J. nat. Rubb. Res., **5(3)**, 182-198

The Mooney Viscosity of Raw Natural Rubber

G.M. BRISTOW*

Non-standard Mooney test parameters have been derived for a range of raw natural rubber grades in an attempt to obtain information other than a simple assessment of viscosity. Increasing the pre-heat time to 5 min and observing the torque–time relation over 1–4 min give readings which prove characteristics of the source materials used in the production of the sample and can distinguish therefore between samples of latex grade (e.g. SMR L) and field coagulum grade (e.g. SMR 10) rubbers.

The potential use of the standard Mooney viscometer to provide more information on raw natural rubber than *ML*1 + 4, 100°C, a simple measure of viscosity, has been noted recently[1]. Such information can be derived from four features:

- The overall shape of the torque-time relation during a test of perhaps 5 min
- A special aspect of the above feature; the extent of the initial stress overshoot as evinced in a maximum torque
- The influence of temperature on the above two features
- Relaxation of torque following cessation of rotor movement at the conclusion of the 4-min test time.

For the effective use of the first three features, to a lesser extent the last feature, the initial pre-heat time must be sufficient to allow the rubber to attain the test (platen) temperature. A period of 5 min, rather than the conventional 1-min pre-heat, has been found to be adequate[1]. This is the only difference to the standard test procedure. As has been shown previously[1], the parameters derived in tests of this type are dependent on the grade (or rather production route) of the rubber in question. Furthermore, the information obtained clearly relates to the rheological characteristics of the rubber and hence inevitably to the processing behaviour noted by the end-user. Although the information is not immediately expressable in terms of basic material properties, consistency in the various Mooney parameters would nevertheless be expected to be indicative of consistency in processing behaviour. The potential practical utility of such parameters is therefore obvious.

The Mooney viscometer was first developed some fifty years ago and has been in continual use ever since. Not surprisingly, therefore, observations relating to the above points have been made in the past, although there seem to have been no systematic studies and certainly no attempt to relate these factors to the production route used for raw natural rubber. The studies reported here have, of course, been stimulated by the current pressure on elastomer producers to supply materials, including natural rubber, with *consistent* processing behaviour in the end-user's factory. As noted above, in practice such *consistency* should be achievable without the need to specify a wide range of fundamental rheological parameters.

In this paper, data for Mooney torque as a function of running time (0–4 min) over a range of temperatures are reported for samples of various grades of natural rubber. Relaxation of torque at the conclusion of the test, is not considered but will be the subject of a subsequent publication.

*Malaysian Rubber Producers' Research Association, Brickendonbury, Hertford SG13 8NL, United Kingdom

EXPERIMENTAL

The samples used were, in the main, normal commercial materials obtained from consumers' stocks. In all cases, before testing the bale rubber was blended and homogenised using the two-roll mill procedure specified for the testing of SMR[2]. A standard Mooney viscometer fitted with a pen recorder was used. As noted above, the rubber was pre-heated in the machine for 5 min and then the torque-time trace recorded over the period 0-4 min. Values of the initial torque, ML_{max}, and torque after 4 min, ML (5 + 4) were obtained from this trace.

RESULTS

Representative torque-time traces for three samples each of SMR L, SMR CV and SMR 10 are given in *Figures 1, 2* and *3* respectively While, in general, torque at a given time usually decreases with temperature, as would be expected, there are much more complex variations in the shape of the torque-time plot. The most obvious feature is the consistency in shape, including the temperature dependence, between the three samples for each grade and the clear differences in shape between the grades. The shape is in essence a 'finger print' of the grade, albeit a qualitative one, and therefore a sample not displaying the appropriate finger print would be suspect. Such complex effects can only result from the interplay of several factors probably having different temperature coefficients, and at this stage a detailed analysis has not proved possible. It should be noted however that an essentially empirical analysis of the torque-time relation of a black-filled system has been proposed by Turner[3]. Further analysis of the data obtained is therefore restricted to a consideration of the parameters ML_{max} and $ML(5 + 4)$ noted above, together with $R = ML_{max}/ML(5 + 4)$ obtained at 70°C – 110°C. Data of this type for a wide range of samples, including those of *Figures 1, 2* and *3*, are given in *Tables 1-5*.

It should be noted perhaps that, while the 4-min test time has been adopted in conformity with conventional practice, the torque or 'viscosity' observed at the 4-min stage is not necessarily an equilibrium value and certainly not an intrinsic material parameter. (Conventionally a 1-min pre-heat, rather than the 5 min adopted here, is used.) Previous studies[1] have shown that, at 100°C at least, the torque falls over the subsequent 4–60-min period at a rate which suggests that oxidation may be a factor. Furthermore, inspection of *Figures 1, 2* and *3* suggests that the proximity of the 4-min torque to an equilibrium or 'true' value almost certainly depends on the temperature of the test.

Examination of *Tables 1-5* reveals that the 'equilibrium' torque or viscosity , $ML(5 + 4)$, decreases with increasing temperature from 70°C to 90°C, as would be expected. At higher temperatures, however, the 'viscosity' is constant or increases. While such an increase might reflect the minor incursion of some crosslinking process, this seems improbable under the conditions of time and temperature involved. It is more likely that the increase is spurious and simply reflects the non-equilibrium nature of the $ML(5 + 4)$ values. The rapid increase in viscosity below *ca* 90°C is paralleled by similar effects noted previously in capillary flow measurements[4]. Representative data for two samples each of the latex grade SMR L and field coagulum grade SMR 10 are plotted in *Figure 4*. The overall dependence of viscosity on temperature for the two grades is broadly similar, including the rise in viscosity above 90°C. There is, however, a tendency for the viscosity increase below 90°C to be more rapid for the latex material. Here again, this is consistent with capillary flow data. The trend is quantified by the grade mean values given in *Table 6*. It is to be noted that the relative increase in viscosity at lower temperatures is particularly marked for the CV grade.

The initial maximum torque, ML_{max} is a more interesting parameter. The dependence of the ratio R [$= ML_{max}/ML(5 + 4)$] on natural rubber grade has been noted previously[1]. More extensive data are presented here in *Tables 1-5*. Unlike $ML(5 + 4)$, ML_{max} shows

COPYRIGHT © MALAYSIAN RUBBER BOARD

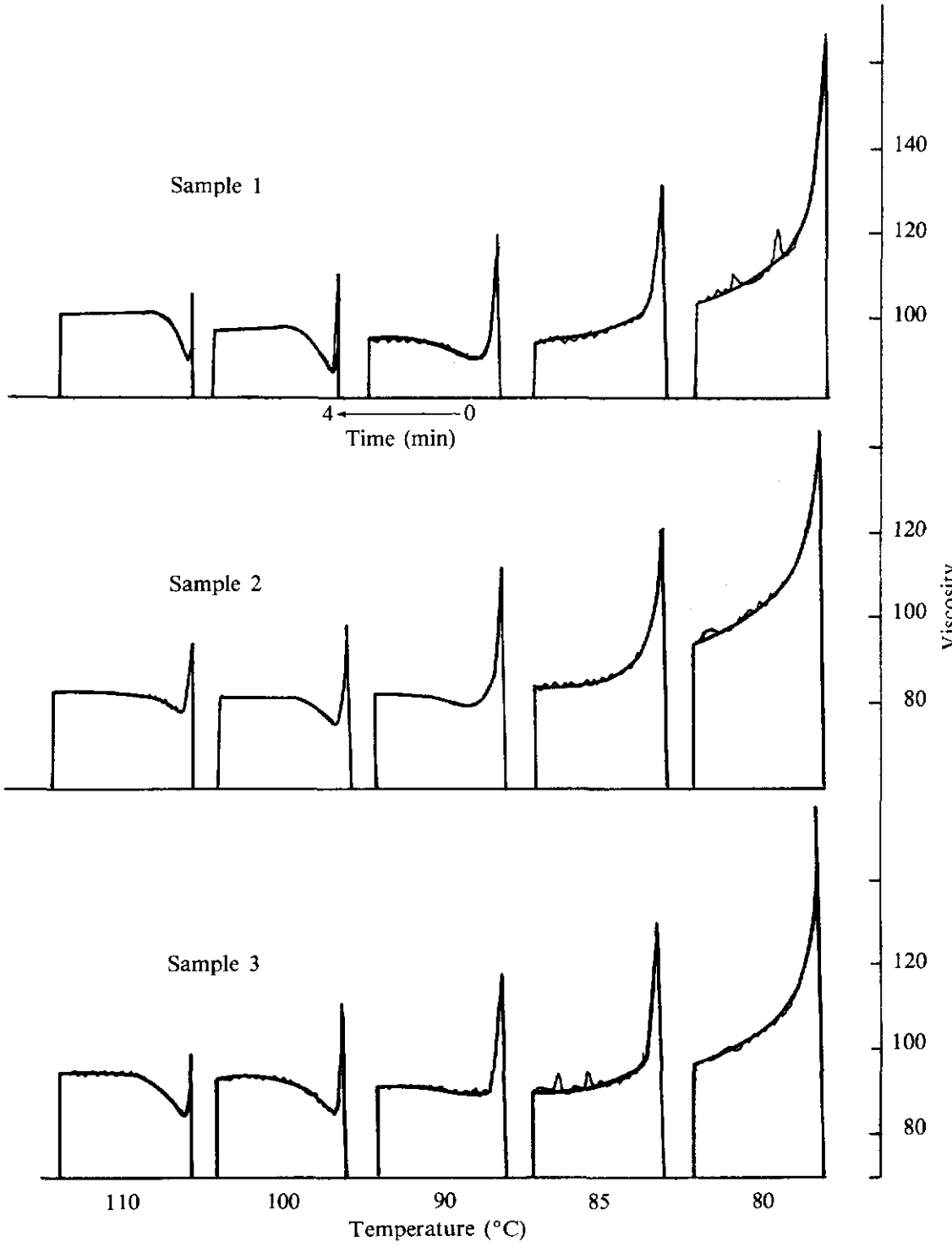

Figure 1. Mooney viscosity as a function of time (0–4 min) after 5-min pre-heat at 80°C–110°C for three samples of SMR L.

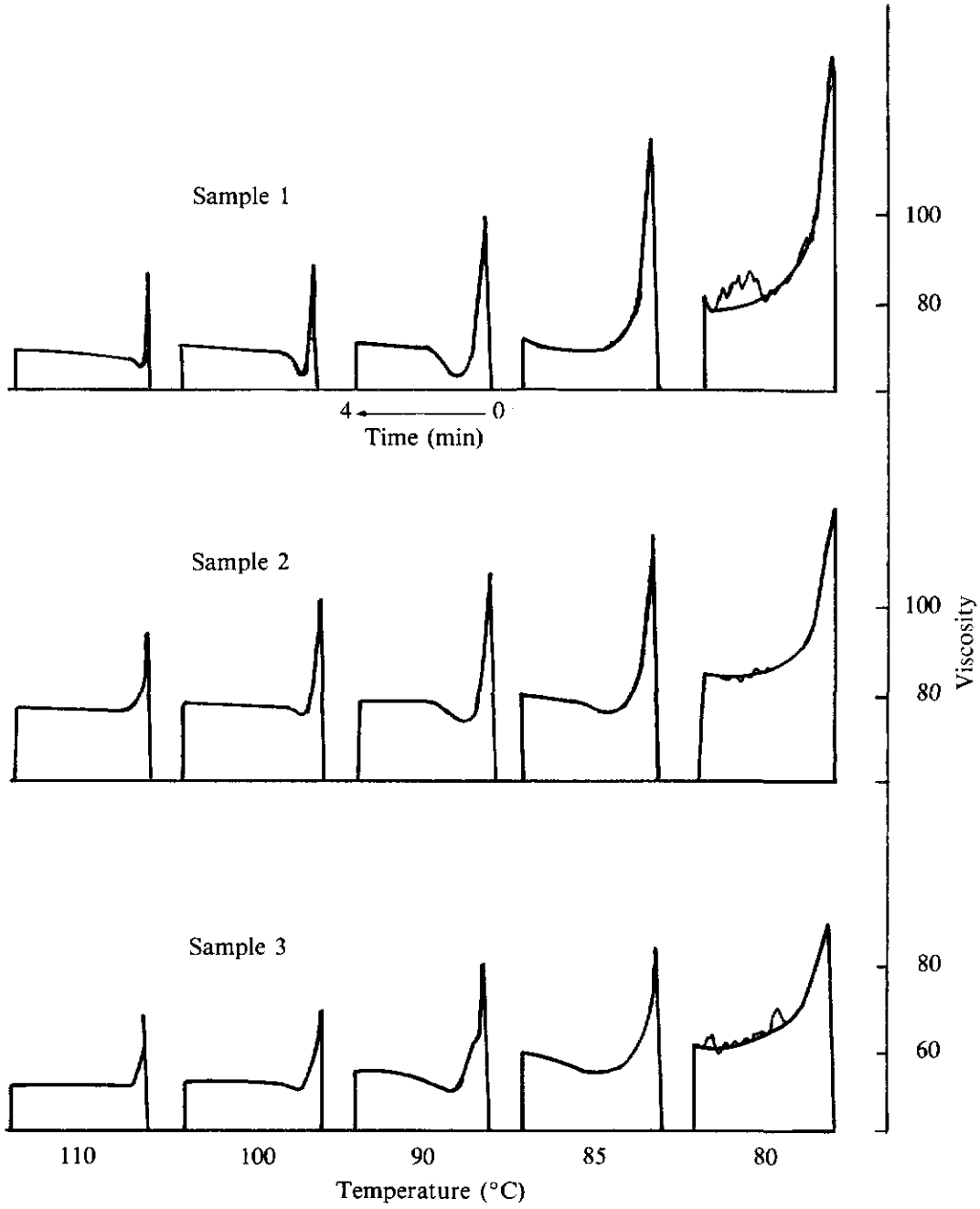

Figure 2. Mooney viscosity as a function of time (0–4 min) after 5-min pre-heat at 80°C–110°C for three samples of SMR CV.

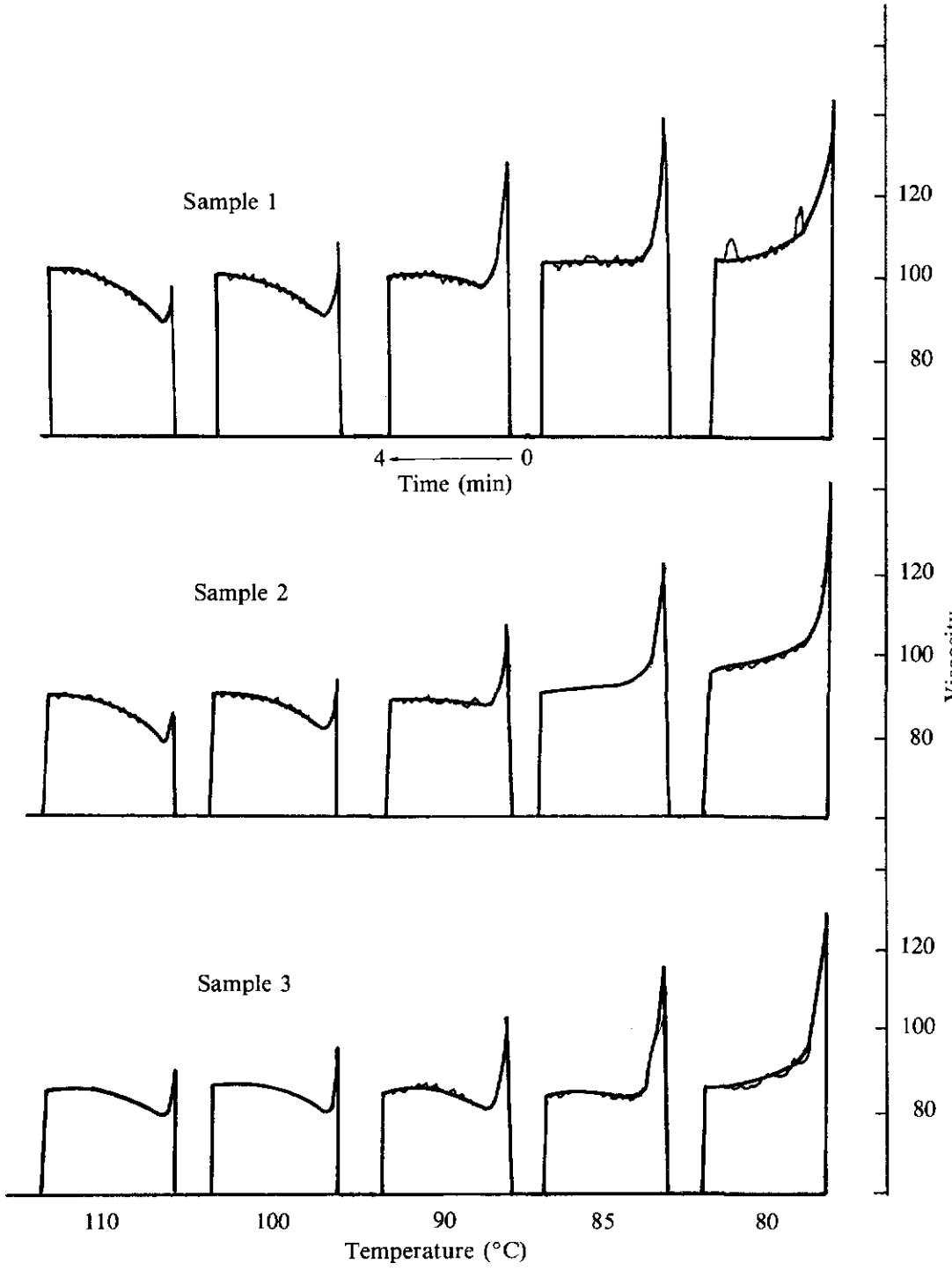

Figure 3. Mooney viscosity as a function of time (0–4 min) after 5-min pre-heat at 80°C–110°C for three samples of SMR 10.

TABLE 1. MOONEY DATA AT 75°C–110°C FOR EIGHT SAMPLES OF SMR L

Temp. (°C)	Sample 1			Sample 2			Sample 3			Sample 4		
	Mx	4′	R	Mx	4′	R	Mx	4′	R	Mx	4′	R
110	99.5	94.5	1.05	90.5	86.5	1.05	95	83	1.14	86	87	0.99
100	108.5	93	1.17	97	87	1.11	99.5	82	1.21	90.5	88	1.03
90	129	90.5	1.43	102.5	85.5	1.20	112	82.5	1.36	98	86	1.14
85	131	91	1.44	111.5	85.5	1.35	121	84	1.44	104.5	84	1.24
80	158.5	98.5	1.61	125	86	1.45	143.5	94	1.53	117.5	83.5	1.41
75	186	118	1.58	144	101	1.43	166.5	111	1.50	132.5	87	1.52
70	>200	143	–	183	121	1.51	195	133	1.47	164.5	105	1.57

TABLE 1. MOONEY DATA AT 75°C-110°C FOR EIGHT SAMPLES OF SMR L (Contd.)

Temp. (°C)	Sample 5			Sample 6			Sample 7			Sample 8		
	Mx	4′	R	Mx	4′	R	Mx	4′	R	Mx	4′	R
110	95.5	84	1.14	85	81	1.05	105	100.5	1.04	101	85.5	1.18
100	100	82.5	1.21	88	82	1.07	111	97.5	1.14	110.5	86	1.29
90	116.5	81.5	1.43	96.5	79.5	1.21	120	95.5	1.26	136	86	1.58
85	120	82	1.46	104	80	1.30	132	94	1.40	150	90	1.67
80	145	91	1.59	118.5	82	1.45	167	104	1.61	180	102	1.77
75	176	116	1.52	132	92.5	1.43	186	125	1.49	>200	122	-
70	>200	144	-	158	112	1.41	>200	150	-	-	-	-

Mx = Initial maximum torque
4′ = ML(5 + 4)
R = Mx/ML(5 + 4)

TABLE 2. MOONEY DATA AT 75°C–110°C FOR ELEVEN SAMPLES OF TSR 10

Temp. (°C)	Sample 1			Sample 2			Sample 3			Sample 4		
	Mx	4′	R	Mx	4′	R	Mx	4′	R	Mx	4′	R
110	99.5	101.5	0.98	109	106.5	1.02	90	92.5	0.97	89	86	1.03
100	108	99	1.09	120	105	1.14	96	93	1.03	94	86.5	1.09
90	121	96.5	1.26	138	104.5	1.32	109	91	1.20	102	85.5	1.19
85	131	96	1.36	149.5	108	1.38	119	92	1.29	116.5	85	1.37
80	145	99	1.46	173	112.5	1.54	132	96	1.39	129	86	1.50
75	178	109	1.63	191	123.5	1.55	149	101	1.48	173	91	1.90
70	>200	127	-	204	142	1.44	181	118	1.52	200	101.5	1.97

TABLE 2. MOONEY DATA AT 75°C-110°C FOR ELEVEN SAMPLES OF TSR 10 (Contd.)

Temp. (°C)	Sample 5			Sample 6			Sample 7			Sample 8		
	Mx	4′	R	Mx	4′	R	Mx	4′	R	Mx	4′	R
110	96	98.5	0.97	97.5	98.5	0.99	113.5	106.5	1.07	93.5	100	0.94
100	105.5	98	1.08	100.5	99	1.02	126.5	107.5	1.18	100	101	0.99
90	121	97	1.25	108	97	1.11	153.5	108	1.42	112.5	98	1.15
85	130.5	98	1.33	120	99	1.21	153	110	1.39	119	100	1.19
80	142	101.5	1.40	128	101.5	1.26	191.5	120	1.60	122	100	1.22
75	176	111	1.59	152.5	111	1.37	>200	135.5	-	146	109	1.34
70	198.5	126	1.58	188	129	1.46	>200	151.5	-	169	117.5	1.44

TABLE 2. MOONEY DATA AT 75°C-110°C FOR ELEVEN SAMPLES OF TSR 10 (Contd.)

Temp. (°C)	Sample 9			Sample 10			Sample 11		
	Mx	4′	R	Mx	4′	R	Mx	4′	R
110	92	94.5	0.97	100.5	103	0.98	87	91.5	0.95
100	102	95	1.07	110	101.5	1.08	95	91	1.04
90	109.5	92	1.19	128	100	1.28	108	90.5	1.19
85	128	93	1.38	139	102	1.36	123	92	1.34
80	141	96	1.47	144	105.5	1.36	132	96.5	1.37
75	154	106.5	1.45	171.5	114	1.50	149	107	1.39
70	184.5	125	1.48	>200	131	-	178	123	1.45

Mx = Initial maximum torque
4′ = ML(5 + 4)
R = Mx/ML(5 + 4)

TABLE 3. MOONEY DATA AT 75°C-110°C FOR TEN SAMPLES OF TSR 20

Temp. (°C)	Sample 1			Sample 2			Sample 3			Sample 4			Sample 5		
	Mx	4′	R	Mx	4′	R	Mx	4′	R	Mx	4′	R	Mx	4′	R
110	83	87	0.95	92	93.5	0.98	105.5	99.5	1.06	92	85.5	1.08	80	83	0.96
100	87	87	1.00	101.5	93.5	1.09	117	98	1.19	96	86.5	1.11	86	89.5	0.96
90	101.5	88	1.15	118	92.5	1.28	138	99	1.39	109	86.5	1.26	95	90.5	1.05
85	109.5	86	1.27	130.5	94	1.39	151.5	104	1.46	121	89.5	1.35	100	91.5	1.09
80	116.5	87	1.34	144	98	1.47	182	111	1.64	136	94	1.45	105	90	1.17
75	134	96	1.40	169.5	107	1.58	>200	123	–	172	106	1.62	125	92.5	1.35
70	168.5	107	1.57	195	121	1.61	>200	140	–	>200	130	–	138	95	1.45

TABLE 3. MOONEY DATA AT 75°C–110°C FOR TEN SAMPLES OF TSR 20 (Contd.)

Temp. (°C)	Sample 6			Sample 7			Sample 8			Sample 9			Sample 10		
	Mx	4′	R	Mx	4′	R	Mx	4′	R	Mx	4′	R	Mx	4′	R
110	94	96	0.98	77	83	0.93	79.5	80.5	0.99	91	95.5	0.95	73	82	0.89
100	99.5	97	1.03	84	88	0.95	85.5	82.5	1.04	94.5	97	0.97	77.5	83	0.93
90	119	98.5	1.21	91.5	89	1.03	94	84	1.12	110	96.5	1.14	83.5	84	0.99
85	124	99	1.25	94	89	1.06	101	85	1.19	118	96.5	1.22	89.5	84.5	1.06
80	136	105	1.30	107.5	89.5	1.20	107	86	1.24	132	99	1.33	93.5	84.5	1.11
75	169	115	1.47	123.5	91	1.36	126	94	1.34	160	106	1.51	103.5	86	1.20
70	192	127.5	1.51	144.5	96	1.51	140.5	104	1.35	187.5	119	1.58	116.5	92	1.27

Mx = Initial maximum torque
4′ = ML(5 + 4)
R = Mx/ML(5 + 4)

TABLE 4. MOONEY DATA AT 75°C–110°C FOR FIVE SAMPLES OF RSS-CV[a]

Temp. (°C)	Sample 1			Sample 2			Sample 3			Sample 4			Sample 5		
	Mx	4′	R	Mx	4′	R	Mx	4′	R	Mx	4′	R	Mx	4′	R
110	76	57	1.33	80	64.5	1.24	106	90	1.18	77	59	1.31	92	75.5	1.22
100	79	58.5	1.35	84	65.5	1.28	114	90	1.27	78	59.5	1.31	97.5	75	1.30
90	87.5	60.5	1.45	92.5	66	1.40	138	89.5	1.54	86.5	60	1.44	113	75.5	1.50
85	96	62	1.55	95.5	68	1.40	151	91.5	1.65	93	61.5	1.51	129	76.5	1.69
80	111.5	63.5	1.76	118	70	1.69	191.5	101.5	1.89	101	62.5	1.62	141	84	1.68
75	126	82	1.54	129	86	1.50	>200	125	–	124	76	1.63	184	107	1.72

[a]Monoclonal samples (*cf* Reference 5)
Mx = Initial maximum torque
4′ = ML(5 + 4)
R = Mx/ML(5 + 4)

TABLE 5. MOONEY DATA AT 75°C–110°C FOR FIVE SAMPLES OF SMR CV[a]

Temp. (°C)	Sample 1			Sample 2			Sample 3			Sample 4			Sample 5		
	Mx	4′	R	Mx	4′	R	Mx	4′	R	Mx	4′	R	Mx	4′	R
110	68	52	1.31	76	58	1.31	107.5	85.5	1.26	67	53.5	1.25	88	71	1.26
100	70	53.5	1.31	83.5	58.5	1.43	115.5	86	1.34	71.5	54	1.32	90	71	1.27
90	80.5	55.5	1.46	88.5	59.5	1.49	137	86.5	1.58	78	55	1.42	100.5	71	1.42
85	84	57.5	1.46	97.5	61	1.60	162	88	1.84	82.5	56	1.47	120	72	1.67
80	90	61	1.48	103.5	65	1.59	191.5	101	1.90	89.5	57	1.57	134.5	79	1.70
75	114	77	1.48	121	83	1.46	>200	128	–	93.5	62	1.51	162	100	1.62

[a]Monoclonal samples (*cf* Reference 5)
Mx = Initial maximum torque
4′ = ML(5 + 4)
R = Mx/ML(5 + 4)

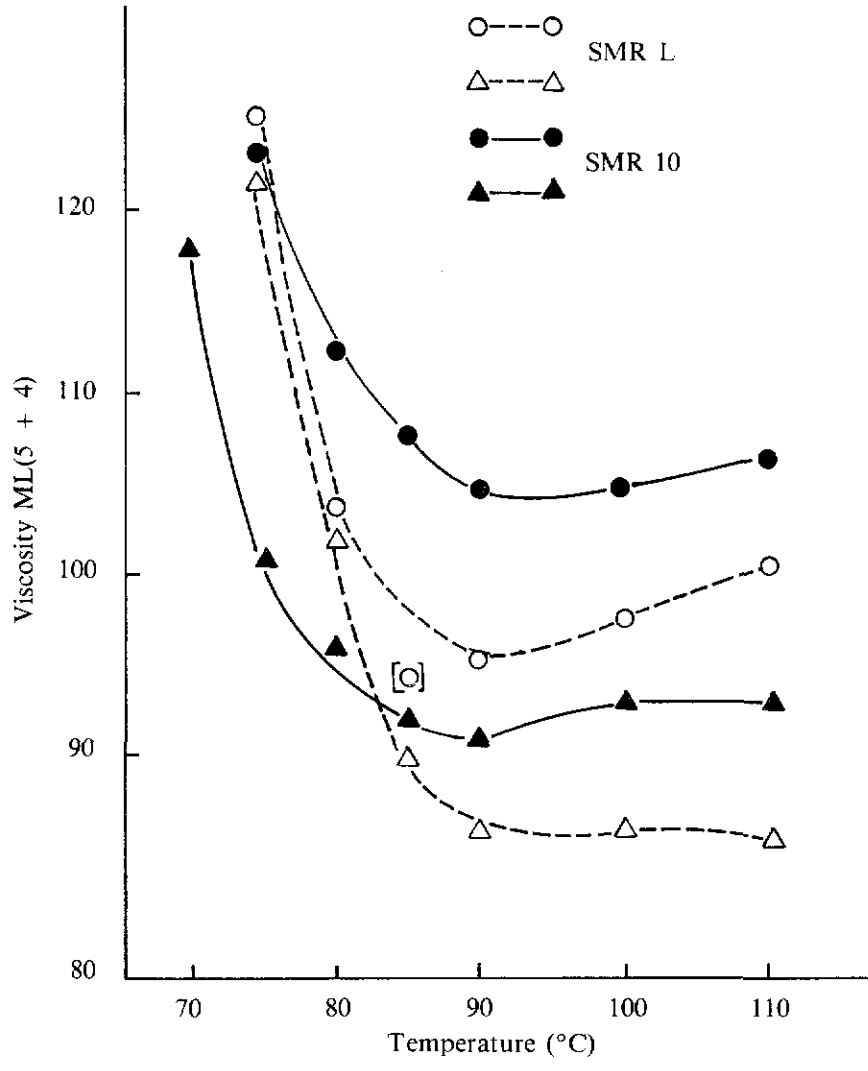

Figure 4. Temperature dependence of ML(5 + 4) for samples of SMR L and SMR 10.

TABLE 6. GRADE MEAN VALUES OF ML(5 + 4)75°C/ML(5 + 4)100°C

Grade	No	Mean	sd
SMR L	8	1.25	0.15
TSR 10	11	1.13	0.06
TSR 20	10	1.13	0.08
SMR CV	5	1.38	0.13
RSS CV	5	1.36	0.06

a monotonic dependence on temperature over the whole range 70°C–110°C and this dependence is large enough to offset small changes in $ML(5 + 4)$ so that the ratio, R, shows a similar temperature dependence. As would be expected, ML_{max} depends upon $ML(5 + 4)$. The ratio R, measured at 100° C, has been noted previously as discriminating between latex and cup lump natural rubber grades. This discrimination is confirmed by the data of *Table 7* which shows further that a

TABLE 7. GRADE MEAN VALUES OF $R = ML_{max}/ML(5 + 4)$ at 75°C–100°C

Temp. (°C)	SMR L 8 samples		TSR 10 11 samples		TSR 20 10 samples		RSS CV 5 5 samples		SMR CV 5 samples	
	Mean	sd	Mean	sd	Mean	sd	Mean	sd	Mean	sd
110	1.08	0.07	0.99	0.04	0.98	0.06	1.26	0.06	1.27	0.05
100	1.15	0.08	1.07	0.05	1.03	0.08	1.30	0.03	1.33	0.06
90	1.33	0.15	1.22	0.09	1.16	0.12	1.47	0.05	1.47	0.07
85	1.41	0.13	1.33	0.07	1.23	0.14	1.56	0.12	1.61	0.16
80	1.55	0.12	1.42	0.11	1.33	0.16	1.73	0.10	1.65	0.16
75	1.50	0.05	1.48	0.10	1.43	0.13	–	–	–	–

similar pattern of behaviour is evident at temperatures other than 100°C. However, normalisation of the data to give the values recorded in *Table 8* shows that improved discrimination cannot be achieved by testing at temperatures other than 100°C.

All the grades tested show considerable within-grade scatter, with coefficients of variation of typically 3%–8%. Such variability clearly limits the use of R as a discriminating test parameter and the source of the scatter is therefore of some interest. The samples tested were commercial materials, for which the precise details of production procedure or even source materials, are unknown. It could well be, therefore, that the value of R is influenced by production factors other than the basic latex or cup lump nature of the source material, such as maturation time of a latex coagulum or time and temperature of drying. This could only be determined using special materials produced by very well characterised procedures.

Variability, or rather apparent variability, also could arise because of the very simple analytical approach adopted. Though ML_{max} clearly must be dependent on $ML(5 + 4)$, there is no physical basis for a simple proportionality, and hence no such basis for a constant value of $R = ML_{max}/ML(5 + 4)$ for samples of one grade differing in viscosity. More generally, R^*

TABLE 8. VALUES OF R RELATIVE TO THE MEAN VALUE AT ONE TEMPERATURE

Temp. (°C)	Mean R	R relative to mean				
		SMR CV	RSS CV	SMR L	TSR 10	TSR 20
110	100	114	113	97	89	88
100	100	113	111	98	91	88
90	100	111	111	100	92	87
85	100	113	109	99	93	86
80	100	107	113	101	92	87

TABLE 9. ANALYSIS OF VARIATION OF ML_{max} WITH ML (5 + 4)

Rubber	Temp. 80°C			Temp. 100°C		
	x	Coeff.[a] corr.	coeff.[b] var. (%)	x	Coeff.[a] corr.	Coeff.[b] var. (%)
SMR L	1.00	0.97	7.6	1.00	0.60	7.3
8 samples	1.67	0.98	3.7	0.83	0.59	1.1
TSR 10	1.00	0.85	8.1	1.00	0.88	5.0
11 samples	1.26	0.82	7.6	1.32	0.88	4.5
TSR 20	1.00	0.92	12.1	1.00	0.79	8.0
10 samples	1.97	0.91	7.8	1.46	0.80	3.0
SMR CV	1.00	0.99	9.8	1.00	0.98	4.5
5 samples	1.39	0.99	3.1	0.97	0.98	4.4
RSS CV	1.00	0.99	6.0	1.00	0.99	2.4
5 samples	1.20	0.99	4.1	0.73	0.82	3.3

[a]Between ML_{max} and [ML(5 + 4)]* for x = 1 or rms value
[b]For $R = ML_{max}/ML(5 + 4)$ or $R^* = ML_{max}/[ML(5 + 4)]^*$

could be equated to $ML_{max}/[ML(5 + 4)]^x$ with x determined by the best fit for a constant value of R^*. The results of such an analysis, which for the data obtained at 80°C and 100°C are given in *Table 9*, are disappointing. While variability in R^* is in many, but not all, cases much less than in R, the values of x required do not conform to any regular pattern and are too variable to be indicative of a viable interpretation of the data.

CONCLUSIONS

The data discussed here were obtained using a standard Mooney 'viscometer' but with 5-min rather than 1-min pre-heating of the test sample. The shape of the torque *versus* time relation over the period 1 min to 4 min and its dependence on temperature has been found to be dependent on the overall process history of the material. Latex rubber (SMR L) and field coagulum (SMR 10) show quite different responses. Quantitative analysis has been restricted to the dependence of viscosity, $ML(5 + 4)$, on temperature; and, the relation between initial maximum torque or stress-overshoot and $ML(5 + 4)$. Both these features appear to depend on the grade. Considerable scatter in behaviour is evident between the several samples of each grade. It is envisaged that this scatter could stem, at least in part, from differences in the detailed process history, such as in the drying temperature. Since the samples studied were normal commercial materials, such differences are quite likely to have been present.

Date of receipt: January 1990
Date of acceptance: April 1990

REFERENCES

1. BRISTOW, G.M. AND SEARS, A.G. (1987) The Use of Novel Parameters in the Assessment of Natural Rubber Processability. *J. nat. Rubb. Res,* **2(1)**, 15.
2. RUBBER RESEARCH INSTITUTE OF MALAYA (1970) RRIM Test Methods for Standard Malaysian Rubbers. *SMR Bull. No. 7.*
3. TURNER, D.M. (1979) Private Communication. Avon Rubber Co. Ltd., Melksham, U.K.
4. BRISTOW, G.M. (1985) Capillary Flow of Raw Natural Rubber, *NR Technol.* **16**, 76.