AD-755 827

HEAT AGEING OF RUBBERS

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Waltham Abbey, England

March 1972

 ERDE TR 91

MINISTRY OF DEFENCE

EXPLOSIVES RESEARCH AND DEVELOPMENT ESTABLISHMENT

TECHNICAL REPORT No. 91

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SUMMARY

The oven ageing behaviour of six rubbers (nitrile, polychloroprene, PVC/nitrile, fluorocarbon, EPDM and natural), in the form of tensile dumb-bells has been measured and attempts have been made to relate the ageing behaviour to conventional rate processes. By fitting to an empirical equation the change of elongation at break the prediction of shelf storage life at ambient temperature can be made for all the rubbers except the fluorinated rubber (Viton).

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Reference: WAC/213/033

1 INTRODUCTION

It is often necessary to predict the safe storage period or working life of a rubber component at the design or pre-production stage. The most commonly accepted way of doing this is to perform accelerated ageing trials on either the component or a test sheet of the same composition in the chosen environment at higher temperatures and fitting the results where possible to an Arrhenius expression:

$$k \text{ (rate of change)} = A \exp(-E/RT)$$

where E = activation energy of the ageing process

T = absolute temperature

R = gas constant

Difficulty is often experienced in fitting the results for rubbers to such an equation. This is hardly surprising due to the complex chemical nature of a rubber vulcanizate. Other factors which add to the difficulty of forecasting a realistic life are:

a manner and environment in which rubber is used,

b thickness of rubber,

c presence or absence of oxygen and ozone,

d rate of diffusion of all degredants (gaseous, liquid or solid),

e ingredients of mix and post vulcanization residues, and

f type of crosslink system

One of the most universal tests used is that of accelerated ageing in a cell oven where rubber samples, usually dumb-bells, are suspended in a free flow of air at various temperatures for different periods of time. Properties measured are tensile strength, elongation at break, modulus and hardness. These results and knowledge of the effects of the variables listed previously, together with previous experience of the ageing of similar materials, usually allows a reasonable estimate of the safe storage or working life to be made. However, one is still faced with the difficulty of deciding in the laboratory the criteria which would govern failure of a component. Thomas et al[1] have in their work arbitrarily defined 10 per cent change in properties such as tensile strength and elongation at break to constitute the safe storage life. Clearly however this cannot easily apply to components such as O-rings and seals since "sealability" is a difficult property to define. In this work the oven ageing behaviour of six different rubbers has been followed.

2 EXPERIMENTAL

2 1 Materials

Rubbers used in this work were:

Nitrile Rubber	Krynac 803 - Polysar UK Ltd
Polychloroprene	Neoprene WRT - Du Pont Ltd
PVC/Nitrile	Breon Polyblend 503 - British Geon
Ethylene/Propylene/Diene Rubber (EPDM)	Nordel - Du Pont Ltd
Fluorinated Rubber	Viton B - Du Pont Ltd
Natural Rubber	Heaveacrumb SMR5 - Natural Rubber Producers' Research Association

Vulcanizates of these rubbers in the form of sheets 150 mm × 150 mm × 2 mm were prepared according to the composition and conditions of cure given in the Appendix.

2 2 Heat Ageing

British Standard type E dumb-bell test pieces were cut from the vulcanized sheets and the width and thickness measured before exposure to the test conditions. Dumb-bells in sets of five were suspended in open glass tubes and exposed to some or all of the following environments:

Hot/dry	Suspended in air at 40, 60, 70, 80, 90, 100 and 150°C
Hot/wet	Immersed in boiled out distilled water at 70 and 90°C
Hot/humid	Suspended above boiled out distilled water at 70 and 90°C
STF	Suspended in iso-octane/toluene, 75/25 v/v mixture at 40 and 60°C.

The charged tubes were placed in a circulating air oven in which the temperatures were within ± 0.5°C of the test temperature. At the end of each exposure period, the required tubes were removed and the contents conditioned at room temperature for 24 hours before testing. Where necessary the test pieces were then dried from superficial liquid and tested for tensile strength, elongation at break and modulus as quickly as possible. The tensile properties were measured by British Standard methods on a Hounsfield Tensometer. No results were discarded except those from obviously faulty specimens; the average values and the percentage changes in original property have been calculated.

3 RESULTS

Ageing results on the six rubbers are given in Tables 5 - 10. Examination of these results shows that under all conditions for all materials elongation at break is the parameter showing the most change with time of ageing. Other properties such as tensile strength, showed quite erratic behaviour. The elongation at break results in the tables have been analysed in terms of conventional rate processes

$$x_0 - x = kt \quad \text{zero order} \qquad (1)$$

$$\log x/x_0 = kt \quad \text{first order} \qquad (2)$$

$$\frac{1}{x} - \frac{1}{x_0} = kt \quad \text{second order} \qquad (3)$$

where x is the property at time t or time zero and k is the rate constant for the reaction.

However only in the case of neoprene do the results conform to rate equations of this type. Results for elongation at break of nitrile rubber appear to fit a first order relationship at 100°C only. The other rubbers give curved plots of property versus time which will not fit any of these relationships.

Taking the results for neoprene and plotting these as a first order relationship, $-\log EB_t/EB_0 = kt$ (where t = time in days or weeks), characteristic values for k are obtained (Fig 1, Table 1).

TABLE 1

Temperature, °C	$\frac{1}{T}$	k	log k
70	2.91	0.0032	-2.495
90	2.75	0.023	-1.638
100	2.68	0.042	-1.377

The value of k at 100°C is in good agreement with the value reported by Thomas et al[1] for the heat ageing of Neoprene WRT. If log k is plotted against 1/T, where T is the absolute temperature, a straight line is obtained (Fig 2), therefore k can be described in terms of temperature by the equation $k = A \exp(-E/RT)$. The values from Fig 2 are given in Table 2.

TABLE 2

Energy of activation E, kJ/mole (kcal/mole)	Pre-exponential factor A
114 (27.2)	1×10^{12}

These values are somewhat different from those obtained by Thomas et al but are well within experimental error for this type of experiment. From this analysis the elongation at break of Neoprene WRT only, can reasonably be described by the pair of equations

$$k = 10^{12} \exp\left(\frac{-27.2 \times 10^3}{RT}\right) \quad (T \text{ in K})$$

$$\text{and} \quad \log EB_t/EB_o = -kt \quad \text{where } t \text{ is in days}$$

With nitrile rubber the plot log EB_t/EB_o vs time only gives a straight line at 100°C. The value of k obtained in this case is 0.05 (within 20 per cent of the value found by Thomas et al). Results at lower temperatures do not fit this relationship and illustrate the great difficulty with accelerated heat ageing. Any predictions in this case from results obtained at 100°C and above greatly underestimate the initial rate of degradation at 70°C but overestimate the rate of loss of EB at longer times where $EB_t \leq 0.8\ EB_o$.

Other empirical relationships have been investigated in an attempt to fit the observed results to a mathematical expression. These include

$$X = X_o - kt^{\frac{1}{2}} \qquad (4)$$

$$X/X_o = A \exp(-kt) \qquad (5)$$

$$X/X_o = A \log t - B \qquad (6)$$

where X is the property at time t and X_o at time zero.

Apart from Viton, which shows little change on ageing and fits none of these relationships, the intermediate ageing results of the other rubbers gave reasonable straight lines with Equations 4 and 5 but the best series of lines were obtained when considering elongation at break and using Equation 6. The initial results again did not fall on the lines but longer time results produced a series of straight lines, in some cases parallel, corresponding to the different temperatures of ageing (Figs 3 - 9). Viton was the only material which did not show this behaviour.

This method of plotting results was first used by Steingiser et al[2] who examined the ageing of flexible polyurethane foams. They defined a term "Threshold Ageing Period" (TAP) to describe the period of time before a rapid loss of properties occurs. Since actual ageing curves tend to be sigmoid and the exact point where rapid ageing commenced was not easily observed, the parallel straight portion of the graphs were extrapolated back to the $EB_t/EB_o = 1$ line (see Fig 3). Fitting these results to an equation of the form (TAP) = A exp(-E/RT) and plotting log TAP vs $\frac{1}{T}$ (K) an apparent activation energy of the process can be determined.

TABLE 3

	E', kJ/mole (kcal/mole)	from Table 2	Ref 1	Ref 3
Nitrile	101 (24.2)	-	77.5 (18.5)	80 ± 8 (19 ± 2)
Neoprene	96.3 (23.0)	113 (27.0)	90.9 (21.7)	80 ± 8 (19 ± 2)
Natural	113 (27.0)	-	-	100 ± 16 (24 ± 4)
EPDM	92 (22.0)	-	-	-
PVC/nitrile	105 (25.0)	-	-	-

In this investigation all the rubbers appear to have similar values for E. It now becomes possible to calculate the TAP at other temperatures and obtain an estimate of the storage life of these rubbers. This gives the results at 20°C in column 1 Table 4. These compare with the percentage retention of elongation given in the second column of control samples of the same rubbers stored in the laboratory at JTRU Australia[4] which show actual changes occurring in storage. These changes show the pattern which would be expected from the TAP values.

TABLE 4

Rubber	TAP at 20°C	% IEB from Ref 4
PVC/nitrile	20 years	105
Neoprene WRT	10 "	104
EPDM	8 "	89
Natural rubber	3 "	85
Nitrile	2 "	83

This method of plotting ageing results only appears to be suitable for elongation at break. Changes in tensile strength appear almost random in nature.

Two qualifications must be added to the results. Firstly, results on rubbers aged under humid or wet conditions give, not surprisingly, different plots and ageing results actually carried out at 40°C on nitrile rubber suggest that its life may be underestimated by calculating TAP from higher temperature measurements. The results for Viton should be discussed separately. This is known to be a material having long storage capability and ageing results in dry air confirm this. However, in wet conditions Viton ages relatively quickly (as indicated by changes in tensile strength and modulus) and these results appear to follow the form suggested, ie $E/E_0 = A \log t - B$.

4 CONCLUSIONS

The ageing results for six rubbers have been presented and analysed. Apart from elongation at break values for Neoprene WRT at 70, 90 and 100°C and nitrile rubber at 100°C the results are not described by conventional rate processes. An alternative method of plotting the results has been examined with some success for elongation at break values and this method allows some estimate of safe storage life at room temperature to be obtained for five materials. Viton shows little change when aged under dry conditions but shows a drastic fall in properties if aged under wet conditions. Predicted ageing behaviour appears to show some correlation with actual ageing results on the same materials stored at JTRU Australia.

5 REFERENCES

1 Thomas D K, Sinnott R, Day J — RAE Tech Report 67118

2 Steingiser S, Darr W C, Saunders J H — Rubb Chem Tech, 37, 38 (1964)

3 Juve A E, Schoch Jr H G — Mater Res Stand, Bull Am Soc Test Mater, 1, 7, 542 (1961)

4 Evans D J, Hollingsworth B L, Ledbury K J, Stokoe A L — ERDE Report No 20/R/68

APPENDIX

ELASTOMER COMPOSITIONS

1 NITRILE

	Parts by weight
Butadiene/acrylonitrile (Krynac 803)	100
GPC black (Ucarb 327)	60
Resin	3
Stearic acid	2
DOP	10
Zinc Oxide	5
Phenol aldehyde amine condensate	1
MBTS	1.5
MC sulphur	1.5

Cure 15 min at 146°C

2 POLYCHLOROPRENE

Neoprene WRT	100
FEF black	30
Silica	20
PBN	2
TMTM	1
Stearic acid	0.5
Oil OM 13	5
Litharge	20

Cure 20 min at 146°C

3 PVC/NITRILE

	Parts by weight
PVC/nitrile (Breon 503)	100
Nitrile rubber (Krynac 803)	83
Zinc oxide	5
Stearic acid	1
ZDC	2
TMTD	2
SRF black	30
Sulphur	1

Cure 30 min at 144°C

4 ETHYLENE/PROPYLENE/DIENE POLYMER

EPDM (Nordel 1700)	100
HAF black	50
Sulphur	1.5
Zinc oxide	0.5
TMTD	1.5
MBT	0.5

Cure 15 min at 165°C

5 FLUORINATED RUBBER

Viton B	100
Magnesium oxide	15
MT black	20
Dicinnamylidene 1-6 hexane diamine	2.5

Cure 1 hour at 150°C. Post cure 24 hours at 200°C followed by 24 hours at 250°C

6 NATURAL RUBBER

	Parts by weight
Smoked sheet	100
HAF black	50
Stearic acid	0.5
Pine tar	4.5
Zinc oxide	5.0
TMTD	2.5
PBN	1.0
MBT	1.0

Cure 15 min at 141°C

TABLE 5

ACCELERATED AGEING OF NITRILE RUBBER

Period of Test	Dry															
	Initial Tensile Strength ITS (MN/m^2)						Initial Elongation at Break IEB (%)						Initial			
													100% (IM 10			
	16.0						540						1.5			
	% ITS						% IEB						% IM 100			
	Ageing Temp (°C)						Ageing Temp (°C)						Ageing Temp			
	40	60	70	80	90	100	40	60	70	80	90	100	40	60	70	80
20 min					99	100					107	112				
1.7 hours			98	95	98	103			109	102	100	102			106	108
2 days			104	95	105	102			105	79	66	61			124	144
4 days				96	108	106					60	61				
1 week	105	92	96	110	125	118	97	95	88	77	51	37	86	101	149	196
2 weeks	103		111	106	124	115	98		77		48	20	88		156	188
4 weeks	106		118		122	126	83		69		34	18	110		168	
10 weeks		96			110			69			32			185		
12 weeks	106		112	116		131	85		65	44	28	5	116		227	425
26 weeks	109		119		133	brittle	77		41		27	brittle	131		350	
30 weeks				115	144					29	10					780
40 weeks					143						7					
52 weeks	109	97	114				76	48	34		8		127	94	480	
104 weeks	115		124				71						252		836	
156 weeks	112						77						226			
260 weeks	103						65						304			

RELATIVE HUMIDITY 10

Period of Test	% ITS			% IEB		
	40°C	70°C	90°C	40°C	70°C	90°C
1 week	100	110	108	94	88	54
2 weeks	101	111	111	89	69	49
4 weeks	103	111	116	91	69	44
12 weeks	102	114	124	91	64	25
26 weeks	103	107	71	84	45	15
52 weeks	109	107	Too brittle	88	33	Too brittle
104 weeks	101	53		67	37	

Dry													Wet (Immersed in Water)			
eak	Initial Modulus at per cent Elongation (MN/m^2)															
	100% (IM 100)						300% (IM 300)									
	1.5						7.6									
	% IM 100						% IM 300						% ITS	% IEB	% IM 100	% IM 300
	Ageing Temp (°C)						Ageing Temp (°C)						Ageing Temp (°C)			
00	40	60	70	80	90	100	40	60	70	80	90	100	70	70	70	70
12					106	93					101	94				
02			106	108	107	108			100	99	104	112				
61			124	144	174	190			113	113	157	180				
61					250	242				147	201	199				
37	86	101	149	196		338	93	96	129	174			111	76	132	130
20	88		156	188			96		138				112	72	137	140
18	110		168				105		161				115	64	142	160
		185			610			150								
5	116		227	425			127		180				110	50	214	178
ttle	131		350										105	41	246	
				780												
	127	94	480				130						87	29	340	
	252		836				156						42	13		
	226						187									
	304						211									

RELATIVE HUMIDITY 100%

% IEB			% IM 100			% IM 300		
40°C	70°C	90°C	40°C	70°C	90°C	40°C	70°C	90°C
94	88	54	92	138	182	92	125	168
89	69	49	99	149	219	97	143	185
91	69	44	114	154	288	104	155	
91	64	25		271			194	
84	45	15		300				
88	33	Too		427				
67	37	brittle		244				

TABLE 6

ACCELERATED AGEING OF NEOPRENE WRT

Period of Test	Dry														
	Initial Tensile Strength ITS (MN/m²)					Initial Elongation at Break IEB (%)					Initial Modulus a (M				
											100% (IM 100)				
	17.8					500					2.1				
	% ITS					% IEB					% IM 100				
	Ageing Temp (°C)					Ageing Temp (°C)					Ageing Temp (°C)				
	40	70	80	90	100	40	70	80	90	100	40	70	80	90	100
1 day				88	102				92	92					
2 days				87	106				89	84					
4 days				86	105				86	78					
1 week	97	95		108	96	100	100		80	72	110	91			176
2 weeks	99	96	111	109	89	96	90	88	76	50	113	113			290
4 weeks	103	104	112	99	81	93	91	75	54	34	119	122	189	295	427
12 weeks	119	104	107	71	brittle	94	79	63	41	brittle	129	172	264	385	britt
26 weeks	107	97	92	56		91	61	40	12		151	241	379		
52 weeks	104	81				83	42				163	320			
104 weeks	105	80				77	36				193	414			
156 weeks															
260 weeks	101					64					236				

RELATIVE HUMIDITY 100%

Period of Test	% ITS			% IEB		
	40°C	70°C	90°C	40°C	70°C	
1 week	100	94	98	94	92	
2 weeks	100	89	95	94	91	
4 weeks	108	101	88	93	97	
12 weeks	106	101	77	98	76	
26 weeks	108	84	20	92	76	
52 weeks	105	71	very soft	88	56	ve
104 weeks	107	81		85	32	

Initial Modulus at per cent Elongation (MN/m²)										Wet (Immersed in Water)			
100% (IM 100)					300% (IM 300)								
2.1					9.7								
% IM 100					% IM 300					% ITS	% IEB	% IM 100	% IM 300
Ageing Temp (°C)					Ageing Temp (°C)					Ageing Temp (°C)			
40	70	80	90	100	40	70	80	90	100	70	70	70	70
								110	122				
								141	142				
								151	166				
10	91			176	100	95		292	148	104	95	121	112
13	113			290	106	112				105	93	118	117
19	122	189	295	427	113	117	163			106	91	120	122
29	172	264	385	brittle	122	151	195		brittle	106	85	124	128
51	241	379			124	184				76	58	143	
63	320				134					50	42	170	
93	414				151					43	40	164	
												145	
36					176								

ELATIVE HUMIDITY 100%

	% IEB			% IM 100		% IM 300	
0°C	40°C	70°C	90°C	40°C	70°C	40°C	70°C
98	94	92	103	106	109	107	101
95	94	91	94	114	102	110	98
88	93	97	74	116	118	111	106
77	98	76	38		175		149
20	92	76	12	106	187	108	128
soft	88	56	very soft	133	208	122	124
	85	32		144	500	130	

TABLE 7

ACCELERATED AGEING OF PVC/NITRILE RUBBER

Period of Test	Dry										
	Initial Tensile Strength ITS (MN/m^2)					Initial Elongation at Break IEB (%)					
	14.6					420					
	% ITS					% IEB					
	Ageing Temp (°C)					Ageing Temp (°C)					
	70	90	100	120	150	70	90	100	120	150	70
1 day	95		100		43	92		90		12	88
2 days	105		106			98		82			85
4 days	103		104			97		81			87
7 days	94		102	105	35	84		70	41	8	97
14 days	110	99			28	98	76			14	79
18 days				38					12		
28 days		86	106		19		72	72		2	111
8 weeks		109	106				66	45			
12 weeks		106	136				52	36			
16 weeks	97					76					
32 weeks											
52 weeks	115			77		66			9		152
104 weeks	115					55					258

	WET (Immersed in Water)												
	% ITS			% IEB			% IM 100			% IM 300			
	70°C	80°C	90°C	70°C	80°C	90°C	70°C	80°C	90°C	70°C	80°C	90°C	70°
2 days	97	96	92	91	86	80	80	81	78	77	79	74	
4 days	87	96	92	90	91	85	74	75	75	70	72	76	
7 days	96	100	99	87	82	80	84	78	80	75	81	84	
14 days	93	97	97	83	80	79	88	93	80	83	89	85	
18 days													
28 days	108	101		74	75		96	83		131	123		[illegible]
8 weeks			93			60			95				
12 weeks	87	72		64	57		89	72					[illegible]
16 weeks			69			47			[illegible]				
32 weeks		82	48		49	28		[illegible]	[illegible]				
52 weeks	79	38		45	19		148						[illegible]
104 weeks	57			26			202						[illegible]

Dry													
...al Elongation at Break IEB (%)				Initial Modulus at per cent Elongation (MN/m²)									
				100% (IM 100)					300% (IM 300)				
420				3.7					11.2				
% IEB				% IM 100					% IM 300				
Ageing Temp (°C)				Ageing Temp (°C)					Ageing Temp (°C)				
90	100	120	150	70	90	100	120	150	70	90	100	120	150
	90		12	88		75			78		80		
	82			85		86			82		90		
	81			87		92			82		101		
	70	41	8	97		95	285		86		108		
76			14	79	80				93	126			
		12											
72	72		2	111	93	99			118	102	138		
66	45				143	212							
52	36				173	350							
		9		152									
				258									

				RELATIVE HUMIDITY 100%											
	% IM 300			% ITS			% IEB			% IM 100			% IM 300		
C	70°C	80°C	90°C	70°C	80°C	90°C	70°C	80°C	90°C	70°C	80°C	90°C	70°C	80°C	90°C
8	77	79	74												
5	70	72	76												
0	75	81	84												
0	83	89	85												
					113	97		77	72		72	67		114	119
	151	123		96			82			79			103		
5						93			65			95			
				84	104	74	71	72	52	76	93	91	106	132	
					97	61		66	24		127	172			
				96	[illegible]		69	26		116	140				
				104			35			289					

TABLE 8

ACCELERATED AGEING OF ETHYLENE/PROPYLENE/DIENE RUBBER

Period of Test	Dry: Initial Tensile Strength ITS (MN/m²)			Initial Elongation at Break IEB (%)			Initial Modulus at per cent Elongation (MN/m²): 100% (IM 100)			300% (IM 300)			Wet (Imme: ITS (MN/m²)		IEB (%)	
	22.0			400			2.4			13.7			22.0		400	
	% ITS			% IEB			% IM 100			% IM 300			% ITS		% IEB	
	Ageing Temp (°C)			Ageing Temp (°C)			Ageing Temp (°C)			Ageing Temp (°C)			Ageing			
	70	100	150	70	100	150	70	100	150	70	100	150	70	90	70	90
½ day			87			82			106			123				
1 day		101	72		84	68		113	110		137			121		96
2 days		102	66		84	55		126	138		140		112	120	68	94
4 days		98	53		72	41		144	205							88
6 days			71			57										
7 days	103	107	49	84	71	30	123	180	330	138			120	101	95	80
10 days			53			45			216							
14 days	106	80	54	81	57	45	112	215	210	150			103	119	92	[illegible]
28 days	88	65	37	72	45	25	137	270	334				107	109	84	[illegible]
8 weeks	95	56	27	76	36	21	149	342					95	94	75	[illegible]
16 weeks	88	47		66	23		181						98	97	[illegible]	[illegible]
32 weeks	90	39		58	14		222						97		70	
75 weeks	86						241						89		69	[illegible]

T ETH/PROPYLENE/DIENE RUBBER

	Dry									Wet (Immersed in Water)							
e	Initial Elongation at Break IEB (%)			Initial Modulus at per cent Elongation (MN/m²): 100% (IM 100)			300% (IM 300)			ITS (MN/m²)		IEB (%)		IM 100 (MN/m²)		IM 300 (MN/m²)	
	400			2.4			13.7			22.0		400		2.4		13.7	
	% IEB			% IM 100			% IM 300			% ITS		% IEB		% IM 100		% IM 300	
)	Ageing Temp (°C)			Ageing Temp (°C)			Ageing Temp (°C)			Ageing Temp (°C)							
0	70	100	150	70	100	150	70	100	150	70	90	70	90	70	90	70	90
7			82			106			123								
2		84	68		113	110		137			121		96		114		122
6		84	55		126	138		140		112	120	88	94	138	132	132	126
3		72	41		144	205							88		131		139
1			57														
9	84	71	30	123	180	330	138			120	101	95	80	114	135	128	137
3			45			216											
4	81	57	45	112	216	210	150			103	119	82	89	136	130	141	148
7	72	45	25	137	270	334				107	109	84	83	136	141	142	159
7	76	36	21	149	342					95	84	75	65	156	147	149	
	65	23		181						98	97	74	64	164	202		
	58	14		222						97		70		178			
				241						89		69		188			

TABLE 9

ACCELERATED AGEING OF VITON B

Period of Test	Dry												Wet (i...		
	Initial Tensile Strength ITS (MN/m^2)			Initial Elongation at Break IEB (%)			Initial Modulus at per cent Elongation (MN/m^2) 100% (IM 100)			Initial Modulus at per cent Elongation (MN/m^2) 300% (IM 300)			ITS (MN/m^2)		IEB (%)
	16.4			340			3.3			15.3			16.4		340
	% ITS			% IEB			% IM 100			% IM 300			% ITS		% IE...
	Ageing Temp °C			Ageing Temp °C			Ageing Temp °C			Ageing Temp °C			Age...		
	70	100	150	70	100	150	70	100	150	70	100	150	70	90	70
1 day		107	107		95	86		106	125		108		95	88	130
2 days		107	106		95	86		100	120		99		86	83	119
4 days		105	103		85	75		122	122				74	77	122
6 days		111	108		91	60		110	114		114				
7 days	101	103	99	102	85	80	112	124	118	109			77	77	130
10 days			92			86									
14 days	106	102	91	104	91	82	111	135	150	111	109		73	76	134
18 days														75	
28 days	113		97	97		91	110			113			75	72	[illegible]
8 weeks	100	102	98	62	84	81	126	124	147				71	65	154
16 weeks													67	46	147
32 weeks	97	99	106	90	82	86	139	154	167	102			42		128
75 weeks	103			92			139			103			25		67

: B

	Dry									Wet (Immersed in Water)							
	Initial Elongation at Break IEB (%)			Initial Modulus at per cent Elongation (MN/m²)						ITS (MN/m²)		IEB (%)		IM 100 (MN/m²)		IM 300 (MN/m²)	
				100% (IM 100)			300% (IM 300)										
	340			3.3			15.3			16.4		340		3.3		15.3	
	% IEB			% IM 100			% IM 300			% ITS		% IEB		% IM 100		% IM 300	
	Ageing Temp °C			Ageing Temp °C			Ageing Temp °C			Ageing Temp °C							
0	70	100	150	70	100	150	70	100	150	70	90	70	90	70	90	70	90
7		95	86		106	125		108		95	88	130	122	91	109	85	83
6		95	86		100	120		99		86	83	118	140	99	98	86	78
3		85	75		122	122				74	77	122	132	100	102	75	76
8		91	60		110	114		114									
9	102	85	80	112	124	118	109			77	77	130	122	104	112	76	77
2			86														
	104	91	82	111	135	150	111	109		73	76	134	132	107	112	68	71
											75						
7	97		81	110			113			75	72	144	158	97	92	61	58
2	62	94	81	126	124	147				71	65	154	152	88	83	49	43
										67	46	147	126	76	81	37	39
	90	92	86	139	154	167	102			42		128		70		30	
	92			139			103			25		87		49			

TABLE 10

ACCELERATED AGEING OF NATURAL RUBBER

Period of Test	Dry												Wet (		
	Initial Tensile Strength ITS (MN/m²)			Initial Elongation at Break IEB (%)			Initial Modulus at per cent Elongation (MN/m²)						ITS (MN/m²)		IEB, (%)
							100% (IM 100)			300% (IM 300)					
	21.7			550			1.5			9.0			21.7		550
	% ITS			% IEB			% IM 100			% IM 300			% ITS		% IE
	Ageing Temp (°C)			Ageing Temp (°C)			Ageing Temp (°C)			Ageing Temp (°C)			Age		
	70	100	150	70	100	150	70	100	150	70	100	150	70	90	70
6 hours		107	81		99	71		122	164		110	140			
12 hours		109	49		98	50		130	162		121				
1 day	103	112	35	101	93	45		154	145		136		109	108	103
2 days		112	10		68	18		209	164		178				
4 days	104	104	10	96	68	3		195			185				
7 days	107	103	8	94		4		236			190		114	96	94
10 days		12			19										
13 days		14			8										
14 days	108	22		93	2		111			114			99	98	102
18 days														83	
28 days	118			93			119			124			98	73	71
6 weeks	119			86			154			141					
8 weeks							155			142			83		55
16 weeks				73			242			174			52		45
32 weeks													20		30

					Wet (Immersed in Water)							
Initial Modulus rer cent Elongation (MN/m²)					ITS (MN/m²)		IEB (%)		IM 100 (MN/m²)		IM 300 (MN/m²)	
100)		300% (IM 300)										
		9.0			21.7		550		1.5		9.0	
100		% IM 300			% ITS		% IEB		% IM 100		% IM 300	
mp (°C)		Ageing Temp (°C)			Ageing Temp (°C)							
0	150	70	100	150	70	90	70	90	70	90	70	90
2	164		110	140								
0	162		121									
4	145		136		109	108	103	99	93	119	108	109
9	164		178									
5			185									
5			190		114	98	94	83	138	132	130	127
		114			99	98	102	78		85		113
						83		75	85	79	112	113
		124			98	73	71	66	205	76	175	102
		141										
		142			83		55		210		110	
		174			52		45		184			
					20		30		130			

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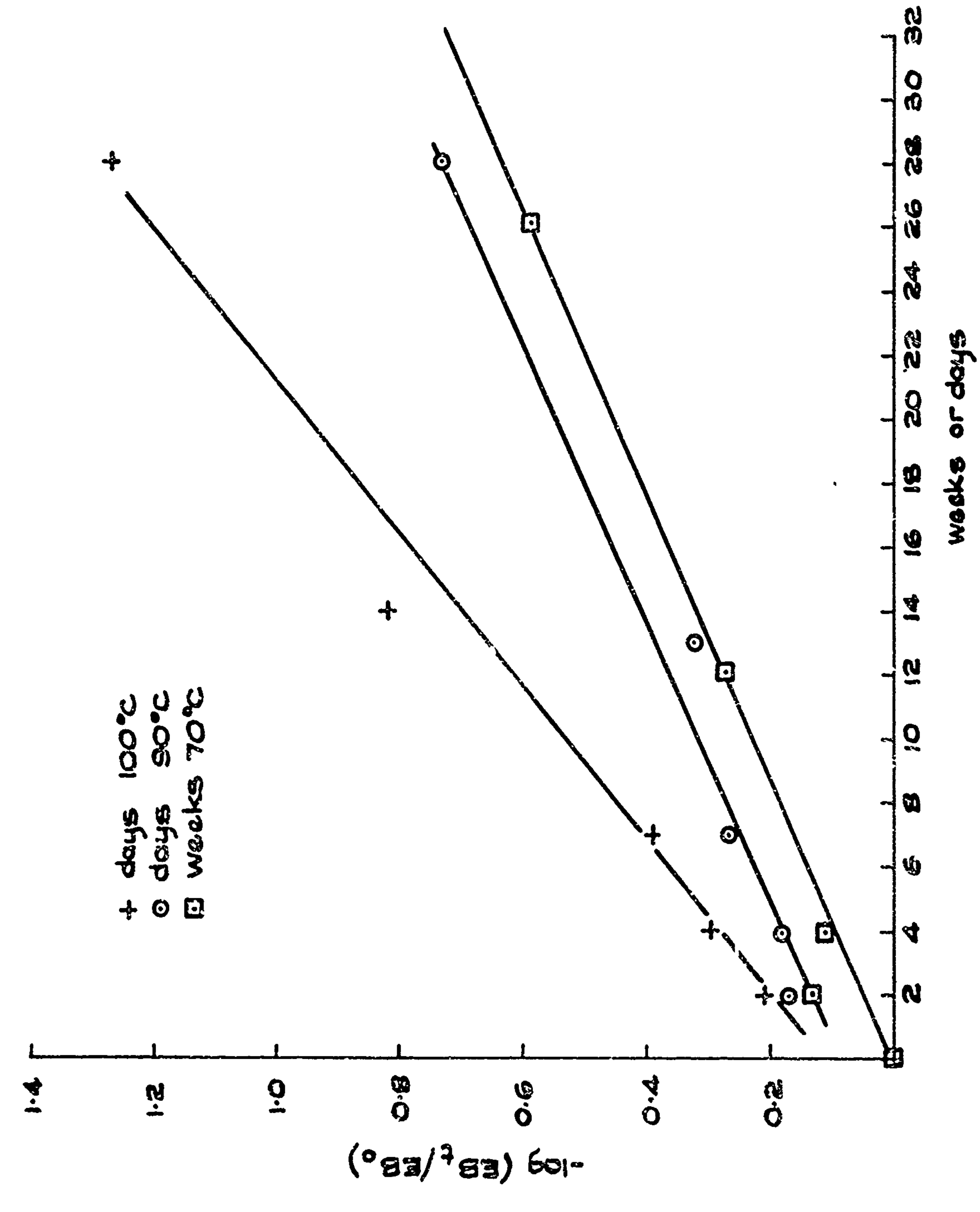
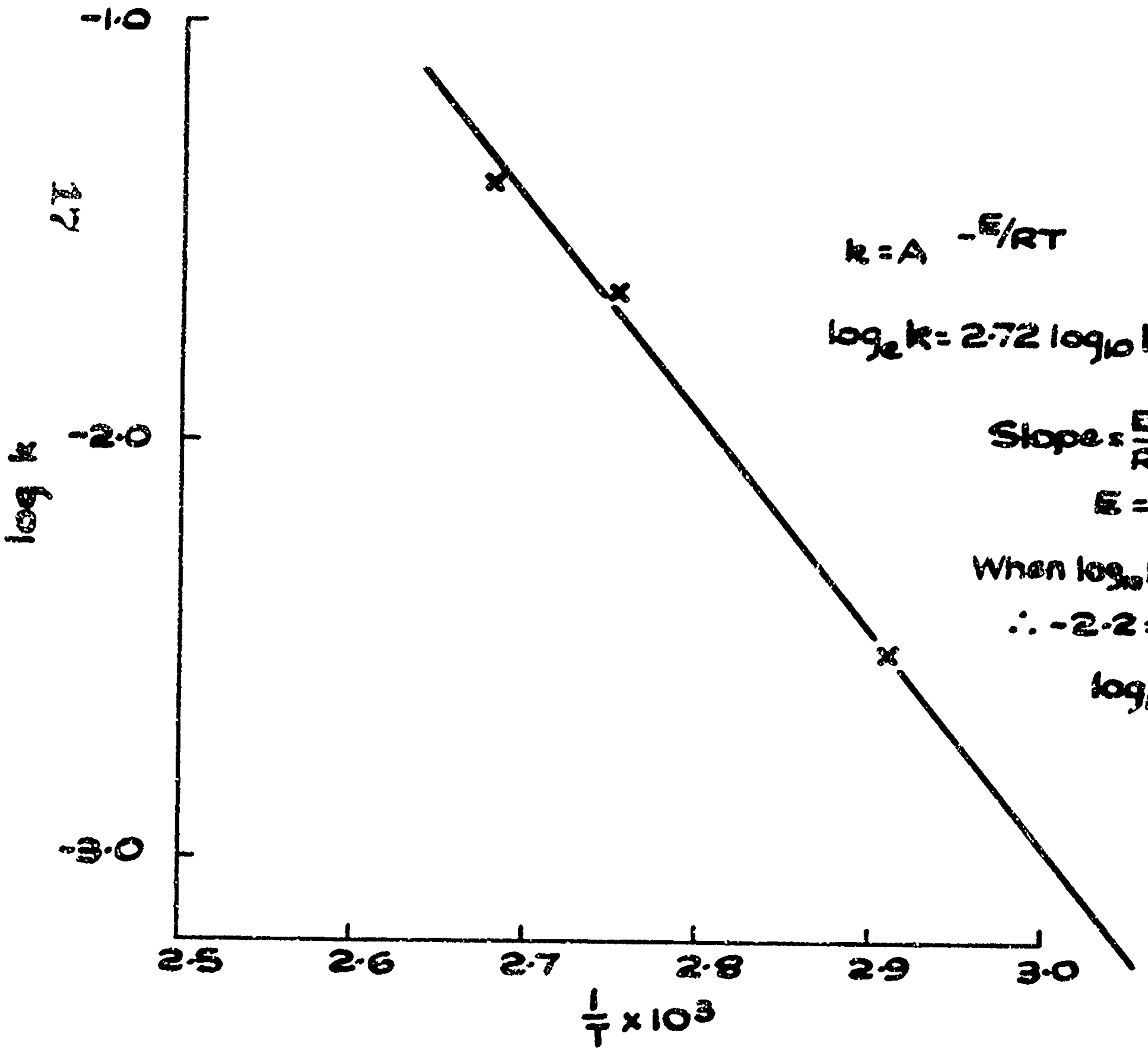


FIG. 1 NEOPRENE WRT CHANGE OF ELONGATION WITH TIME OF AGEING.



$k = A^{-E/RT}$

$\log_e k = 2.72 \log_{10} k = 2.72 [\log_{10} A] - E/RT$

$\text{Slope} = \frac{E}{R} = 5.0 \times 2.72 \times 10^3$

$E = 5.0 \times 2.72 \times 2 \times 10^3 = 27.2 \text{ kcal/mole}$

When $\log_{10} k = 2.2 \quad \frac{1}{T} = 2.85 \times 10^{-3}$

$\therefore -2.2 = \log_{10} A - 2.85 \times 10^{-3} \times 5 \times 10^3$

$\log_{10} A = 12$

$A = 1 \times 10^{12}$

FIG. 2 NEOPRENE WRT. LOG k (RATE CONSTANT) VS $\frac{1}{T}$.

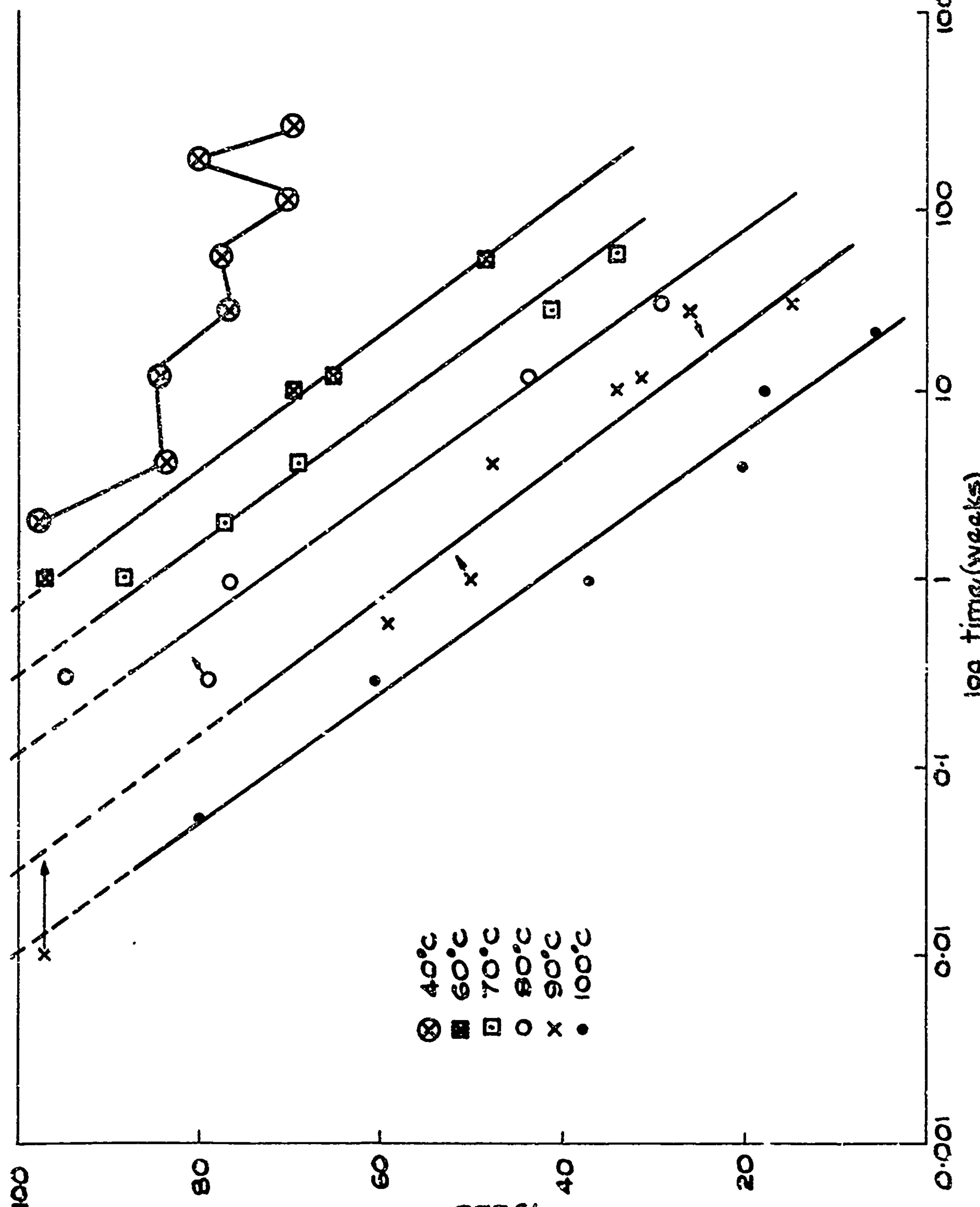


FIG. 3 NITRILE RUBBER DRY. EB VS LOG TIME.

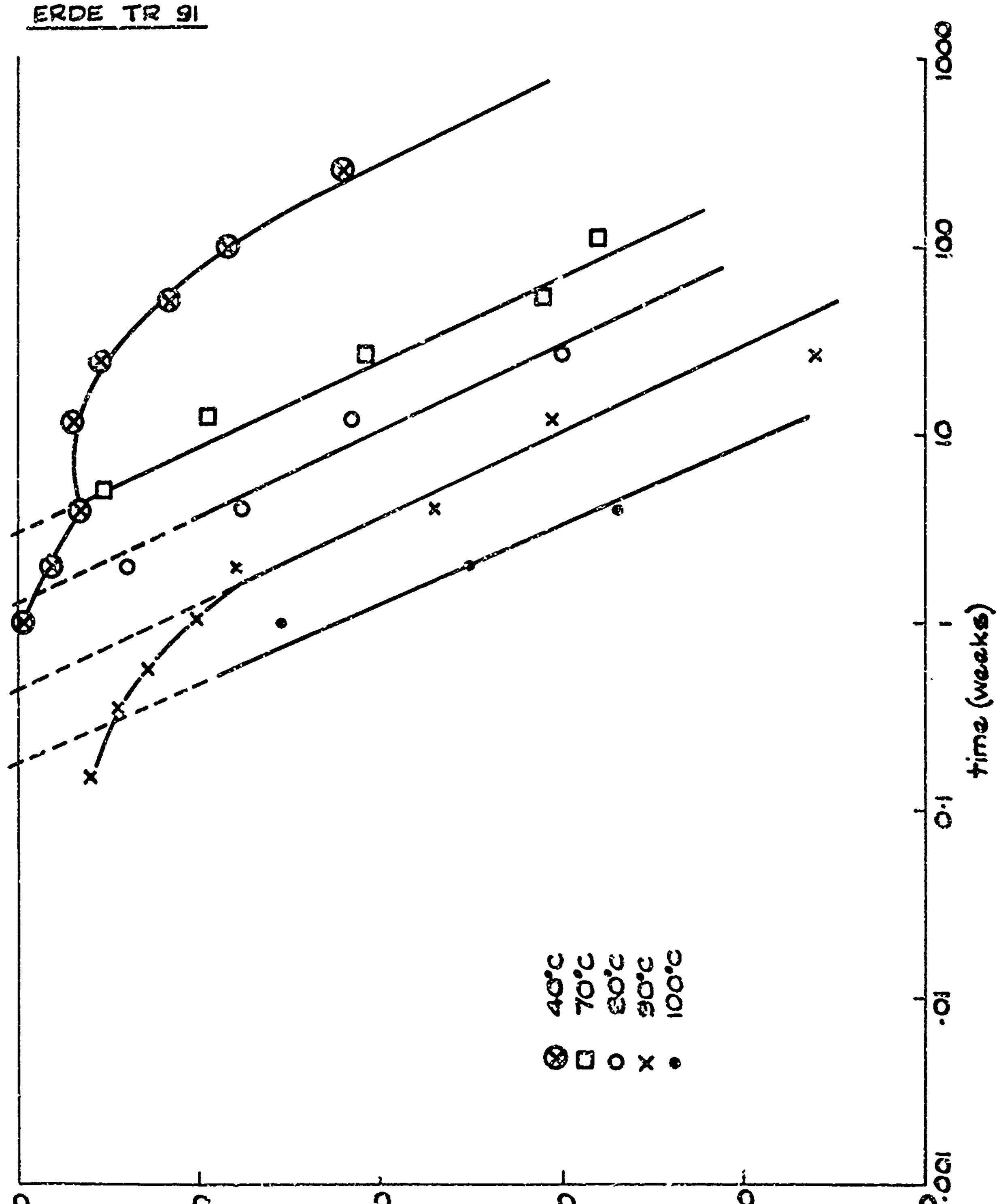


FIG. 4 NEOPRENE DRY. EB VS LOG TIME

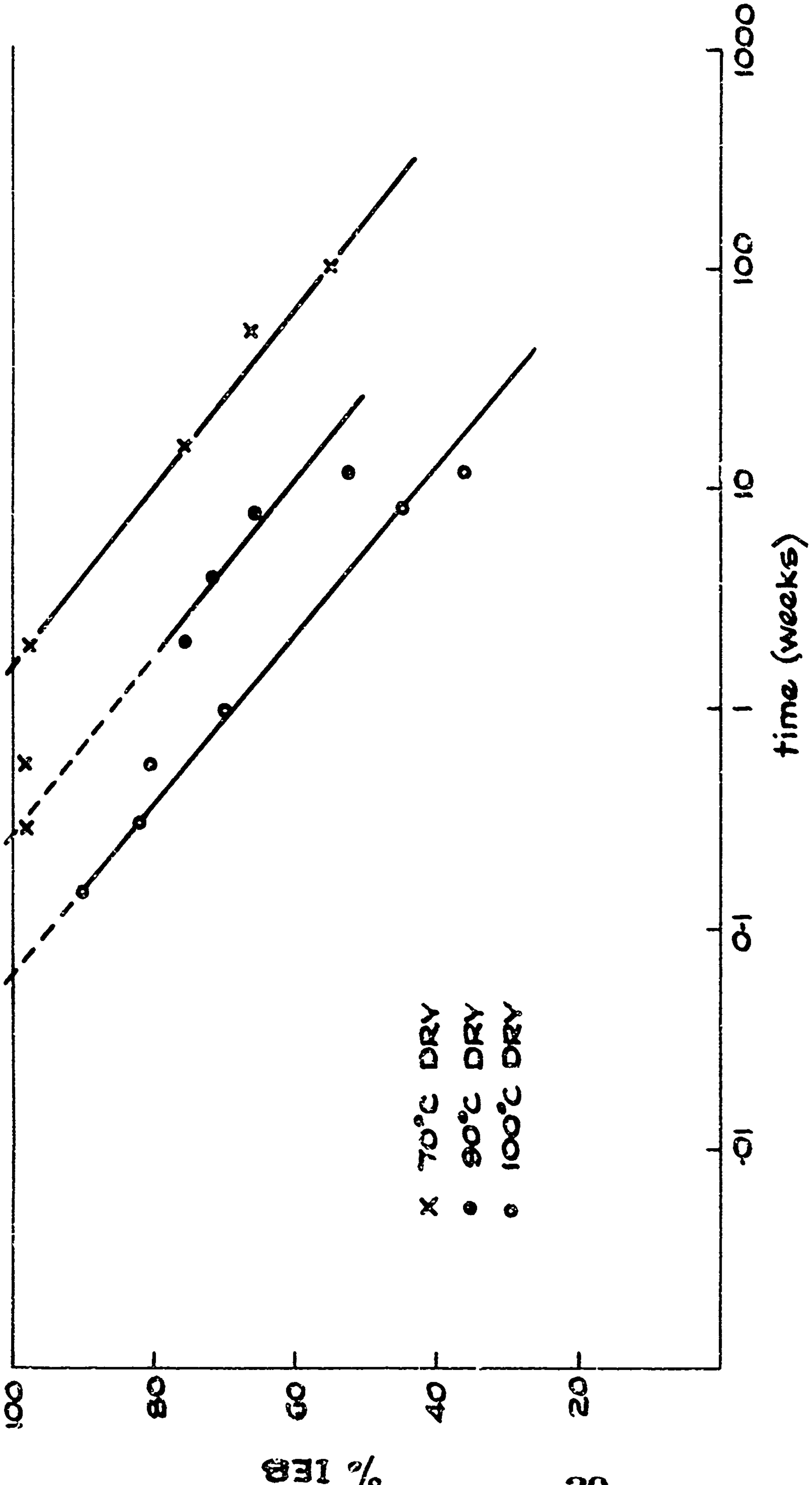


FIG. 5 PVC/NITRILE (DRY). EB VS LOG TIME.

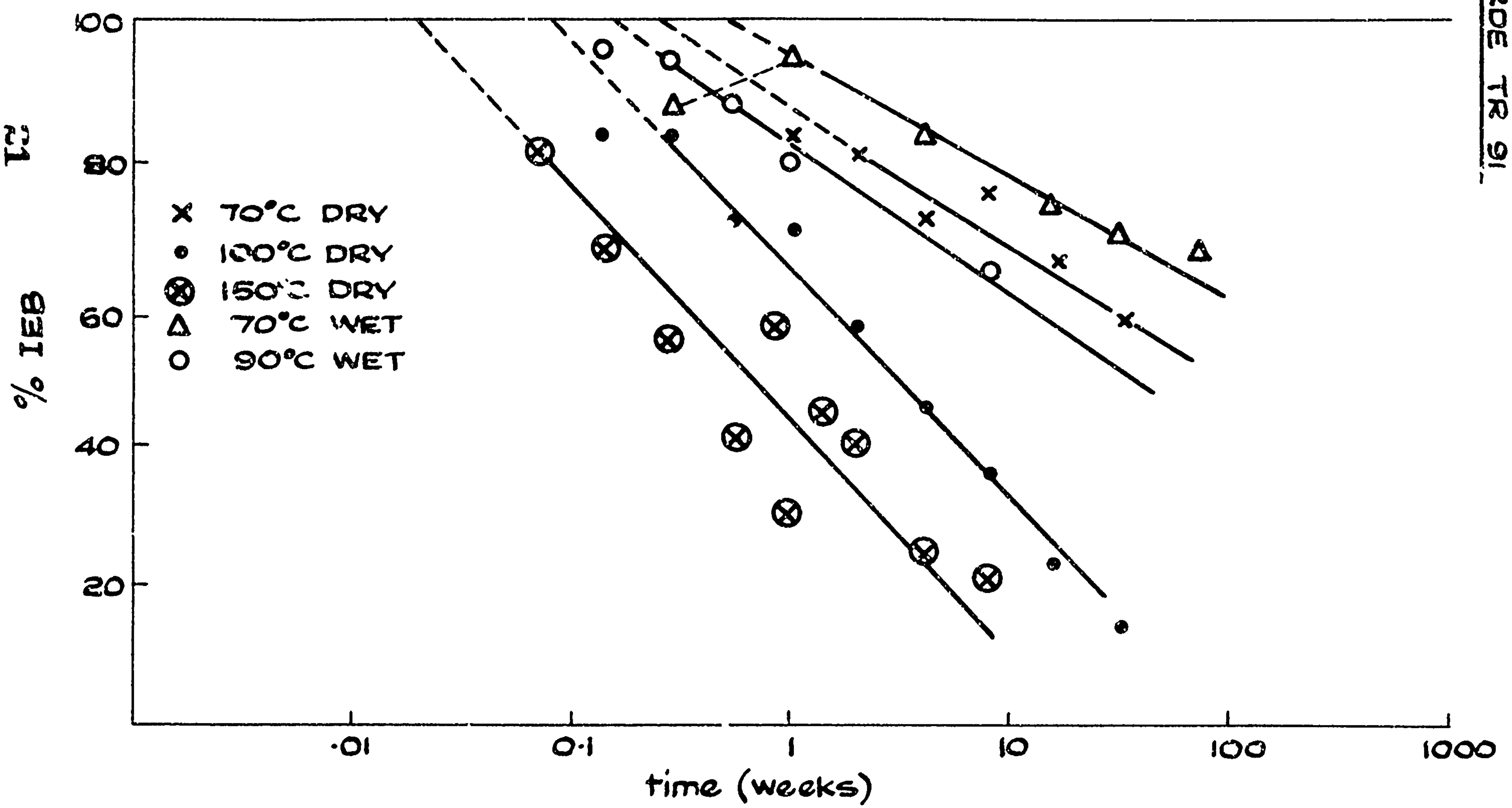


FIG. 6 EPDM (WET AND DRY). EB VS LOG TIME.

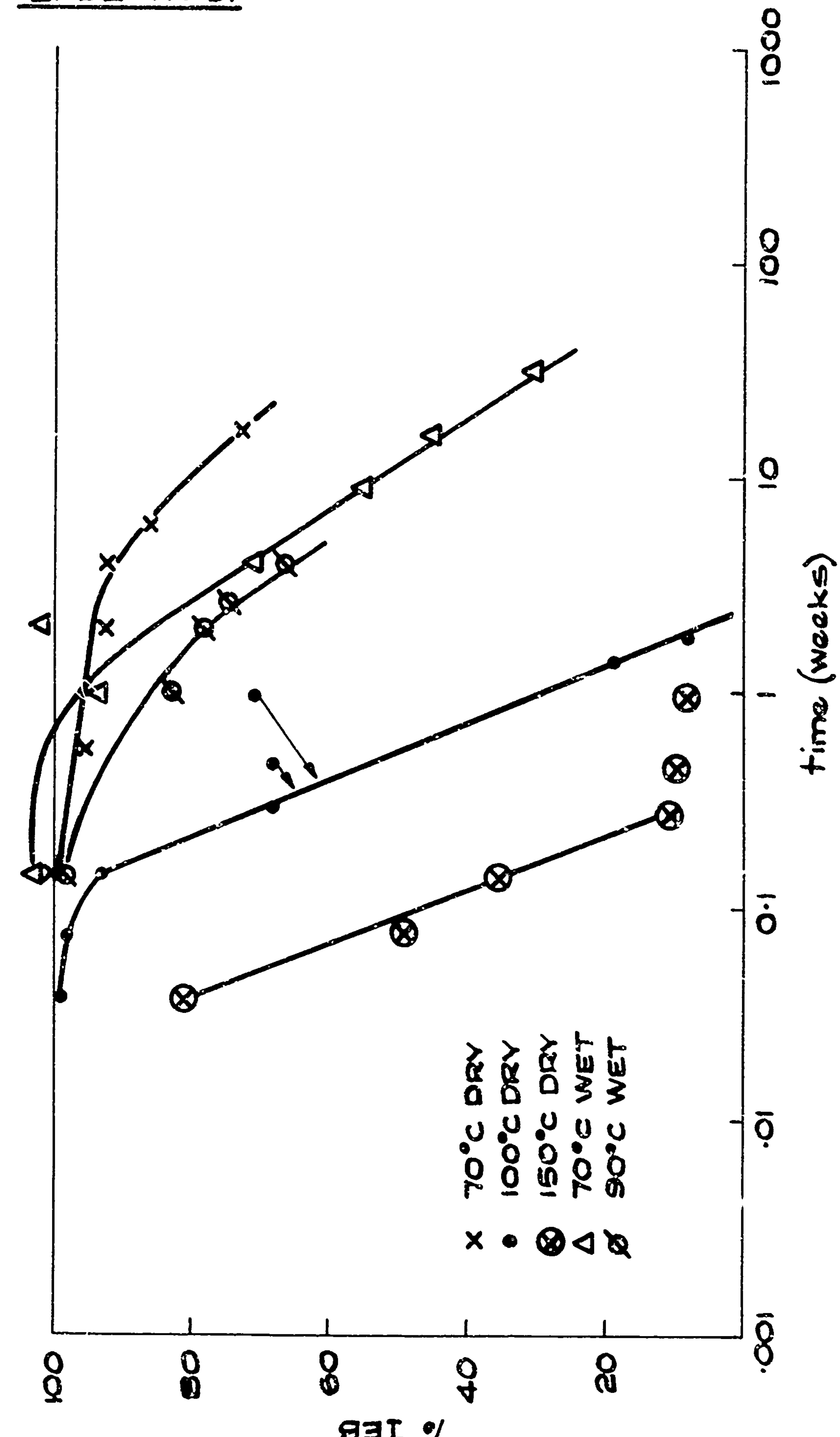


FIG. 7 NATURAL RUBBER. EB VS LOG TIME

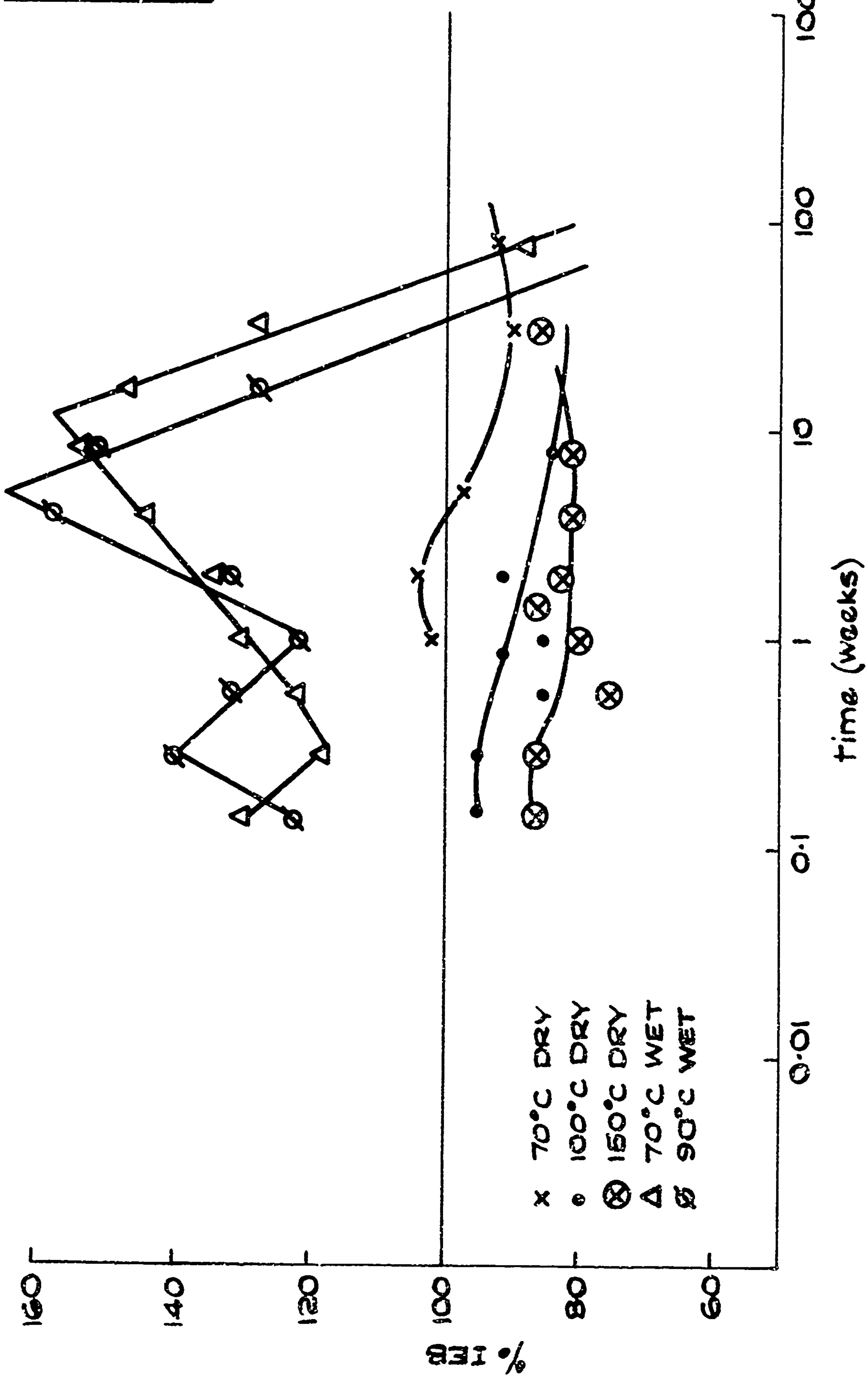


FIG. 8 VITON (DRY AND WET). EB VS LOG TIME.

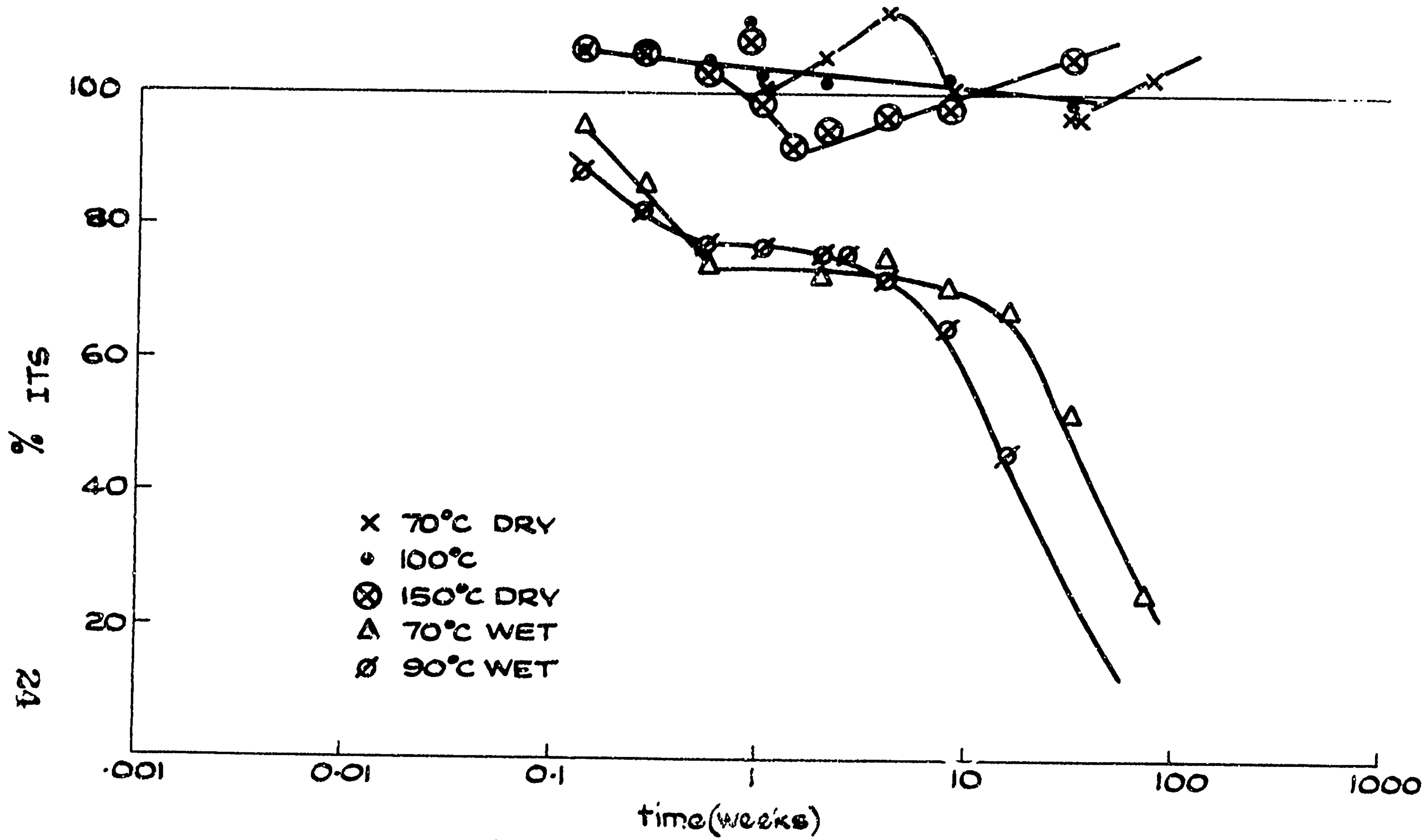


FIG. 9 VITON (DRY AND WET). TENSILE STRENGTH VS LOG TIME.