FACTORS AFFECTING RESULTS OBTAINED WITH THE MOONEY VISCOMETER

By

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PREFACE

The factors discussed in this paper do not by any means cover all that might be written on the care and operation of Mooney viscometers. It has been assumed that such things as periodic fluctuations, general overhaul, and detailed specifications for parts and adjustments have been adequately covered in the manuals issued by the manufacturer of the viscometer and by the Rubber Reserve Company. It is hoped that this discussion of the cleaning, calibration, dimensions, adjustments of the viscometer, and the preparation of the test piece, together with the errors that are likely to result if correct practices are not followed, will lead to more uniform results and a better understanding of the operation of the machine.

E. U. Condon, Director.
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ABSTRACT

Experience with the operation of the Mooney viscometer in 18 laboratories indicates the need for better reproducibility of results. A study of the available data from these laboratories along with numerous experiments made at the National Bureau of Standards have shown the factors that must be considered in improving the reproducibility. If uniformity in the values of Mooney viscosity on the same sample with different viscometers and in different laboratories is to be obtained, the methods of adjustment that must be followed and the precautions that must be taken are cleaning, mechanical calibration, dimensions of dies, die holders, and rotors, die closures, and preparation of test pieces. Each of these items and the errors which may result from maladjustments and lack of precautions in the use of this instrument are discussed.

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I. INTRODUCTION

Studies of the results obtained with Mooney viscometers in measurements of viscosity of synthetic rubbers indicate the need for better and more definite instruction of operators of such viscometers. Recent reports of tests made on standard bales of raw GR-S at 18 different laboratories show that readings for individual tests vary over a range of approximately 12 units and that averages obtained by the different laboratories vary over a range of 4.5 units. If suitable precautions are taken, individual values from different laboratories need not vary by more than 3 units, and the average values from the different laboratories should vary by less than 1 unit.

In order to attain this degree of agreement in different laboratories, it is most essential that precautions be taken in caring for and adjusting Mooney viscometers with respect to the following five items:
(1) The machine must be clean; particularly, there must be no rubber in the bearings on the vertical shaft, (2) the machine must be mechanically calibrated to rigid specifications, (3) the dies, die holders, and rotors must be of specified dimensions, (4) the die closures must be carefully adjusted to rigid specifications, and (5) the test pieces used in the machine must be carefully prepared and selected for accuracy of dimensions and freedom from air. A well-constructed machine with all operating parts machined to proper tolerance is of course implied.

This paper describes the precautions that must be taken and the methods of adjustment that must be followed if uniformity in the values of Mooney viscosity are to be obtained on the same samples with different viscometers and in different laboratories. The variations that may result if the outlined procedures are not closely followed are discussed.

II. EFFECTS OF INADEQUATE OR INFREQUENT CLEANING OF THE MOONEY VISCOMETER

It is of the utmost importance that certain parts of the Mooney viscometer be kept absolutely free from rubber at all times. Results obtained from tests made on machines that have rubber in the bearings can never be depended upon, and tests made on machines operated with the serrations on the dies and rotors clogged with rubber are very apt to give erroneous results.

1. EFFECTS OF RUBBER AROUND THE TOP OF THE VERTICAL SHAFT

In AA and earlier model viscometers by far the most important cleaning operation, and the one which has been the hardest to bring to the realization of Mooney operators, is the removal of rubber from around the top of the vertical shaft (see fig. 1 for location of parts). The lower die should be removed, and any rubber that has leaked past the rotor stem should be cleaned out so that it will not be forced into or work itself into the top bearing. No definite rule can be established for the regularity with which this cleaning operation should be performed because of the variables involved. The amount of rubber that will leak past the rotor stem will depend on the clearance between the rotor stem and the lower die and upon the plasticity of the rubber being tested. The amount of rubber that can safely be permitted to leak into the chamber at the top of the vertical shaft before cleaning depends chiefly on the clearance between the top of the vertical shaft and the bottom of the lower die. Frequently, maintenance personnel in replacing the bronze bearing in the upper part of the housing have not realized the importance of this clearance and have left the bearing too long, so that the clearance was reduced to less than \( \frac{3}{16} \) inch. In fact, one case was encountered in which it was so small that not more than one test could be made without obtaining erroneous results. The design of this instrument calls for a clearance of \( \frac{1}{4} \) inch. It is recommended that it be not less than \( \frac{3}{32} \) inch on any machine. It should be borne in mind also that it is desirable to have the top of the vertical shaft as high as practicable in order to give the necessary support to the rotor.
A well-oiled felt washer at the top of the vertical shaft has been found quite helpful in preventing the rubber from working its way into the bearings. Some laboratories have not found these washers to be of any help simply because their cleaning periods were so infrequent that rubber was forced into the felt washer rendering it useless, in time destroying it. Proper cleaning and oiling permitted the use of one felt washer at the National Bureau of Standards for a period of more than 6 months without the necessity of removing it or the vertical shaft.

Normally a small amount of rubber leaks past the rotor stem each time a test is made. Assuming sufficient clearance between the top of the vertical shaft and the lower die, this rubber will lodge on the top of the shaft without affecting the viscometer reading. As rubber is forced into the space between the die and the shaft, it will be rolled between the two without materially affecting the results. Then, as more rubber is forced past the rotor stem it will completely fill the space between the die and the shaft and will finally be forced down into the bearing. Should this happen, the only way in which the machine can be properly cleaned is by removing the vertical shaft and cleaning thoroughly both the shaft and the bearing. If this is not done, the results at best are bound to be unreliable.

Mechanical calibration designed to compensate for the increased friction due to the rubber in the bearing should never be tolerated. In most cases where this is done, high values of viscosity will be
obtained because the increased pressure due to additional leakage of rubber during a test increases the friction still more. The errors resulting from this condition have been found to be as much as 6 units.

When the felt washer is not used at the top of the vertical shaft, any rubber that leaks past the lower die is likely to find its way into the bearing, and it is never certain just how much the results are being affected by this condition.

The only way in which to be sure that the results are not affected is to have a felt washer at the top of the shaft, keep it well oiled, and above all, remove the lower die and clean out the rubber before it has been forced into the felt. Approximately 10 minutes are required to remove the lower die, clean out the rubber, oil the felt, and replace the die. On the other hand, 2 hours or more are required to remove the vertical shaft and perform the necessary cleaning operation after rubber has been forced into the bearing, and it is never certain just when the excess rubber begins to affect the readings.

The design of the new NBS model Mooney viscometer alleviates some of the difficulties of cleaning required in the earlier designs by increasing the size of the cavity at the top of the vertical shaft, by using a better bearing shield at the top of the shaft, and by using a sealing washer in the lower die. This does not mean that rubber leakage through the lower die has been eliminated nor does it mean that cleaning is no longer necessary. The frequency of cleaning, however, has definitely been decreased. Oil at the top of the shaft is not recommended for the NBS models, but otherwise the above discussion applies to these models as well as to others.

2. EFFECT OF RUBBER IN THE SERRATIONS OF THE DIES AND ROTORS

It is highly important that rubber that sticks to the dies and rotors and fills the serrations of these parts should be removed before proceeding with the next test. The greatest errors will be experienced when a test on rubber of low viscosity is followed by one of higher viscosity. The error is caused by shearing of the rubber that remains in the dies and on the rotor near the surface of these parts rather than shearing of the sample itself. Insufficient data are available to show the extent of such error, but low readings have been observed in cases where the operator has failed to clean the rubber from the serrations on the rotor and in the die cavity.

3. EFFECT OF RUBBER AROUND THE PLUNGERS

The frequency with which the plungers require cleaning depends on the clearance between the plungers and plunger holes in the top die, the kind of samples being tested, and the precautions taken to keep the plungers from becoming clogged. Rapidly working the plungers up and down several times after every few tests will tend to keep them free and in good working order. In fact, if this procedure is performed regularly, the viscometer can be operated continuously for weeks without having to remove the plungers for cleaning.

Inoperative plungers will cause the viscometer reading to be low. Where there is no plunger action at all, the error introduced may be as much as 3 units.
Oil should never be applied to the plungers because it causes the rubber that leaks past the upper die to become sticky and more difficult to remove from the plunger parts.

III. MECHANICAL CALIBRATION

Mechanical calibration of Mooney viscometers, although somewhat tedious, is a straightforward procedure that should not give undue trouble. It is, nevertheless, a very important step that must be taken in order to insure correct readings of viscosity.

There has been considerable discussion as to whether it is sufficient to calibrate viscometers at the normally specified reading of 100 or whether they should be calibrated at or near the reading that will be obtained from the rubber tested. Actually, it makes no difference as long as the U-spring is not stressed beyond its elastic limit, since the calibration curve for the U-spring will then be a straight line. A few cases have been found where an error was introduced as a result of permanent set taken by the U-spring when rubbers having a high viscosity were tested. This means that in these particular cases the spring was stressed beyond its elastic limit. Consequently, the results would not be dependable, regardless of the point at which the calibration was made. Any U-spring designed for use on the viscometers that does not give a straight-line relation between load and deflection should not be used.

IV. EFFECTS OF DIMENSIONS OF DIES, DIE HOLDERS, AND ROTORS

The dimensions of the die cavity and the dimensions of the rotor head are very important from the standpoint of attaining uniform results with different Mooney viscometers.

Mooney\textsuperscript{1} gave the following equation for the Mooney viscometer reading in terms of constants for a particular instrument and the average viscosity of the sample being tested.

\[
G = \frac{\eta_m(Q \omega)}{181.44 g P} \left( \frac{4 \pi \Omega}{a} \int_0^R r^3 dr + \frac{2 \pi h R^3 \Omega}{b} \right),
\]

which may be reduced to

\[
G = \frac{2 \pi \eta_m Q \omega}{181.44 g P} \left( \frac{R}{2a} + \frac{h}{b} \right),
\]

where all the terms are expressed in cgs units and

- $G =$ Gage reading
- $\eta_m =$ Average viscosity
- $Q =$ Angular velocity of rotor in radians/sec.
- $g =$ Gravitational acceleration
- $P =$ Pitch radius of worm gear
- $R =$ Radius of rotor
- $a =$ Vertical clearance between rotor and stator above or below the rotor
- $h =$ Thickness of rotor
- $b =$ Effective radial clearance between rotor and stator.

\textsuperscript{1} Melvin Mooney. A shearing disk plastometer for unvulcanized rubber. Ind. Eng. Chem. Anal. Ed. 6, (No. 2) 147 (1934).
1. EFFECT OF DIMENSIONS OF ROTORS

For the purpose of calculating the effect of sizes of rotor on the viscometer reading, we may assume the average viscosity \( \eta_m \) to be a constant that will give a gage reading of 50 for rotors and dies of specified dimensions. Then, since \( g, p, \) and \( \Omega \) are constant, we may write

\[
G = KD\left(\frac{D}{4a} + \frac{h}{b}\right),
\]

where \( K \) is a constant, \( D \) is the diameter of the rotor, and \( D, a, h, \) and \( b \) are all measured in inches. Now, since the inside diameter

![Figure 2. Calculated effect of thickness and diameter of rotor on viscometer reading.](image)

- Curve 1—thickness.
- Curve 2—diameter.
of the die holder is to be 2.000 inches, and the total depth of the chamber is to be 0.418 inch, we may write

\[ a = \frac{0.418 - h}{2} \]  

and by Mooney's reasoning,

\[ b = \frac{1}{2} \left( \frac{3a + 2.000 - D}{2} \right) \]

to a close approximation.

If we now assume values for \( D \) and \( h \) and calculate \( G \), the curves shown in figure 2 are obtained. Thus we see that the viscometer reading for the sizes of rotors considered will vary at the rate of approximately 0.25 unit per 0.001 inch change in thickness of the rotors and approximately 0.15 unit per 0.001 inch change in the diameter of the rotor.

In order to check the theory presented above, tests on Vistanex were made with four available rotors and the results compared with the calculated value for each rotor. These comparisons are shown in table 1.

For purposes of calculation, 49.5 and 55.5, the values obtained with rotor No. 1 were assumed to be the correct gage readings for rotor No. 1, and were used in calculating the constant \( K \) in eq 2 and the expected results for rotors Nos. 2, 3, and 4. It should be noted that the calculated gage reading is related to the effective values of \( D \) and \( h \), and that these may or may not be the same as the measured values. This is particularly true where the rotor has burred edges, as was the case with rotors Nos. 3 and 4.

<table>
<thead>
<tr>
<th>Table 1.—Effect of rotor dimensions on viscometer reading</th>
</tr>
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<tbody>
<tr>
<td>Rotor number</td>
</tr>
<tr>
<td>--------------</td>
</tr>
<tr>
<td>Rotor dimensions, in inches:</td>
</tr>
<tr>
<td>Diameter ( D )</td>
</tr>
<tr>
<td>Burr diameter ( b )</td>
</tr>
<tr>
<td>Thickness ( h )</td>
</tr>
</tbody>
</table>

Viscometer reading:

<table>
<thead>
<tr>
<th>Sample 1—</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Observed value</td>
<td>49.5</td>
<td>51</td>
<td>52</td>
</tr>
<tr>
<td>Calculated value based on:</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(1) Diameter</td>
<td>49.8</td>
<td>50.0</td>
<td>52.2</td>
</tr>
<tr>
<td>(2) Burr diameter</td>
<td></td>
<td>51.4</td>
<td>53.5</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sample 2—</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Observed value</td>
<td>55.5</td>
<td>55.5</td>
<td>57</td>
</tr>
<tr>
<td>Calculated value based on:</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(1) Diameter</td>
<td>55.8</td>
<td>56.1</td>
<td>58.5</td>
</tr>
<tr>
<td>(2) Burr diameter</td>
<td></td>
<td>57.6</td>
<td>60.0</td>
</tr>
</tbody>
</table>

\(^1\) Burrs were formed at the corners of the rotor during the chromium-plating operation. The burr diameter is taken as the maximum diameter of the rotor including the burrs.

2. EFFECT OF DIMENSIONS OF DIE CAVITIES

Changes in the depth and the inside diameter of the die holders affect the gage reading insofar as they affect changes in the values of \( a \) and \( b \), respectively. If the rotor head is assumed to be 1.500 inches
in diameter and 0.218 inch thick and the dimensions of the die cavity are permitted to vary, eq 3 and 4 become

\[ a = \frac{T - 0.218}{2} \]  
\[ b = \frac{1}{2} \left( 3a + d \right) - \frac{1.500}{2} \]  

where \( T \) is the total depth of the die cavity and \( d \) is the diameter of the cavity. From an analysis of eq 2, 3, 4, 5, and 6, it can be shown that the change in the viscometer reading with an increase in \( T \) is essentially equal to the change in reading resulting from a decrease in \( h \) equal to the increase in \( T \), and vice versa. The change in reading is approximately 0.25 unit per 0.001 inch change in the total depth of the die cavity, whereas the change in reading resulting from a change in the diameter \( d \) is only about 0.15 unit per 0.010 inch change in \( d \). It will be noted from eq 2 and 6 that the effect of changing the inside diameter \( (d) \) of the die cavity is much less than the effect of changing the diameter \( (D) \) of the rotor.

As the variations in the dimensions of the rotors studied are not as great as those encountered in different plants, the variations in viscometer readings shown in table 1 are not as great as those found in actual practice. However, they do indicate the conformance of observed data to theory, and the curves shown in figure 2 indicate the importance of using only rotors, dies, and die holders that conform to the specified dimensions. The dimensions and tolerances usually recommended are given below as a matter of interest:

**Rotor:**

- Head diameter: 1.500 \( +0.001 \) \( -0.001 \) in.
- Head thickness: 0.218 \( +0.001 \) \( -0.001 \) in.

**Dies:**

- Total thickness: 0.509 \( +0.000 \) \( -0.001 \) in.
- Thickness from bottom to shoulder: 0.406 \( +0.000 \) \( -0.001 \) in.

**Die holders:**

- Thickness from shoulder to closing face: 0.312 \( +0.000 \) \( -0.001 \) in.
- Inside diameter: 2.000 \( +0.010 \) \( -0.000 \) in.

### 3. EFFECT OF POSITION OF ROTOR HEAD

The Mooney viscometer readings are affected not only by the dimensions of the die cavity and the rotor but also to some extent by the position of the rotor in the die cavity. It is intended that the rotor head shall operate in the exact center of the die cavity. In the A and AA model viscometers, however, it is impractical if not impossible to maintain the rotor in this position because of the rapid wear which takes place between the shoulder on the rotor stem and the lower die. This difficulty is not present in the NBS model. As long as the dimensions of the various parts are such that the rotor will be properly positioned to begin with, one can be reasonably sure that the correct position will be maintained.

The rotor head can be off the central position by 0.01 inch without materially affecting the results. But no more than 0.01 inch should be permitted. Where the rotor is off center by 0.03 or 0.04 inch the
viscometer readings may be increased by 2 or 3 units. A very practical way to check the position of the rotor is to compare the thickness of the cross sections of the portions of the test piece from above and below the rotor.

The curve in figure 3 shows the effect of displacing the rotor head from the central position of the die cavity. This curve was calculated from eq 2, 3, and 4 and correlates reasonably well with experience.

![Figure 3](image)

**Figure 3.**—Calculated effect of vertical displacement of the rotor in the die cavity.

V. ADJUSTMENTS

Correct adjustment of the die closure is without doubt the most difficult to attain of the five items discussed in this paper. It has not been found possible to set forth a method of adjustment that is free from the personal factor, nor has it been possible to convince all operators of the importance of careful adjustment. Several different methods of adjustment have been tried, none of which are entirely satisfactory. Considerable progress has nevertheless been made.

Correct adjustment of the die closure is considered to be a condition such that when the mating surfaces of the die holders are forced together there will be a uniform pressure at all points between the two surfaces. The pressures will not be great enough to cause undue strain in any of the parts, and yet the pressures will be great enough to eliminate practically all of the leakage from the die cavity between the closing surfaces. The following method is suggested for making this adjustment.

1. **ADJUSTMENT OF PLUNGERS AND DIES**

Replace the upper die and plungers, being careful to aline the die with the plungers. Place the die holder (TD in fig. 4) over the die and hold it firmly in place while applying the four cap screws that hold the die holder and die in place, being careful to tighten opposite screws evenly. Do not tighten the cap screws too much. Just bring them up snug.
If the plungers do not operate freely after the die and the plungers have been tightened in place, loosen the studs in the die holder and in the plunger clamp and shift them into a position that will permit the plungers to operate freely.

Check the operation of the plungers by rapidly raising and releasing the plungers several times. (Recheck the operation of the plungers frequently throughout the day, as they tend to pick up rubber and become inoperative very quickly, especially when compounded samples are tested. Clean them whenever necessary, as indicated in section II, 3.)

Oil the felt washer around the top of the vertical shaft in models A and AA, as outlined in section II, 1. Replace the lower die. Start the motor, insert the rotor and aline the die by shifting it to the posi-

![Figure 4.—Mooney viscometer.](image-url)
tion at which the magnitude of the periodic fluctuations of two per minute is a minimum. Place the die holder on the die and tighten the four cap screws as was done on the upper die, being sure that the fluctuations are not increased by this operation.

Make a pattern of the die closure (C) by placing a piece of thin, (not thicker than 0.0015 inch) soft tissue paper between the dies and closing the machine. Adjust the four cap screws in the die holders until a continuous pattern of uniform intensity is obtained. Do this by tightening the cap screws adjacent to the part of the die holder which produced a pattern of high intensity, and loosening those adjacent to the part which produced a pattern of low intensity. Figure 5 shows a good die holder pattern and examples of poor patterns. If a perfect pattern cannot be obtained in this manner, remove the four cap screws from the lower die holder (BD), turn the die holder one-quarter turn and proceed as before. If, after trying each position of the die holder, a pattern of uniform intensity has not been obtained, replace the die holder with a new one. To aid in relocating the correct position of the die holders when replacing them after cleaning, place a reference mark on them.

2. ADJUSTMENT OF PLATENS

Make the following adjustments, with the platens at the temperature of the test:

(1) Adjust the position of the bridge so that the distances $X_1$ and $X_2$ in figure 4 are equal when the dies are closed, by adjusting the guide rod nuts 1, 2, 3, and 4 on guide rods GR$_1$ and GR$_2$. 

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure5.png}
\caption{Typical patterns of die closures.}
\end{figure}

A, Good die pattern; B, poor because of cocked die holder; C, poor because of badly worn die holder; D, poor because of nonuniform tightening of die holder or inequality in thickness around die from bottom to shoulder.
(2) Adjust the position of the link (L), which connects the top platen (TP) with the lower lever arm (A2) so that the link is on dead center when the dies are closed; i.e., so that the four pins P1, P2 (not shown—connects L with A1), P3, and P4 all lie on the same center line. Do this by adjusting the stop bolts SB1 and SB2. Turn the stop bolt (SB1) out of the guide rod (GR1) to swing the link forward at the top. Turn the stop bolt (SB2) out of the lever arm (A2) to swing the pin P3 toward the back and the pin P4 toward the front of the machine.

(3) Mark the position of two adjacent corners of the hexagon nuts 1 and 3 on the bridge with a pencil for reference. In all subsequent adjustments involving the nuts on guide rods GR1 and GR2, loosen nuts 2 and 4, then make the necessary adjustments by turning nuts 1 and 3. Be careful to turn both nuts exactly the same amount with reference to the pencil marks placed on the bridge, and then tighten nuts 2 and 4. Place four springs between the platens, one at each corner. The plunger springs originally supplied on the first AA models are suitable. If these are not available, similar springs may be made by winding ¾-inch spring wire on a 0.665-inch-diameter rod. The finished spring should be about 2½ inches long and have a ½-inch pitch. Adjust the height of the bridge so that after the top platen has been raised and lowered with the springs in place, a 0.002-inch feeler gage can just be passed between the closing surfaces of the die holders at some one point but so that a 0.004-inch feeler gage cannot be passed between the die holders at any point. Loosen nuts 2 and 4. Turn nuts 1 and 3 down exactly one-twelfth turn (one-half of one face of the nuts) and tighten nuts 2 and 4 to give the correct die pressure.

(4) Recheck items (1) and (2) above. Also check the die pattern as described in section V, 1. Make further adjustments if necessary. If it is found necessary upon rechecking to adjust for items (1) and (2), make readjustments as specified in item (3).

Adjustment of the die closure as outlined above will produce a total deformation of parts of from 0.004 to 0.006 inch, which is distributed throughout the dies, die holders, platens, guide rods, connecting link, bridge, and lever system. The amount of this deformation is determined by the adjustments and the fact that the guide rods have 11 threads per inch. Thus the bridge will be raised or lowered 0.091 inch per turn of nuts 1 and 3. As all play in pins P1, P2, P3, and P4 is assumed to be taken up by the action of the springs between the platens, and as the bridge and upper platen assembly will be moved down approximately 0.008 inch by turning nuts 1 and 3 down one-twelfth turn, the difference between 0.008 inch and the clearance between the mating surfaces (0.002 to 0.004 inch) will be from 0.004 to 0.006 inch, and this must be accounted for by actual deformation of parts.

It is difficult to show a direct correlation between viscometer readings and the amount of deformation that would take place by a particular adjustment. It is well known, however, that as the die closure becomes loosen the reading will decrease, and as it becomes tighter the reading will increase. It is not definitely known whether the increased reading in the latter case is due to increased pressure on the test piece or increased friction in the machine due to deformation of parts. Probably the reading is influenced by both. As the closing pressures are decreased the reading will be influenced princi-
pally by the amount of the leakage that takes place. Actual cases have been observed where maladjustment of the die closures resulted in errors of plus or minus 5 units, depending on whether the closures were too tight or too loose. It should also be noted that even though the pressure on the die closure is too high, the reading may be low in cases where a badly worn die holder or poorly adjusted die holders permit undue leakage.

VI. PREPARATION OF TEST PIECES

The viscosity value obtained for a given material is dependent not only on the condition of the viscometer but upon the preparation of the test piece as well. The test pieces should be not less than ½ inch thick, so that one test piece below the rotor and one above the rotor will completely fill the die cavity with sufficient overflow to force the rubber into the corners of the die cavity and to force out as much air as possible. The test pieces should also be as free from air as it is practical to make them, and they should be free from pockets, which may trap air against the rotor or die surfaces. Furthermore, the test

![Figure 6](image_url)

*Figure 6.—Effect of time of milling on viscosity of different rubbers.*
piece placed below the rotor should have a hole punched through its center and be slipped over the rotor stem. Test pieces should never be cut and slipped around the rotor stem, as this method frequently traps air in the die cavity.

The statement has been made that air works out of the test piece and escapes through the plunger holes during operation of the machine. The writer has observed, however, that very little if any air escapes from the die cavity once the test piece is in the machine and the dies are closed. Normally part of the trapped air will be absorbed by the specimen and the remainder will collect in small pockets near the center of shear. This may be readily verified by observing the cross section of test pieces that contained considerable air at the time they were placed in the machine. Air in the test pieces may lower the whole viscosity time curve by 1 or 2 units, and will usually cause a much greater drop during the first 2 minutes of operation.

With the exception of GR–I, and possibly GR–M, suitable test pieces seldom can be prepared directly from bales of rubber obtained commercially. Some milling is required in order to eliminate at least part of the air and to knit the sample together. It is important, however, that rigid specification for milling be followed in order to get reproducible results between samples of the same material. In general, the Mooney viscosity is quite sensitive to milling, particularly during the first part of the milling operation. Figure 6 shows the effect of milling on the Mooney viscosity for natural rubber, GR–M, GR–S, and GR–I.

In testing compounded stocks not only the extent of milling and amount and kinds of compounding ingredients will affect the viscosity but also the mill temperature. Figure 7 shows the relationship between Mooney viscosity and mill-roll temperature for X–179 GR–S compounded in accordance with the specification for GR–S issued by the Rubber Reserve Company.

WASHINGTON, June 26, 1945.