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STORAGE LIFE OF FULLY COMPOUNDED RUBBER STOCKS. PART 2: 12 MONTHS' STORAGE TESTING. PART 3: SUBSEQUENT AGEING BEHAVIOUR

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Explosives Research and Development Establishment

Prepared for:

Defence Research Information Centre

April 1973

UNLIMITED

ERDE TR 92

Procurement Executive Ministry of Defence

Explosives Research and Development Establishment – Rocket Propulsion Establishment

Explosives Research and Development Establishment

Technical Report No. 92

Storage Life of Fully Compounded Rubber Stocks

Part 2. 12 Months' Storage Testing

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DOCUMENT CONTROL SHEET

(Notes on completion overleaf)

Overall security classification of sheet ....

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1. DRIC Reference (if known)	2. Originator's Reference	3. Agency Reference	4. Report Security Classification
	ERDE TR 92		Unlimited

5. Originator's Code (if known)	6. Originator (Corporate Author) Name and Location
445050	Explosives Research and Development Establishment Waltham Abbey Essex England
5a. Sponsoring Agency's Code (if known)	6a. Sponsoring Agency (Contract Authority) Name and Location

7. Title STORAGE LIFE OF FULLY COMPOUNDED RUBBER STOCKS
PART 2: 12 MONTHS' STORAGE TESTING
PART 3: SUBSEQUENT AGEING BEHAVIOUR

7a. Title in Foreign Language (in the case of translations)

7b. Presented at (for conference papers). Title, place and date of conference

8. Author 1, Surname, initials	9a Author 2	9b Authors 3, 4...	10. Date	pp	ref
Ledbury K J	Richards R W	Stokoe A L	4.1973	54	1[illegible]

11. Contract Number	12. Period	13. Project	14. Other References

15. Distribution statement

15. Descriptors (or keywords)

Storage, Ageing test (materials), Elastomers, Compression molding, Injection molding, Curing

(TEST)

continue on separate piece of paper if necessary

Abstract Part 2. Processing and cure characteristics have been determined for ten fully compounded rubber stocks representative of natural and rubber mixes stored for periods up to twelve months.

Part 3. Ten fully compounded rubber stocks, five of natural and five of synthetic rubber, have been stored at room temperature and 40°C for various times and vulcanisates prepared from them by compression and injection moulding have been subsequently aged at 80°C for up to 16 weeks. The ageing characteristics of the vulcanisates have been investigated.

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Technical Report No 92

April 1975

Storage Life of Fully Compounded Rubber Stocks

Part 2
12 Months' Storage Testing

Part 3
Subsequent Ageing Behaviour

by

K J Ledbury
R W Richards
A L Stokoe

SUMMARY

Part 2 Ten fully compounded rubber stocks representative of natural and synthetic rubber mixes have been stored for periods up to twelve months and their processing and cure characteristics determined. Results show that after storage at room temperature, satisfactory mouldings may be produced from most of the stored stocks, both by injection and compression moulding, having similar properties to those produced from unstored materials.

Part 3 Ten fully compounded stocks, five of natural and five of synthetic rubber, have been stored at room temperature and 40°C for various times and vulcanisates prepared from them by compression and injection moulding have been subsequently aged at 80°C for up to 16 weeks. The general ageing characterictics of the vulcanisates prepared from the stocks are not altered to any significant degree by storage, provided that each stock is given its optimum cure.

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PART 2

12 MONTHS' STORAGE TESTING

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Reference: WAC/213/034

1 INTRODUCTION

It has been shown[1] that fully compounded natural rubber stocks can be stored at room temperature for up to 6 months and then injection moulded or compression moulded to give vulcanizates with good physical properties. It was suggested that these fully compounded stocks could be granulated and bagged ready for use as are thermoplastics. Already certain rubber compositions are being marketed in this form, for example the silicone rubbers, Viton E60C (fluorinated rubber), together with other ready to use compounds such as the thermolastics, polyurethanes,[2] powdered natural[3] and nitrile rubbers.[4] Other products on similar lines are the specially compounded master batches of carbon black, sulphur or accelerator and also master batches of carbon black and oil prepared from the latex emulsions before coagulation.

There is very little published knowledge available[5,6,7] on the life of fully compounded rubbers and the properties of the products produced. Therefore it was decided to investigate changes which take place during storage using a natural rubber composition containing a conventional sulphur/accelerator curing system (high sulphur/low accelerator) with and without carbon black as a filler, an efficient vulcanizing system (low sulphur/high accelerator) and one essentially sulphurless curing system. Other elastomers which were included in the investigation were one formulation only of nitrile, polychloroprene, SBR, EPDM and butyl rubbers.

2 EXPERIMENTAL

Five natural, one nitrile, one polychloroprene, one SBR, one EPDM and one butyl rubber compositions were compounded in a 5 kg internal Banbury mixer omitting the sulphur. The batch was then sheeted out and allowed to cool before mixing in the sulphur in the usual manner on a mill (200 mm × 400 mm). 2 batches of each rubber composition were cut up and blended on the mill and then sheeted out to approximately 6 mm thick. The compounding was carried out at NRPRA because of their facilities for making larger batches than normally made in the laboratory. The materials were subsequently granulated into 6 × 6 × 6 mm pieces by a commercial firm. Due to the preparation circumstances the initial moulding and testing of all stocks were delayed for approximately 2 weeks after mixing. The compositions of the rubber compounds and other preparation details are given in Appendix A.

The granulated rubbers were received in polyethylene bags and these were stored in a test room, the temperature of which was $23^{\circ} \pm 2^{\circ}C$ with a relative humidity of $65^{\circ} \pm 5^{\circ}C$. For accelerated or warm storage, the granules of each rubber mix were spread out in trays approximately 300 × 250 × 30 mm to a depth of 10 mm and each formulation placed in a separate air circulating oven at $40^{\circ} \pm 2^{\circ}C$. In each instance, the mixes were withdrawn at prescribed intervals and tested as described below.

The Wallace/Shawbury curometer, the Mooney viscometer and the Brabender plastograph were used to measure plasticity, scorch or cure times at 120, 150 or 180°C. In addition the Monsanto rheometer was used to make similar measurements at 150 and 180°C. This instrument measures the torque acting on a disc oscillating at 1.7 Hz which is embedded in the rubber compound set in a cavity mould. A record of the change in modulus with time during vulcanization is obtained and from the graph, values of viscosity, processability or scorch time, cure rate, cure time as well as maximum modulus can be estimated. The tendency for reversion can also be noted.

The gel content of the rubber mixes was estimated by extracting a weighed sample with benzene and determining the extracted material. In most instances small amounts of carbon black passed through the filter thimble into the extract. The carbon black was estimated by the nitric acid method and the weight substracted from the extract. The method proposed by Gessler[8] was also tried but due to the extraction of carbon black this method proved unsatisfactory.

Compression moulding was carried out in ASTM moulds 150 x 150 x 3 mm or a mould 100 x 75 x 3 mm depending on the requirements and vulcanized at 150°C for the stated times, using stock which had been passed 5 times through the tight nip of a 6-inch diameter laboratory mill. This was necessary since the granulated material would not coalesce into a homogeneous sheet due to the talc coating applied during granulation.

Injection moulding was carried out on a 4 oz Ankerwerke screw injection moulding machine using the granulated materials without further treatement under the conditions described in Appendix B. The rubber was moulded into 2 plaques, 100 x 75 x 3 mm, using a flash gate.

British Standard type E dumb-bells were cut from the compression and injection moulded test sheets and the physical properties determined using a Hounsfield E-type testing machine.

3 RESULTS AND DISCUSSION

3 1 Visual Examination

The granules were initially liberally coated with talc which prevented them from sticking together. However this obscured any blooming of sulphur, antioxidant, etc which otherwise might have been noted. After approximately 6 months' storage the granules did appear to stick together but in no case were they difficult to separate. After a few weeks at room temperature or a few days at 40°C most of the granules appeared to be stiffer or harder but it was still possible to carry out all the tests using the methods described. No further change appeared to take place except with the neoprene which became fairly hard and had an elastic feel after 12 months' storage at room temperature. At 40°C storage 3 sets of granules felt fairly hard and appeared cured, the polychloroprene after 4 weeks, nitrile after 8 weeks and the natural rubber without carbon black after 16 weeks.

3.2 Scorch Properties

Scorch values measured by the various instruments are summarised in Figure 1 and Tables 1 - 4.

The scorch times obtained on the stored stocks show that mix A, a conventional natural rubber/sulphur mix changes very little on storage whereas mix B, an unfilled gumstock, shows a rapid decrease in scorch time. Mix C barely changes up to 6 months but then shows a rapid fall in scorch time. Mix D had a low initial scorch time of 7 minutes but this only decreased to 5½ minutes after one year. Mix E appeared to show an initial increase in scorch time followed by a slight decrease but after 12 months the value was still higher than the original.

Nitrile and polychloroprene mixes F and G show a marked decrease in scorch times. The polychloroprene mix started off with a low scorch time and after 12 months was not processable.

Mixes H, I and J (SBR, EPDM and butyl) showed little change in scorch time.

At 40°C changes in scorch times were accelerated. No results could be obtained on polychloroprene at 8 weeks, nitrile at 16 weeks, natural rubber gumstock and TMTD cured NR after 24 weeks. Most other mixes showed a tendency to become more scorchy. The Shawbury curometer can also be used to estimate scorch times. A value of 0.9 a_o, where a_o is the initial separation of the cure curve, can be taken as a value similar to the Mooney 5 point rise.

Results obtained using the curometer at 120°C show good agreement with those from the Mooney.

Scorch times estimated from the Brabender curves are lower than those from the curometer or Mooney. However this is almost certainly due to heat build up within the relatively larger sample of rubber under test and the higher rates of shear involved. Values of scorch time estimated from Monsanto Rheometer traces at 120°C for the unstored stocks are in good agreement with the Mooney scorch values with the exception of mix J. However after storage the scorch times from the rheometer traces are considerably different at times for mixes B, E and J to the values obtained with the Mooney viscometer. No explanation can be offered for this difference at present.

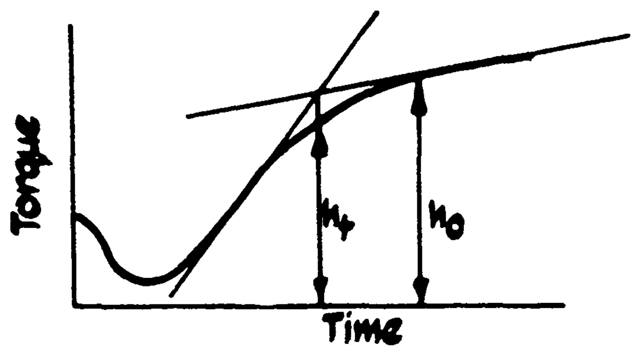
3.3 Viscosity

The Mooney plasticities and the Brabender viscosities both increase progressively and in approximately parallel lines for the natural rubber mixes over the storage period of 12 months. (Tables 2 and 3).

The nitrile rubber (F) shows only a slight change but the polychloroprene mix (G) had increased very rapidly in viscosity over the 12 months' storage period. Both instruments (Mooney and Brabender) showed this change. For mixes H (SBR), I (EPDM) and J (Butyl) the Brabender viscosities are almost unchanged after 12 months' storage. The Monsanto minimum torque values (Table 4) show similar changes to those observed with the Mooney. The natural rubber mixes appear to show a more rapid and larger increase in viscosity as measured by this instrument than the Mooney, the viscosity then staying constant after 6 months. Polychloroprene again shows a larger increase in viscosity whereas nitrile, although showing a rapid loss of scorch time in the 12 month period shows little change in viscosity. Mixes H, I and J again show little change.

3.4 Cure Times

The normal method of calculating optimum cure time from the Shawbury curometer is rather laborious; however it was found that the direct visual reading of the time to reach the narrowest point of the cure curve was almost as accurate (± 3 per cent) as the recommended method. This simpler visual method was therefore adopted. The Monsanto cure times were taken as the time where the tangent to the steepest part of the curing curve intersected that of the flattest part of the curve (see Fig 2).



The ratio of h_t/h_o was found to be close to 80 per cent cure for all the rubber mixes tested. The average of 26 results was 81 with a maximum reading of 88 and a minimum of 68. This method was adopted because most of the synthetic rubbers had not reached a true cure plateau after 50 to 60 minutes' curing at 150°C.

The optimum cure times at 150°C plotted in Fig 2 show that the cure times from both instruments correlate fairly well although the Shawbury results are more erratic. After storage at room temperature only one rubber stock (J,butyl) had a significantly higher optimum cure time at 150°C than the original material. The cure time (at 150°C) for maximum tensile strength also does not appear to change with period of storage. These results are confirmed by the tensile properties at various cure times of compression moulded sheets produced at 150°C (Table 5). On the other hand after storage at 40°C, 7 of the 10 rubbers had increased cure times after 16 weeks' storage.

Instrumental measurements of cure times at 180°C show only small decreases or little change to have occurred in the actual times after storage at either room temperature or 40°C (Fig 2). However measurements of maximum tensile strength versus cure time on the injection moulding machine show a different pattern of behaviour (Fig 3). Initially the natural rubber mixes A - D show rather sharp peaky curves. After storage the rubber stocks give a wider cure curve and the optimum cure time moves to much longer times. Nitrile rubber shows little change in peak position, polychloroprene cannot be moulded after 12 months' storage at room temperature but the 6 months result suggests an increase in optimum cure time. In SBR an optimum cure time defined as the position of maximum tensile strength is not reached but the results show a movement of the cure curve to longer times. With EPDM the optimum time appears to be very much shorter than that suggested by either the Monsanto or the cureometer. The results for butyl agree better with the instrumental results and show little change. Very little change in the shape of the cure curve occurs with the synthetic rubber stored stocks.

3 5 Tensile Properties

The materials stored for 12 months and compression moulded at 150°C (Table 5) show little change in ultimate tensile strength (UTS), elongation at break (E_b), 300 per cent modulus (M_{300}) or hardness at the optimum cure with the exception of the polychloroprene which shows a reduction of about 25 per cent in UTS and E_b. However, 7 of the rubbers show a marked increase in M_{100}; these include 3 natural rubbers and all the synthetics with the exception of SBR which shows a significant reduction in M_{100} value (Table 5). With the injection moulded stored stocks some differences become apparent depending on how the optimum cure is defined (Fig 3, Table 6). If this is taken as the point of highest tensile strength then the natural rubber mixes A - C show a slight fall (10 per cent) in tensile strength after storage at room temperature accompanied by a fall in M_{100} and M_{300}. A more rapid fall occurs with these mixes after storage at 40°C. Mixes D - J show little significant change in optimum tensile strength but all show some decrease in M_{100} and/or M_{300} with the exception of polychloroprene which shows no change.

After storage at 40°C, the nitrile and polychloroprene rubbers could not be moulded, mix D showed a drastic fall in UTS, the remainder remaining almost unchanged.

In general, although the time of optimum cure changes, the tensile properties at optimum cure are not greatly affected by storage at room temperature. The actual values of tensile strengths found on the injection moulded stocks were similar to the values obtained from the compression moulded sheets. This is in agreement with the findings of Wheelan[10] and Ruby.[11]

3 6 Sulphur Content

The "free" sulphur content determined by a cold extraction method is given in Table 7 and compared to the total initial sulphur content. In all cases a marked fall in "free" sulphur content occurred and this fall was linear with time for many of the stored stocks. These results indicate that sulphur has been lost or possibly the accelerator or accelerator fragments are becoming attached to the polymer chain and may no longer be available for the curing reaction. This could be one reason for the observed decreased rate of cure and increase optimum cure time at 180°C.

3 7 Benzene Extractables

The benzene extractables are given in Table 8. These figures indicate that in all the rubbers as mixed there is a small amount of non-extractable material (gel). In the black natural, nitrile and polychloroprene mixes the gel content increases significantly with time of storage whereas with SBR, EPDM and butyl the gel content remains unchanged. The unfilled natural rubber shows unusual behaviour with the benzene extract first decreasing rapidly over the first 6 months (ie increased gel), then increasing after 12 months back to the theoretical value. Storage at 40°C increases the rate of gel formation with the result that the benzene extractables are low (high gel content) for mixes B, D, F and G after 16 weeks' storage. Mixes H, I and J show little change in gel content after storage at 40°C.

4 CONCLUSIONS

The results show that many fully compounded rubber mixes can be stored at room temperature as chips for periods up to 12 months and subsequently processed by injection moulding at 180°C to give satisfactory mouldings with only small changes in properties. Remilling the stored stock for a minimum time and compression moulding at 150°C produces rubber samples having almost identical properties to the originals. Compression or injection moulding the same stock give samples with similar properties. Storage at 40°C produces much quicker changes in the stored materials and after 16 weeks nitrile and chloroprene rubbers cannot be processed.

Instrumental techniques have shown that certain changes have occurred in the stored stocks; these are mainly a reduction in scorch time and a change in cure rate although actual cure times at 150°C do not appear to have changed as much as those at 180°C. Sulphur determinations and benzene extracts also show that chemical changes have occurred in many of the stored stocks. Further work will be directed towards the assessment of the ageing behaviour of mouldings produced from stored rubber mixes.

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APPENDIX A

COMPOSITION OF NATURAL RUBBER MIXES

Mix No	A	B	C	D	E
Natural Rubber SMR5	100	100	100	100	100
Zinc Oxide	5	5	5	5	5
Stearic Acid	2	2	2	2	2
Phenyl β-Naphthylamine	1	1	1	1	1
Carbon Black FEF	25	-	25	25	25
Sulphur	2.5	2.5	0.33	-	2.5
Cyclohexylbenzthiazole sulphenamide (CBS)	0.5	0.5	5.0	-	-
Tetramethylthiuram disulphide (TMTD)	-	-	0.5	2.5	-
Morpholinothiobenzthiazole (MOR)	-	-	-	-	0.55
	136	111	138.8	135	136

COMPOSITION OF SYNTHETIC RUBBER MIXES

Mix No	F	G	H	I	J
Nitrile Rubber (Krynac 803)	100	-	-	-	-
Neoprene WRT	-	100	-	-	-
Butadiene/Styrene (SBR) Intol 1500	-	-	100	-	-
Ethylene/Propylene Diene (EPDM) Intolan 104A	-	-	-	100	-
Butyl Rubber (Butyl 301)	-	-	-	-	100
Zinc Oxide	5	5	5	5	5
Stearic Acid	2	0.5	2	1	2
Phenyl β-Naphthylamine (PBNA)	1	-	1	-	-
Carbon Black FEF	25	25	25	25	25
Sulphur	2	-	1.75	1.5	1.5
Cyclohexylbenzthiazyl sulphonamide (CBS)	2	-	1.25	-	-
Mercaptoimadazoline (NA 22)	-	0.5	-	-	-
Tetramethylthiuram disulphide (TMTD)	-	0.5	-	-	-
Magnesium Oxide	-	4	-	-	-
Zinc Diethyldithiocarbamate (ZDC)	-	-	-	2	2
Mercaptobenzthiazoledisulphide (MBTS)	-	-	-	0.5	0.5
Petroleum Jelly	-	-	-	-	5
	137	135.5	136	135	141

INJECTION MOULDING CONDITIONS

Barrel Temp (°C)	80 - 90
Mould Temp (°C)	180
Injection time (s)	1 - 2
Follow up pressure (MN/m^2)	3 - 5
Injection Speed	Max
Plasticisation time (s)	10 - 18
Back pressure	Nil
Screw speed (r/min)	37
Mould dwell time	Various according to cure time

TABLE 1

SHAWBURY CUROMETER

SCORCH TIMES AT 120°C
(min)

Rubber Mix	Before Storage	Storage at Room Temperature			Storage at 40°C				
		3 months	6 months	12 months	2 weeks	4 weeks	8 weeks	16 weeks	24 weeks
A (NR)	22	25	35	15	29	31	18	13	13
B (NR)	36	44	42	16	34	19	17	4	No reading obtainable
C (NR)	23	23	14	9	20	20	22	19	3
D (NR)	4	5	6	6	6	6	6	6	No reading obtainable
E (NR)	22	32	36	31	47	44	32	33	27
F (NBR)	21	21	24	5	21	12	2	No reading obtainable	
G (CR)	13	9	9	4	8	6	No reading obtainable		
H (SBR)	78	50	66	71	62	64	46	69	29
I (EPDM)	12	9	11	9	9	8	7	7	4
J (IIR)	21	18	20	14	19	16	18	17	11

TABLE 2

MOONEY PLASTICITY AND SCORCH TIME

Mix	Storage at Room Temperature							
	Mooney Plasticity				Mooney Scorch (min)			
	Period of Storage							
	0	3 months	6 months	12 months	0	3 months	6 months	12 months
A	38	22	45	45	23	23	19	18
B	27	30	36	42	48	27	22	16
C	28	37	35	43	22	24	15	8½
D	39	49	54	64	7	6	6½	5½
E	44	45	47	47	24	34½	32+	31
F	46	-	-	46	31	-	-	10
G	61	-	-	144	7	-	-	4
H	57	-	-	54	32+	-	-	32+
I	76	-	-	80	11	-	-	12
J	55	-	-	58	15	-	-	13

TABLE 3

BRABENDER PLASTOGRAPH VALUES

Mix	r/min	Temp of Circulating Oil (°C)	Original			12 months' Storage at Room Temp		
			Minimum Viscosity (m/g)	Temp at Min Visc (°C)	Scorch Time (min)	Minimum Viscosity (m/g)	Temp at Min Visc (°C)	Scorch Time (min)
A	60	100	1400	105	58	2100	110	19
	52	120	1500	120	16	2025	126	10
B	60	100	1200	108	125	1800	110	20
	52	120	1400	120	36	1850	122	9½
C	60	100	1100	102	38	1600	108	15
	52	120	1100	140	15	1700	148	4½
D	60	100	1630	107	9	2200	110	5½
	52	120	1000	146	4½	2300	148	3½
E	60	100	1700	110	30	2200	116	27
	52	120	1550	144	10½	1900	150	13
F	60	100	1900	108	40	2100	108	12
	52	120	1500	140	19	1750	140	8
G	60	100	2000	140	9½	2800	150	7 Cured
	52	120	2100	140	3½	3300	154	3 Cured
H	60	100	2000	138	75	2000	140	54
	52	120	1800	148	36	1600	148	35
I	60	100	2900	150	6½	3000	146	6½
	52	120	2600	146	6	2500	150	6
J	60	100	1750	136	13	1750	136	13
	52	120	1700	142	8	1400	140	8½

TABLE 3 Contd

BRABENDER PLASTOGRAPH

Mix	r/min	Temp of Circulating Oil (^oC)	Original		
			Minimum Viscosity (m/g)	Temp at Min Visc (^oC)	Scorch Time (min)
A	52	100	1500	108	55½
		120	1500	120	16
		130	1600	110	8
		150	1600	138	4
		180	1600	146	1½
A	52	100	1500	108	55½
	60	100	1400	105	57
	100	100	1600	126	10
	140	100	1800	140	3
B	60	100	1200	108	125
	100	100	1300	118	45
	140	100	1600	132	9

TABLE 4

MONSANTO RHEOMETER

Mix	Period of Storage Room Temp	Minimum Torque at 120°C	Scorch Time at 120°C (min)
A	2 weeks (0)	6	26
B	" "	5.5	46
C	" "	5.5	25
D	" "	9	11
E	" "	8	26.5
F	" "	7	36
G	" "	14	8.5
H	" "	14	60+
I	" "	18.5	13.5
J	" "	12.7	24
A	6 months	15	25.5
B	" "	13	25.5
C	" "	11	19.5
D	" "	-	-
E	" "	15.5	47.5
F	" "	10	28
G	" "	-	-
H	" "	14	60+
I	" "	19	10.5
J	" "	-	-
A	12 months	16	21
B	" "	11.5	16
C	" "	11	13
D	" "	16	10
E	" "	10.5	45
F	" "	10	11.5
G	" "	26	6.7
H	" "	13.5	60+
I	" "	18	14.2
J	" "	12.8	25

Mix	Period of Storage 40°C	Minimum Torque at 120°C	Scorch Time at 120°C (min)
A	16 weeks	16	21
B	" "	14.5	11
C	" "	10	31.5
D	" "	-	-
E	" "	13	55
F	" "	-	-
G	" "	-	-
H	" "	14.5	60+
I	" "	-	-
J	" "	-	-

Mix	Period of Storage 40°C	Minimum Torque at 150°C	Scorch Time at 150°C (min)
A	16 weeks	13.5	2.4
B	" "	12.8	2.5
C	" "	9	4.05
D	" "	-	-
E	" "	11.5	4.5
F	" "	-	-
G	" "	-	-
H	" "	11	8
I	" "	-	-
J	" "	-	-

TABLE 5

COMPARISON OF PRESS CURES BEFORE AND AFTER STORAGE AT ROOM TEMPERATURE (NATURAL RUBBERS)

Mix	Cure Time at 150°C (min)	Tensile Strength after Storage (MN/m²)		Elongation at break after Storage (%)		Modulus at 100% E after Storage (MN/m²)		Modulus at 300% E after Storage (MN/m²)		Hardness after Storage (BS°)	
		0 m	12 m	0 m	12 m	0 m	12 m	0 m	12 m	0 m	12 m
A	10	23.1	25.3	560	580	0.12	0.47	5.5	5.7	48	51
	15	25.3	24.6	510	560	0.53	0.54	8.9	6.1	56	55
	20	25.9	26.0	490	580	0.58	0.52	9.7	5.9	56	52
	30	26.0	24.3	500	555	0.63	0.53	9.8	6.1	56	52
B	10	21.0	15.4	690	612	not recorded	not recorded	0.2	1.0	36	41
	15	22.1	17.7	680	650		0.11	0.4	1.1	39	40
	20	24.5	19.0	655	650		0.11	0.9	1.2	43	40
	30	20.7	16.1	570	605		0.24	1.4	1.4	45	38
C	10	21.1	25.2	530	505	0.13	0.69	5.5	7.5	48	56
	15	25.0	20.4	510	455	0.33	0.88	7.6	8.6	54	57
	20	24.6	25.3	505	535	0.30	0.68	7.5	7.5	55	57
	30	25.0	26.7	500	515	0.38	0.79	8.0	7.5	55	57
D	10	24.0	25.6	610	555	0.10	0.46	4.5	6.6	47	50
	15	21.7	18.6	580	460	0.12	0.56	4.5	7.6	48	52
	20	21.9	25.0	560	510	0.12	0.54	5.4	7.4	48	49
	30	23.9	24.4	600	540	0.09	0.32	5.1	5.9	48	48
E	10	25.7	24.5	595	540	0.12	0.45	6.0	6.3	52	50
	15	25.7	26.4	590	555	0.12	0.59	6.4	7.0	52	53
	20	26.2	26.5	585	565	0.21	0.57	7.0	6.5	54	52
	30	26.6	25.5	565	550	0.40	0.74	7.7	7.2	56	54

TABLE 5 Contd

COMPARISON OF PRESS CURES BEFORE AND AFTER STORAGE AT ROOM TEMPERATURE (SYNTHETIC RUBBERS)

Mix	Cure Time at 150°C (min)	Tensile Strength after Storage (MN/m^2)		Elongation at Break after Storage (%)		Modulus at 100% E after Storage (MN/m^2)		Modulus at 300% E after Storage (MN/m^2)		Hardness after Storage (BS°)	
		0 m	12 m	0 m	12 m	0 m	12 m	0 m	12 m	0 m	12 m
F	10	12.6	16.3	370	420	0.5	1.5	6.9	9.6	58	63
	15	14.4	13.6	400	410	0.7	1.2	9.4	7.9	58	64
	20	14.9	15.7	380	425	1.0	1.4	10.2	8.9	60	65
	30	13.7	15.5	350	405	0.9	1.5	10.6	9.5	63	63
G	5	14.7	-	560	-	1.2	-	6.5	-	60	-
	10	17.0	14.5	425	330	2.0	2.6	10.9	12.7	61	64
	15	-	12.2	-	290	-	2.4	-	-	-	68
	20	17.2	16.0	380	330	2.1	2.3	12.7	14.0	65	67
	30	17.0	9.3	350	215	2.1	2.9	14.0	-	66	70
H	10	11.2	16.4	455	775	0.7	0.4	5.2	2.6	57	50
	15	-	12.8	-	880	-	0.2	-	1.5	-	48
	20	13.5	17.3	445	740	0.8	0.5	7.0	3.0	59	54
	30	9.7	11.3	350	490	0.9	0.7	7.2	4.5	60	58
I	5	9.2	-	480	-	0.7	-	3.8	-	65	-
	10	6.7	7.8	320	335	0.9	1.0	6.1	6.6	65	68
	15	-	7.5	-	255	-	1.6	-	-	-	70
	20	6.3	6.2	260	225	1.2	1.7	-	-	67	70
	30	5.5	5.4	235	190	1.2	1.9	-	-	66	71
J	10	18.0	14.1	780	750	0.1	0.1	1.9	1.3	44	39
	15	17.2	15.7	740	710	0.1	0.1	1.8	2.1	43	45
	20	17.3	14.9	705	630	0.2	0.3	2.5	2.5	46	47
	30	14.4	-	605	-	0.3	-	3.3	-	49	-

TABLE 6

TENSILE PROPERTIES OF INJECTION MOULDED STORED STOCKS

MIX A

Period of Storage	Cure Time (s)	Tensile Strength (MN/m^2)	Elongation at Break (%)	Modulus at 100% E (MN/m^2)	Modulus at 500% E (MN/m^2)	Hardness (BS°)
0	20	21.2	540	0.3	5.0	48
	30	21.6	510	0.5	6.5	50
	40	21.6	545	0.4	5.8	49
	45	22.9	535	0.5	6.4	53
	60	20.1	520	0.5	5.5	51
	90	14.4	500	0.3	4.4	47
	120	7.3	375	0.2	4.6	43
	180	12.1	490	0.1	3.4	41
6 months at Room Temp	20	7.2	500	-	1.6	31
	40	19.5	590	0.1	3.7	43
	60	21.1	510	0.5	6.2	52
	100	21.6	515	0.6	6.1	53
	160	19.6	480	0.6	6.5	54
	240	15.1	450	0.5	5.8	51
12 months at Room Temp	30	10.1	485	-	2.6	36
	40	16.3	520	-	3.8	41
	50	18.6	495	-	5.4	42
	80	18.6	480	0.2	5.9	45
	120	21.0	485	0.2	6.6	48
	180	20.3	490	0.2	6.3	49
	240	17.5	480	0.2	5.5	48
16 weeks at 40°C	30	8.1	490	0.1	2.1	34
	40	7.8	485	0.1	2.1	34
	60	13.6	495	0.3	3.3	40
	100	17.5	515	0.4	5.0	44
	240	63.9	465	0.5	5.2	49
24 weeks at 40°C	30	6.8	525	-	1.3	< 30
	40	8.3	525	-	1.8	< 30
	60	10.9	515	-	2.8	35
	100	12.7	480	0.1	4.2	41
	150	14.1	455	0.3	5.2	47
	240	14.5	475	0.3	4.9	46

TABLE 6 Contd

TENSILE PROPERTIES OF INJECTION MOULDED STORED STOCKS

MIX B

Period of Storage	Cure Time (s)	Tensile Strength (MN/m^2)	Elongation at Break (%)	Modulus at 100% E (MN/m^2)	Modulus at 300% E (MN/m^2)	Hardness (BS^o)
0	30	15.7	670	N.R.	0.8	38
	40	16.7	710	"	1.2	40
	45	13.6	720	"	1.4	38
	60	14.1	710	"	0.7	40
	90	10.9	760	"	0.4	38
	120	5.3	640	"	0.2	37
	180	6.1	450	"	N.R.	<30
6 months at Room Temp	30	5.6	645	"	0.4	<30
	40	16.2	750	"	0.4	30
	60	18.4	690	"	0.9	36
	90	20.0	655	0.1	1.2	41
	120	11.8	540	0.1	1.6	42
	180	12.0	550	0.2	1.7	41
12 months at Room Temp	30	9.8	605	N.R.	0.1	<30
	40	14.7	665	"	0.2	32
	60	17.7	650	"	0.7	40
	90	16.6	655	"	0.7	38
	120	15.3	575	"	0.9	38
	180	14.5	595	"	0.8	38
16 weeks at 40°C	60	6.8	640	"	1.0	31
	90	7.2	585	0.1	1.1	37
	120	9.6	60	0.1	1.1	37
	180	9.1	63	0.1	1.4	37
24 weeks at 40°C	Could not be moulded					

TABLE 6 Contd

TENSILE PROPERTIES OF INJECTION MOULDED STORED STOCKS

MIX C

Period of Storage	Cure Time (s)	Tensile Strength (MN/m^2)	Elongation at Break (%)	Modulus at 100% E (MN/m^2)	Modulus at 300% E (MN/m^2)	Hardness (BS°)
0	30	24.1	546	0.2	5.8	49
	40	25.5	555	0.4	5.9	47
	45	25.4	500	0.6	7.5	53
	60	25.9	530	0.5	7.0	51
	90	25.4	525	0.5	7.0	50
6 months at Room Temp	40	19.6	530	0.4	5.2	51
	60	21.1	490	0.6	6.4	53
	90	20.2	455	0.8	8.0	55
	130	17.6	430	0.7	7.8	55
	180	25.1	555	0.6	5.9	55
12 months at Room Temp	40	17.0	515	N.R.	3.9	44
	60	21.9	520	0.2	5.5	48
	90	20.0	465	0.3	7.0	50
	130	17.1	435	0.3	6.8	52
	180	21.3	490	0.2	6.1	52
16 weeks at 40°C	40	7.3	445	0.1	2.7	40
	60	10.7	490	0.1	3.2	40
	90	22.5	515	0.6	6.4	52
	130	22.6	505	0.6	6.8	54
	180	19.0	425	0.9	9.3	54
24 weeks at 40°C	40	5.9	250	1.0	-	54
	60	8.8	330	0.5	5.9	48
	130	7.0	270	1.1	-	59
	180	8.5	280	0.9	-	58

TABLE 6 Contd

TENSILE PROPERTIES OF INJECTION MOULDED STORED STOCKS

MIX D

Period of Storage	Cure Time (s)	Tensile Strength (MN/m²)	Elongation at Break (%)	Modulus at 100% E (MN/m²)	Modulus at 300% E (MN/m²)	Hardness (BS°)
0	15	15.0	540	0.3	3.4	47
	20	16.6	550	0.2	3.9	44
	30	21.0	550	0.3	4.6	48
	40	20.0	540	0.2	5.2	49
	50	20.0	525	0.2	5.5	49
	60	19.9	505	0.3	6.3	48
	80	19.7	550	0.3	4.9	47
	90	18.9	545	0.2	4.7	46
	100	18.3	545	0.2	4.5	45
	120	16.1	525	0.1	4.0	45
	180	14.0	510	N.R.	3.9	43
6 months at Room Temp	20	14.1	515	0.2	3.[illegible]	47
	25	12.4	530	0.1	3.5	46
	30	16.0	550	0.3	3.8	47
	40	18.1	555	0.3	4.2	48
	60	21.5	535	0.4	5.9	51
	100	20.8	530	0.5	5.9	52
	160	18.9	470	0.6	6.8	52
12 months at Room Temp	20	7.3	450	N.R.	2.4	32
	30	13.0	500	N.R.	4.0	41
	40	16.6	450	0.1	6.6	44
	100	20.7	505	0.1	6.1	46
	160	20.0	475	0.2	6.1	46
16 weeks at 40°C	40	3.2	212	0.9	3.1	55
	100	5.1	224	1.1	4.1	55
24 weeks at 40°C	Could not be moulded					

TABLE 6 Contd

TENSILE PROPERTIES OF INJECTION MOULDED STORED STOCKS

MIX E

Period of Storage	Cure Time (s)	Tensile Strength (MN/m^2)	Elongation at Break (%)	Modulus at 100% E (MN/m^2)	Modulus at 300% E (MN/m^2)	Hardness (BS°)
0	20	13.7	390	0.6	7.2	54
	30	22.2	525	0.4	6.5	54
	40	13.9	425	0.5	7.4	51
	60	14.7	455	0.5	5.9	52
	120	11.3	420	0.3	5.4	52
	180	13.0	500	0.1	4.0	43
6 months at Room Temp	40	11.6	520	0.2	2.7	39
	45	19.3	565	0.3	4.4	47
	60	19.9	560	0.4	4.7	47
	100	20.8	515	0.5	6.2	54
	160	20.2	510	0.6	6.0	54
12 months at Room Temp	40	6.9	475	N.R.	1.7	<30
	60	14.0	485	-	4.0	39
	90	20.2	505	-	5.8	45
	120	19.1	465	0.2	6.8	47
	200	20.3	505	0.2	6.4	48
16 weeks at 40°C	30	6.6	480	0.1	1.8	<30
	40	7.8	510	N.R.	1.8	32
	60	9.9	510	N.R.	2.5	35
	100	20.8	530	0.4	5.5	49
	160	13.6	412	0.7	8.1	51
24 weeks at 40°C	40	5.7	490	N.R.	1.4	31
	60	12.1	530	0.1	2.8	37
	100	17.5	550	0.2	4.2	44
	160	19.6	535	0.4	5.2	49
	180	19.4	530	0.4	5.4	49

TABLE 6 Contd

TENSILE PROPERTIES OF INJECTION MOULDED STORED STOCKS

MIX F

Period of Storage	Cure Time (s)	Tensile Strength (MN/m^2)	Elongation at Break (%)	Modulus at 100% E (MN/m^2)	Modulus at 300% E (MN/m^2)	Hardness (BS°)
0	30	14.9	465	0.8	6.8	59
	40	15.6	450	0.8	6.5	57
	60	15.8	445	1.0	7.6	59
	90	14.7	415	1.0	7.9	59
	180	14.5	400	1.1	8.3	59
6 months at Room Temp	30	15.5	455	1.1	7.1	67
	40	14.7	440	1.1	7.2	68
	60	17.7	480	1.3	7.9	65
	90	16.4	435	1.4	8.4	65
	130	15.1	400	1.5	9.0	66
	180	15.4	400	1.5	9.1	68
12 months at Room Temp	30	15.5	460	0.7	7.4	61
	40	15.7	480	0.6	6.4	60
	60	16.8	505	0.5	5.7	58
	90	15.0	470	0.6	6.3	58
	130	13.2	415	0.6	6.7	59
	180	13.6	415	0.6	6.9	59
	240	12.8	360	1.0	8.8	60
16 weeks at 40°C	Could not be moulded					

TABLE 6 Contd

TENSILE PROPERTIES OF INJECTION MOULDED STORED STOCKS

MIX G

Period of Storage	Cure Time (s)	Tensile Strength (MN/m^2)	Elongation at Break (%)	Modulus at 100% E (MN/m^2)	Modulus at 300% E (MN/m^2)	Hardness (BS^o)
0	30	16.2	535	1.1	7.5	54
	50	18.3	430	2.1	11.6	61
	80	17.7	385	2.3	12.8	63
	120	16.3	315	2.8	15.5	64
	180	15.9	280	3.2	-	65
6 months at Room Temp	30	17.1	510	1.9	9.0	68
	40	17.9	440	2.5	11.4	69
	50	18.4	445	2.6	11.6	71
	80	17.2	420	2.5	12.5	67
	120	19.3	355	3.1	15.7	70
	180	17.8	320	2.9	16.5	74
12 months at Room Temp	Could not be moulded					
16 weeks at 40°C	Could not be moulded					

TABLE 6 Contd

TENSILE PROPERTIES OF INJECTION MOULDED STORED STOCKS

MIX H

Period of Storage	Cure Time (s)	Tensile Strength (MN/m^2)	Elongation at Break (%)	Modulus at 100% E (MN/m^2)	Modulus at 300% E (MN/m^2)	Hardness (BS°)
0	60	14.7	480	0.7	6.3	54
	90	17.0	455	1.1	8.0	55
	130	17.1	485	0.8	7.2	55
	180	17.6	460	1.1	8.3	56
	240	19.1	480	1.0	7.5	55
6 months at Room Temp	60	17.0	660	N.R.	0.3	32
	90	17.7	695	0.5	3.6	52
	130	18.0	690	0.6	3.9	51
	180	18.7	540	1.5	6.5	59
	240	17.1	485	1.6	7.6	62
12 months at Room Temp	120	8.2	675	N.R.	1.5	46
	150	14.8	700	0.1	3.1	49
	180	16.4	665	0.3	3.9	50
	240	17.9	635	0.4	4.5	54
	300	16.6	565	0.5	5.2	56
	330	18.1	580	0.5	5.3	55
16 weeks at 40°C	90	5.8	930	N.R.	0.6	40
	130	13.2	795	0.3	1.9	48
	180	17.5	690	0.7	3.8	53
	240	18.4	640	0.8	4.6	54
24 weeks at 40°C	90	Too sticky to handle				
	130	3.6	810	0.1	0.6	35
	180	11.0	780	0.1	1.6	47
	240	15.4	720	0.4	2.7	53

TABLE 6 Contd

TENSILE PROPERTIES OF INJECTION MOULDED STORED STOCKS

MIX I

Period of Storage	Cure Time (s)	Tensile Strength (MN/m^2)	Elongation at Break (%)	Modulus at 100% E (MN/m^2)	Modulus at 300 % E (MN/m^2)	Hardness (BS°)
0	30	9.6	515	0.6	3.6	58
	50	8.4	405	0.6	4.7	60
	80	7.8	295	1.0	-	63
	120	7.8	255	1.3	-	66
	180	6.8	270	1.0	-	64
6 months at Room Temp	30	16.6	745	0.6	2.3	61
	50	12.0	500	0.8	4.7	67
	80	9.3	350	1.3	7.0	71
	120	10.6	320	1.6	9.5	72
	180	10.2	295	1.7	-	73
12 months at Room Temp	30	12.5	615	N.R.	2.6	60
	50	10.1	355	1.1	7.8	63
	80	9.0	280	1.5	-	65
	120	8.9	285	1.1	-	66
	180	8.0	240	1.1	-	66
16 weeks at 40°C	20	11.4	805	0.5	1.6	58
	30	12.6	600	0.6	3.0	62
	50	11.2	460	1.0	5.3	64
	80	9.3	350	1.2	7.0	65
	120	9.5	305	1.6	9.5	67
24 weeks at 40°C	20	8.4	680	0.5	1.7	60
	25	12.8	640	0.6	2.6	61
	30	11.9	660	0.6	2.4	60
	50	10.7	445	0.7	4.3	65
	80	9.8	345	1.1	7.3	68
	120	8.7	310	1.3	8.2	68

TABLE 6 Contd

TENSILE PROPERTIES OF INJECTION MOULDED STORED STOCKS

MIX J

Period of Storage	Cure Time (s)	Tensile Strength (MN/m^2)	Elongation at Break (%)	Modulus at 100% E (MN/m^2)	Modulus at 300% E (MN/m^2)	Hardness (BS^o)
0	40	13.5	735	N.R.	0.9	35
	60	16.4	740	N.R.	1.5	39
	90	16.3	710	N.R.	2.0	42
	130	16.6	700	N.R.	1.9	41
	200	16.5	650	N.R.	2.6	43
6 months at Room Temp	40	12.9	795	N.R.	0.7	<30
	60	15.1	780	N.R.	1.0	35
	90	16.6	740	N.R.	1.6	35
	130	16.5	700	0.1	2.1	43
	200	12.3	580	0.4	2.7	45
	300	15.5	600	0.4	3.3	47
12 months at Room Temp	60	14.5	765	N.R.	0.6	38
	90	15.8	730	N.R.	1.0	41
	130	15.0	675	N.R.	1.3	43
	150	14.9	660	N.R.	1.6	43
	200	14.2	640	N.R.	1.9	44
16 weeks at 40°C	40	11.5	765	N.R.	0.5	32
	60	12.9	740	N.R.	0.9	35
	90	16.3	755	N.R.	1.5	39
	130	16.5	725	N.R.	1.8	41
	200	16.2	645	0.3	2.8	45
24 weeks at 40°C	40	6.7	780	N.R.	0.2	<30
	60	9.9	740	N.R.	0.5	<30
	90	11.5	700	N.R.	1.0	34
	130	16.5	735	N.R.	1.4	37
	200	15.8	680	0.1	2.0	44

TABLE 7

"FREE" SULPHUR DETERMINATION ON STORED STOCKS

Mix	Total Sulphur present (%)	Added Sulphur as S (%)	"Free" Sulphur determined after storage (%) for 4 m Duplicate results		"Free" Sulphur determined after storage (%) for 12 m Duplicate results	
A	1.92	1.83	1.61	1.55	1.03	1.07
B	2.36	2.26	1.92	1.87	1.03	1.11
C	0.78	0.24	(0.39 (0.29	(0.28 (0.25	0.08	0.04
D	0.99	0	0.15	0.10	0.07	0.06
E	1.95	1.83	1.53	1.64	0.91	0.92
F	1.82	1.46	-	-	-	-
G	0.32	0	0.097	0.096	0.11	0.10
H	1.50	1.28	1.33	1.27	1.04	1.10
I	1.78	1.11	0.94	0.97	1.23	1.01
J	1.70	1.06	(0.34 (0.41	(0.89 (1.01	0.56	0.44

TABLE 8

BENZENE EXTRACT OF STORED RUBBERS

Mix	Benzene Extract (%)					
	Before Storage	Theoretical	Storage at Room Temp		Storage at 40°C	
			Period of Storage		Period of Storage	
			6 months	12 months	8 weeks	16 weeks
A	65.6 66.1	76	58.1 56.4	47.1 47.5	61.4 62.3	35.3 36.7
B	82.2 94.9	93	46.6 47.9	92.2 88.1	62.1 63.5	19.4 17.4
C	62.1 65.4	78	34.8 34.5	49.5 53.5	63.7 63.5	30.6 29.5
D	68.7 62.4	78	55.7 54.2	30.5 32.7	15.2 16.6	15.0 13.6
E	58.0 60.4	78	55.1 56.6	30.6 36.5	51.6 52.8	44.8 46.3
F	68.3 68.2	77	65.2 65.4	47.5 65.0	7.9 21.7	4.3 6.8
G	66.6 65.2	75	59.2 57.9	47.5 46.3	7.9 8.4	4.3 5.8
H	52.4 62.4	76	60.8 61.4	67.8 68.7	66.8 67.7	69.6 76.1
I	71.5 75.0	76	72.0 74.5	75.3 74.9	63.0 62.8	75.3 75.1
J	74.9 78.1	78	75.5 75.2	71.2 68.5	76.6 70.9	74.3 75.4

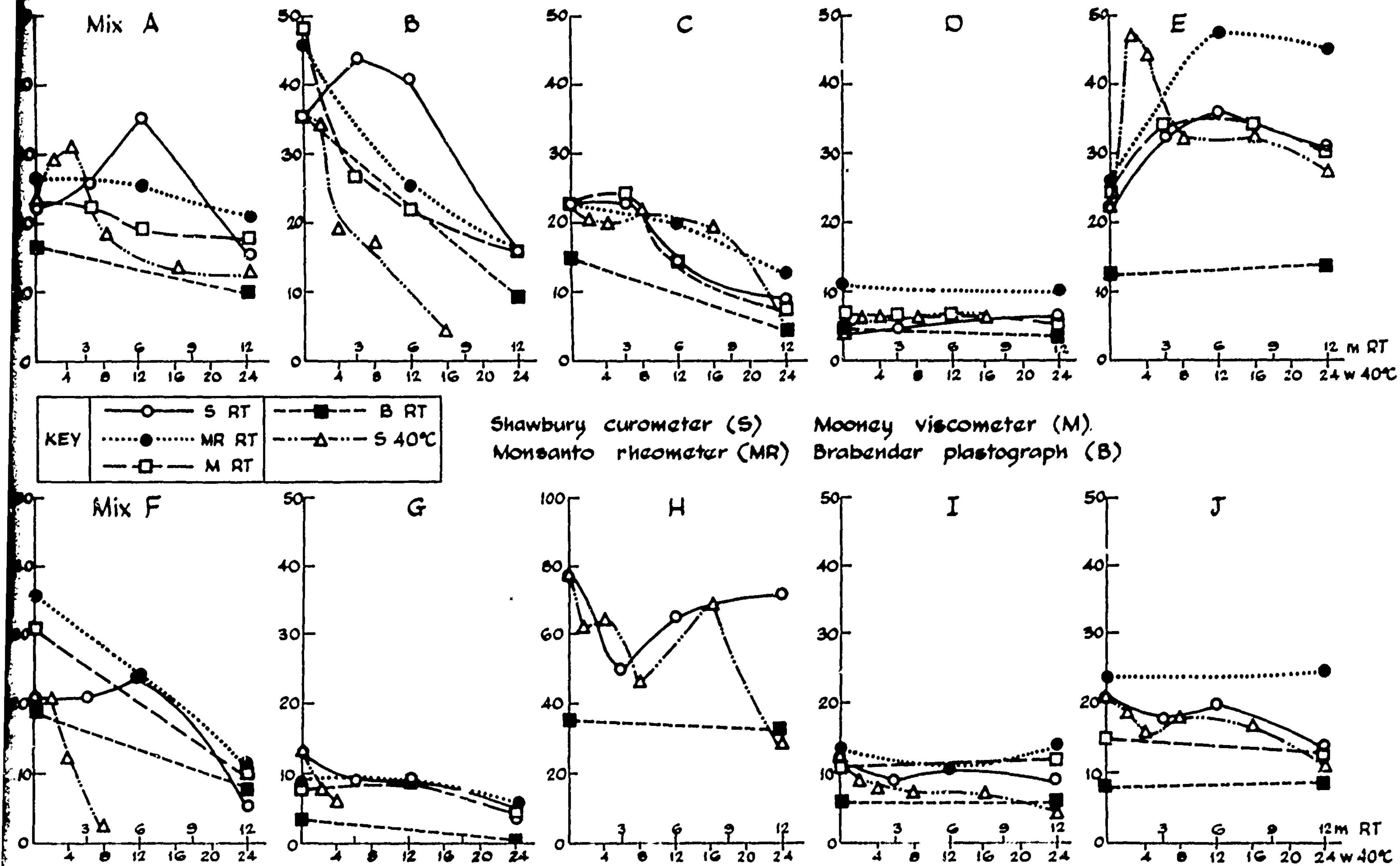

FIG. 1

SCORCH TIMES AT 120°C

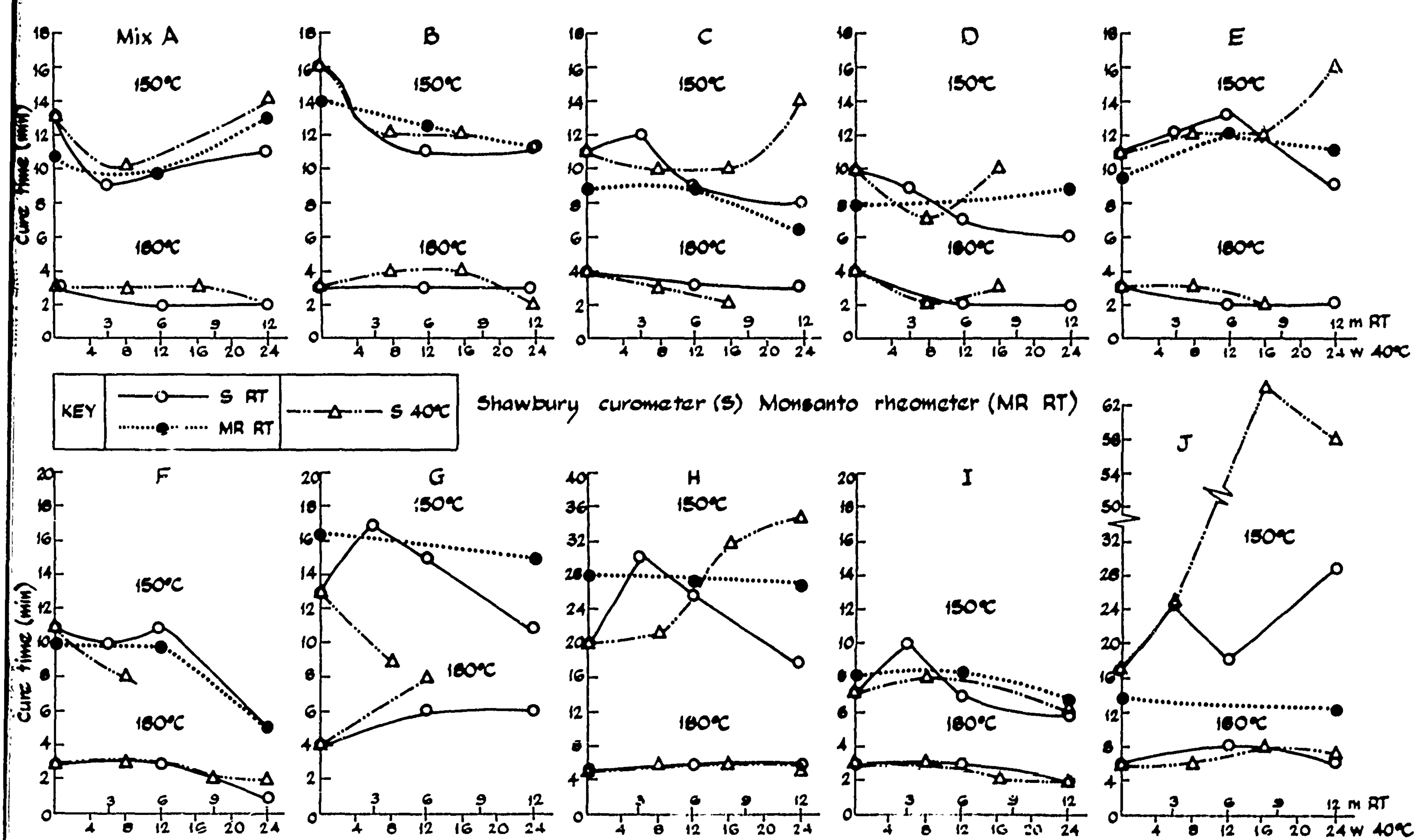

FIG. 2 CURE TIMES AT 150 AND 180°C

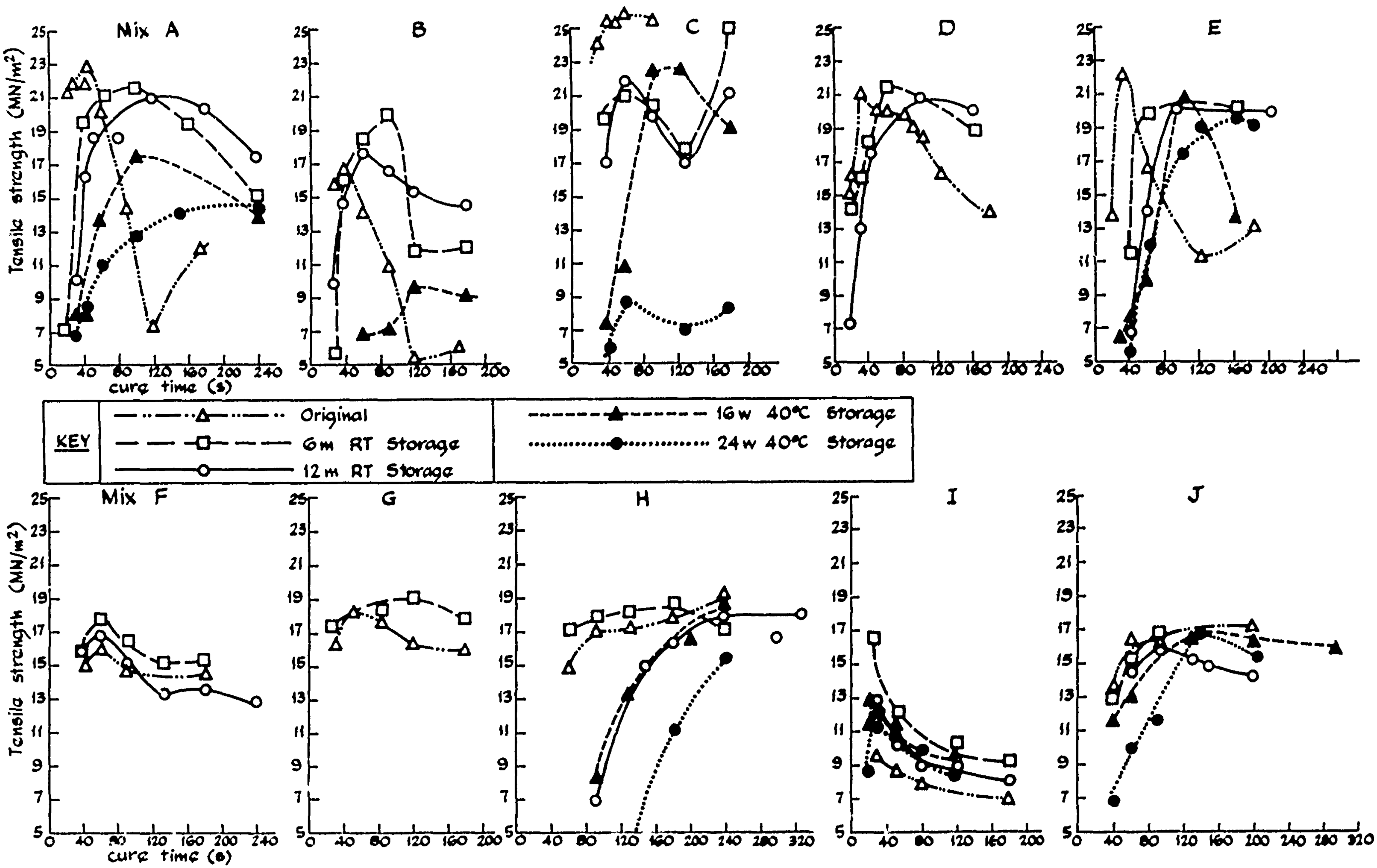

FIG 3 TENSILE STRENGTH V INJECTION MOULDING CURE TIME

PART 3

SUBSEQUENT AGEING BEHAVIOUR

1 INTRODUCTION

Parts 1[1] and 2 have shown that fully compounded rubber stocks can be satisfactorily stored for up to 12 months at room temperature and 24 weeks at 40°C before vulcanisation, depending upon the type of rubber and vulcanising system used. The present work was undertaken to investigate the effect of storage of the stocks on the subsequent ageing characteristics of their vulcanisates.

2 EXPERIMENTAL

Each stock was injection and compression moulded after the selected storage period and three of the injection moulded vulcanisates were chosen to undergo accelerated heat ageing from the range of curing times given in Table 6 of Part 2. The times chosen were such that one vulcanisate was below maximum tensile strength, one was as near as possible to maximum and one was possibly overcured. The compression moulded samples were given optimum cures as defined by the Shawbury Curometer. The composition of the mixes (A - J) and the methods of obtaining the vulcanisates are given in Part 2.

Type E dumb-bells were cut from the vulcanisates and measurements of their width and thickness were made before exposure. Five dumb-bells were suspended in open glass tubes and placed in an air circulating oven at 80° ± 1°C for intervals of 2, 5, 8, or 5, 8 and 16 weeks.

After exposure the test pieces were conditioned for about 24 hours at room temperature before testing. The physical tests, tensile strength, elongation at break, and modulus were carried out on a Tensometer E-type testing machine and hardness on a Wallace micro-hardness tester according to British Standard methods. The average of five results is reported.

3 RESULTS AND DISCUSSION

All of the results are given in Tables 1 - 10, and, in addition, the effect of heat ageing on vulcanisates of one particular cure (optimum) of each mix is shown in Figures 1 - 2 by plotting the changes in tensile strength and elongation at break with time.

The natural rubber mixes were chosen mainly on the basis of their sulphur/accelerator ratio, mixes A, B and E having a conventional system of high sulphur and low accelerator, mix C a low sulphur and high accelerator ratio (commonly known as an efficient vulcanisation (EV) system), and mix D being an essentially sulphurless cure using only the sulphur bearing accelerator TMTD. The main features of these systems are that the high sulphur vulcanisates give good processing and medium ageing properties, the EV systems good processing and ageing characteristics but poor resilience and abrasion resistance, and the TMTD system good ageing but poor processing properties.

Test pieces from injection moulded plaques of the natural rubber stocks were cut before ageing trials were started. On heat ageing, the vulcanisates prepared from unstored mixes A, B and E behaved as expected and showed more rapid deterioration than both the EV mix, C, and the TMTD mix, D. After storage of the stocks the same general trends were maintained.

For all of the injection moulded vulcanisates it would appear that storage of the stocks for 6 months at room temperature does not significantly change the ageing characteristics. However, after 12 months' storage of the stocks at room temperature, the high sulphur mixes for both natural and synthetic rubbers showed better ageing properties; the EV systems of natural rubber aged well and showed no significant change in their rate of deterioration from the original material. The enhancement of the ageing properties after 12 months' storage may be due to a slight undercure, or to the formation of mono- and di-sulphide crosslinks in preference to polysulphides as suggested by Cunneen[2] in his review of oxidative ageing. This would be in keeping with the reduction of sulphur content of the rubbers as shown in Table 7 of Part 2. Storage of the unvulcanised stocks for 16 weeks at 40°C gave slightly enhanced ageing properties. These differences were not quite so pronounced as those of vulcanisates prepared from stocks stored for 12 months at room temperatures.

Compression moulded vulcanisates prepared from stock which had not been stored showed better ageing properties than those which had been injection moulded. Both compression and injection moulded vulcanisates prepared from the high sulphur mixes A, B and E stored for 12 months at room temperature showed higher rates of deterioration than those compression moulded from the initial unstored material. The ageing characteristics of the vulcanisates compression moulded from the low sulphur/high accelerator stocks C and D, stored for 12 months at room temperature, were similar to those press cured from the initial unstored material and deteriorated less than those prepared by injection moulding. After storage at 40°C for 16 weeks, those stocks which could be press cured gave vulcanisates with poorer ageing properties than those prepared from both unstored stocks and stocks stored for 12 months at room temperature, and in general were poorer than the injection moulded vulcanisates.

The difference in ageing characteristics of press cured vulcanisates compared with those of injection moulded samples of stocks stored for 12 months at room temperature may be explained by the possibility of an overcure, or that any rubber/sulphur/accelerator complexes formed during storage were not broken down at 150°C (press cure temperature) but were decomposed at 180°C, the cure temperature for injection moulding, or that the antioxidant was not suitable for injection moulding.

4 CONCLUSIONS

The storage of stocks for 6 months at room temperature does not significantly affect the ageing characteristics of subsequently injection moulded vulcanisates. Most of the injection moulded vulcanisates prepared from stocks stored for 12 months at room temperature showed slightly enhanced ageing properties over those prepared from unstored stocks, but some press cured samples had poorer characteristics. The injection mouldable vulcanisates of stocks stored at 40°C for 16 weeks of the three natural rubbers A, C and D, and the three synthetic rubbers H, I and J, had ageing characteristics similar to those prepared from the unstored stocks. The properties of the press cured materials showed a somewhat faster rate of deterioration.

The general ageing characteristics, which are dependent on the type of rubber and the sulphur/accelerator ratio, are not altered to any significant degree by storage of the rubber stock, provided that the vulcanisates are given their optimum cure.

5 REFERENCES

1	Ledbury K J, Sims D, Stokoe A L	ERDE Technical Report No 44, August 1970
2	Cunneen J I	Rubb Chem Technol, 1968, 41, 1, 182-208.

TABLE 1

Mix Number	Period of Storage	Cure Time	Tensile Strength (MN/m²) Ageing Period (weeks)				Elongation at Break (%) Ageing Period (weeks)				Modulus at 100% E (MN/m²) Ageing Period (weeks)				Modulus at 300% E (MN/m²) Ageing Period (weeks)				Hardness (BS°) Ageing Period (weeks)			
			0	2	5	8	0	2	5	8	0	2	5	8	0	2	5	8	0	2	5	8
A	0	20 s	21.2	10.6	2.4	2.8	540	350	165	70	0.3	0.7	0.9	-	5.0	7.3	-	-	48	46	45	60
		40 s	22.0	6.6	3.1	2.8	545	280	175	90	0.4	0.7	1.0	-	5.8	-	-	-	49	42	39	63
		60 s	20.2	4.0	2.6	2.6	520	205	145	50	0.4	0.9	1.3	-	5.5	-	-	-	51	42	52	71
A	6 months at room temperature	40 s	19.5	9.8	1.9	3.3	590	395	140	60	0.2	0.4	1.1	-	3.8	5.8	-	-	44	44	47	75
		60 s	21.2	10.8	2.9	3.2	570	370	155	125	0.5	0.7	1.3	2.3	6.3	7.5	-	-	52	45	50	56
		100 s	21.7	5.4	3.5	4.1	515	265	145	105	0.6	0.7	1.7	3.9	6.2	-	-	-	53	43	51	61
A	12 months at room temperature	80 s	18.9	20.8	7.5	2.5	480	450	375	195	0.2	0.5	0.1	0.5	6.0	7.3	4.9	-	45	47	38	41
		120 s	21.2	20.2	5.6	2.5	485	455	310	160	0.2	0.8	0.1	0.7	6.7	9.6	5.4	-	48	49	39	42
		180 s	20.2	19.6	5.2	3.0	490	445	260	170	0.2	0.7	0.5	0.9	6.4	8.9	-	-	49	50	41	44
A	16 weeks at 40°C	40 s	7.9	8.5	3.0	1.9	485	435	285	135	0.1	0.2	0.2	1.1	2.1	4.1	-	-	34	30	30	42
		60 s	13.6	11.3	6.4	2.2	495	385	370	115	0.3	0.1	0.8	1.7	3.3	3.9	7.3	-	40	<30	51	50
		100 s	17.5	7.2	3.0	2.9	515	400	190	60	0.4	0.2	1.0	-	5.0	4.2	-	-	44	30	41	67
A	24 weeks at 40°C	100 s	14.4	11.7	4.6	2.4	485	465	365	225	0.1	0.2	-	0.3	4.7	4.6	3.2	-	43	36	<30	<30
		180 s	17.2	10.8	5.5	2.4	495	410	350	125	0.3	0.3	0.6	1.6	5.9	5.9	6.7	-	47	41	46	42
		240 s	16.6	Not done	6.2	4.5	460	Not done	285	235	0.4	Not done	0.7	0.7	6.5	-	-	-	47	Not done	34	36
A Press cured	0	20 min	22.6	23.4	18.2	12.1	470	450	400	310	0.9	1.7	1.7	1.7	9.6	13.0	12.2	11.6	60	64	56	51
A Press cured	12 months at room temperature	20 min	26.0	22.4	5.8	4.0	580	540	265	170	0.5	0.8	0.8	1.3	5.8	20.4	-	-	52	50	36	52
A Press cured	16 weeks at 40°C	15 min	16.9	Not done	1.4	1.9	495	Not done	100	20	0.3	Not done	1.3	-	5.4	-	-	-	50	Not done	51	73

TABLE 2

Mix Number	Period of Storage	Cure Time	Tensile Strength (MN/m²)				Elongation at Break (%)				Modulus at 100% E (MN/m²)				Modulus at 300% E (MN/m²)				Hardness (BS°)			
			Ageing Period (weeks)				Ageing Period (weeks)				Ageing Period (weeks)				Ageing Period (weeks)				Ageing Period (weeks)			
			0	2	5	8	0	2	5	8	0	2	5	8	0	2	5	8	0	2	5	8
B	0	40 s	16.7	9.8	0.5	1.0	710	430	184	90	Not recorded	0.3	Not recorded	-	1.2	2.7	-	-	40	49	30	39
		60 s	14.0	7.4	0.8	0.9	710	390	180	80	"	0.2	"	-	0.7	2.6	-	-	40	40	31	47
		90 s	10.9	4.9	1.0	0.9	765	405	220	85	"	0.1	"	-	0.4	1.9	-	-	38	33	<30	44
B	6 months at room temperature	40 s	16.2	7.2	0.9	1.4	750	405	190	94	"	0.2	0.2	-	0.4	2.6	-	-	30	40	32	44
		90 s	19.9	6.2	0.8	0.8	655	400	185	90	0.1	0.2	0.2	-	1.2	2.5	-	-	41	38	<30	45
		120 s	11.8	7.1	0.7	0.5	540	435	170	85	0.1	NR	0.2	-	1.6	2.0	-	-	42	37	<30	35
B	12 months at room temperature	40 s	14.6	18.2	10.6	0.8	665	505	440	230	Not recorded	0.3	Not recorded	Not recorded	1.8	2.9	2.5	-	32	45	41	33
		60 s	17.7	19.6	10.6	0.6	650	510	480	225	"	0.2	"	"	0.7	2.9	2.5	-	40	43	40	33
		90 s	16.6	14.9	11.2	2.8	655	485	455	390	0.1	0.2	"	"	0.7	2.6	1.7	1.0	38	41	38	33
		120 s	15.2	15.9	11.4	0.6	575	505	505	250	Not recorded	0.1	"	"	0.9	2.3	1.4	-	38	41	37	33
B Press cured	0	20 min	12.4	13.1	13.3	6.3	490	435	410	330	0.3	0.5	0.6	0.6	1.9	3.6	4.5	4.5	58	53	47	42
B Press cured	12 months at room temperature	15 min	17.7	15.3	11.4	0.9	650	510	510	225	0.1	0.3	0.1	0.2	1.1	2.2	1.7	-	40	42	31	31.5
		20 min	19.0	13.6	3.2	1.2	650	495	370	200	0.1	0.4	0.1	0.2	1.2	2.5	1.6	1.1	40	38	35	< 30

TABLE 3

Mix Number	Period of Storage	Cure Time	Tensile Strength (MN/m²) Ageing Period (weeks) 0	5	8	16	Elongation at Break (%) Ageing Period (weeks) 0	5	8	16	Modulus at 100% E (MN/m²) Ageing Period (weeks) 0	5	8	16	Modulus at 300% E (MN/m²) Ageing Period (weeks) 0	5	8	16	Hardness (BS°) Ageing Period (weeks) 0	5	8	16
C	0	40 s	25.4	19.3	19.5	10.2	555	440	425	305	0.4	0.9	0.9	[illegible]	6.0	9.0	9.4	9.7	47	57	65	62
		60 s	25.8	18.8	18.7	12.4	530	415	405	335	[illegible]	1.3	1.1	1.3	7.0	9.6	10.3	9.7	51	60	68	66
		90 s	25.4	19.5	16.3	15.5	525	450	405	345	0.5	0.8	0.7	1.0	7.0	8.3	8.7	9.7	50	57	64	64
C	6 months at room temperature	60 s	21.1	18.4	18.2	11.8	490	415	405	370	0.6	[illegible]	1.1	1.1	6.4	9.8	9.7	8.4	53	60	67	70
		90 s	20.2	20.1	17.3	13.8	455	440	415	355	0.8	1.3	1.0	[illegible]	8.0	9.2	9.0	9.3	55	61	67	[illegible]
		130 s	17.5	19.3	16.6	15.0	430	440	380	380	0.7	1.1	[illegible]	1.0	7.8	8.8	10.4	9.1	55	60	68	[illegible]
C	12 months at room temperature	40 s	17.0	17.1	14.3	10.5	515	440	385	330	Not recorded	0.5	0.6	0.7	3.9	7.3	8.3	8.7	43	55	55	56
		60 s	21.8	21.1	18.0	18.1	520	460	425	400	0.2	0.6	0.7	0.1	5.5	8.0	8.6	9.9	48	55	56	55
		90 s	20.0	21.6	19.5	16.4	465	470	440	375	0.3	0.5	0.7	0.1	7.0	7.7	8.5	10.2	50	56	56	56
		130 s	17.1	18.0	16.7	16.8	435	450	410	415	0.3	0.4	0.6	0.8	6.9	7.6	8.2	8.6	52	55	55	54
C	16 weeks at 40°C	60 s	10.8	12.9	8.9	7.6	490	131	250	270	0.1	1.3	1.6	0.9	3.2	9.6	-	-	40	57	68	67
		90 s	22.3	17.7	13.3	10.8	515	400	320	330	0.6	1.3	1.6	1.2	6.4	9.9	12.0	9.3	52	58	66	68
		130 s	19.0	15.1	15.9	11.4	425	310	365	Not recorded	0.9	1.2	1.1	Not recorded	9.3	8.4	10.8	Not recorded	54	59	68	Not recorded
C Press cured	0	30 min	24.9	23.1	22.6	18.0	490	425	425	380	0.8	1.6	1.6	1.8	8.2	10.7	11.4	11.6	61	61	61	67
C Press cured	16 weeks at 40°C	15 min	24.4	17.3	15.1	6.3	545	430	340	225	0.5	1.1	1.2	1.3	5.8	8.1	11.0	-	57	60	[illegible]	56
C Press cured	12 months at room temperature	15 min	20.4	18.5	13.4	1.0	455	390	345	285	0.9	1.4	1.6	1.7	8.4	10.0	10.4	-	57	58	64	60
		20 min	25.4	21.0	19.0	16.2	535	450	415	400	0.7	1.2	1.4	1.5	6.9	8.7	9.5	9.3	57	57	60	58

TABLE 4

Mix Number	Period of Storage	Cure Time	Tensile Strength (MN/m²) Ageing Period (weeks) 0	5	8	16	Elongation at Break (%) Ageing Period (weeks) 0	5	8	16	Modulus at 100% E (MN/m²) Ageing Period (weeks) 0	5	8	16	Modulus at 300% E (MN/m²) Ageing Period (weeks) 0	5	8	16	Hardness (BS°) Ageing Period (weeks) 0	5	8	16
D	0	30 s	21.1	21.7	19.2	11.7	545	485	435	370	0.3	0.6	0.6	0.4	4.6	8.0	8.3	7.5	48	51	61	52
		50 s	20.2	19.6	19.8	10.0	525	470	485	430	0.2	0.6	0.4	0.1	5.5	7.5	6.9	5.0	49	52	60	55
		90 s	18.8	16.8	15.6	9.2	545	485	480	380	0.2	0.4	0.3	0.1	4.7	6.3	6.0	5.7	46	50	49	58
D	6 months at room temperature	40 s	18.2	20.7	18.8	12.6	555	455	445	390	0.3	0.8	0.5	0.5	4.2	8.9	7.9	7.5	48	56	63	55
		60 s	21.8	20.3	16.3	12.2	555	460	405	350	0.4	0.7	0.6	0.7	5.9	8.3	8.0	8.7	51	55	64	71
		100 s	21.0	19.8	16.4	13.3	530	460	390	360	0.5	0.7	0.6	0.4	5.9	8.1	9.1	8.8	52	54	61	56
D	12 months at room temperature	40 s	16.6	21.4	20.8	13.4	450	455	440	352	0.1	0.4	0.5	0.6	6.6	8.5	9.6	9.6	44	54	52	53
		100 s	20.7	23.4	20.4	17.4	505	510	465	395	0.1	0.3	0.2	0.6	6.1	7.1	7.7	9.3	46	50	51	53
		160 s	20.0	23.2	23.6	16.7	475	505	485	410	0.2	0.4	0.2	0.4	6.8	7.4	8.7	8.4	46	50	50	50
D	16 weeks at 40°C	40 s	3.2	-	-	-	210	-	-	-	0.9	-	-	-	3.1	-	-	-	55	-	-	-
		100 s	5.1	-	-	-	225	-	-	-	1.1	-	-	-	4.1	-	-	-	55	-	-	-
			Not aged as appearance and physical properties rather poor																			
D Press cured	0	20 min	23.8	24.4	25.1	19.2	540	475	480	400	0.5	1.1	1.2	1.4	7.0	10.3	10.2	11.7	53	55	55	[illegible]
D Press cured	12 months at room temperature	15 min	18.5	15.4	15.5	13.1	465	355	365	350	0.5	1.3	1.2	1.1	7.4	11.8	10.7	9.9	52	52	53	56
		20 min	24.8	22.5	23.0	16.3	510	460	455	395	0.5	0.9	0.9	0.9	7.3	9.1	9.4	9.3	49	69	54	50
D Press cured	16 weeks at 40°C	15 min	12.6	7.4	7.0	1.3	380	255	230	165	0.5	1.0	1.1	0.3	7.4	-	-	-	55	61	71	31

TABLE 5

Mix Number	Period of Storage	Cure Time	Tensile Strength (MN/m²) Ageing Period (weeks)				Elongation at Break (%) Ageing Period (weeks)				Modulus at 100% E (MN/m²) Ageing Period (weeks)				Modulus at 300% E (MN/m²) Ageing Period (weeks)				Hardness (BS°) Ageing Period (weeks)			
			0	2	5	8	0	2	5	8	0	2	5	8	0	2	5	8	0	2	5	8
E	0	30 s	22.1	5.88	3.43	2.87	525	220	165	30	0.29	1.37	1.35	-	6.5	-	-	-	54	53	56	77
		40 s	13.9	6.99	2.89	2.93	425	245	125	40	0.49	1.30	1.90	-	7.35	-	-	-	51	52	51	76
		60 s	14.7	5.39	2.49	3.95	455	205	105	55	0.49	1.37	2.29	-	5.93	-	-	-	52	50	56	76
E	6 months at room temperature	40 s	11.6	13.9	2.79	2.21	520	410	200	45	0.17	1.02	1.25	-	2.71	8.46	-	-	38	52	45	92
		60 s	19.8	11.3	2.40	3.05	560	400	150	70	0.39	0.638	1.20	-	4.68	6.65	-	-	47	44	41	66
		100 s	20.7	6.9	2.49	3.64	515	320	120	110	0.54	0.568	1.87	0.28	6.23	6.40	-	-	54	42	51	61
E	12 months at room temperature	60 s	14.0	21.3	14.1	9.19	485	550	490	435	-	0.265	-	-	2.99	5.76	4.61	4.30	39	44	39	32
		90 s	20.1	22.1	12.4	5.41	505	515	440	310	-	0.430	-	-	5.87	7.56	6.36	5.01	45	47	40	38
		120 s	19.1	20.2	12.05	3.81	465	460	390	240	0.23	0.510	2.75	0.314	6.80	8.72	6.95	-	47	50	47	39
E	16 weeks at 40°C	60 s	9.92	14.0	4.02	3.20	510	420	230	156	-	0.757	0.942	1.51	2.55	7.48	-	-	35	50	39	50
		100 s	20.8	10.5	3.69	3.32	530	370	205	110	0.41	0.686	1.10	2.71	5.51	7.29	-	-	49	46	46	56
		160 s	13.6	11.2	7.64	3.00	410	320	310	76	0.64	0.745	1.24	-	8.13	7.12	9.30	-	51	55	56	66
E	24 weeks at 40°C	100 s	18.00	-	16.1	4.6	520	-	445	293	0.24	-	0.677	0.470	4.8	-	-	-	47	-	41	30
		160 s	19.7	14.0	16.2	3.04	516	415	425	160	0.36	0.618	0.947	1.37	5.79	-	-	-	50	47	52	43
		180 s	24.8	-	10.3	2.55	530	-	335	160	0.57	-	0.873	1.18	7.64	-	-	-	50	-	44	41
E Press cured	0	15 min	29.2	25.7	20.9	13.3	520	485	440	340	0.83	1.53	1.59	1.65	10.61	12.05	12.00	11.3	57	63	55	50
E Press cured	12 months at room temperature	20 min	26.8	20.4	6.94	3.47	565	465	280	155	0.27	0.971	1.05	1.93	6.43	9.19	-	-	52	52	58	53
E Press cured	16 weeks at 40°C	15 min	18.7	-	1.32	1.82	470	-	95	90	0.54	-	-	-	6.16	-	-	-	57	-	55	59

TABLE 6

Mix Number	Period of Storage	Cure Time	Tensile Strength (MN/m^2) Ageing Period (weeks)				Elongation at Break (%) Ageing Period (weeks)				Modulus at 100% E (MN/m^2) Ageing Period (weeks)				Modulus at 300% E (MN/m^2) Ageing Period (weeks)				Hardness (BS°) Ageing Period (weeks)			
			0	5	8	16	0	5	8	16	0	5	8	16	0	5	8	16	0	5	8	16
F	0	40 s	15.6	13.1	13.6	11.3	480	275	260	140	0.755	2.22	2.20	6.27	6.47	-	-	-	57	72	81	90
		60 s	15.8	14.8	10.9	12.2	440	295	230	145	1.04	2.22	2.43	6.72	7.67	-	-	-	59	73	80	89
		90 s	14.7	14.6	13.2	12.8	415	275	255	155	4.43	2.04	2.22	6.83	7.95	-	-	-	59	72	81	89
F	6 months at room temperature	60 s	17.6	13.9	14.8	13.4	480	255	255	155	1.29	2.70	2.83	6.35	7.85	-	-	-	65	73	81	88
		90 s	16.4	13.8	13.3	13.5	435	260	250	145	1.37	2.65	2.50	7.29	8.36	-	-	-	65	73	82	89
		130 s	15.1	13.3	12.0	12.3	400	230	230	140	1.44	2.95	2.75	6.84	9.05	-	-	-	66	74	82	91
F	12 months at room temperature	30 s	15.5	12.2	10.8	-	440	285	260	-	0.735	1.53	1.57	-	7.36	-	-	-	61	68	68	-
		40 s	15.7	14.9	17.1	13.9	480	330	300	240	0.608	1.27	1.53	2.62	6.36	12.1	13.9	-	60	68	70	72
		60 s	16.5	14.2	13.9	13.6	505	325	320	245	0.480	1.34	1.27	2.28	5.72	11.9	12.5	-	58	67	69	72
		90 s	15.0	14.3	14.0	13.8	470	340	305	255	0.578	1.28	1.42	2.37	6.32	11.1	13.4	-	58	67	69	71
F Press cured	0	20 min	15.5	15.4	15.8	14.9	335	280	280	205	1.99	2.62	2.84	4.75	13.5	-	-	-	66	68	69	78
F Press cured	12 months at room temperature	20 min	15.7	15.2	17.3	15.7	425	340	340	275	1.40	1.88	2.45	2.62	8.94	12.7	14.7	-	65	67	67	72
F Press cured	16 weeks at 40°C	15 min	7.58	8.95	8.72	10.6	270	225	180	95	1.40	2.58	3.10	-	-	-	-	-	64	73	83	81

TABLE 7

Mix Number	Period of Storage	Cure Time	Tensile Strength (MN/m²) Ageing Period (weeks)				Elongation at Break (%) Ageing Period (weeks)				Modulus at 100% E (MN/m²) Ageing Period (weeks)				Modulus at 300% E (MN/m²) Ageing Period (weeks)				Hardness (BS°) Ageing Period (weeks)			
			0	5	8	16	0	5	8	16	0	5	8	16	0	5	8	16	0	5	8	16
G	0	30 s	16.2	17.5	15.6	16.0	535	230	180	75	1.08	6.12	7.69	-	7.47	-	-	-	54	81	90	98
		50 s	18.2	16.3	14.1	16.5	430	210	160	70	1.11	6.30	7.78	-	11.6	-	-	-	61	82	92	98
		120 s	16.3	16.3	16.6	15.7	315	200	180	65	2.84	6.86	8.75	-	15.5	-	-	-	64	82	92	98
G	6 months at room temperature	30 s	17.1	16.5	14.6	15.2	510	210	160	70	1.94	6.19	8.14	-	9.00	-	-	-	68	82	92	98
		50 s	18.3	15.3	14.1	16.7	445	200	155	75	2.62	6.01	8.01	-	11.6	-	-	-	71	83	91	98
		120 s	19.2	16.7	14.1	14.9	355	200	155	150	3.06	6.72	8.32	-	15.7	-	-	-	70	82	93	98
G Press cured	0	10 min	17.0	14.8	14.3	12.8	425	190	160	15	1.92	6.59	8.80	-	10.9	-	-	-	61	82	93	99
G Press cured	12 months at room temperature	20 min	16.0	14.6	13.1	13.1	325	235	160	100	2.26	3.80	7.17	13.5	14.0	-	-	-	67	73	80	91

TABLE 8

Mix Number	Period of Storage	Cure Time	Tensile Strength (MN/m²) Ageing Period (weeks)				Elongation at Break (%) Ageing Period (weeks)				Modulus at 100% E (MN/m²) Ageing Period (weeks)				Modulus at 300% E (MN/m²) Ageing Period (weeks)				Hardness (IRH°) Ageing Period (weeks)			
			0	5	8	16	0	5	8	16	0	5	8	16	0	5	8	16	0	5	8	16
H	0	60 s	14.7	8.4	7.8	7.0	480	155	130	145	0.69	3.74	4.40	4.31	6.26	-	-	-	54	76	82	87
		120 s	17.1	11.3	11.0	9.1	485	220	235	185	0.84	2.62	2.70	2.89	7.20	-	-	-	55	72	77	83
		180 s	17.6	11.4	11.8	10.1	460	275	240	200	1.08	1.76	2.50	3.13	8.27	-	-	-	56	68	74	82
H	6 months at room temperature	90 s	17.7	8.4	9.72	9.7	695	170	165	170	0.53	3.14	3.82	3.84	3.61	-	-	-	52	75	83	84
		120 s	18.0	9.8	9.62	10.2	690	190	175	150	0.63	3.22	3.72	4.31	3.92	-	-	-	51	73	82	84
		180 s	18.6	10.6	10.4	9.6	560	215	195	175	1.44	2.96	3.23	3.73	6.56	-	-	-	59	73	82	84
H	12 months at room temperature	180 s	16.4	12.0	13.3	11.6	665	225	220	190	0.31	2.98	3.38	4.39	3.91	-	-	-	50	69	74	73
		240 s	17.9	14.7	15.2	14.4	635	250	285	225	0.41	2.86	2.46	3.97	4.51	-	-	-	54	68	70	71
		300 s	16.6	13.8	13.3	11.6	565	280	240	210	0.45	2.27	3.38	3.74	5.15	-	-	-	56	66	71	70
H	16 weeks at 40°C	90 s	5.9	11.1	-	9.9	930	225	-	185	-	2.74	-	2.34	6.18	-	-	-	40	73	-	84
		130 s	13.3	11.2	-	10.5	795	240	-	185	0.25	2.79	-	3.49	1.845	-	-	-	48	73	-	85
		240 s	18.2	12.1	-	11.5	640	255	-	245	0.81	2.48	-	2.46	4.58	-	-	-	54	71	-	83
H	24 weeks at 40°C	130 s	13.6	-	10.0	8.9	740	-	255	200	0.33	-	2.05	2.84	2.52	-	-	-	48	-	66	71
		180 s	16.0	-	10.8	9.7	670	-	245	210	0.43	-	2.53	2.66	3.50	-	-	-	55	-	68	71
		240 s	19.9	-	11.4	10.1	640	-	250	205	0.53	-	2.71	2.52	4.75	-	-	-	58	-	67	71
H Press cured	0	30 min	13.8	9.0	8.1	9.2	445	205	175	140	0.81	2.50	3.06	5.05	6.97	-	-	-	59	73	85	74
H Press cured	12 months at room temperature	20 min	17.3	8.1	10.1	10.0	740	220	205	190	0.47	2.36	3.12	3.38	3.00	-	-	-	54	61	72	71
		30 min	11.4	10.7	8.7	7.6	490	235	200	175	0.66	2.46	2.88	3.21	4.55	-	-	-	58	83	75	73
H Press cured	16 weeks at 40°C	30 min	14.2	8.6	7.4	8.1	440	210	180	155	0.91	2.34	2.41	3.50	7.30	-	-	-	59	70	81	71
H Press cured	24 weeks at 40°C	Not enough material																				

TABLE 9

Mix Number	Period of Storage	Cure Time	Tensile Strength (MN/m²) Ageing Period (weeks) 0	5	8	16	Elongation at Break (%) Ageing Period (weeks) 0	5	8	16	Modulus at 100% E (MN/m²) Ageing Period (weeks) 0	5	8	16	Modulus at 300% E (MN/m²) Ageing Period (weeks) 0	5	8	16	Hardness (BS°) Ageing Period (weeks) 0	5	8	16
I	0	50 s	8.4	5.6	5.8	5.1	405	165	145	140	0.64	2.48	2.86	2.84	4.66	-	-	-	60	77	83	84
		80 s	7.8	6.1	6.3	5.6	295	160	165	140	0.95	2.62	2.64	2.93	-	-	-	-	63	77	84	85
		120 s	7.8	5.8	5.0	5.8	255	192	165	140	1.31	1.85	2.14	3.07	-	-	-	-	66	73	80	87
I	6 months at room temperature	50 s	12.0	7.6	5.9	6.3	500	175	145	140	0.83	2.95	2.96	3.40	4.64	-	-	-	67	76	85	86
		80 s	9.2	7.2	6.1	6.0	350	170	140	150	1.27	3.02	3.00	3.19	7.09	-	-	-	71	76	86	86
		120 s	10.6	8.1	6.4	6.4	320	190	155	150	1.58	2.79	2.89	3.11	9.50	-	-	-	72	75	85	85
I	12 months at room temperature	30 s	12.5	7.0	7.0	6.9	615	185	170	155	-	1.81	2.68	3.07	2.55	-	-	-	60	72	76	74
		50 s	10.1	8.7	9.1	7.5	355	180	170	150	1.05	3.20	3.75	3.67	7.75	-	-	-	63	72	76	74
		80 s	9.0	8.3	8.9	7.5	280	170	175	150	1.48	3.11	3.57	3.70	-	-	-	-	65	71	76	72
I	16 weeks at 40°C	30 s	12.7	5.6	4.1	4.0	600	140	110	105	0.56	3.53	3.38	3.55	2.99	-	-	-	62	77	85	86
		50 s	11.2	6.0	6.1	6.9	460	150	140	125	1.00	3.32	3.12	4.20	5.34	-	-	-	64	76	86	87
		80 s	9.3	7.1	6.8	6.5	350	165	150	135	1.17	3.11	3.40	4.00	6.98	-	-	-	65	77	86	87
I	24 weeks at 40°C	20 s	9.2	-	7.9	6.3	640	-	185	140	0.53	-	2.61	3.41	1.70	-	-	-	58	-	73	76
		25 s	12.3	-	7.4	6.6	570	-	175	160	0.87	-	2.87	2.82	4.69	-	-	-	61	-	73	77
		30 s	13.8	-	6.5	7.6	650	-	175	165	0.72	-	2.53	3.12	3.58	-	-	-	62	-	73	76
I Press cured	0	5 min	9.3	6.0	5.4	5.8	480	175	155	150	0.68	2.54	2.61	2.82	3.88	-	-	-	65	75	84	72
I Press cured	12 months at room temperature	10 min	7.8	4.3	4.6	5.7	335	135	140	150	1.02	2.78	2.81	2.98	6.65	-	-	-	68	85	73	73
		20 min	6.2	6.2	5.6	5.0	275	175	140	180	1.70	2.47	3.08	3.28	-	-	-	-	70	77	75	76
I Press cured	16 weeks at 40°C	10 min	8.3	5.5	6.6	6.3	360	150	145	140	1.16	3.11	3.00	3.43	6.24	-	-	-	68	77	86	74
I Press cured	24 weeks at 40°C		Not enough material																			

TABLE 10

Mix Number	Period of Storage	Cure Time	Tensile Strength (MN/m²) Ageing Period (weeks) 0	2	5	8	16	Elongation at Break (%) Ageing Period (weeks) 0	2	5	8	16	Modulus at 100% E (MN/m²) Ageing Period (weeks) 0	2	5	8	16	Modulus at 300% E (MN/m²) Ageing Period (weeks) 0	2	5	8	16	Hardness (BS°) Ageing Period (weeks) 0	2	5	8	16
J	0	30 s	16.4	9.4	7.6	8.5	-	740	435	380	375	-	-	0.47	0.52	0.79	-	1.51	4.97	5.13	5.63	-	39	52	[illegible]	[illegible]	-
		90 s	16.3	9.2	8.3	8.7	-	710	435	410	360	-	-	0.53	0.56	0.84	-	1.96	4.87	5.09	6.00	-	42	52	56	[illegible]	-
		130 s	16.6	9.0	9.1	5.6	-	700	435	405	390	-	-	0.35	0.57	0.76	-	1.91	4.42	5.48	5.58	-	41	52	54	[illegible]	-
J	6 months at room temperature	60 s	15.1	11.0	9.1	8.5	-	780	485	430	410	-	-	0.61	0.71	0.89	-	0.99	4.27	4.81	5.01	-	35	52	[illegible]	57	-
		90 s	16.1	8.9	9.1	7.8	-	735	425	440	370	-	-	0.74	0.64	1.04	-	1.55	4.82	4.82	5.65	-	35	54	54	57	-
		130 s	16.5	10.8	8.0	8.2	-	700	465	390	365	-	0.147	0.62	0.73	0.99	-	2.05	4.63	5.30	5.89	-	43	54	56	56	-
J	12 months at room temperature	60 s	14.5	16.2	11.8	11.7	-	765	635	515	450	-	-	-	0.15	0.73	-	0.59	2.78	3.69	5.31	-	38	47	47	55	-
		90 s	15.8	13.5	11.7	8.0	-	730	580	510	390	-	-	0.12	0.12	0.59	-	0.96	2.91	3.90	4.80	-	41	47	47	55	-
		130 s	15.0	12.5	10.7	9.5	-	675	530	465	475	-	-	0.12	0.12	0.63	-	1.33	3.56	4.21	5.20	-	43	48	49	54	-
J	16 weeks at 40°C	40 s	11.5	13.5	9.2	8.6	-	765	540	405	395	-	-	0.42	0.78	0.89	-	0.49	3.76	5.82	5.45	-	32	50	55	54	-
		90 s	16.3	11.5	8.4	7.0	-	755	475	365	335	-	-	0.62	0.87	1.01	-	1.48	4.79	6.12	5.84	-	39	52	55	54	-
		130 s	16.5	11.4	8.0	7.7	-	725	470	355	350	-	-	0.64	0.90	1.00	-	1.76	4.83	6.39	6.00	-	41	52	55	54	-
J	24 weeks at 40°C	90 s	11.5	-	-	12.1	9.9	690	-	-	520	400	-	-	-	0.57	0.785	0.87	-	-	4.00	4.21	38	-	-	48	57
		130 s	14.6	-	-	13.2	9.3	700	-	-	515	455	-	-	-	0.67	0.775	1.39	-	-	4.73	5.47	38	-	-	48	54
		200 s	17.9	-	-	9.6	7.0	690	-	-	430	350	0.118	-	-	0.74	0.745	2.63	-	-	4.98	5.42	42	-	-	52	54
J Press cured	0	15 min	17.2	-	9.5	7.5	7.4	740	-	430	370	340	0.108	-	0.80	0.94	0.726	1.79	-	5.10	5.50	5.83	47	-	[illegible]	54	54
J Press cured	12 months at room temperature	20 min	14.9	-	11.5	8.9	6.7	630	-	475	400	345	0.294	-	0.75	1.24	0.756	2.52	-	4.65	5.56	5.45	47	-	51	54	55
J Press cured	16 weeks at 40°C	20 min	17.0	-	7.8	8.1	6.3	650	-	330	370	340	0.196	-	0.76	0.86	0.654	2.72	-	5.86	5.62	5.89	48	-	56	56	52
J Press cured	24 weeks at 40°C	Not enough material																									

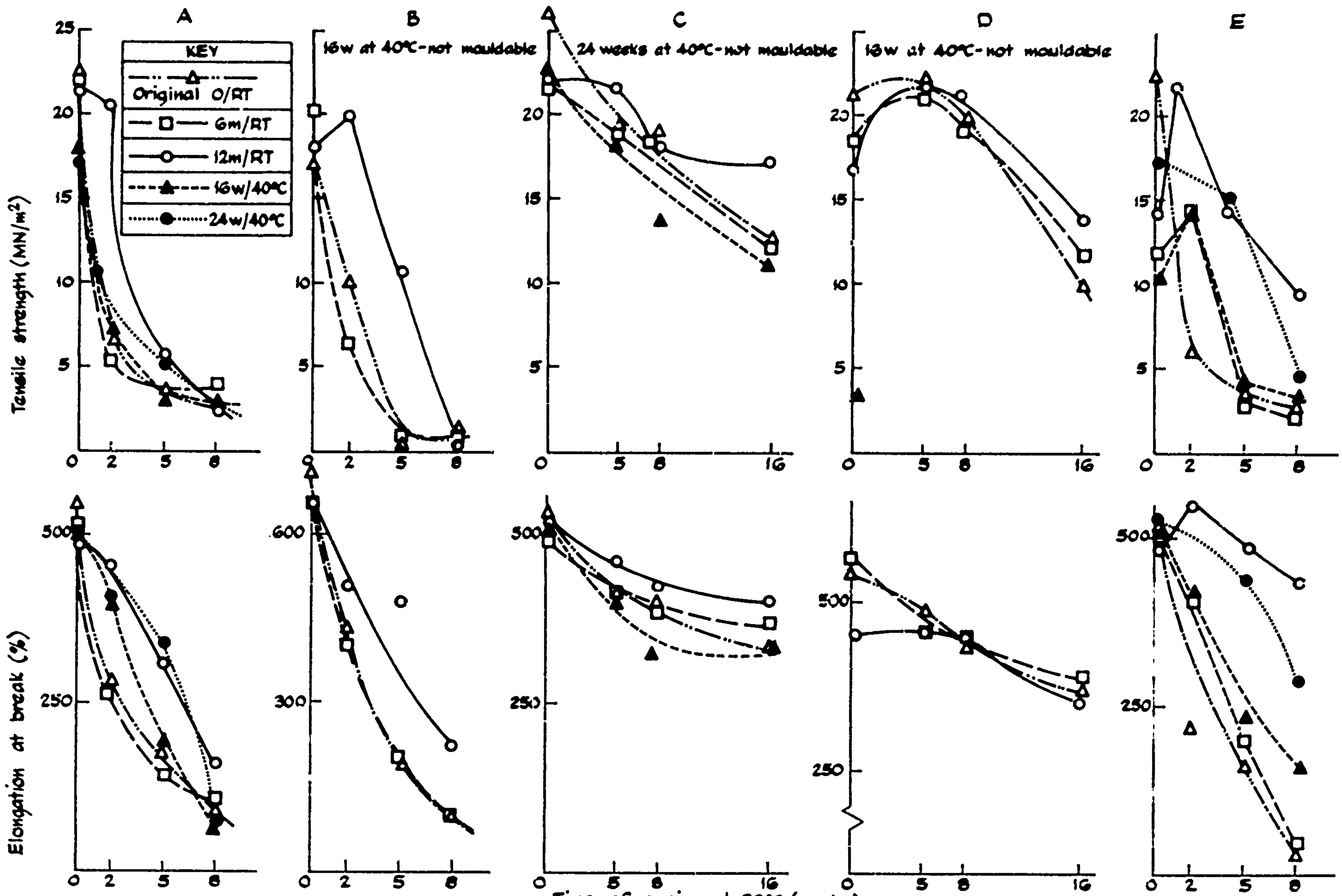

FIG. 1(a) AGEING OF INJECTION MOULDED NATURAL RUBBER VULCANISATES

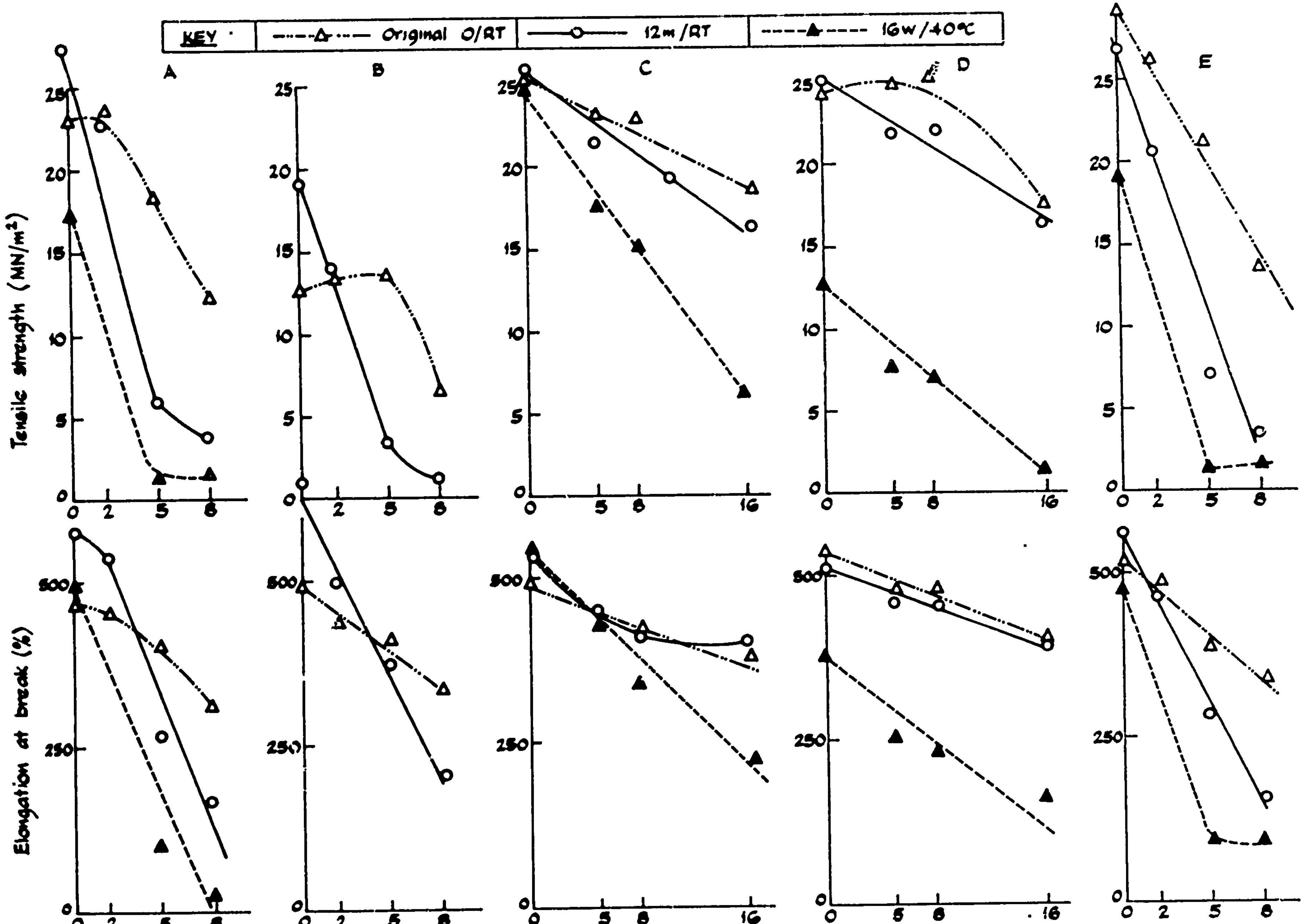

FIG. 2(a) AGEING OF PRESS CURED NATURAL RUBBER VULCANISATES

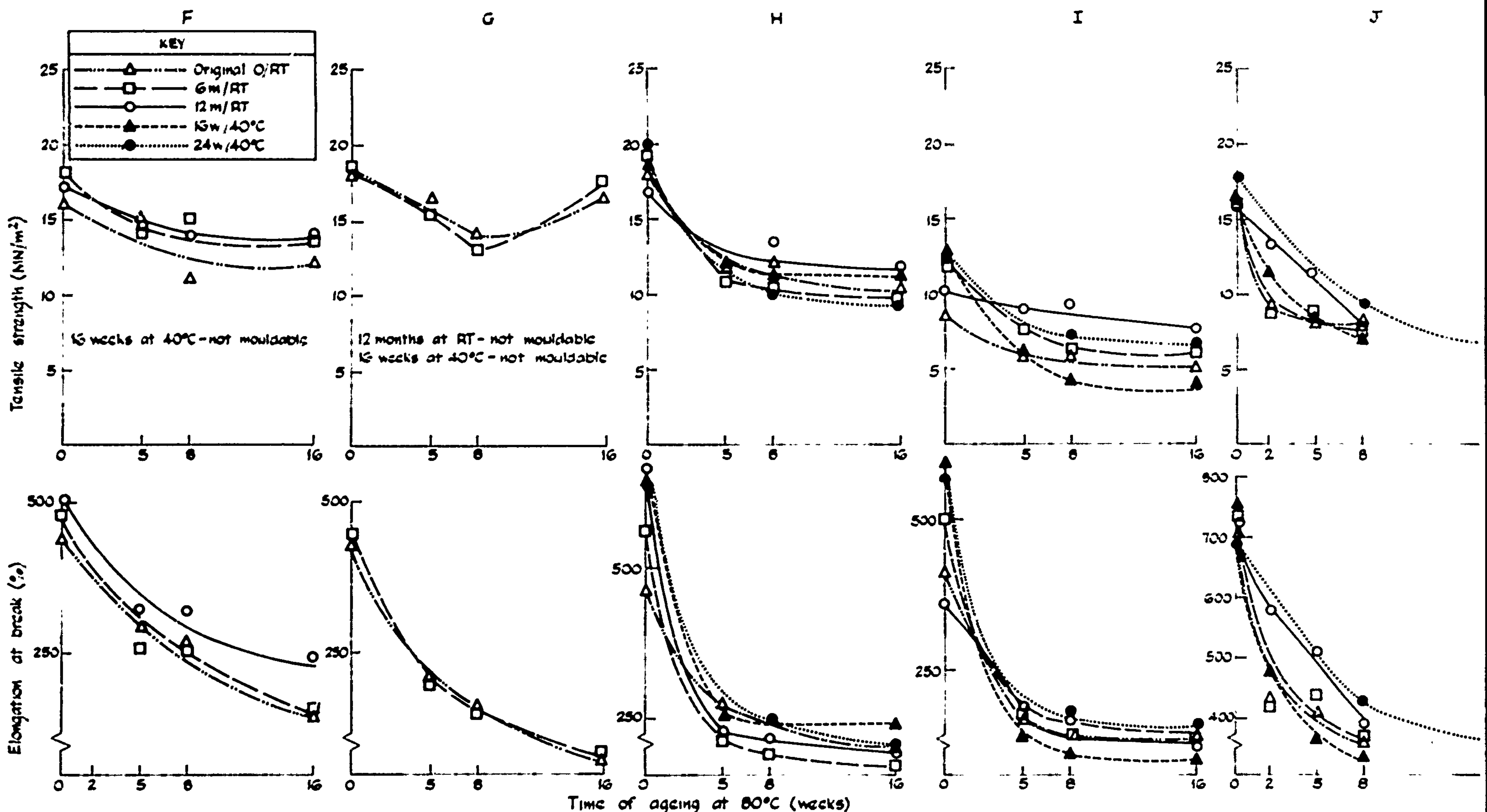

FIG. 1 (b) AGEING OF INJECTION MOULDED SYNTHETIC RUBBER VULCANISATES

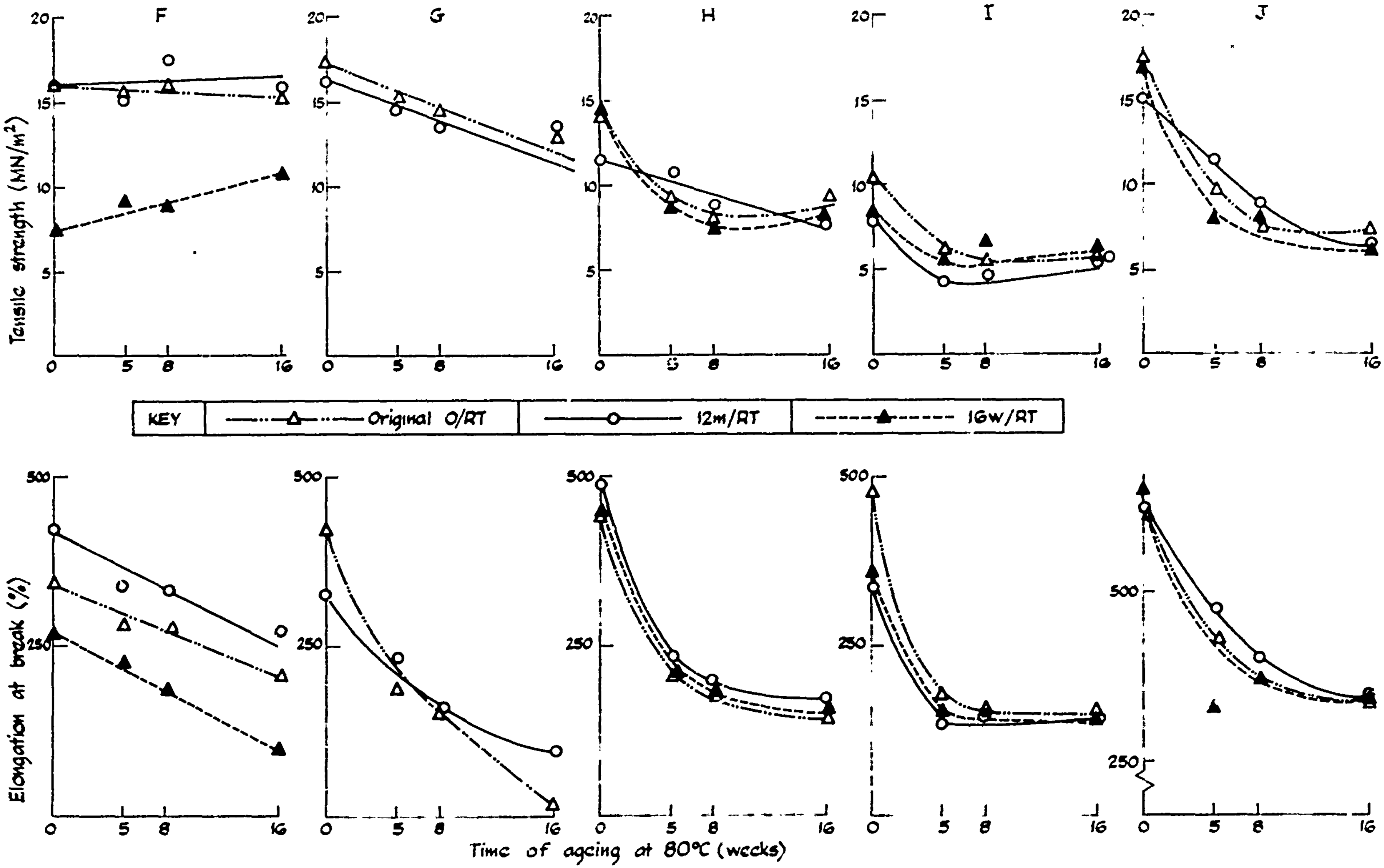

FIG. 2(b) AGEING OF PRESS CURED SYNTHETIC RUBBER VULCANISATES

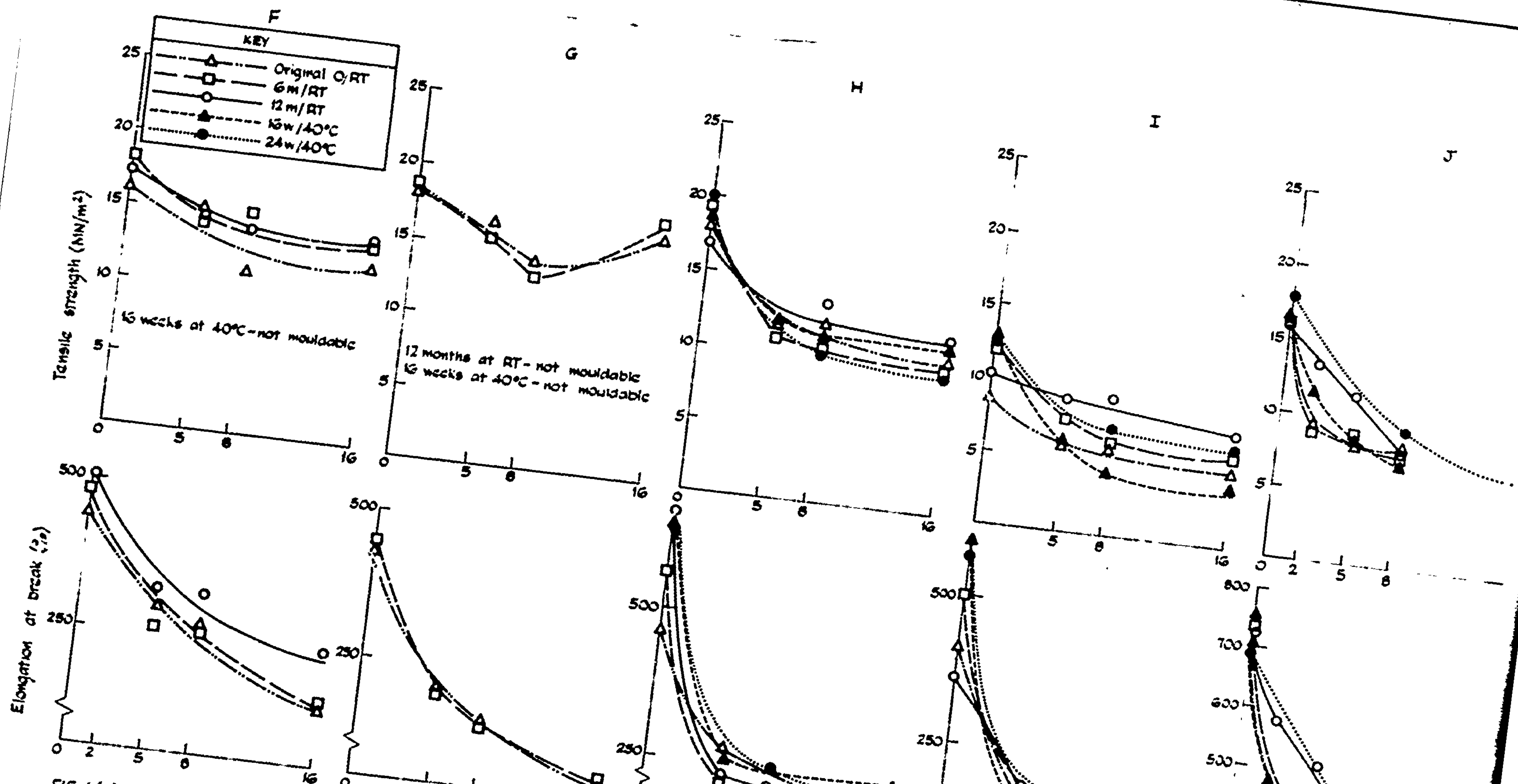

FIG. 1(t)

AGEING OF INJECTION MOULDED SYNTHETIC RUBBER VULCANISATES

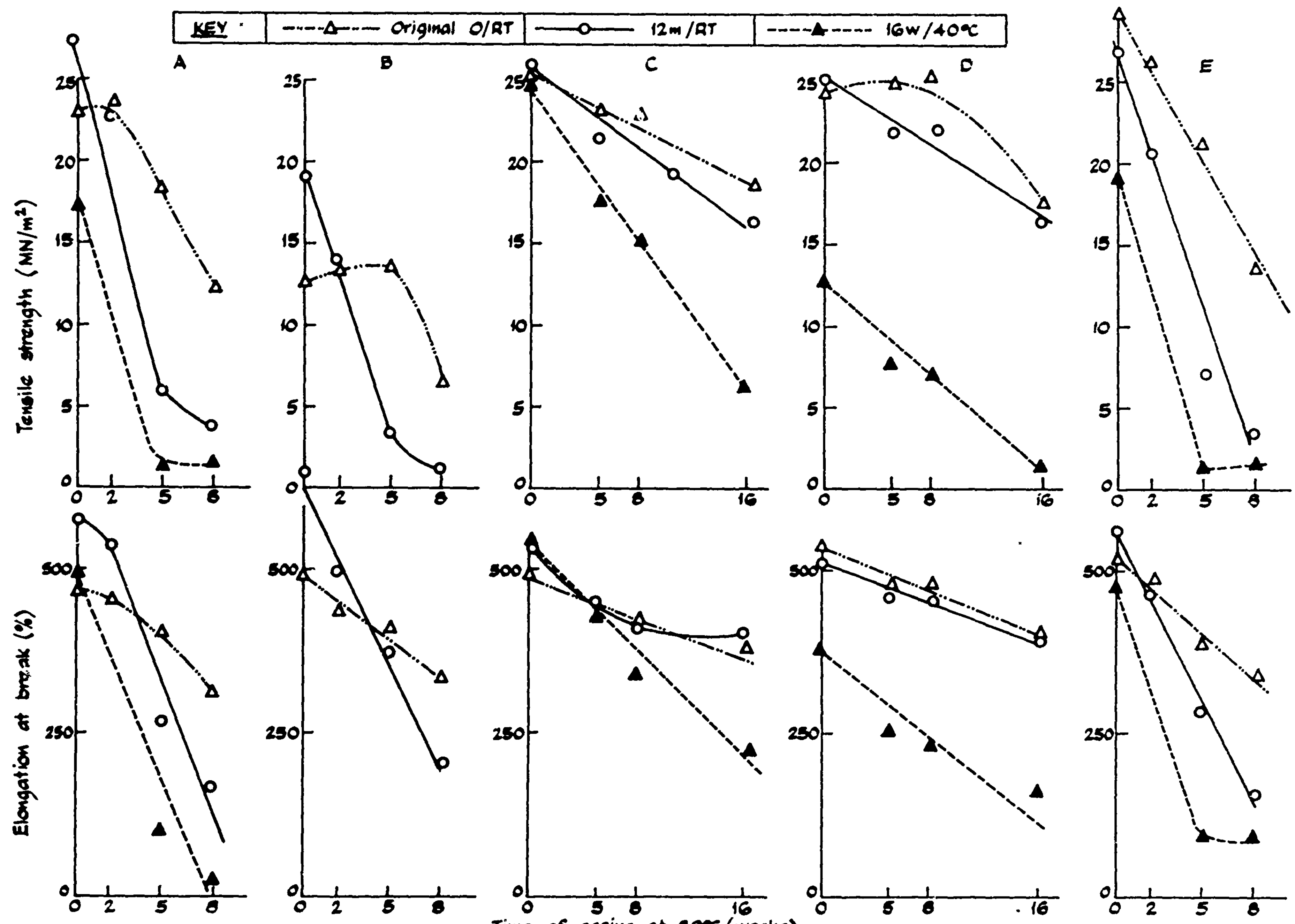

FIG. 2(a) AGEING OF PRESS CURED NATURAL RUBBER VULCANISATES