹	•
6 pts DADPM	а 1	-	2930	•	· · · ·
2 mls Accel. 983-C por 2 gm Polymer	1300	2250	2310	2330	2000
2 mls 983+C + 0 4 pts DADPN	1430	2720	3150	3260	3200
2 mls 983-C+ 4 pts DADPM+ 2 pts ZnO	2400	4630	6020	6130	6300
l pt Mg0	3510	" •	6300	н. Sa.	
2.5 pts Zn0	2940		4270	-	•

Table I

1. Conversion - 52.5%; acid content - 11.8%

			Cornell Series No. 9	· ·			
Specimen No.	Polymer No.	Additives	Curing Agents	Curing Conditions	Primer	No. of Coats	Approx. Thickness in Mils
9 4	A-37 l	2 pts Royal Spectra Car- bon Black	5 pts Zn0	Room Temp.	Bostik	1 . 1 .	8 • •
8	AK-20 ²	Nora	lo pts Zimate ⁵ / 2 pts DADPM ⁴	1 hr. at 120° C	C-MA	14	0°0 8
0 6	AK-20	2 pts Royal Spectra	2 mls. Accel 983-C ⁵ per 2 gms pclymer	Room Temp.	C-MA	4	0.8
	AK-2 0	Here		Кот Тещ.	Bostik	₩	8.5
<u>म</u> 8	AK-20	None	2 mls Accel. 983-C per 2 gms Polymer	Room Temp.	Bostlk	Ω ۴-۴	0°0
£ч 65	AK- 20	None	2 mls Accel. 983-C ver 2 gms Polymer	Rcom Temp.	C-MA	12	4 7 .
	AK-20	5 pts Hydro- phobic Silica	5 pts Zn0	Rcom Temp.	Bostik	12	7.5
9H	AK-20	None	1_pt Mg0	Room Temp.	C-MA	1.3	0.9
16	AK-20	None	2 mls 983-C/2gm Polymer / 4 pts DAD	Room Temp. PM	Bostik	1.5	0.0
6	AK-2 0	Kone	2 mls 983-C/2gm pol mer / 4 pts DADPM / 2 pts ZnO	^y Room Temp.	Bostik		
- 00 0 - 00 0 - 00 0	MA Copol	ymer with conve ymer with conve fluttatogaria	rsion of 55.0% and act rsion of 52.5% and act dt0:	d content of 12.7%. d content of 11.3%.			

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Table II

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p-p¹-Diaminodephenylmethane. Goodyear Accelerator 983-C ້ທີ

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Aging Characteristics of the C-MA Copolymer Films						
Specimen	Curing System	Tensile psi	Elong %	ASTM Set %	Recovery %	
84	10-Zimate, 2-DADPM 6% Ludox (latex)	2180 3960	830 890 -	27 19	4 6	
80	10-Zimate, 2-DADPM 10 p. Hydrophobic Silica	457 0 4670	680 870	31 25	7 / 10	
BD	lo-Zimate, 2-DADPM 20 p. Hydrophobic Silica	4170 4530	750 790	35 37	13 18	
8E	10-Zimate, 2-DADPM S.4% Ludox (latex)	3620 4030	540 600	114 45	66 37	
8F	10-Zimate 3.4% Ludox (latex)	3810 3590	550 600	79 79	65 41	
86	10-Zimate, 2-DADPM 3.5% Ludox (latex)	4560 4200	810 810	24 18	6 6	
81	1% Mg0	2880 3900	870 890	18 12	3 3	
A K-2 0	1% Mg0	4840 5020	985 910	25 20	5 3	
Gates	· · · · · · · · · · · · · · · · · · ·	2800 2800	700 840	20 7	7 4	
Goodyear		2000 2500	690 900	126 3 8	33 12	

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Table III

1. All specimens, except Gates' and Goodyear's cured 1 hr. at 120°C. Time between the two series: 2 months (original test on the upper line)

Run No.	Specimen	Curing System ¹	Aging Cune Days	Load Kg/cm ²	l min. Comp.	lO min. Comp.
3	Gates	B en	R.T	3.38	1.15	1.3
3	C-MA-T	10-Zimate, 2-DADPM	1 hr. 7 at 120°C	9.18	8.5	12.9
7	C-MA-T	10-Zimate, 2-DADPM	^N 64	9.85	6.4	86
6	Goodyear	ан 9 • • • • • • • • • • • • • • • • • • •	R.T. 6	33.1	0.15	0.22
· 8	AK- 20	10-Zimate, 2-DADPM 10-Hydrophobic Silica	l hr. 60 at 120°C	6.20	3م3	4,5
· .9	AK-2 0	10-Zimate, 2-DADPM 20-Hydrophobic Sili	# 60 ca	7.44		1.75
10	9E		R.T. 18	8.81	10.5	16.7
11	9 A		R.T. 10	8.12	3.6	4.7
12	9 F	аналанан аланан алан Колтон аланан	R.T. 20	8.32	~ 16	~30
13	9J		R.T. 19	9.45	7.4	10.3
14	9D		R.T. 14	7.63	3.5	5.3
15	90		R.T. 23	5.63	~16.5	~ 36
16	9 B	and the second secon Second second	R.T. 20	5.89	7.2	9.9
1,	Curing syst	ems for the 9th Rain	Erosion test	series	indicat	bd

Table IV

Creep Behavior of the C-MA Copolymers

in Table I

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