

NOTES

**A Simplified Method for Determining the Glass Transition Temperature of Elastomers\***

In the characterization of any new elastomer, one of the most important properties to be determined is the glass transition temperature. A variety of methods of measurement have been devised, based mainly on measurements of the coefficient of expansion as a function of temperature. Although some of these measurements are capable of considerable precision, they are generally time consuming and are not suitable as a routine test. It would be an advantage to have a technique that involves a minimum of both time and skilled operation.

One standard and widely used test of the low temperature properties of elastomers is the Gehman low temperature torsion flex test.<sup>1</sup> The results are usually expressed in terms of  $T_2$ ,  $T_5$ , etc., or in terms of the "freezing point" obtained by extrapolation of the steep portion of the curve. It has been found, however, that a more characteristic quantity that may be derived from the Gehman curve is the "inflection temperature," i.e., the temperature at which an inflection takes place in the steep portion of the curve. It has been found that there is a close relation between the inflection temperature and the glass transition temperature determined from dilatometer measurements. For best correlation it is preferable that the Gehman test be carried out with  $\theta = 160^\circ$  in the rubber region where the modulus is little affected by temperature. In all data reported here the test was made with  $\theta = 160^\circ$  at  $25^\circ\text{C}$ . When this was not convenient, a simple calculation<sup>1</sup> adjusted the data to this value. It is clear that if a material with a glass transition close to  $25^\circ\text{C}$ . is to be used, then the initial torsion flex readings must be made at a higher temperature.

TABLE I

Polymer	$T_g$	$T_{inf}$
SBR	-57	-43
Natural rubber	-72	-57
Natural rubber (tread stock)	-68	-54
High <i>cis</i> poly(butadiene)	-102	-90
Poly(1-methoxy butadiene)	-17	-4
Ethylene propylene copolymer #1	-40	-25
#2	-37	-20
Butyl rubber	-72	-58
Hypalon 20	-28	-14
Polyester urethane rubber #1	-29	-14
#2	-36	-23
#3	-44	-29
#4	-48	-34
#5	-53	-39
#6	-56	-40

\* Contribution #245 from these laboratories.

In Table I, results are given for a variety of elastomers. The Gehman curves were all run on either raw polymer or cured gum stocks. One example of a carbon black loaded tread stock is also included. The glass transition temperatures were determined using a dilatometer, usually with silicone oil as the confining fluid. The dilatometer was carefully designed to keep the rubber/fluid ratio as high as possible and hence increase the sensitivity of the measurements.

The data from Table I are plotted in Figure 1. It may be seen that there is a good correlation between  $T_g$  and  $T_{inf}$  with the former being  $15^\circ\text{C}$ . lower than the latter. Hence, from the Gehman curve  $T_g$  may be estimated with accuracy.

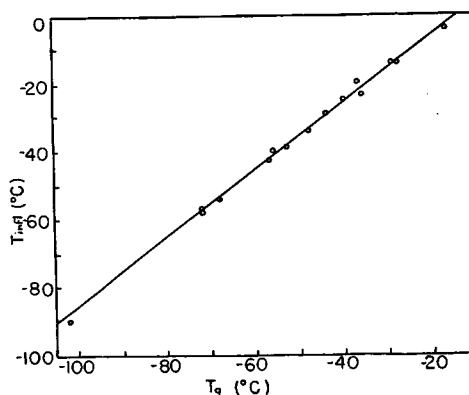


Fig. 1. Relation between glass transition temperature and inflection temperature.

A particular application of the simplified technique is in the determination of the composition of copolymers. Once a suitable calibration curve of  $T_g$  vs. composition has been established, copolymer composition may be estimated readily from the inflection temperature. For example, polyethylene and polypropylene differ by about  $100^\circ\text{C}$ . in their  $T_g$  values,<sup>2</sup> hence the composition of their copolymers may be readily determined from the inflection temperatures.

The author would like to express his appreciation to Mr. F. S. Farson, Dr. T. F. Yen, and Dr. R. E. Cunningham for preparing many of the polymers and to the Physical Testing Laboratory for the torsion flex data.

References

1. ASTM D1053-54T.
2. Dannis, M. L., *J. Appl. Polymer Sci.*, **1**, 121 (1959).

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Received January 6, 1960