# Importance of Experimental Parameters on Rapid Plasticity Testing for PRI (Plasticity Retention Index)

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## Abstract

Rapid plasticity is one of the basic tests used in the natural rubber industry and is the measurement of compression of a specimen of known thickness at a known temperature under a predetermined load for a known time. The Plasticity Retention Index is a measure which reflects the resistance of natural rubber to thermal oxidation. Several parameters affect the end result, such as platen parallelism, platen temperature, force, time, the centralisation of the sample, the type of paper/material used between the platens and sample and the ageing temperature and time.

This paper aims to demonstrate the effect of these parameters on the final end result.

Experimental work was carried out using the current Rapid Plastimeter (P12E) and the new Wallace Cogenix Rapid Plastimeter (P14). The new P14 enabled the platen temperature and test times to be accurately varied and controlled. The effects of sample positioning and the type and thickness of paper used between the platens and sample was also investigated.

The P14 instrument enables tighter temperature control, allowing the effects of temperature to be investigated.

## Introduction

Use of the equipment and parameters specified in the current standard define Rapid Plasticity (ISO 2930:1995 - ref. 1). In general, a plastic material is one which, when subjected to a force, continues to deform as long as the force acts with much (or all) of the deformation remaining when the force is removed (ref. 2). Rapid Plasticity is defined by measuring the compression of a specimen of specified thickness at a specified temperature under predetermined loading for a specified time. Samples are sandwiched between a carrier paper to protect the platens from contamination. One Wallace Rapid Plasticity unit represents a change in thickness of 0.01mm (ref. 3). The current standard specifies a method for the rapid determination of the plasticity of raw rubber and non-vulcanised rubber compound.

Rapid Plasticity has remained one of the basic tests in the Rubber Industry for the past 50 years. The original Wallace Rapid Plastimeter (P1 - developed in 1951) used steam-heated platens and relied on a dial gauge, a stopwatch and the skill of the operator to produce results. The arrival of the microprocessor lead to the P12 instrument - first introduced in 1984. The P12 retained the steam driven platens but used a sensor to measure distance and the microprocessor to time the test; the load was spring-driven. The current production instrument (P12E) was introduced in the late 1980's and uses electrically heated platens[1] and an accurate load application method; the P12E still relies on operator skill to calibrate the platen gap. The new P14 Rapid Plastimeter builds on the previous instruments and includes several new

[1] The temperature is not indicated; it is fixed by adjusting the temperature set point on calibration.

features. These include the use of platinum resistance sensors to measure the temperature of both platens; operator error is reduced by a semi-automatic gap-calibration procedure.

The Plasticity Retention Index of a sample is the ratio of the rapid plasticity numbers after ageing at 140°C and before ageing, multiplied by 100. The PRI test requires the use of a plastimeter and ageing oven. The current production oven (O12) was introduced to accurately heat the samples to 140°C. However, the temperature is not indicated but is fixed by adjusting the temperature set point. The new O14 (to complement the new P14) makes use of a platinum resistance thermometer together with a 3 term temperature controller to accurately control the temperature to 140° ± 0.2°C. The standard (ref. 1) requires an ageing time of 30 mins ± 0.25 mins.

The standard (ref. 1) specifies limits and acceptable ranges for each of the parameters affecting the end result; some of these originate from the era of the P1. This paper investigates some of these parameters and limits with the advent of more accurate instrumentation. A practical appreciation of their effects may be used to produce accurate, consistent results. This paper incorporates previously unpublished work from H W Wallace and surveys other literature on the subject.

## Method

All data were taken with a resolution of 0.1 plasticity units and recorded with a resolution of 0.5 units. A median result was taken from 3 measurements.

### Platen Temperature Variation

Platen temperature dependence was studied by adjusting the temperature of both top and bottom platens either side of the standard temperature (100°C). The temperature was measured using the temperature probe mounting supplied in the instruments' calibration kit, which is traceable to national standards.

### Sample Centralisation and Platen Parallelism

Sample centralisation was investigated by placing samples at various off-centre positions on the platen face. The affect of platen parallelism was studied, by adjusting the tilt (fig. 1) of the top platen from 0° to 0.65° and 1.3° respectively.

### Operator Dependence and Instrument to Instrument Variation

Operator result dependency was tested by using 5 members of staff. Their instrument knowledge ranged from an operator with several years of inspection experience through to one with no previous experience.

Instrument to instrument variation was examined by analysing the historical inspection/calibration reports produced on P12E instruments that used common rubber batch samples.

### Sample Paper Dependence

Testing was carried out by using different types of material and paper between the platens and sample. Materials tested include standard paper, photocopier paper, tracing paper, toilet tissue, aluminium foil and brown paper. In addition to varying the type of material, each was tested at the standard size (35mm square) and at 16 and 14.5mm square.

Load Variation

The load applied to the sample was investigated by adjusting the force either side of the standard force (100N).

## Results

The natural rubber used to perform the tests normally has a graduation in plasticity value across the sheet. Since the same sample cannot be tested more than once, the results had to be made using different, but adjacent, samples of the test rubber. Some variation in the results can be attributed to these variations in the sample. Previous experience with a number of instruments suggests that the variation with similar material is normally about ±1.5 plasticity units across the sheet.

Platen Temperature Variation

The temperature specified by the standard is 100 ±1 °C. At 101°C and 99°C, the results were comparable to the standard results at 100°C. Individual results did however, appear to exhibit slightly more fluctuations than usual about the median. At 102°C (1°C above the top limit) the results were approximately 1 plasticity unit lower. At 98°C (1°C below the bottom limit) there was a 1 unit increase in plasticity. At 95°C there were more fluctuations in the results and approximately a 1.5 unit increase in the plasticity number (table 1).

Previous work (D. A. Hills, ref. 4) enforces these findings; the results showed that the plasticity number will be increased by approximately 2 units for a drop in temperature of 6°C from the standard 100°C platen temperature. Reliable results will only be obtained if the platen temperatures are kept within close limits.

Sample Centralisation and Platen Parallelism

Centralisation of the sample is important. Generally, it is unlikely that the operator will place the sample exactly in the centre every time. A few millimetres deviation from this point does not measurably influence the results. However, once the top platen compresses the edge of the sample (corresponding to about 5 mm off-centre) the plasticity number drops by approximately 1 unit - the result of using the same compression over a reduced sample surface area. Results will continue to drop as less of the sample is used.

With 0.65° tilt on the top platen (from the standard parallel position - see fig. 1), results are consistently reduced by 1 plasticity unit. Tilting the platen to 1.3° tilt reduces the result by 2 units (see table 2). The results will eventually drop to zero as the tilt is increased.

Operator Dependence and Instrument to Instrument Variation

Note that it was necessary to train the inexperienced operators (for about 5 minutes) before testing. It is virtually impossible for an untrained operator to produce a reliable result. The results were then consistent between operators (see table 3), with a variation of ±0.5 plasticity units.

The instrument to instrument variation was examined by analysing the last 10 inspection/calibration reports produced on P12E instruments. The same batch of rubber was used in all cases. The variation was found to be minimal and can be seen in the distribution curve (figure 2).

Sample Paper Dependence

Tissue paper or cigarette paper of about 22g/m$^2$ cut into two equal pieces should be used (refer to ISO standard, ref. 1). However, it is known that the recommended method is not always used. Measurements have been made using different sizes and types of material between the rubber sample and platen.

The instrumental results are very dependent on this parameter. When the size of the material is halved (using the recommended standard cigarette paper), the paper does not rest on the shield around the bottom platen. This reduces the test results by about 4 units. Thus, for this reason, the position of the paper becomes more important if larger sample papers are used.

The other materials used (10 in total) made a difference to the final result (see table 4). The differences ranged from a few units below to more than 10 units above the standard result. It can be seen that although the numerical value of the result varies between different material types for the first group of materials, the results are reasonably consistent for any one material.

Load Variation

The load specified in the standard is 100 ±1 N. At 101 N and 99 N, there was no significant variation from the standard results (when applying a force of 100 N). At 102 N (1 N above the top limit) the results were about the same as at the top limit. At 103 N (2 N above the top limit) the results were approximately 1.5 units below the standard result. At 98 N (1 N below the bottom limit) the results were approximately the same as at the bottom limit. A force of 97 N (2 N below the bottom limit) gives results approximately 1.5 units above the standard result. These results are tabulated in table 5. This demonstrates that it is important to check the load calibration.

## Conclusion

It has been demonstrated that several parameters can affect the plasticity value obtained from a given sample. Results are very repeatable, provided these parameters are controlled. In practice, it has often been found that results are simultaneously affected by the variation of more than one parameter.

Tight temperature control is important. The platens of the obsolete P1 and older P12 instruments were steam heated – leading to large temperature variations in some cases. The P12E platens are electrically heated, and must be checked and adjusted regularly (which is often not the case). The measurement method and lack of care in setting the P12E's platen temperatures can lead to significant variations from the 100 ±1 °C specified in the standard. The new P14 deals with these problems by using an accurate sensor to measure the platen temperature, and by incorporating a calibration point near to the platen surfaces. Care must be taken to ensure that the same temperatures are used for both platens, since this will effect the end results.

Centralisation of the sample is important, but normal care is adequate. Lack of platen parallelism also affects the result. This is particularly so when the alternative platen sizes allowed in the standard are used – i.e. the parallelism must be reset every time the platen has been changed. In addition, care must be taken to ensure that the correct platen sizes are used as this will effect the results obtained.

Once trained, operator dependence is not important. The instrument to instrument variation is low and within the spread of a 'uniform' sample sheet.

The dependence of the result on the sample paper used is important. Use of the paper recommended by the standard will ensure that results are consistent. Other sample carriers may be used (after validation) and will produce different, but consistent results. The paper size is also important and if larger sample papers are used, the effect of the heat-shield can lead to significantly low readings.

Sample size is important. The standard must be adhered to; other sizes must not be used. The sample preparation equipment supplied with the instrument ensures this.

The load is accurately applied only in the P12E and P14 instruments – with a calibrated weight arm applying the load. It is worth noting that the geographical variation in gravity can lead to differences of up to 0.5%. This accounts for almost half of the allowable tolerance in the standard. Therefore it is recommended that the weight arm is calibrated locally at any particular location in the world.

Use of the P1, timed by the operator, can lead to highly significant variations in the final result. Figure 3 demonstrates that a variation of ±1 second in the time of application of the test force can make a difference to the final result by as much as one plasticity unit. The crystal controlled timing of the P12, P12E and P14 ensure that the result timing is accurate and exact.

Regular servicing of the instrument will ensure that all parameters are set accurately within the limits specified in the standard to maintain accurate and reliable results.

PRI Measurement

The PRI test requires the use of a plastimeter and ageing oven and so in addition to the results being affected by the parameters discussed above, the PRI result may also be affected by the oven. As in the case of the requirements for tight temperature control of the plastimeter platens, the ageing oven also requires tight temperature control. Previous work (E.D. Farlie and H. W. Greensmith - ref. 5) indicates that a temperature variation of ±1°C from the standard 140°C can produce a difference in the PRI value of ±3 units for a PRI of approximately 50. As a result, the standard (ref. 1) specifies the tolerance to be ±0.2°C to maintain the accuracy. The standard also states that a variation of ±0.5°C may be used but this may impair the accuracy of the results. The new oven, O14, uses an accurate platinum resistance thermometer to measure and control the temperature.

Additionally, previous work (ref. 5) also suggests that rubber plasticity falls continuously but at a decreasing rate with increasing ageing time. Consequently, a variation in the ageing time of a few minutes is likely to effect the result by a few units (the exact deviation is dependent on the type of rubber sample). The standard (ref. 1) specifies the ageing time within close limits (± 0.25 mins) to ensure reliability and repeatability.

**References**


1. ISO 2930, Determination of the plasticity retention index (PRI) of raw natural rubber, 1995.
2. RABRM Bulletin, Special Issue on Plasticity (1954). Vol. 8, No. 4.
3. McGarry, B. M., Rapid Plasticity and Plasticity Retention Index Testing (1994).
4. Hills. D. A. (1964), Rubber Journal, Testing with the Wallace Rapid Plastimeter.
5. Farlie. E. D. & Greensmith. H. W., Transactions of I.R.I, 1966, 42, Part III.


**Tables and Figures**

| Temperature (°C) | Median Results (plasticity number) | | |
|---|---|---|---|
| 102 | 31.0 | 30.5 | 31.0 |
| 101 | 30.5 | 31.5 | 31.5 |
| 100 | 31.0 | 31.0 | 32.0 |
| 99 | 31.5 | 31.0 | 31.5 |
| 98 | 32.0 | 32.0 | 32.0 |
| 95 | 32.0 | 32.5 | 33.0 |

Table 1. Effect of platen temperature

| Angle of tilt (°) | Median Results (plasticity number) | | | |
|---|---|---|---|---|
| 0.00 | 33.0 | 32.5 | 33.0 | 32.5 |
| 0.65 | 32.0 | 32.0 | 32.0 | 31.5 |
| 1.30 | 31.0 | 31.0 | 31.5 | 31.0 |

Table 2. Effect of platens not being parallel

| Operator | Results (plasticity number) | | |
|---|---|---|---|
| 1 | 33.0 | 33.0 | 33.0 |
| 2 | 33.0 | 32.5 | 33.0 |
| 3 | 33.0 | 32.5 | 32.0 |
| 4 | 32.0 | 32.5 | 33.0 |
| 5 | 33.0 | 33.5 | 33.0 |

Table 3. Effect of different operators

| Material no. | Results (plasticity number) | | |
|---|---|---|---|
| Cigarette paper | 36.5 | 37.0 | 36.0 |
| Hand towel paper | 33.0 | 35.0 | 34.0 |
| Hard toilet tissue | 35.0 | 37.0 | 36.5 |
| Wrapping film | 38.0 | 38.0 | 36.0 |
| Toilet tissue | 46.5 | 44.0 | 45.0 |
| Photocopier paper | 53.0 | 43.0 | 49.0 |
| Tissue paper | 52.0 | 53.0 | 51.5 |
| Tracing paper | 49.0 | 57.0 | 52.0 |
| Brown paper | 59.5 | 58.0 | 57.0 |
| Mylar film | 32.5 | 33.0 | 34.0 |
| Cling film | 23.0 | 22.0 | 23.0 |

Table 4. Effect of using different material in place of the cigarette paper

| Force (N) | Median Results of a typical sample (plasticity number) |
|---|---|
| 103 | 30.5 |
| 102 | 31.0 |
| 101 | 31.0 |
| 100 | 32.0 |
| 99 | 32.0 |
| 98 | 32.0 |
| 97 | 33.5 |

Table 5. Effect of force on platens

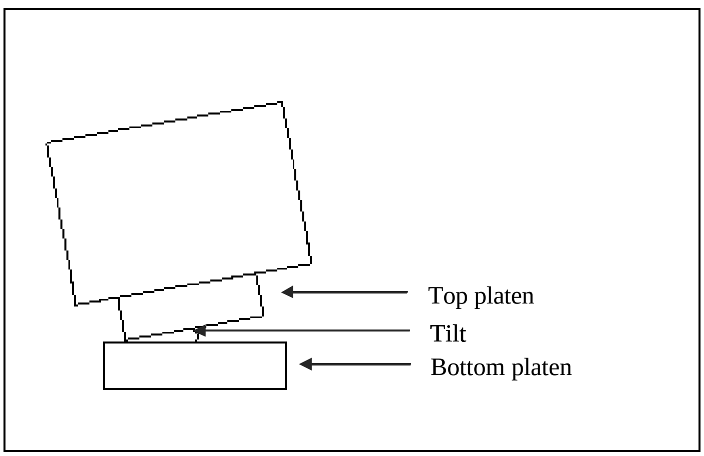


Figure 1. Diagram (exaggerated) showing top platen at an angle to the bottom platen

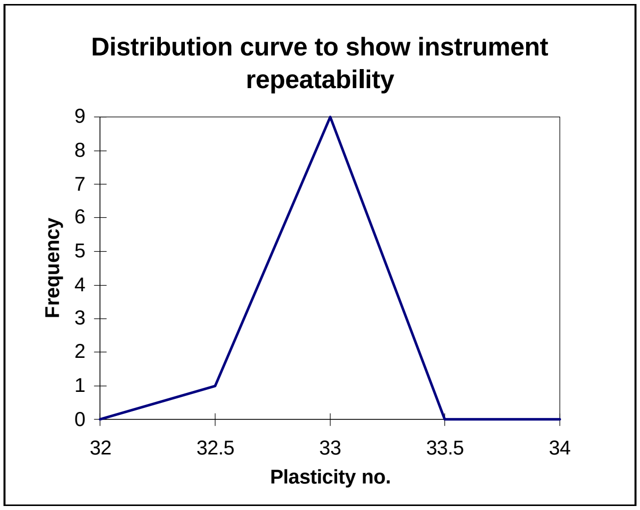

Figure 2.  Graph showing instrument to instrument variation

Figure 3.  Graph to show the effect of time of application of test force on the plasticity number