

Comparison of Viscosities of Rubbers from the McKee Worker-Consistometer and from the Mooney Viscometer¹

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Viscosities of three GR-S rubbers and three GR-I rubbers were measured at 100° C with the McKee worker-consistometer, a piston-type capillary viscometer, and with the standard Mooney viscometer, a rotating disk instrument. Rates of shear from several tenths to several hundred sec⁻¹ were covered with the consistometer and from 0.1 sec⁻¹ to several sec⁻¹ with the Mooney viscometer. The procedure for preparing the samples and taking the measurements with the Mooney viscometer was selected as giving the best possible simulation of the conditions of the standard Mooney measurement, consistent with a valid indication of the temperature of the sample and as nearly as possible a like state of degradation at all rates of shear. The viscosities from the consistometer were calculated, using the Weissenberg-Rabinowitsch differentiation method for reducing observed flow data to fundamental quantities. The agreement of the results from the two instruments in their common range varies for the different rubbers, being better for the GR-S's. The best agreement is within about 2 percent, the uncertainty of the measurements, and the worst is within about 20 percent. The results from both instruments for X-518 GR-S agree with results obtained by Treloar for a "Standard GR-S" with a Piper and Scott biconical rotor modification of the Mooney viscometer. As the disagreements shown are not the same for all the rubbers they cannot be the result of constant instrumental differences, but must involve factors such as hydrostatic pressure, elasticity, and slippage at instrument surfaces.

I. Introduction

The McKee worker-consistometer [1],² which was recently adapted to measurements of the viscosities of raw rubbers [2, 3], is a piston-type capillary viscometer. Probably the most commonly used instrument for measuring such viscosities is the standard Mooney viscometer [4], which is based on a rotating disk. The results obtained with the two instruments for a given rubber should agree if, for both instruments, the method used for interpreting the observed data and for reducing them to fundamental quantities is truly applicable to the situation concerned. In order to check the applicability of the methods used it is necessary to compare results from the two instruments. Such comparison has been made with the results for three GR-S and three GR-I rubbers at 100° C.

II. Materials and Apparatus

The six rubbers for which viscosities were measured are listed, with descriptive data, in table 1.

The worker-consistometer used and its operation are described in reference [2, 3]. Rates of shear lower than those mentioned in reference [2, 3] were obtained by increasing the number of gears between the driving motor and worm gear.

The standard Mooney viscometer and its operation are described in reference [4]. However speeds other than the standard 2 rpm are reported here. In unreported measurements on the GR-S rubbers with this apparatus and with a biconical rotor Mooney viscometer [6], these two instruments gave identical viscosities for a given rubber if the samples were treated similarly in the two instruments.

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² Figures in brackets indicate the literature references at the end of this paper.

TABLE 1. Rubbers investigated

Rubber	Origin	Polymerization temperature	Standard Mooney reading ^a
		°C	
X-518 GR-S.....	Standard reference lot...	50	49
X-558 GR-S.....	do.....	5	59
X-478 GR-S.....	Production sample.....	5	64
Y-105 GR-I-17.....	Standard reference lot.....	-90	61
GR-I-18 B-13.....	Production sample.....	-90	78
GR-I-18 D.....	do.....	-90	81

^a 100° C, 2 rpm, large rotor, 4-min readings, GR-S samples prepared according to Taylor [5], GR-I's tested as received.

For this reason it was felt that the standard Mooney viscometer was satisfactory for the present comparisons, and that it was not necessary to use the biconical instrument, which has a more nearly uniform rate of shear throughout the flow field.

III. Measurements and Treatment of Data

1. Worker-Consistometer

Measurements were made in the worker-consistometer with a 50-hole shearing element at reciprocal rates of piston travel from about 13.6 hr/in. to 1 min/in. These rates correspond to rates of shear from about 0.3 to 500 sec⁻¹. The procedure in these measurements is as follows: A fresh sample of rubber (not mill-massed) is placed in the instrument and forced through the shearing element 10 times at a nominal rate of shear of 100 sec⁻¹. During these passes the apparent viscosity of the rubber increases. Previous measurements [2] show that the viscosity of the rubber at a given rate of shear remains nearly constant for the second 10 passes. The data at all rates reported are obtained between the tenth and the twentieth passes on one

filling of the instrument. In this instrument the differences between results for GR-S samples mill-massed for 25 passes and samples that have not been mill-massed are small enough to be ignored.

The observed pressure drop across the capillary (P) and volume rate of flow (Q) are converted to shearing stress at the capillary wall (s_w) and nominal rate of shear (D) by the usual relations

$$s_w = RP/2L, \text{ and } D = 4Q/\pi R^3, \quad (1)$$

where R and L are the radius (0.0188 cm) and length (0.635 cm), respectively, of the capillary. The actual rate of shear at the capillary wall ($\dot{\gamma}_w$) is obtained from the relation

$$\dot{\gamma}_w = (D/4) (3 + d \log D/d \log s_w). \quad (2)$$

This relation is the result of the differentiation method for reducing observed data to fundamental quantities. This method is described by Mooney [7] and others. The term $d \log D/d \log s_w$ is obtained for a given value of D by plotting $\log D$ against $\log s_w$ over the range available and measuring the tangential slope at the point concerned. In this paper viscosity (η) is defined as the ratio of shearing stress to rate of shear, regardless of whether the flow is Newtonian. According to this usage the quantities called viscosities are not material constants for non-Newtonian substances, but are merely helpful ratios, the numerical values of which vary with the rate of shear or the shearing stress. This usage has been adopted by Burgers and Scott-Blair [8] and by Reiner [9], and has been used by numerous other authors. The ratio $s_w/\dot{\gamma}_w$ is thus the viscosity corresponding to the rate of shear $\dot{\gamma}_w$.

2. Mooney Viscometer

When a fresh sample of any of the GR-S rubbers used (mill-massed according to Taylor [5]) is placed in a Mooney viscometer and the instrument started, the viscosity first rises to a maximum and then decreases continually with further shearing. This decrease results from molecular degradation. Viscosity results varying by as much as a factor of 2 can be obtained, depending on the procedure used in taking the measurements [10, 11]. The data reported here for the GR-S rubbers in the Mooney viscometer were obtained by a procedure that meets the following three requirements: (1) the rubber should be allowed to come to the temperature of the instrument before data are taken; (2) the data at all rates of shear should be taken on material in as nearly as possible a uniform state of degradation; and (3) conditions should simulate as nearly as possible those of the standard Mooney measurement. The procedure selected is as follows. A fresh, mill-massed sample is placed in the instrument, which has previously been heated to 100° C. Ten minutes is allowed for the rubber to reach the temperature of the instrument. The viscometer is then started at 2 rpm. At the end of 4 min the gage reading is recorded, and the speed is reduced to 1.5 rpm. As

soon as the gage reading becomes steady at the new speed (less than 1 min) it is recorded and the speed reduced again. Further readings are made at successive speeds of 1.0, 0.5, and 0.2 rpm. The speeds used correspond to rates of shear from 0.103 to 1.03 sec⁻¹. As GR-I rubbers do not show the above molecular degradation [5, 12], this procedure is not followed so exactly with them, and measurements are made at higher rates of shear.

For the Mooney viscometer the observed gage reading (G) and rate of revolution (W) are converted to a volume average viscosity (η) and rate of shear ($\dot{\gamma}$) by the approximate method given by Mooney [4]. Mooney found that a purely theoretical analysis had to be empirically corrected to give agreement with results from the "rubber rheometer" [13], a viscometer of more nearly uniform rate of shear developed by him. This correction is used here. The numerical relations used here are

$$\eta = 34,000G/W, \text{ and } \dot{\gamma} = 0.516W \quad (3)$$

IV. Results

Results on the six rubbers with the worker-consistometer and with the Mooney viscometer, used according to the procedures described above, are given in figures 1 through 6 as logarithmic plots of viscosity at 100° C versus rate of shear. These figures also show the viscosities calculated from the standard Mooney readings.

For X-518 GR-S (fig. 1) the two curves agree

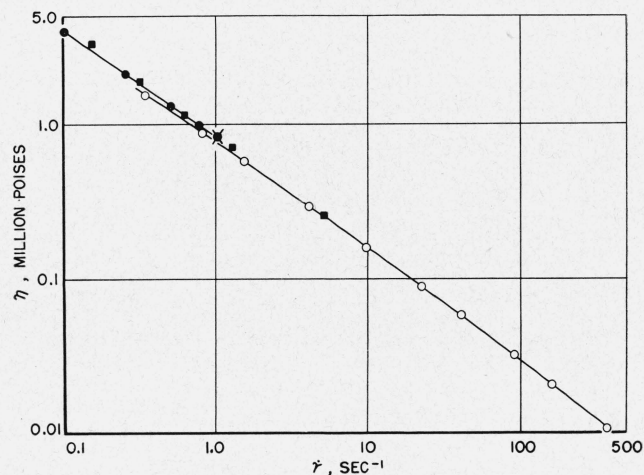


FIGURE 1. Viscosity of X-518 GR-S versus rate of shear.

■, Treloar; X, standard Mooney value; ○, worker-consistometer values; ●, Mooney values.

within 10 percent over their common range. The standard Mooney value indicated by the x agrees with the Mooney curve within 3 percent. The two curves have slightly different slopes in their common region. However, an extension of the Mooney curve to slightly higher rates of shear appears to show better agreement with the worker-consistometer curve.

For X-558 GR-S (fig. 2) all comparable data agree within the uncertainty of the measurements.

For X-478 GR-S (fig. 3) the only Mooney observation is the standard Mooney value. This value agrees with the worker-consistometer curve within the uncertainty of the measurements.

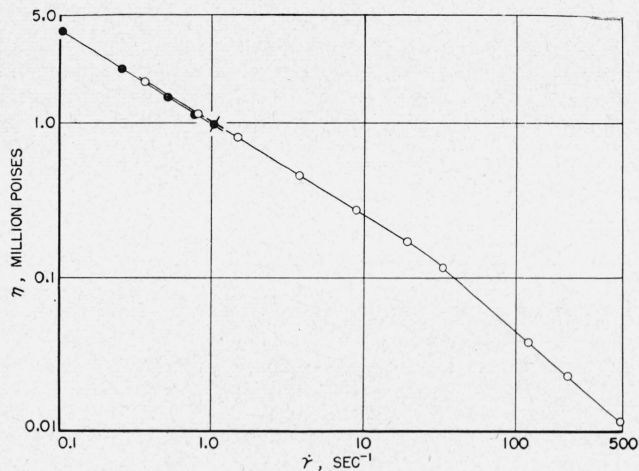


FIGURE 2. Viscosity of X-558 GR-S versus rate of shear.

X, Standard Mooney value; O, worker-consistometer values; ●, Mooney values.

The results for the three GR-I rubbers (figs. 4, 5, and 6) are sufficiently similar so that they may be mentioned collectively. For these rubbers the standard Mooney values are in complete agreement with the present Mooney curves. This result would be expected since no degradation occurs under the conditions of the experiments [5, 12], and it is presumably degradation that causes discrepancies here for the GR-S rubbers. In the region around 1 sec^{-1} the Mooney values are all about 20 percent higher than the corresponding worker-consistometer values. A Mooney curve and a corresponding worker-consistometer curve do not have identical slopes

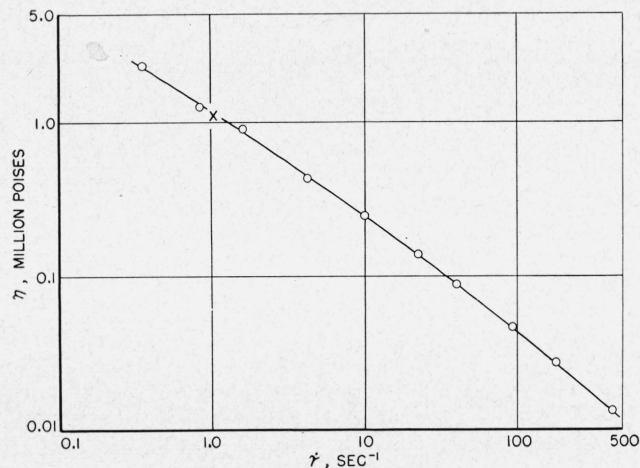


FIGURE 3. Viscosity of X-478 GR-S versus rate of shear.

X, Standard Mooney value; O, worker-consistometer values.

over their common region in every case. However, from figure 5 it appears that an extension of the Mooney curve to higher rates of shear may coincide with the worker-consistometer curve.

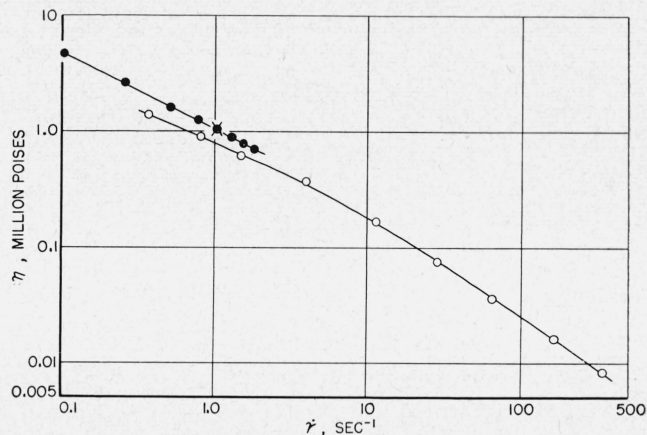


FIGURE 4. Viscosity of Y-105 GR-I-17 versus rate of shear.

X, Standard Mooney value; O, worker-consistometer values; ●, Mooney values.

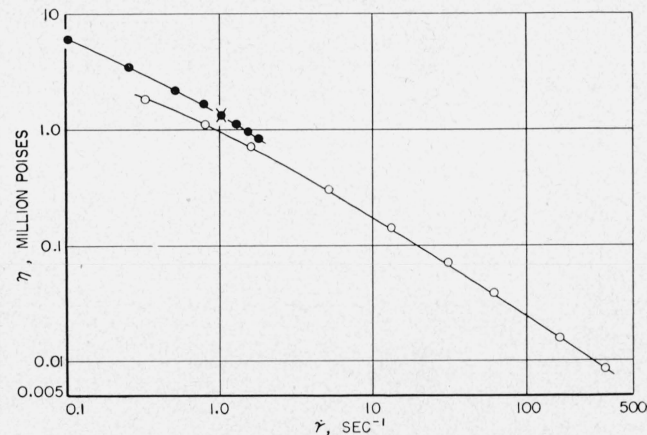


FIGURE 5. Viscosity of GR-I-18 B-13 versus rate of shear.

X, Standard Mooney value; O, worker-consistometer values; ●, Mooney values.

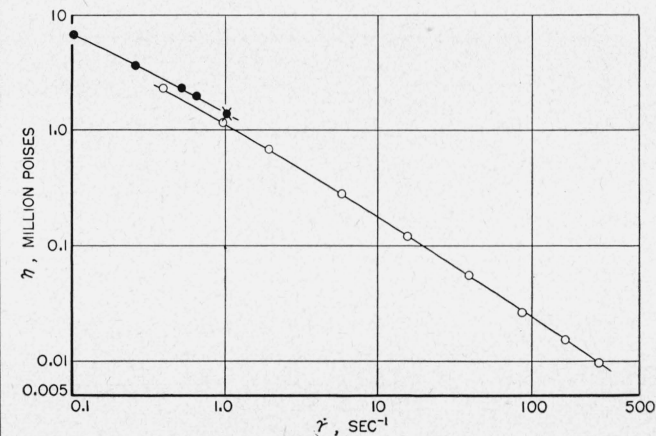


FIGURE 6. Viscosity of GR-I-18 D versus rate of shear.

X, Standard Mooney value; O, worker-consistometer values; ●, Mooney values.

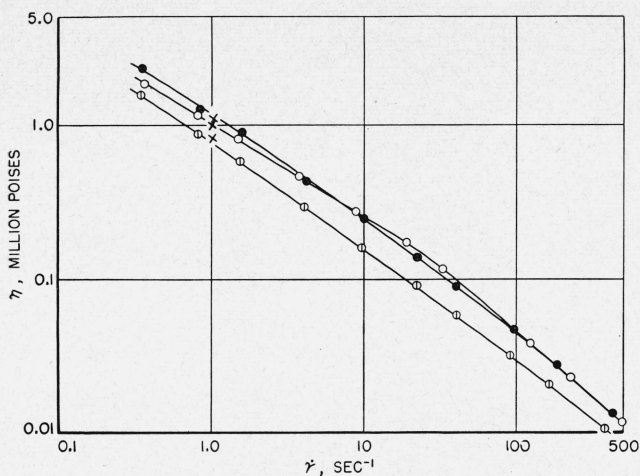


FIGURE 7. Worker-consistometer data for GR-S rubbers. X, Standard Mooney values; O, X-558 GR-S; ●, X-478 GR-S; □, X-518 GR-S.

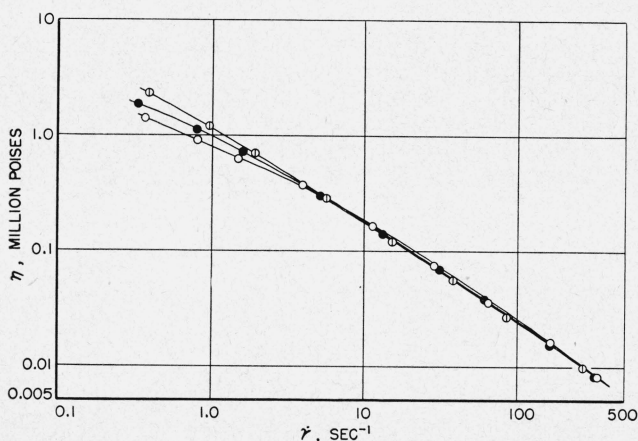


FIGURE 8. Worker-consistometer data for GR-I rubbers. ○, D GR-I; ●, B-13 GR-I; □, Y-105 GR-I.

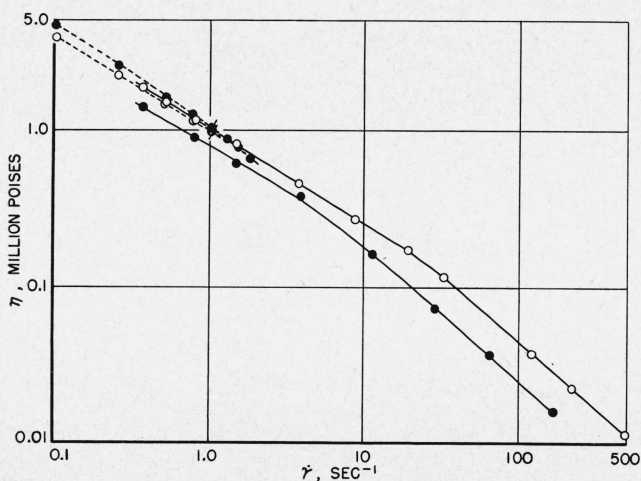


FIGURE 9. Flow curves for GR-S and GR-I rubbers. ○, X-558 GR-S; ●, Y-105 GR-I; ---, Mooney values; —, worker-consistometer values; x, standard Mooney values.

Figure 7 is a composite plot of the worker-consistometer data for the GR-S rubbers. It is included as a demonstration of the differences in the flow curves for GR-S rubbers. The plots for X-518 GR-S and for X-478 GR-S have approximately the same shapes, but different locations. The plots for X-558 GR-S and for X-478 GR-S have roughly the same locations, but different shapes.

Figure 8 is a similar composite plot for the GR-I rubbers. It is interesting that although the flow curves for these three rubbers are separated at lower rates of shear they are nearly coincidental at higher rates of shear.

In order to give a direct comparison of the data for a GR-S rubber with that for a GR-I, the results for X-558 GR-S and for Y-105 GR-I are plotted together in figure 9. The viscosity of the GR-I rubber shows the most rapid decrease at lower rates of shear than does that of the GR-S.

V. Discussion

Since the differences shown in figures 1 to 6 between the results from the two instruments for identical rubbers are not the same for all the rubbers, there is no constant instrumental difference between the instruments.

In the case of the GR-S rubbers the variation of the differences may represent varied degrees of degradation produced in different rubbers by the same procedure. Treloar [10] shows what large variations may be produced in the location of a flow curve by procedural differences. Unreported data of our own confirm this fact and also show that large differences in the slopes may be thus produced.

The differences between the results from the two instruments for the GR-I rubbers show a large scale similarity within the group. In all three cases the Mooney values are about 20 percent higher than the worker-consistometer values. Degradation cannot be a factor in these differences. However, since the differences do not show sufficient uniformity to be attributed completely to constant instrumental differences, they are apparently also contributed to by properties of the rubbers other than viscosity, such as elasticity, and adhesion to the viscometer surfaces. These properties may vary for the different rubbers and thus produce varied discrepancies. Known differences and variations in the hydrostatic pressures, and possible ones in the temperatures in the two instruments, would also result in differences that would not be related to the flow mechanics and that would vary for different rubbers having unlike pressure and temperature coefficients of viscosity. Another possible source of difference is the flow analysis for the Mooney viscometer, which is only approximate and may not take adequate account of the change of viscosity with rate of shear. Therefore the Mooney results on two rubbers having different flow curves may well compare differently with worker-consistometer results for the same two rubbers.

In the case of figure 1, a tentative comparison with literature can be made. Treloar [10] gives flow data obtained with a biconical rotor Mooney viscometer [6] for a rubber described as a "standard GR-S". Although this rubber is not described further, and the preparation of the sample is described only as "a preliminary light milling", the results probably represent a material similar to that treated in figure 1. Treloar's data have been expressed in the form of figure 1 and are plotted on that figure (■). Good agreement with the present results is shown, but it may be entirely fortuitous in view of the extreme dependence of results on the preparation of the sample.

VI. Conclusion

It can be concluded that in the worst cases there is agreement within about 20 percent between results from the worker-consistometer and those from the standard Mooney viscometer used according to the procedure described here. For two of the rubbers investigated the differences are within the limits of uncertainty of the measurements. The procedure used was selected as giving the best possible simulation of the conditions of the standard Mooney measurement, consistent with a valid indication of the temperature of the sample. The degree of agreement shown is within reasonable expectations in view of the confusing factors mentioned above.

It has also been demonstrated that the flow curves

for different rubbers of a given type can be of noticeably different characters.

Many of the calculations and all of the drawings for this paper were made by Miss H. V. Belcher.

VII. References

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