Statistical Evaluation of Technical Classification Strain Results Obtained at Six Testing Stations

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Technical Classification (TC) strain is a measure of the rate of cure of rubber. The strain results at six testing stations are compared for the years 1958–62. The test errors or reproducibility variation for routine strain (normal procedure) for the different stations do not exceed 5% coefficient of variation. After correction for Mooney viscosity 40 and cross-sectional area, the test error is decreased and the coefficient of variation falls below 3%. The masterbatches of all the stations are similar. For the routine TC strain values the stations in Malaya and Ceylon agree closely, the Indonesian stations tend to give strain values 2.6 units less, and the Vietnam station underestimates by 6.7 units on the average. This may lead to inconsistencies and classification difficulties if constant marginal limits are maintained by these stations for the three types (Red, Yellow and Blue) of rubber.

The station differences are established, but to a smaller extent, even when strain values are corrected for variation in Mooney viscosity and for cross-sectional area of the test piece.

The TC scheme for the technical classification of natural rubber was put forward in 1949 to give consumers some guidance on the behaviour of their raw material during processing and manufacture and also to exclude technically abnormal rubbers. The scheme was not designed to replace the visual grading system; instead the TC symbol was to be superimposed in existing types and grades with the guarantee of a certain technical uniformity within each class.

With international collaboration among the natural rubber producing countries, TC rubber was first marked in 1951 by Malaya, Vietnam, Cambodia and Indonesia. Testing stations were set up in these countries and also in Ceylon and Thailand. The last two, however, never reached the stage of marketing TC rubber.

Certain modifications such as the reduction in the number of classes and alteration in class limits were made during the early years of the operation of the scheme until it was finally stabilised in 1954, since when TC rubber has been marked and exported in three classes: Red, Yellow and Blue Circle. The classification is decided by the strain (or modulus) test and gives an indication of the rate of cure of the rubber.

The world production of TC rubber rose rapidly to a maximum of about 65000 tons in 1955. Indonesia (which was the smallest producer of TC rubber) ceased production in 1962 and Vietnam in 1963 so that only Malaysia and Cambodia are still marking TC rubber at the time of writing. Since 1959 Malaya has recorded a fairly steady output of 30000–40000 tons. Approximately 80% of this is Yellow Circle rubber (with medium curing characteristics), which has in the course of time become the most popular class with consumers. It is believed that TC rubber is most useful to those small consumers who do not do their own initial control testing and have no facilities for blending their rubber to give a uniform starting material.

As common class limits were internationally agreed upon, the R.R.I.M. has functioned as the international co-ordinator for TC rubber and testing stations since the inception of the scheme. In this capacity, it has organised and conducted annual cross-check experiments in

COMMUNICATION 394

which interested TC laboratories were invited to participate.

The technical classification (TC) strain results obtained during the period 1958–62 at six testing stations in South East Asia and Ceylon are compared in this paper. TC strain is a measure of the rate of cure of rubber: it is defined as the extension of a standard cured specimen of rubber produced by a tensile stress of 5 kg per square centimetre of cross-sectional area and is expressed as percentage of the original length of the specimen. Of the stations, two are in Malaya (A and B), one in Ceylon (C), two in Indonesia (D and E) and one in Vietnam (F). The station in Vietnam normally uses the modulus test (i.e. stress in kg per sq. cm at 100% extension) on ring-test pieces, but for comparison with the other stations using the strain test, modulus values are converted to strain using a standard curve. The stations in Indonesia usually correct their *rapid** strain for a Mooney viscosity value of 40 as a routine procedure. The stations in Malaya and Ceylon use their rapid strain results for routine purposes without making any corrections. In the first study described here, the 'routine strain values' (i.e. the values obtained by routine procedures) of the six stations are compared.

The procedures at the stations are examined for the different sources of variation in mixing, curing and testing. The variation, measured by the variance, is defined as the mean value of the squares of the deviations of any set of observations from their true mean and it is measured as units square. The standard deviation is equal to the positive square root of the variance. The coefficient of variation (c.v.) is obtained by expressing the standard deviation as a percentage of the mean. Further, the stations are compared to determine strain level differences (bias). This type of bias can be serious in that it leads to misclassification.

A second study is carried out on the 'corrected strains', obtained from rapid strain values adjusted for Mooney 40 and for the cross-sectional areas of the test pieces.

* In the normal strain test, mixing, curing and testing occupy three days: in the rapid strain test the same operations are completed in one day.

EXPERIMENTAL METHOD

Mixing and Sampling Procedure

One station in Malaya (A) supplied three types of rubber (Red, Yellow and Blue) and a quantity of masterbatch to each of the other stations once a year. The stations were requested to mix in duplicate each of these rubbers using the masterbatch supplied. In addition, at each station a set of duplicate mixes was made for the three rubbers using the local masterbatch. Thus there were four mixes for each type of rubber (12 in all) and for each mix the Mooney value was determined. Stations were requested to adjust their mixing procedures to give a Mooney viscosity between 35 and 45 for the compound.

Two set specimens were cut out from each Red or Blue rubber mix and four specimens from each Yellow rubber mix. The 32 specimens were cured in two sets of cures so that each cure contained one specimen from each mix of Red and Blue rubbers and two specimens from each mix of Yellow rubber. The cured test pieces were tested for rapid strain value and all test pieces were sent to Station A. The cross-sectional areas of these test pieces were measured for cavity corrections at Station A.

Available Data

All six stations provided data for the five-year period, 1958–62, with the exceptions that:

1. Station F in Vietnam provided values from only one mix from each masterbatch combination with the three types of rubber in 1959. Occasionally, three ring test pieces were cured from a single mix but only two of the values obtained were used in the statistical analysis, being converted from modulus to strain for this purpose.
2. One of the stations in Indonesia (E) provided no data for one year (1961).

Prior to 1958 these stations took part in inter-laboratory cross-checking on tests, but the data are less reliable than for the succeeding years, which are analysed here.

Method of Estimation: Test Errors in the Stations

For each station the data for each year are analysed for the different sources of variations in the tests. The three types of rubber mixed with the A masterbatch and local masterbatch provide a total of 6 combination groups. These groups are examined for different sources of reproducibility variations. The following components are examined:

(1) Mix variation $= \sigma_2^2$
(2) Cure variation $= \sigma_1^2$
(3) Test piece variation (including interaction of mix and cure) $= \sigma_0^2$

Each of these in any particular year is derived from mean square with 6 degrees of freedom for (1) and (3) and approximately 5 degrees of freedom for (2). These three sources together make up the total error of testing a single test piece for a combination group.

The different components vary considerably from year to year and consequently the analysis averaged over the available years provides the best estimate of the values for these components.

The duplicate test pieces of the four mixes from Yellow rubber provide the variation component expected from test-piece variations in curing and testing under similar mixing and curing conditions. Values of this source of variation (σ_t^2) for the different stations over the available years are given in *Tables 1* and *2*.

Knowing the three major elements of error variation it is possible to estimate the standard error of a strain mean. When only one test piece is available from one mix and one cure, the reproducibility error of the strain observation would be the square root of the total variation, $\sqrt{(\sigma_0^2+\sigma_1^2+\sigma_2^2)}$.

ROUTINE STRAIN ANALYSIS: RESULTS

The coeffcient of variation (c.v.) of the total test error (*Table 1(b)*) varies from 5% of the mean value for station A to 1.5% for station C. The annual variation in the total test error (*Table 1(a)*) at each station is also very high. The mix component of variation is appreciably large only for the stations E and F. In relation to the high variations in σ_0^2 for stations A and D, the other two sources of variation reduce to zero.

Comparison of Local Masterbatches with A Masterbatch

Since for any given station and year the same type of rubber is mixed with both the local masterbatch and A masterbatch, it is valid to compare the two masterbatches in any one year. The masterbatches of the different stations agree closely with the A masterbatch. Only in station B is there a slight tendency for the A masterbatch to give a higher mean strain value than the corresponding local masterbatch.

Estimation of True Strain Values

The stations differ among themselves in respect of strain means, even for the A masterbatch when used with the three types of rubber supplied. The local masterbatches used at different stations may not obtain their ingredients from the same sources, but it is possible to consider them as one group because the differences are negligible in terms of their influence on strain.

The six rubber/masterbatch combinations are averaged over the stations for each year. If we consider the annual means of the stations as true values it is possible to plot the true values against observed strain values. Within a particular station, y denotes the true strain, x denotes the observed strain and $y=a+bx$ is the regression line fitted to the data for the particular station, where a and b are constants. Although the observations for station F are more variable than the others, all the linear regressions are highly significant. Comparison of the slopes or the coefficients of x between stations indicates that the slopes of all the stations can be represented by a common slope.

When the common slope has been estimated, it is applied to all stations and the constants a (or intercepts on Y axis) are obtained for each station. The stations A, B and C agree closely for these intercepts and so a common regression relationship is possible for these stations. Similarly the stations D and E, both from Indonesia can be grouped to give a single relation-

TABLE 1. ANALYSIS OF ROUTINE STRAIN VALUES (IN SQUARE UNITS OF STRAIN)

TABLE 1(a). TOTAL TEST ERROR VARIATION (REPRODUCIBILITY) FOR DIFFERENT YEARS

Years	Station					
	A	B	C	D	E	F
1958	32.52	3.29	0.86	12.63	9.20	5.28
1959	7.43	3.19	1.56	4.92	10.40	—
1960	25.45	3.79	1.30	8.26	1.69	0.80
1961	25.75	4.76	2.86	11.14	—	14.41
1962	3.87	3.42	0.83	13.71	3.52	2.81

TABLE 1(b). TEST ERRORS AVERAGED OVER THE YEARS

Description of components	Station					
	A	B	C	D	E	F
Mix variation (σ_2^2)	0	0.22	0.73	0	2.56	1.32
Cure variation (σ_1^2)	0	1.08	0.22	0	1.22	0
Test piece variation (including interaction of mix and cure) (σ_0^2)	16.95	2.03	0.50	9.01	1.73	4.25
Total test error (reproducibility)	16.95	3.33	1.45	9.01	5.51	5.57
c.v. based on mean strain of 83 units	5.0%	2.2%	1.5%	3.6%	2.8%	2.8%
Test piece variation for Yellow rubber (σ_t^2)	4.75	1.58	0.73	6.13	1.90	0.80*

* Based on data for one year only (1960).

ship, but station F (Vietnam) cannot be grouped with any others. Thus we obtain the following linear relationships to estimate the true strain (y) values from observed strain (x) values.

Stations	*Relationship*	
A, B and C	$y=3.16+0.9373x$	...(1)
D and E	$y=5.78+0.9373x$	...(1)
F	$y=9.86+0.9373x$	...(1)

Estimation of Bias

If the bias of a station is defined as the amount to be added to the observed value to obtain the true strain, we have the following equation for bias.

Let z denote the bias, then

$$y=x+z$$

but $y=a+bx$

$$\therefore \quad z=a+(b-1)x$$

We can estimate the biases of the various stations from the equations given above as follows:

Stations	*Bias relationships*	
A, B and C	$z=3.16-0.0627x$	...(2)
D and E	$z=5.78-0.0627x$	...(2)
F	$z=9.86-0.0627x$	...(2)

The above equations are plotted in *Figure 1* as parallel lines.

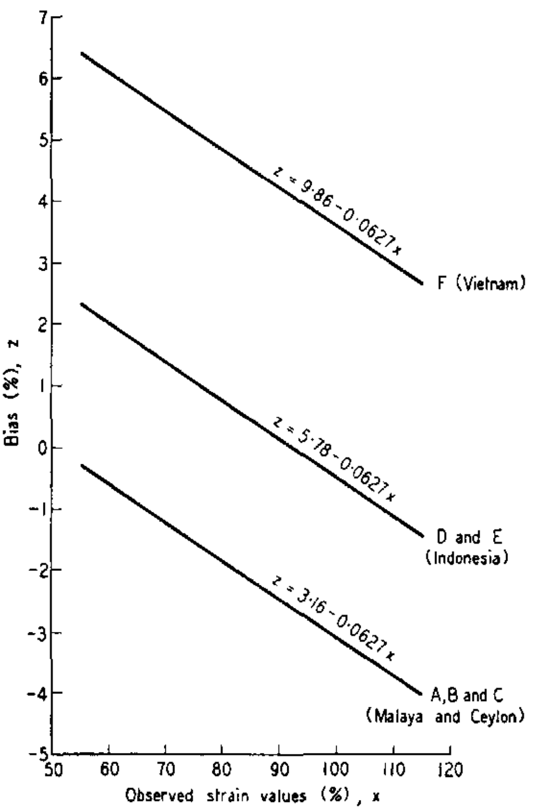

Figure 1. Bias of stations, routine strain (or usual procedure).

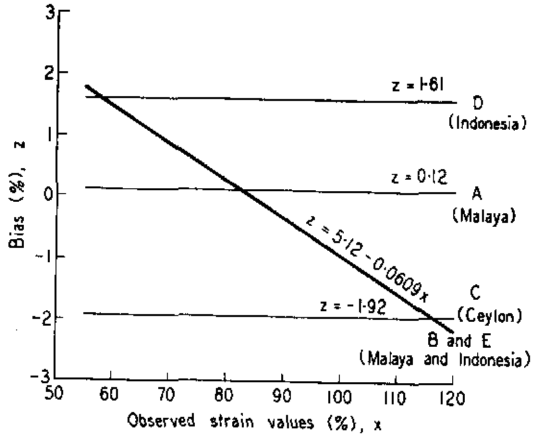

Figure 2. Bias of stations, corrected strain.

Relative to stations A, B, C which agree closely, the strain values of the Indonesian stations (D and E) are 2.6 units lower and the Vietnam station underestimates by as much as 6.7 units. This can lead to discrepancies among the stations in classifying rubber. The amount of bias depends on the observed strain value of the rubber, but the station differences are constant and do not depend on observed strain. It is possible to estimate approximately the strain value for any particular station by using the strain value given by another known station for the same rubber.

CORRECTED STRAIN ANALYSIS: RESULTS

The five stations A, B, C, D and E which use dumb-bell shaped test pieces can be compared on the basis of rapid strain corrected for Mooney viscosity 40 and cross-sectional area differences.

Test Errors

The components of variation are computed and given in *Table 2*. The reproducibility variations are comparable among the different stations, ranging between 2.2% and 2.7% of mean strain value.

For station A, the test-piece variation for Yellow rubber is only 1.04 compared with 4.75 obtained with uncorrected strain (*Table 1(b)*). Further, the significant reduction in the total error variation establishes the need mainly for correction for cross-sectional area.

In station D, correction for cross-sectional area significantly decreases the test piece variation and also the total test error. In station C, there is slight increase in error variabilities.

Estimation of True Strain Values

The mean strain value of all stations is assumed to be the true strain value. The estimation equations can be simplified as:

Stations	*Regression lines to estimate true strain*
A	$y=0.12+x$
B	$y=5.12+0.9391x$
C	$y=-1.92+x$
D	$y=1.61+x$
E	$y=5.12+0.9391x$

TABLE 2. ANALYSIS OF CORRECTED STRAIN VALUES (IN SQUARE UNITS OF STRAIN)

TABLE 2(a). TOTAL TEST ERROR VARIATION (REPRODUCIBILITY) FOR DIFFERENT YEARS

Years	Station				
	A	B	C	D	E
1958	5.26	3.54	4.50	4.71	6.30
1959	4.56	1.65	4.94	0.86	6.67
1960	3.17	6.78	3.89	2.28	12.20
1961	—	2.84	5.29	3.42	—
1962	2.94	4.24	0.66	8.47	2.02

TABLE 2(b). TEST ERRORS AVERAGED OVER THE YEARS

Description of components	Station				
	A	B	C	D	E
Mix variation (σ_2^2)	0.29	2.11	0	0.83	0.45
Cure variation (σ_1^2)	0.75	0.99	0.20	0.45	0.82
Test piece variation (including interaction of mix and cure) (σ_0^2)	2.27	0.70	3.13	2.03	3.79
Total test error (reproducibility)	3.31	3.80	3.33	3.31	5.06
c.v. based on mean strain of 83 units	2.2%	2.3%	2.2%	2.2%	2.7%
Test piece variation for Yellow rubber (σ_t^2)	1.04	1.26	2.13	0.79	0.66

where y denotes the true strain value estimates and x denotes the observed strain values.

Estimation of Bias

The equations for bias are easily obtained from the above equations:

Stations	*Bias equation* (z)
A	$z=0.12$
B	$z=5.12-0.0609x$
C	$z=-1.92$
D	$z=+1.61$
E	$z=5.12-0.0609x$

These equations are plotted for the different stations in *Figure 2*. This figure shows that the station A has negligible bias, and that for the stations B and E the variable bias, falls numerically within 2 units. Station D which has a positive bias of 1.61 units, generally underestimates by this amount in observed strain value. Station C has a negative (1.92 units) bias and over-estimates the true strain.

Comparison of the various bias values relative to test error shows that the bias is as high as the standard error of a determination of strain. Relative to station C, station D underestimates strain by 3.5 units.

The pattern of inter-station variation for corrected strain differs from that obtained for routine procedure (*Figure 1*) where the stations A, B and C differed from D and E by 2.6 units. The correction has not eliminated the station differences.

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