RESULTS OF A ROUND ROBIN TEST SERIES TO EVALUATE COMPLEX MODULI OF A SELECTED DAMPING MATERIAL

by

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ABSTRACT

A Round Robin test series was conducted to establish the current state of the art in the measurement of complex moduli of polymeric damping materials as functions of frequency and temperature. Several laboratories participated, using a variety of test systems, providing data over the temperature range from -50°C to +100°C, and the frequency range from 1E-5 Hertz to over 10000 Hertz. The paper briefly describes the selection of the test material, the test methods used, and the means used to establish the accuracy of each data set and identify random and systematic errors. The results show the manner in which data from different test techniques can differ and sources are identified for some of these differences. Frequency-temperature analysis of the data is discussed.

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INTRODUCTION

In recent years it has become apparent that the routine process of measuring the complex modulus properties of polymeric materials has been conducted far too frequently without sufficient care and attention to ensuring that the data was free of errors. The outcome has been particularly unfortunate for those concerned with the application of existing data to the design of damped structures, but it has affected other users too, such as those interested in relating the observed behavior of the complex modulus properties with molecular structure. Clearly, if the data being used for comparison is erroneous, the conclusions of the research may also be erroneous. It was against this background that the Round Robin test series was organized. Round Robins have been conducted before, and have rarely been completely successful. This one may have been somewhat more successful than most, and some of the conclusions are interesting and may be of value to those concerned with improving the accuracy of future complex modulus testing.

ORGANIZING THE ROUND ROBIN

The Round Robin was organized by the author, then at the Materials Laboratory, Wright Patterson Air Force Base, Ohio, in conjunction with an international cooperative program. Many organizations in the USA and abroad were contacted, initially through a government to government cooperative program and later on a more informal basis. These organizations are summarized in Table 1. The material selected for the testing was a commercial polymer based damping material, available in sheets of various thicknesses from which various specimen geometries could be accomodated from EAR Corporation, Indianapolis, Indiana. The material identity (EAR C-1002) was not divulged to the participating organizations, though doubtless many could recognize the material. Each laboratory was requested to provide their specimen size requirements and cut specimens approximating those sizes were sent to each participant, along with limited guidelines concerning the test conditions. The main requirements are summarized in Table 2. The tests were conducted by each laboratory, as resources and time permitted, and the results were returned to the author for analysis and evaluation. Some results are still awaited, so the analysis described, in part, in this paper is still incomplete. Further analysis, and the writing of the final report, is yet to be accomplished.

ANALYSIS OF THE TEST DATA

The test data provided by each laboratory covered a wide range of test systems, as summarized on an anonymous basis in Table 3, and over a wide frequency range, as illustrated in Figure 1. The very wide frequency range provided a unique basis for evaluating the accuracy of the various data sets.

WICKET PLOT ANALYSIS

The first task was to evaluate the accuracy of each data set. This has not been a simple task in the past, but the relatively recent emergence of the Wicket Plot as a means of identifying possible errors has greatly simplified the matter. The Wicket Plot is a graph of log (loss factor)

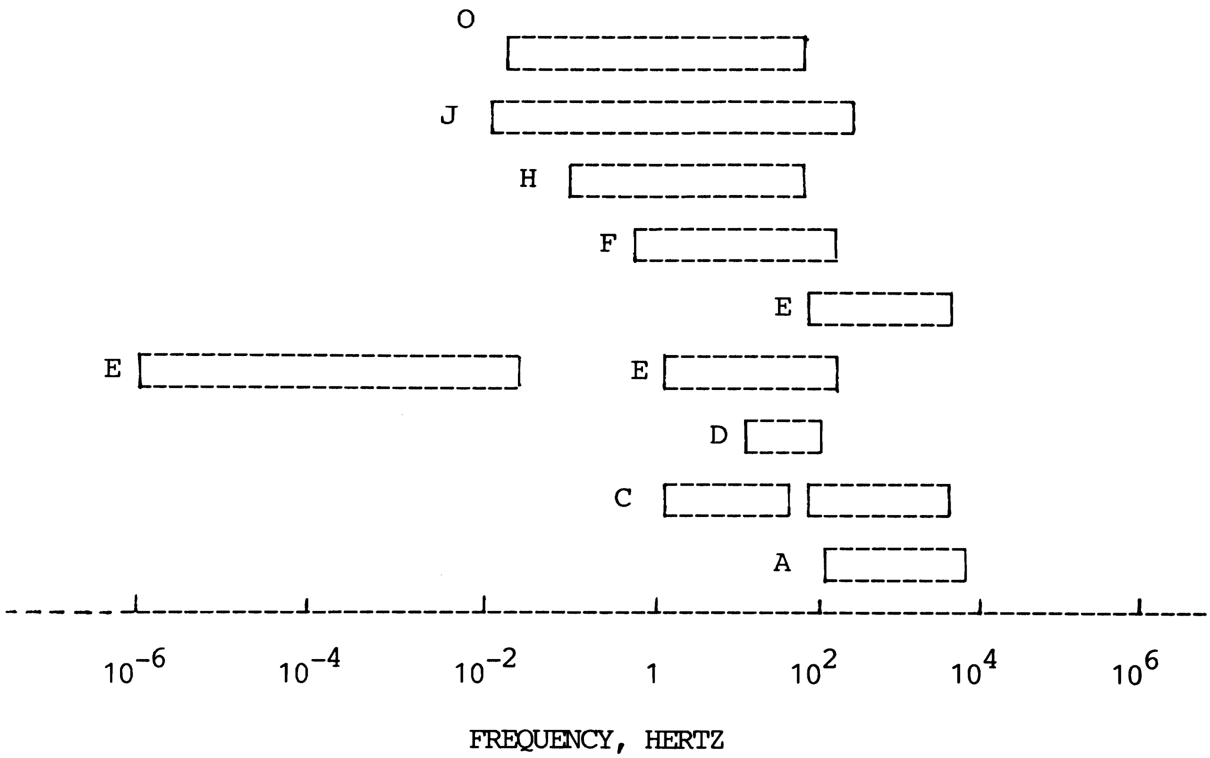

Figure 1. Frequency ranges evaluated by each laboratory

versus log (modulus) and differs from the Cole-Cole plot, which is a linear scale plot of imaginary modulus versus real modulus, in that the use of the logarithmic scale effectively accomodates the very wide range of values of the shear or Young's modulus exhibited by most polymeric materials. A linear scale may display one, or even two decades, of modulus values but only a logarithmic scale can effectively display the four or five decades often encountered. The Wicket plot is, theoretically, a unique curve for each polymer provided that no non-stationary, or time dependent, processes are taking place, and the material complex modulus behavior is thermorheologically simple, so any deviations from such a unique curve must be due to errors or time-dependent effects and in this way one may determine whether frequency-temperature analysis (reduced variables) is likely to be useful before expending often considerable effort in vain.

Representative wicket plots, for some of the test data, are shown in Figures 2 to 17, for Laboratories A,C,D,E,F,H,M,N and O in the shear and extensional modes of deformation. Each figure illustrates the types of error which may arise for the various test systems. These include random scatter, especially for the beam tests conducted by Laboratory A, deviations from the main sequence due to high specimen stiffnesses in direct stiffness test systems (Laboratories C,D,E,F,H,J,M,N and O) and deviations due to high frequency (inertia) effects in direct stiffness systems.

VARIATION OF COMPLEX MODULI WITH FREQUENCY

The Wicket Plot is the Occam's razor of complex modulus data quality analysis, in the sense that an inadequate Wicket plot at once implies that the data is suspect. However, to pass the Wicket plot test is not to guarantee the quality of the data, since temperature and frequency have been eliminated in the Wicket plot and any errors in these dimensions must also be checked. Figures 18 and 19 show typical plots of modulus and loss factor versus frequency, again on a log-log scale, at - 10°C and - 20°C, respectively. It is seen that some of the errors highlighted in the Wicket plots are reflected here also, and are in some cases more clearly seen. These plots, along with data for other temperatures, form the basis for constructing master plots of modulus and loss factor versus reduced frequency. Figure 20 shows such a master plot, generated by manual estimates of the shift factors. While the scatter is evident, the agreement between results for so many laboratories is quite remarkable. The corresponding shift factors are plotted against 1/T in Figure 21. The results lie fairly close to an Arrhenius shift relationship, of the form $\log \alpha(T) \propto 1/T$, but the small deviations may be significant. The scatter, unfortunately, is too great to provide a definitive settlement of the matter.

CONCLUSIONS

The purpose of this paper has been to present the interim conclusions of a major Round Robin test series to measure the complex modulus properties of a commercial damping material sample by several test techniques, at several test locations, over wide frequency and temperature ranges. Many laboratories participated, and several more are expected to complete their tests in the near future. It is clear that all the test techniques used can produce accurate data over specific ranges of modulus and frequency, but that outside of these ranges major errors can readily be encountered. The Wicket Plot has emerged as an effective tool for detecting some of these errors. It is recommended that more than one test technique be used if data is required over a wide range of moduli, as would occur over any temperature range wide enough to encompass the rubbery, transition and glassy regions of the material behavior. A full report of all the Round Robin tests will be issued when all testing and analysis has been completed.

NOMENCLATURE

A	Load carrying area (direct stiffness tests)
E	Young's Modulus (MPa)
G	Shear Modulus (MPa)
h	Thickness of shear specimen (direct stiffness tests)
h_1	Thickness of metal beams (beam tests)
h_2	Thickness of polymer layer (beam tests)
L	Length of extensional specimen (direct stiffness tests)
Q	Activation energy
R	Universal Gas Constant
T	Absolute temperature (°K)
T_A	Activation temperature (=Q/R, °K)
η	Loss factor
$\alpha(T)$	Shift factor

TABLE 1. PARTICIPATING ORGANIZATIONS

Admiralty Research Establishment, Holton Heath, Poole, England (J.R. House)
Anatrol Corporation, Cincinnati, Ohio (A.D. Nashif)
CSA Engineering Inc., Palo Alto, California (C. Johnson)
DCAN Toulon, Toulon Naval, France (M. Khoury)
Dow Corning Corporation, Midland, Michigan (N. Langley)
Institue of Sound & Vibration Research, Southampton, England (R.C. Drew)
Lehigh University, Bethlehem, Pennsylvania (L. Sperling)
MTS Corporation, Minneapolis, Minnesota (K. Biegler)
National Physical Laboratory, Teddington, England (G. Dean)
Naval Underwater Systems Center, New London, Connecticut (W. Maciejewski)
Polymer Laboratories Inc., Amherst, Massachusetts (J.C. Duncan)
TA Instruments, Cincinnati, Ohio (J. Schilthuis)
Tufts University, Medford, Massachusetts (D. Walker)
University of Dayton Research Institute, Dayton, Ohio (M.L. Drake)
University of Manchester, Manchester, England (G.R. Tomlinson)
Wright Laboratories, Materials Laboratory (D.I.G. Jones)
Wright Laboratories, Flight Dynamics Directorate (L.C. Rogers)

TABLE 2. ROUND ROBIN TEST REQUIREMENTS

Temperature range:	-20°C to +90°C in steps of 10°C (later amended to -50°C low temperature)
Frequency range :	Tester's option
Temperature soak :	At least 30 minutes before testing at each temperature.
Test condition :	Isothermal
Pre-strain :	None (preferably)
Strain amplitude :	Less than 0.1 percent (linear range)
Test methods :	Testers option
Units :	SI (preferably)
Bonding :	Tester's option

TABLE 3. TEST SYSTEMS USED BY ROUND ROBIN PARTICIPANTS

LAB	TEST SYSTEM	COMMENTS
A	7 in. Oberst Beam (E) 7 in. Sandwich Beam (G)	Limited range for extensional data at low moduli. Random scatter in shear data.
B	Direct Stiffness (E)	Resonances in test range. No usable data.
C	Direct Stiffness (HFSMA)(G) Polymer Labs DMTA (E)	Errors in dual cantilever configuration.
D	Direct Stiffness (G,E)	Some scatter at low moduli
E	10 in. F-F Homogeneous beam(E) 10 in. Oberst beam (E) 6 in. C-F homogeneous beam(E) 10 in. C-F sandwich beam (G) Resonance (E,G) Direct stiffness (G,E) Metravib Viscoanalyzer (G,E) Creep/Relaxation(G,E) Low frequency impedance	Round Robin test program within the main test program. Widest temperature and frequency ranges.
F	Direct stiffness (G)	Limited number of temperatures and limited data at high modulus values.
G	None	Analysis only.
H	Polymer Labs DMTA (G,E)	Scatter at low moduli, high modulus offset and limited range of Young's moduli.
I	No test data yet received.	
J	MTS 831.50 Elastomer Test system	High modulus offset, some discrepancies, and limited temperature range.
K	None	Analysis only.
L	Fitzgerald system	Testing in progress
M	Metravib Viscoanalyzer (E,G)	Some scatter and high modulus offset for extensional and shear data.
N	Direct stiffness systems	Testing completed
O	Rheometrics RDA2 system (G) Rheovibron (E)	High modulus offset and some erroneous data at low modulus values. Considerable scatter at low moduli, high modulus offset, and only two test frequencies.
P	No data yet received.	
Q	No data yet received	

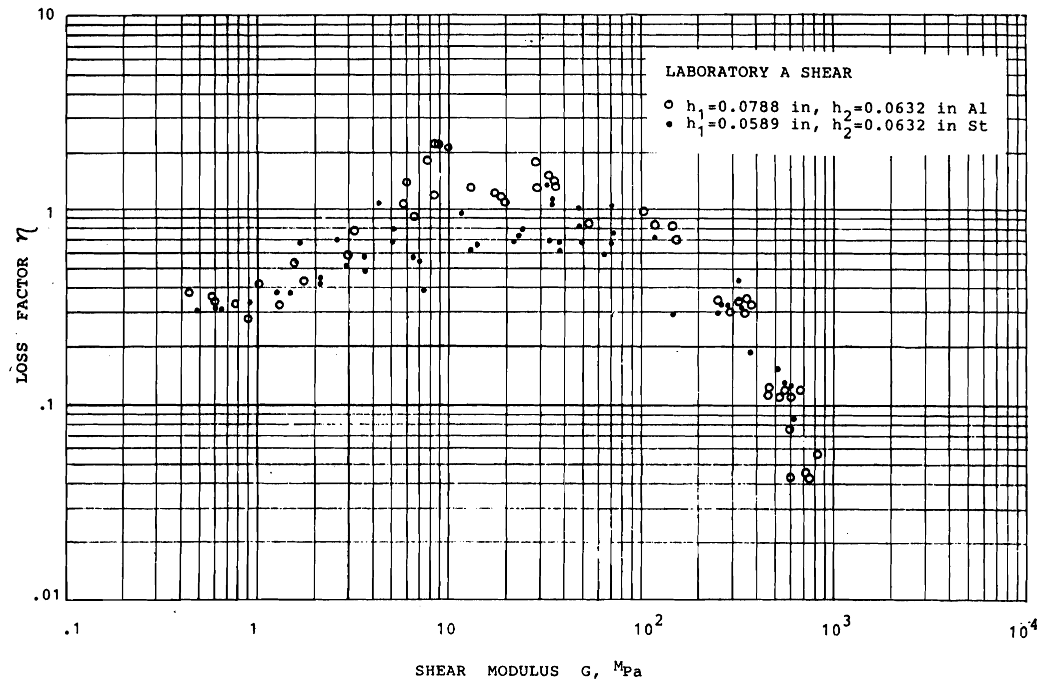

Figure 2. Wicket Plot for Laboratory A Shear Data (o h_1 = 2 mm, h_2 = 1.61 mm, Aluminum; • h_1 = 1.50 mm, h_2 = 1.61 mm, Steel)

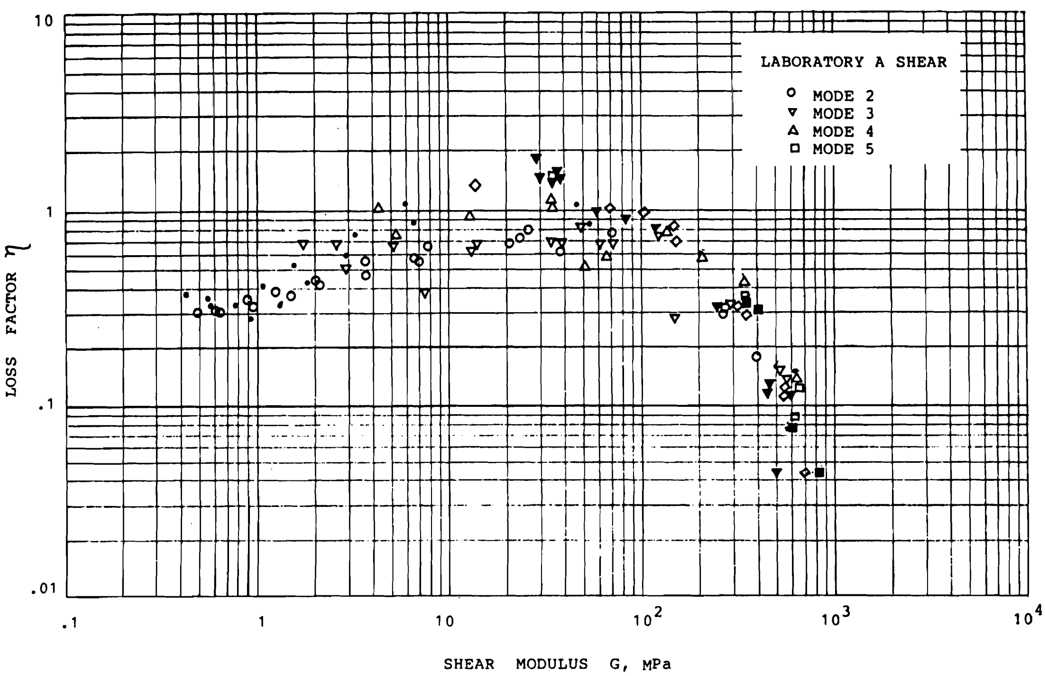

Figure 3. Wicket Plot for Laboratory A Shear Data (o Mode 2, ▽ Mode 3, △ Mode 4, ◻ Mode 5)

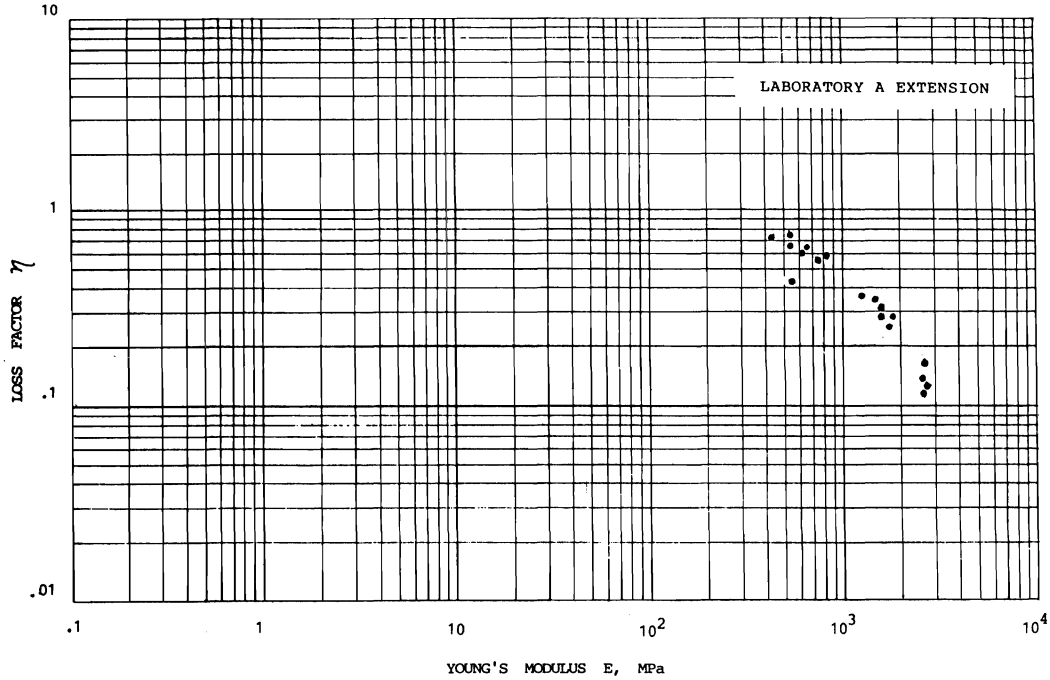

Figure 4. Wicket Plot for Laboratory A Extensional Data (h_1 = 1.52 mm, h_2 = 1.61 mm, Steel Oberst Beam)

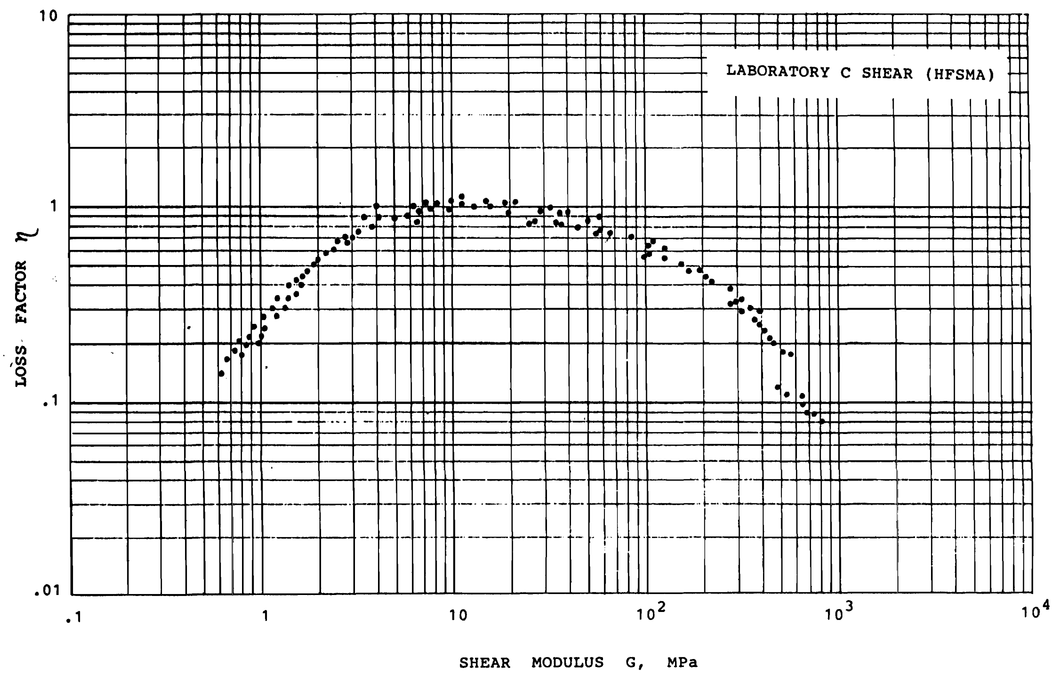

Figure 5. Wicket Plot for Laboratory C Shear Data (Direct Stiffness Method/HFSMA, thickness = 1.9 mm, diameter = 10 mm, A/h = 41.34 mm)

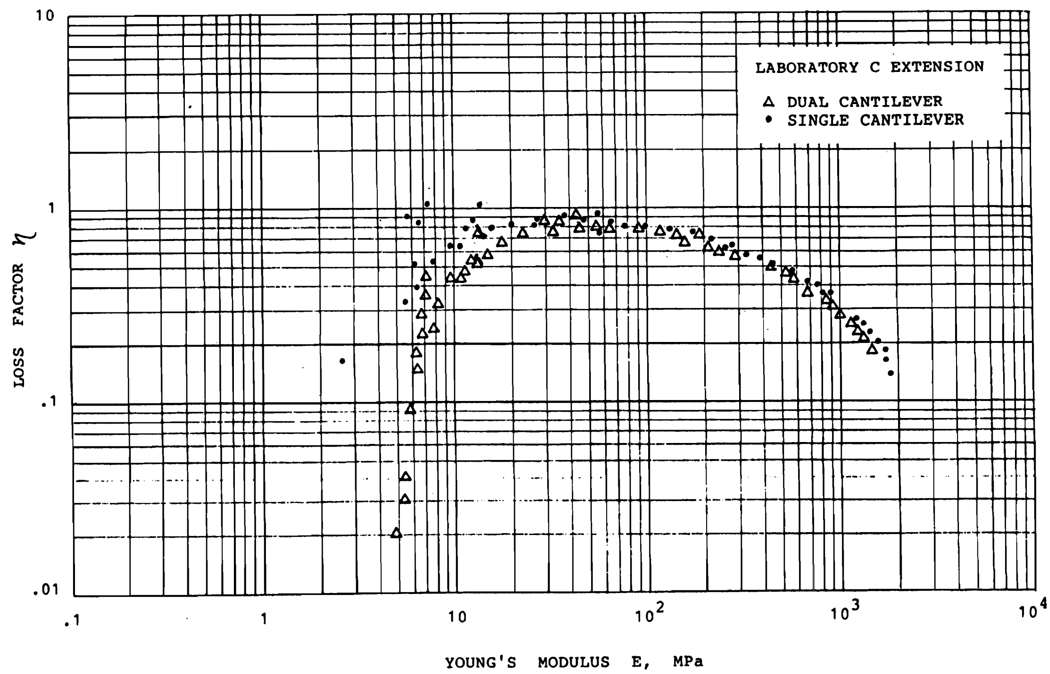

Figure 6. Wicket Plot for Laboratory C Extensional Data (Direct Stiffness Technique/PL DMTA, flexural mode, 1.24 x 12.21 x 8 mm sample)

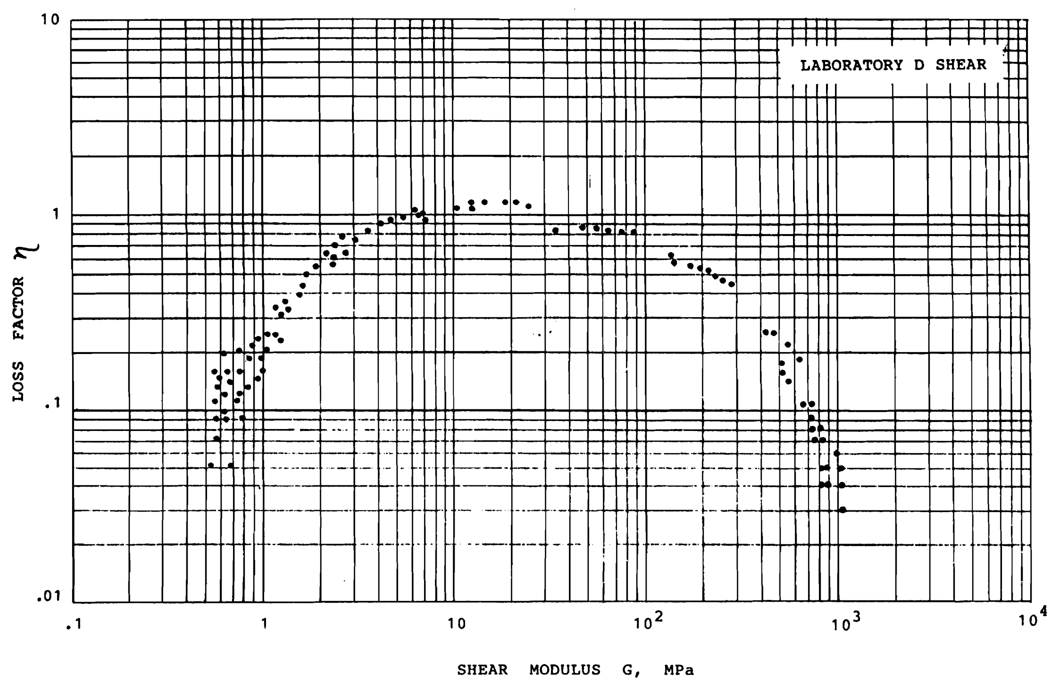

Figure 7. Wicket Plot for Laboratory D Shear Data (Direct Stiffness Method/Double Lap Shear Specimen, thickness = 12 mm, Net Area = 321.86 mm^2, A/h = 26.82 mm)

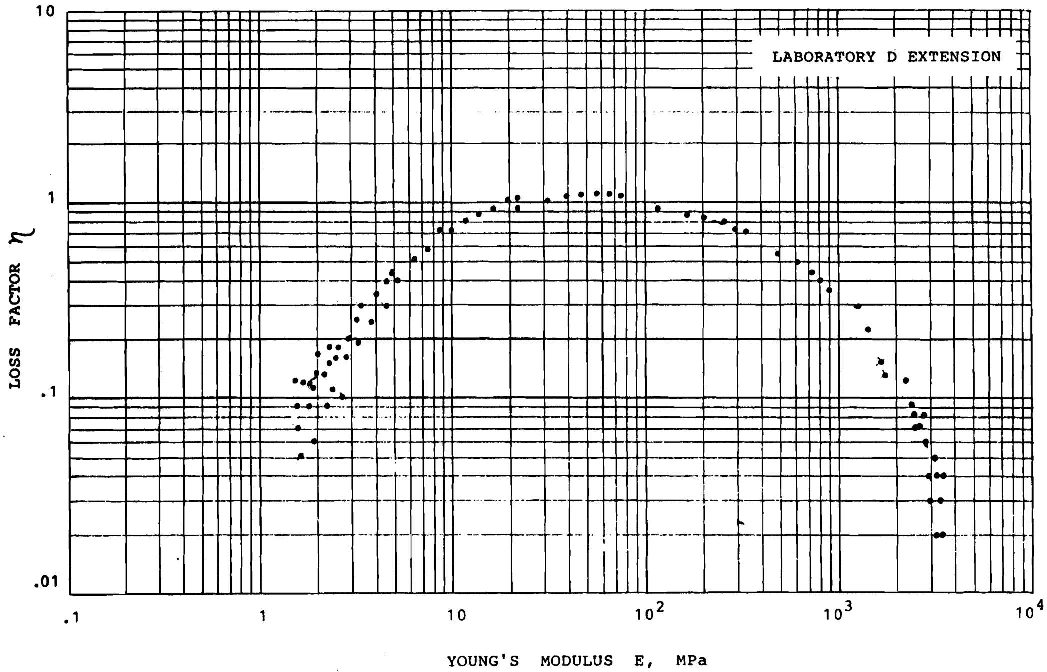

Figure 8. Wicket Plot for Laboratory D Extensional Data (Direct Stiffness Method, Length = 17.5 mm, area = 160.93 mm², A/h = 9.20 mm)

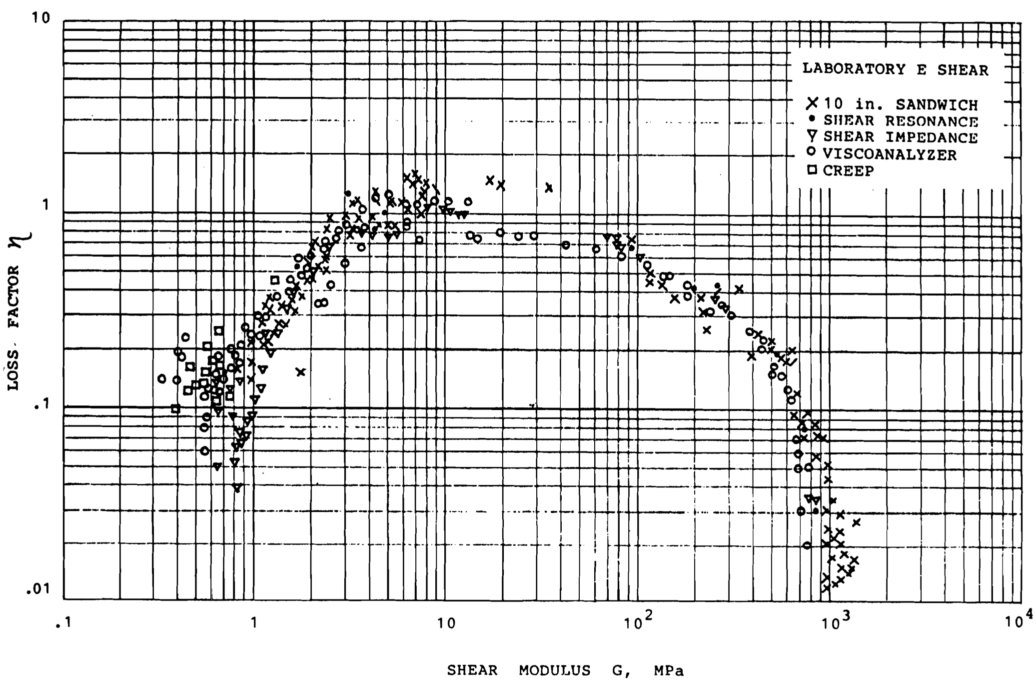

Figure 9. Wicket PLot for Laboratory E Shear Data (X 10 inch sandwich Cantilever, • Resonance, ∇ Impedance, o Viscoelasticimetre, □ Creep)

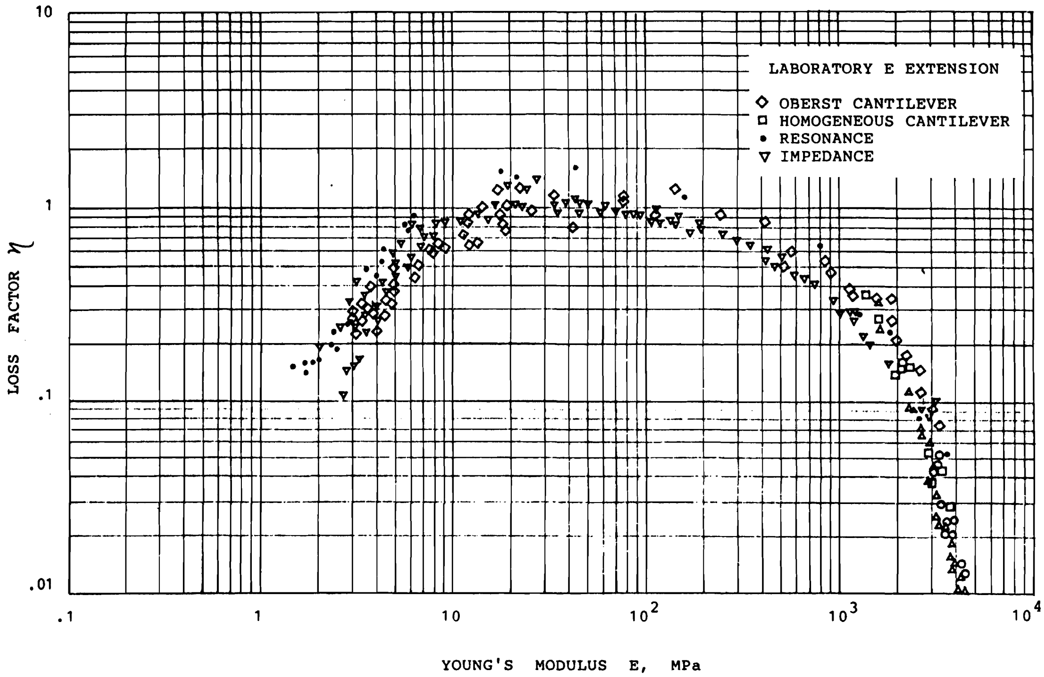

Figure 10. Wicket Plot for Laboratory E Extensional Data (◇ Oberst Cantilever, □ Homogeneous Cantilever, △ Homogeneous Free-Free, • Resonance, ▽ Impedance: L = 44 mm, Area = 196.63 mm^2, A/L = 4.47 mm)

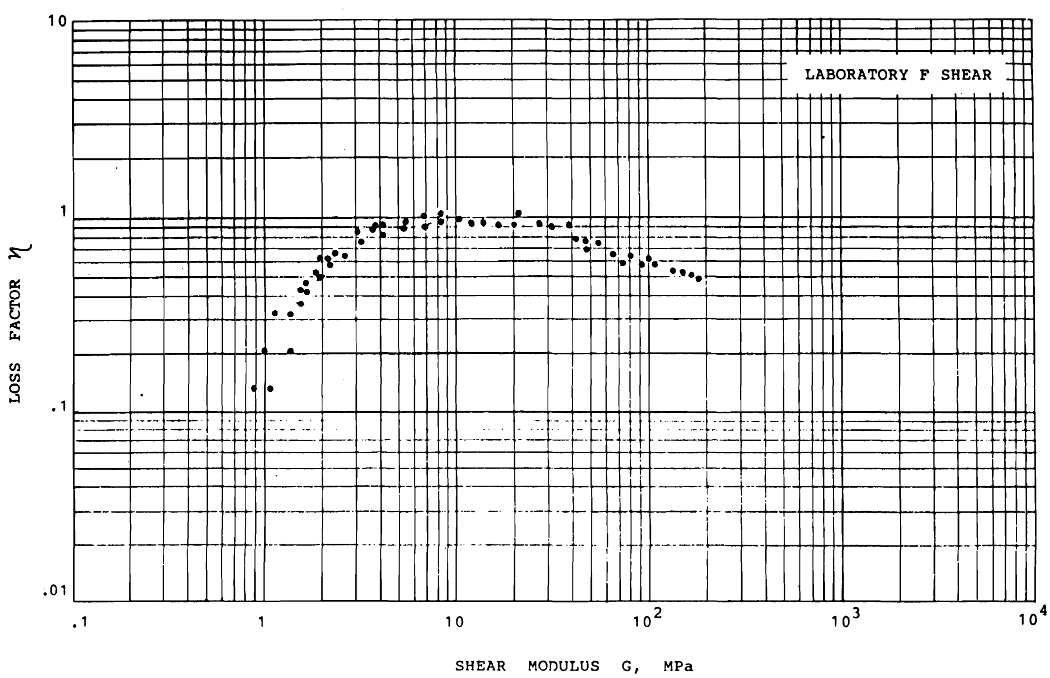

Figure 11. Wicket Plot for Laboratory F Shear Data (Direct Stiffness)

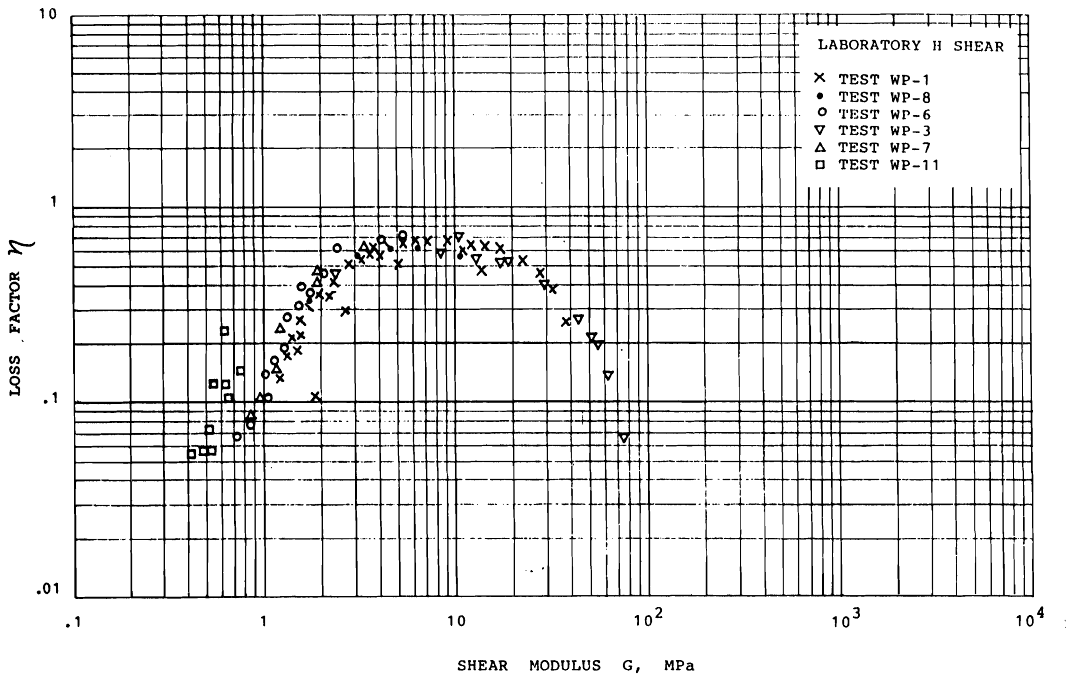

Figure 12. Wicket Plot for Laboratory H Shear Data (Direct Stiffness Method/PL DMTA, × Test WP-1, • Test WP-8, o Test WP-6, ▽ Test WP-3, △ Test WP-7, □ Test WP-11)

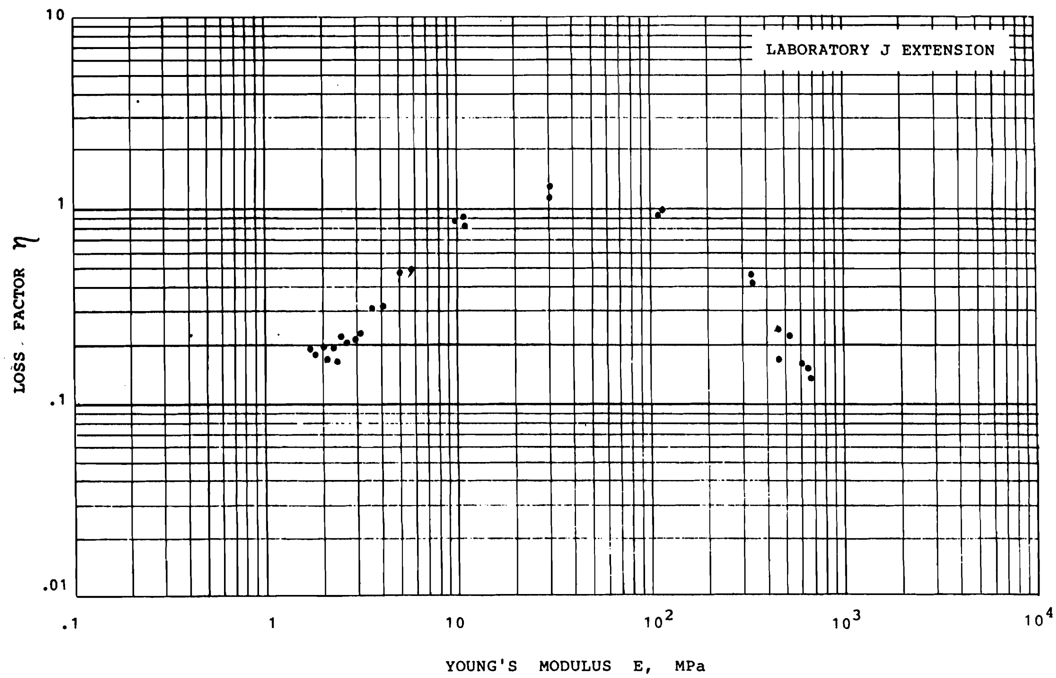

Figure 13. Wicket Plot for Laboratory J Extensional Data (Direct Stiffness Method, thickness = 12.4 mm, Area = 490.0 mm^2, A/h= 35.59 mm)

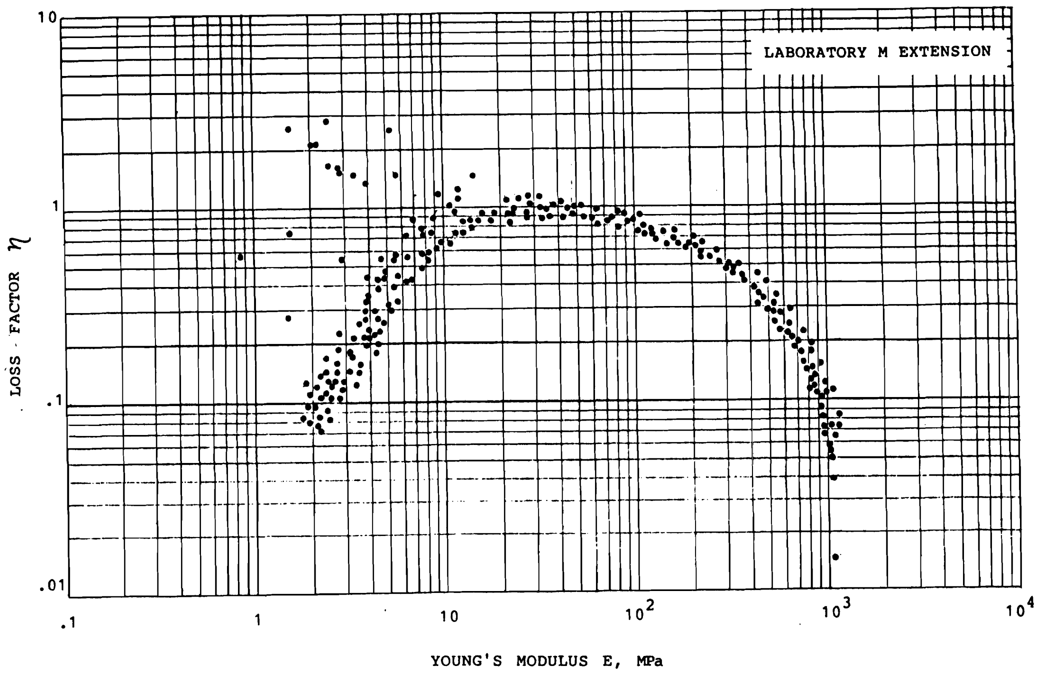

Figure 14. Wicket Plot for Laboratory M Extensional Data (Direct Stiffness Method/Viscoelasticimetre, length = 31.28 mm, Area = 447.08 mm^2, A/h = 14.29 mm)

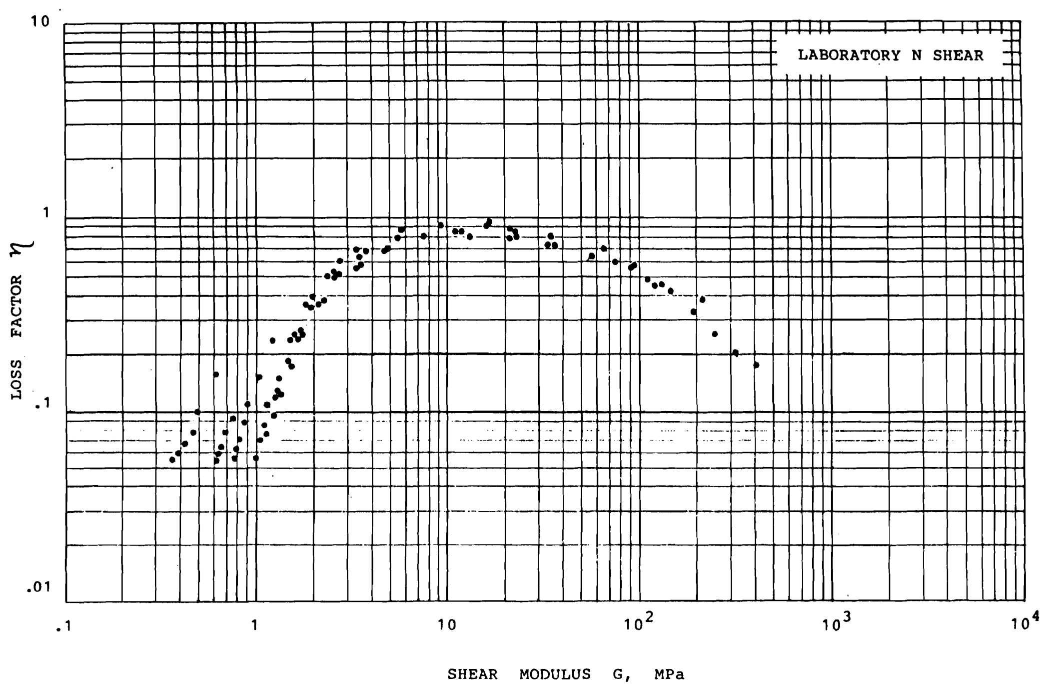

Figure 15. Wicket Plot for Laboratory N Shear Data (Direct Stiffness)

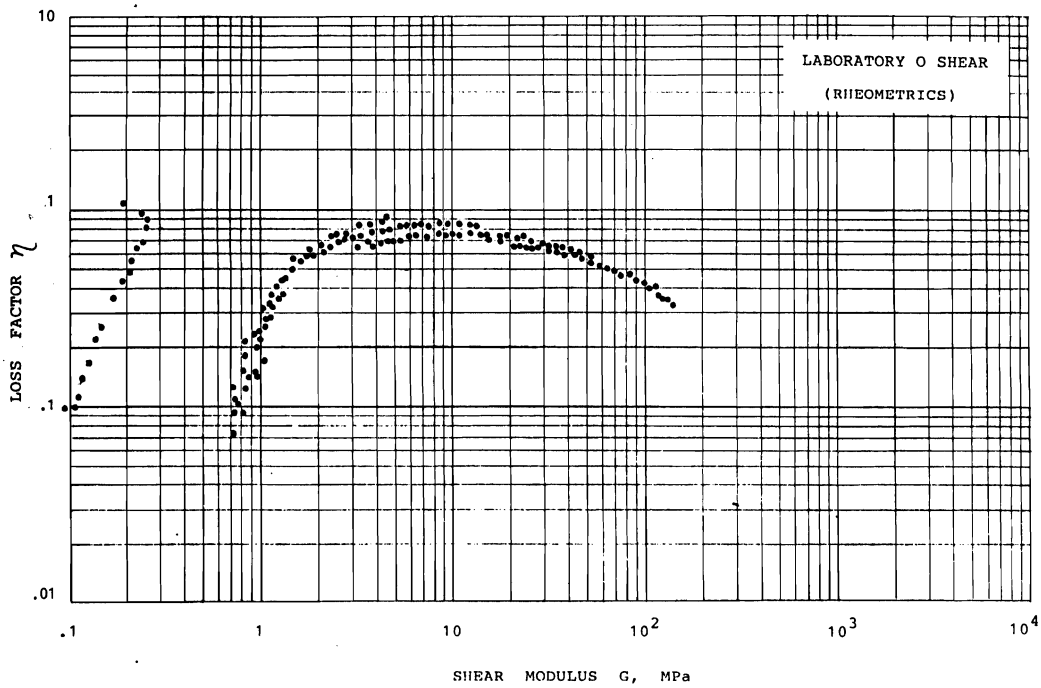

Figure 16. Wicket Plot for Laboratory O Shear Data (Rheometrics Analyzer thickness = 3 mm, Area = 49.02 mm^2, A/h = 16.43 mm)

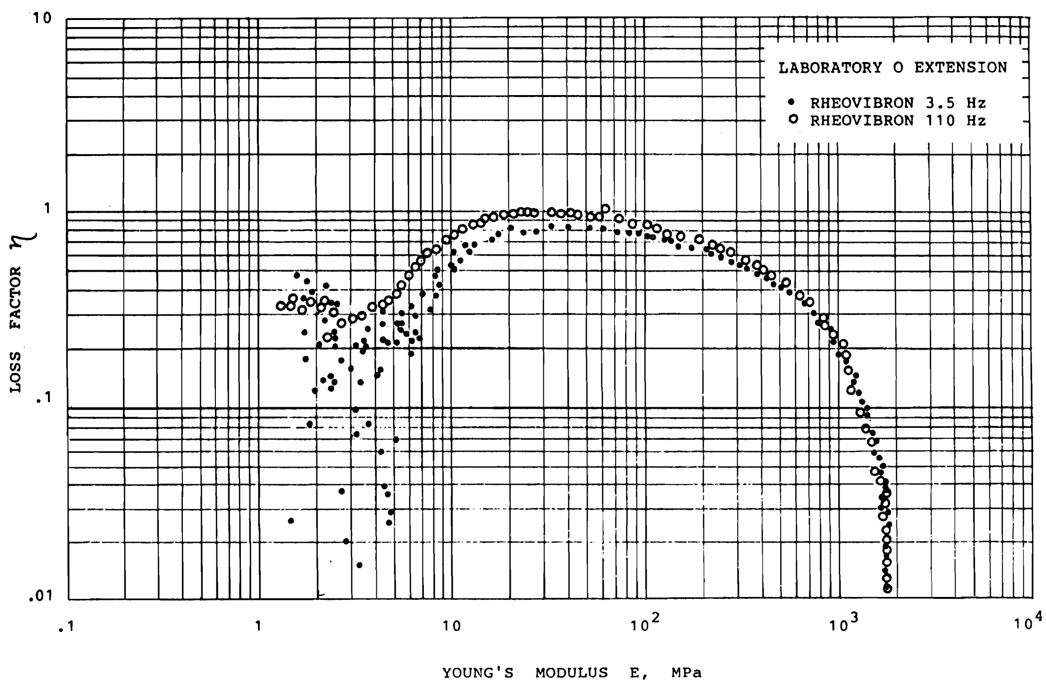

Figure 17. Wicket Plot for Laboratory O Extensional Data (Rheovibron Test System, L = 16.52 mm, Area = 24.36 mm^2, A/L = 1.47 mm)

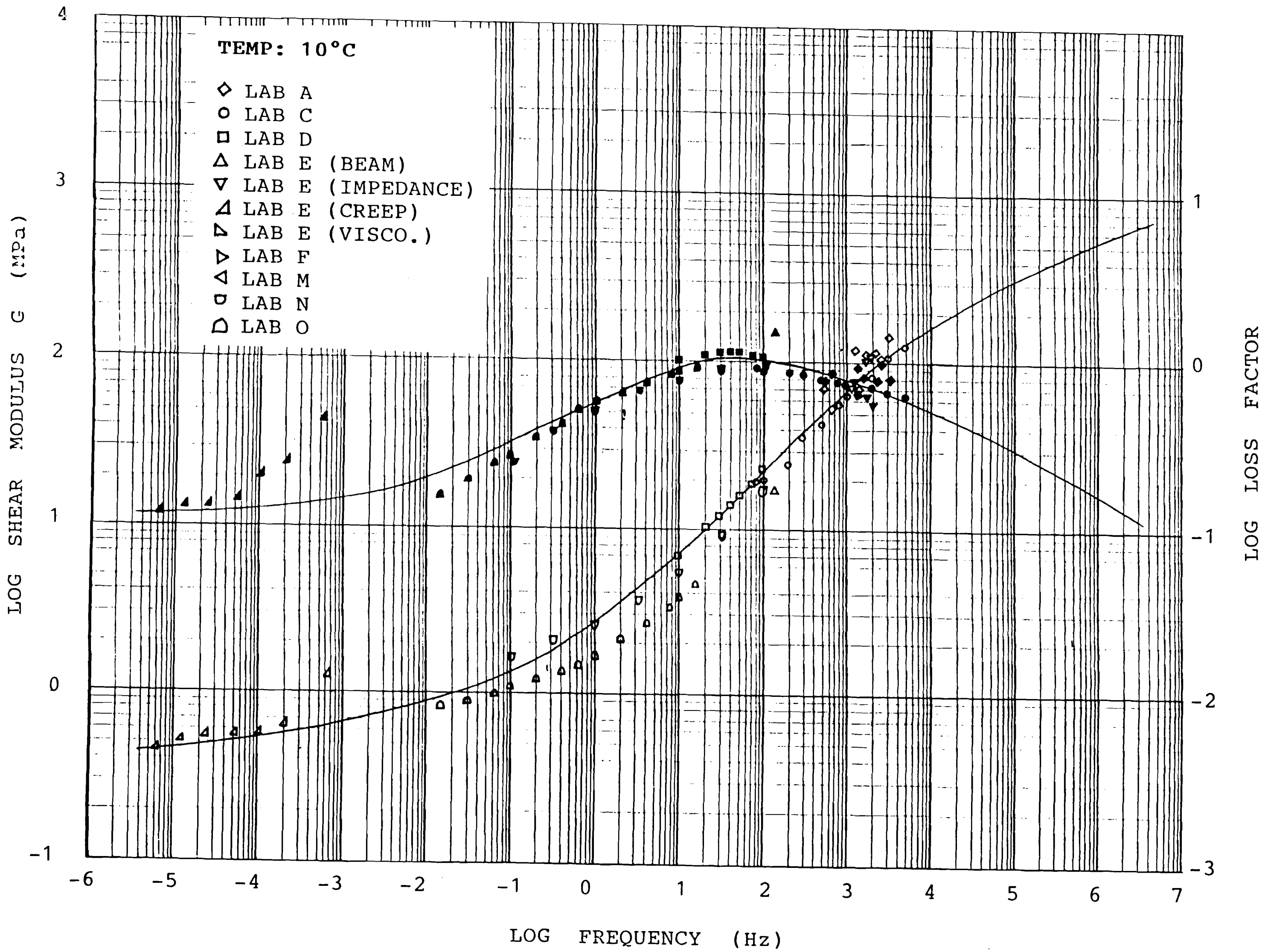

Figure 18. Plot of Shear Modulus and Loss Factor versus Frequency at + 10° (◇ Lab A, ○ LabC, □ Lab D, △ Lab E (beam), ▽ Lab E (Impedance), ◿ Lab E (Creep), ◺ LabE (Viscoelasticimeter), ▷ Lab F, Ʊ Lab N, ◁ Lab M, Ω Lab O)

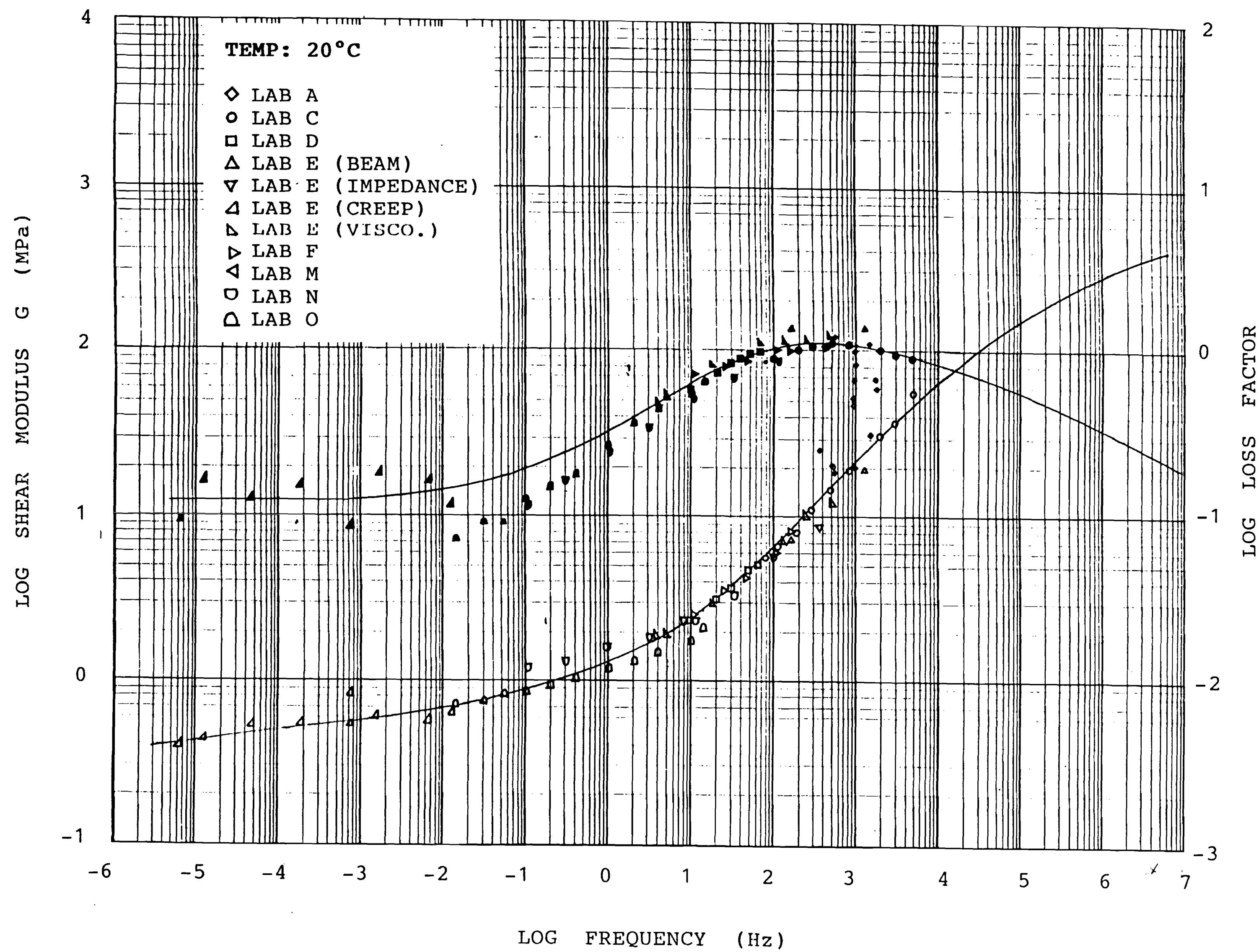

Figure 19. Plot of Shear Modulus and Loss Factor versus Frequency at + 20° (◇ Lab A, ○ LabC, □ Lab D, △ Lab E (beam), ▽ Lab E (Impedance), Lab E (Creep), LabE (Viscoelasticimeter), ▷ Lab F, Lab N, ◁ Lab M, Lab O)

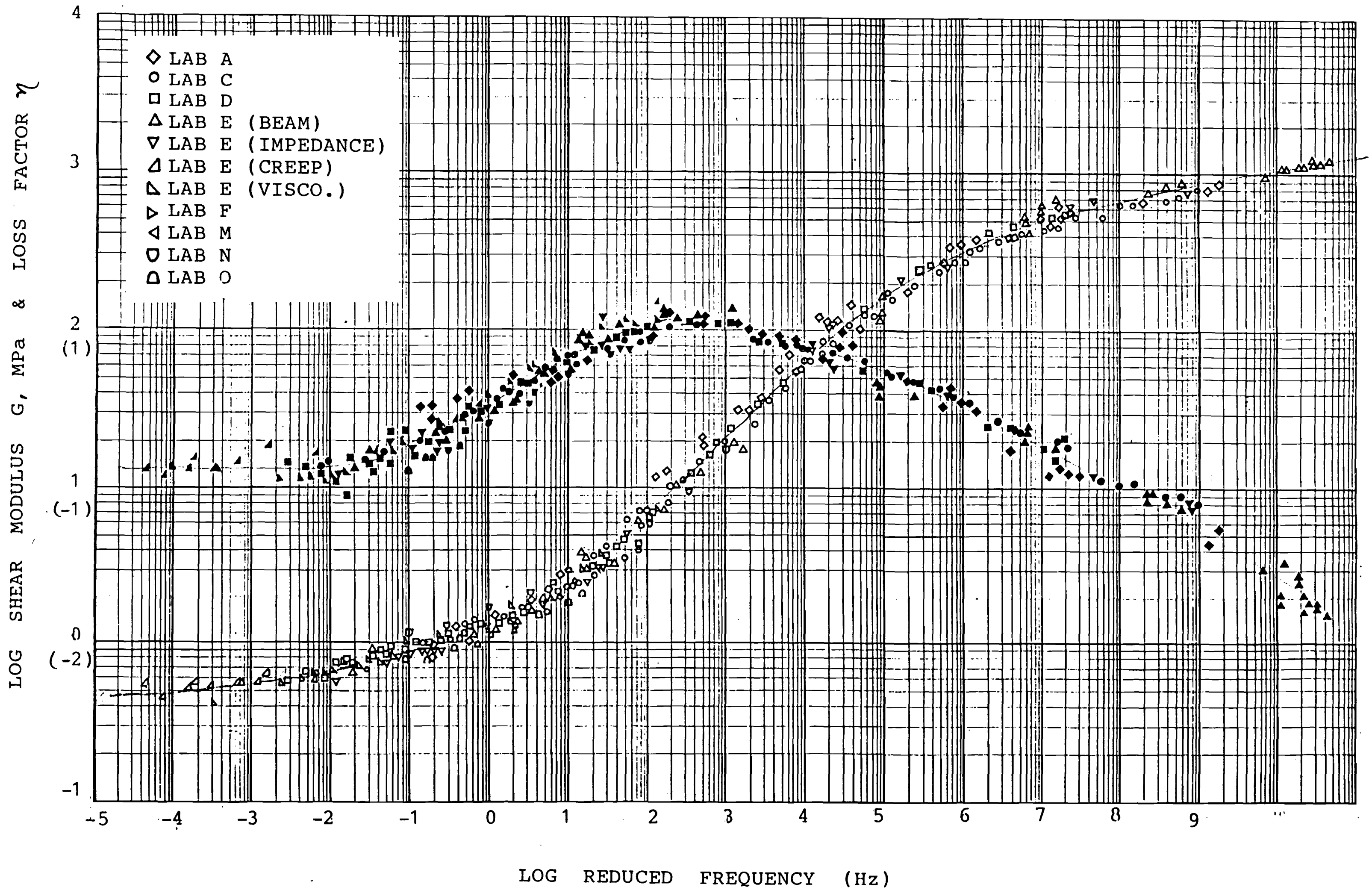

Figure 20. Master plots for All Laboratories (Shear)

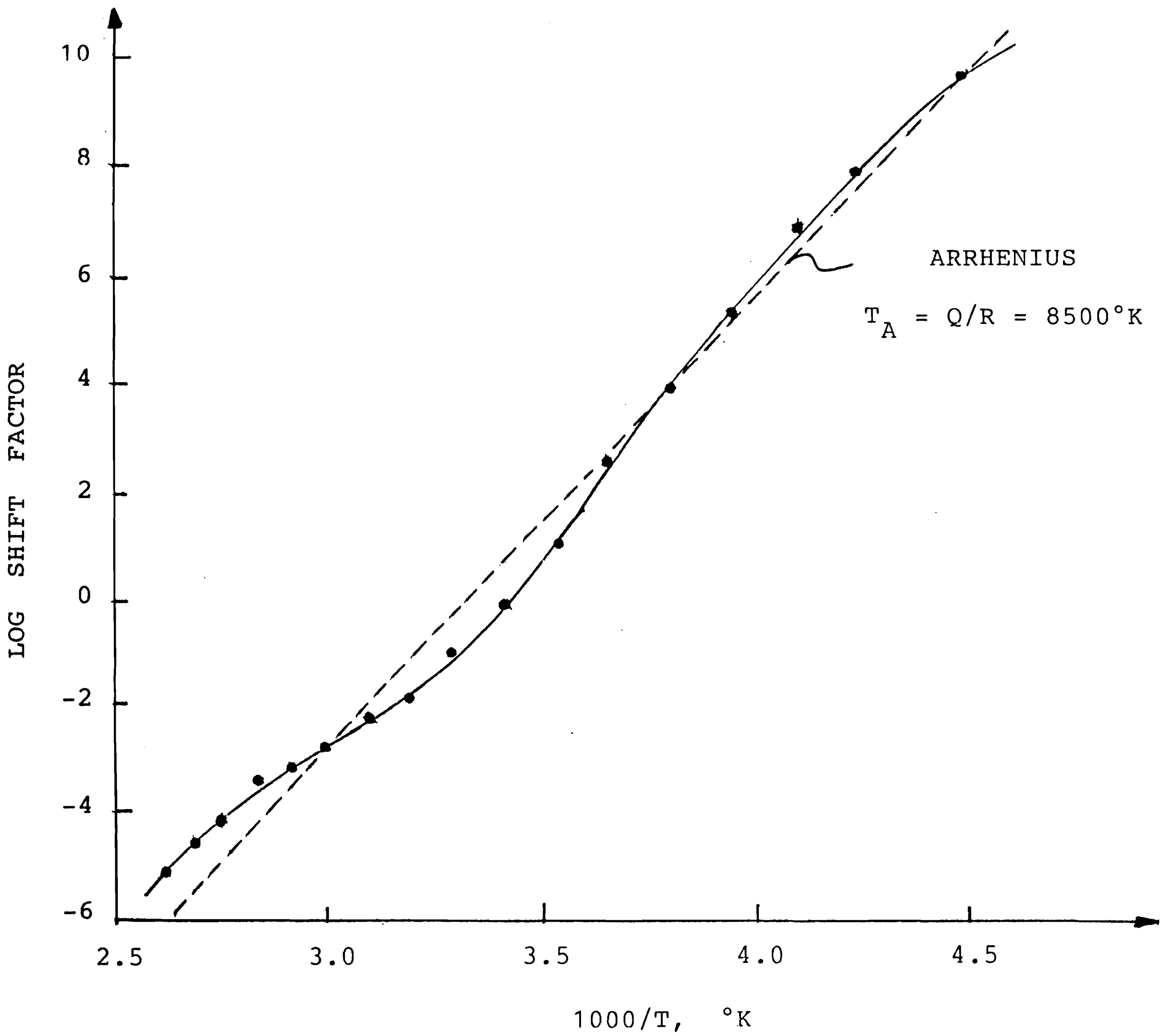

Figure 21. Plot of Log (Shift Factor) versus 1000/T