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NAVSWC TR 90-450

**DYNAMIC VISCOELASTIC PROPERTIES OF
SELECTED NATURAL-NEOPRENE RUBBER BLENDS**

**BY DR. WALTER M. MADIGOSKY
RESEARCH AND TECHNOLOGY DEPARTMENT**

30 SEPTEMBER 1990

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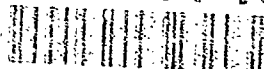
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FOREWORD

The purpose of this report is to document two of the dynamic viscoelastic properties (shear modulus and loss factor) of viscoelastic rubber blends. They are determined as a function of frequency and temperature. Applying the time-temperature superposition principle to the data, master curves are constructed and Williams-Landel-Ferry (WLF) shift constants are determined.

This work was done during FY89-90 with funds and materials provided by David Taylor Research Center (Code 1908), Bethesda, Maryland.

Approved by:

Carl E. Mueller

CARL E. MUELLER, Head
Materials Division

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CONTENTS

	<u>Page</u>
INTRODUCTION	1
RUBBER BLEND FORMULATIONS AND THEIR BASIC RHEOLOGIC CHARACTERISTICS	2
RESULTS OF DYNAMIC MECHANICAL MEASUREMENTS	3
CONCLUSIONS	4
REFERENCES	23
DISTRIBUTION	(1)

ILLUSTRATIONS

<u>Figure</u>		<u>Page</u>
1	SAMPLE SPECIFIC GRAVITY VERSUS NEOPRENE-NATURAL RUBBER RATIO	5
2	SAMPLE MODULUS VERSUS NEOPRENE-NATURAL RUBBER RATIO FOR DIFFERENT ELONGATIONS	6
3	LOG E' AND TAN (δ) VERSUS TEMPERATURE AT VARIOUS FREQUENCIES FOR SAMPLE 9107	7
4	LOG YOUNG'S MODULUS AND LOG LOSS FACTOR VERSUS FREQUENCY FOR SAMPLE 9107	8
5	LOG E' AND TAN (δ) VERSUS TEMPERATURE AT VARIOUS FREQUENCIES FOR SAMPLE 9108	9
6	LOG YOUNG'S MODULUS AND LOG LOSS FACTOR VERSUS FREQUENCY FOR SAMPLE 9108	10
7	LOG E' AND TAN (δ) VERSUS TEMPERATURE AT VARIOUS FREQUENCIES FOR SAMPLE 9109	11
8	LOG YOUNG'S MODULUS AND LOG LOSS FACTOR VERSUS FREQUENCY FOR SAMPLE 9109	12
9	LOG E' AND TAN (δ) VERSUS TEMPERATURE AT VARIOUS FREQUENCIES FOR SAMPLE 9110	13
10	LOG YOUNG'S MODULUS AND LOG LOSS FACTOR VERSUS FREQUENCY FOR SAMPLE 9110	14
11	LOG E' AND TAN (δ) VERSUS TEMPERATURE AT VARIOUS FREQUENCIES FOR SAMPLE 9111	15
12	LOG YOUNG'S MODULUS AND LOG LOSS FACTOR VERSUS FREQUENCY FOR SAMPLE 9111	16
13	LOG E' AND TAN (δ) VERSUS TEMPERATURE AT VARIOUS FREQUENCIES FOR SAMPLE 9112	17
14	LOG YOUNG'S MODULUS AND LOG LOSS FACTOR VERSUS FREQUENCY FOR SAMPLE 9112	18

TABLES

<u>Table</u>		<u>Page</u>
1	FRACTIONAL COMPOSITION OF SAMPLES	19
2	INTERMEDIATE BATCH COMPOSITION FOR SAMPLES 9107-9109	19
3	INTERMEDIATE BATCH COMPOSITION FOR SAMPLES 911-112	20
4	RHEOLOGIC PROPERTIES OF SAMPLES	20
5	DIMENSIONS OF SAMPLES USED IN DMTA APPARATUS ..	21
6	ESTIMATED GLASS TRANSITION TEMPERATURES AND WLF COEFFICIENTS FOR SIX NEOPRENE- NATURAL RUBBER BLENDS	21

INTRODUCTION

In support of the underwater warfare program of the United States Navy, the Naval Surface Warfare Center (NAVSWC), White Oak Laboratory, has conducted a research and development program for the formulation and characterization of polymers suited to undersea system applications. Over a period of time, formulations of innovative polymers have been undertaken, and the fundamental viscoelastic properties of these materials have been measured.¹⁻⁵ To date the polymer formulations and measurement efforts have been applied to natural and synthetic rubbers, urethanes and silicones. This report provides the formulation specifications and the results of measurements made on six different neoprene-natural rubber blends.

The formulation and mixing of each of the six different blends followed a specific sequence to ascertain if there were differences in rheologic properties which could be traced to the actual process of the mixing and curing of the rubber compound. The neoprene-natural rubber blends discussed in this report were compounded by Burke Industries, San Jose, California.

Both static and dynamic property measurements were made on the rubber samples. The static measurements included:

- Specific gravity
- Young's modulus at various elongations
- Tensile strength
- Elongation at break, and
- Hardness

Dynamic mechanical measurements were performed using a Polymer Laboratories Dynamic Mechanical Thermal Analyzer to obtain the damping factor ($\tan \delta$) and the dynamic storage modulus (E') of the materials. The automated measurement technique produced a set of data over a temperature range of -70 to +30 degrees centigrade at discrete frequencies between 1 and 30 hertz. From the resulting data, the glass transition and the Williams-Landel-Ferry (WLF) equation constants of the polymer samples were estimated.

Section 2 of this report provides a detailed description of the sample formulations. Section 3 provides a synopsis of the dynamic measurement technique and the resulting measurements.

RUBBER BLEND FORMULATIONS AND THEIR BASIC RHEOLOGIC CHARACTERISTICS

Among the polymers investigated by the NAVSWC research staff are samples prepared from blends of natural and neoprene rubber. The six samples discussed herein were made with different portions of natural and neoprene rubber and with different blending procedures. Table 1 provides a summary of the ingredients by weight, constituting the various samples.

Each of the final blends listed in Table 1 was prepared from intermediate mixes of the primary ingredients. Then the zinc oxide, Santocure NS, and sulfur were added to intermediate mixes to arrive at the sample specifications listed in Table 1.

In preparing samples 9107, 9108, and 9109, both the natural rubber and the neoprene were added together to produce the intermediate mixes shown in Table 2, where the M on the batch identification number indicates an intermediate formulation.

After these intermediate mixes were prepared, the final blends were obtained by adding the zinc oxide, Santocure, and sulfur to achieve the composition by weight given in Table 1 for samples 9107, 9108, and 9109.

Preparation of samples 9110, 9111, and 9112 had another variation in the formulation process. For these samples, the intermediate polymers listed in Table 3 were prepared. These intermediate polymers differed in neoprene and natural rubber content. The intermediate polymers labeled with an A contained only neoprene and no natural rubber, while the polymers labeled with a B contained only natural rubber and no neoprene. Appropriate portions of each of these intermediate polymers were blended to obtain the final sample formulations provided in Table 1 for samples 9110, 9111, and 9112.

All of the blends listed in Table 1 were cured at a temperature of 160 degrees centigrade for a period of 12 minutes.

Burke Industries, the compounder of these neoprene-natural rubber blends, performed measurements of some of the samples' fundamental properties. The results on three different specimens of each blended polymer are listed in Table 4. The values listed in Table 4 are the mean values obtained from the three specimens of each blend.

Figures 1 and 2, pages 7 and 8, provide a plot of the average values of these properties versus the ratio of neoprene to natural rubber in each blend. There it is seen that each of the properties is essentially a linear function of this ratio.

RESULTS OF DYNAMIC MECHANICAL MEASUREMENTS

The dynamic mechanical properties of the six natural neoprene rubber samples were measured using the Dynamic Mechanical Thermal Analyzer (DMTA) manufactured by Polymer Laboratories, Ltd., of Shropshire, England. The DMTA applies a sinusoidal force to a "clamped beam" sample of the material under test and measures the amplitude and phase of the resulting strain. The temperature of the sample and the frequency of the applied force are varied to obtain data for a predetermined set of temperatures and frequencies. The principles of operation of the DMTA apparatus are presented and discussed by Dlubac, et. al.⁶

The results of calculations involving the experimental data depend significantly upon the actual configuration of the sample under measurement. Of particular importance are the length, width, and thickness of the samples as clamped in the apparatus. Table 5 provides the specifications of the configurations of the samples as tested.

--What about, "Table 5 provides the configuration specifications of the samples tested."?

The NAVSWC DMTA apparatus is connected to a Hewlett-Packard Model 9816 minicomputer which automatically collects the data from the DMTA. The resulting data is both stored and plotted as graphs of modulus E' and $\tan \delta$ versus temperature and frequency. From the collected and displayed data, the glass transition temperature and WLF coefficients of the samples are estimated.

Figures 3 through 14 provide a display of the data collected by the DMTA Hewlett-Packard test apparatus. Figures 3, 5, 7, 9, 11, and 13 present plots of measured storage modulus E' and loss factor $\tan \delta$ as a function of temperature for four discrete frequencies. Figures 4, 6, 8, 10, 12, and 14 provide plots of the resulting time-temperature shifted curves and $\log \tan \delta$ versus frequency for each of the samples.

Table 6 provides the estimates of the glass transition temperature and the WLF coefficients of the six samples. For this report the glass transition temperature was taken as the point, on the $\log E'$ versus temperature graph, at which the modulus becomes frequency independent, that is, the temperature where the four different frequency curves overlap. The plots of modulus E' versus temperature show the expected shifting of the glass transition temperature as a function of the relative portions of neoprene and natural rubber. In all cases the estimated transition temperatures are between -70 and -50 degrees centigrade.

The WLF coefficients were interactively determined by choosing values of the coefficients which provided the visual best fit of the various sections of the plot of the Young's modulus versus \log frequency. Figures 2, 4, 6, 8, 10, 12, and 14 are the result of this interactive procedure for determining the WLF coefficients. The reference temperature for all cases was 10 degrees centigrade.

CONCLUSIONS

The results of the static measurements show that there is no discernible effect on static rheologic properties due to intermediate steps in the compounding of the neoprene-natural blends. The static measurements of specific gravity and the moduli of the samples show a simple linear relation with the ratio of neoprene to natural rubber in the sample. These static properties are not dependent upon whether the polymers are mixed separately in intermediate batches or are mixed together in a one-step process. Similarly, the dynamic measurement of the viscoelastic properties of the samples showed there are no temperature shifts due to the polymer mixing procedures. Comparison of Figure 3 with Figure 11, Figure 5 with Figure 9, and Figure 7 with Figure 13, shows that those polymer samples, mixed as intermediate batches, have curves which are nearly identical in amplitude and shape.

Examination of the loss factor, $\tan \delta$, as a function of temperature, shows that the neoprene-natural rubber blends exhibit the bimodal shape expected of two immiscible polymers. Distinct transition temperatures for each of the constituent polymers occurs in the vicinity of -50 degrees centigrade and -20 degrees centigrade on all of the samples tested. There is no large shift of the two peaks toward each other nor formation of a single transition peak as might be expected if the two polymers were miscible or completely mixed.⁷

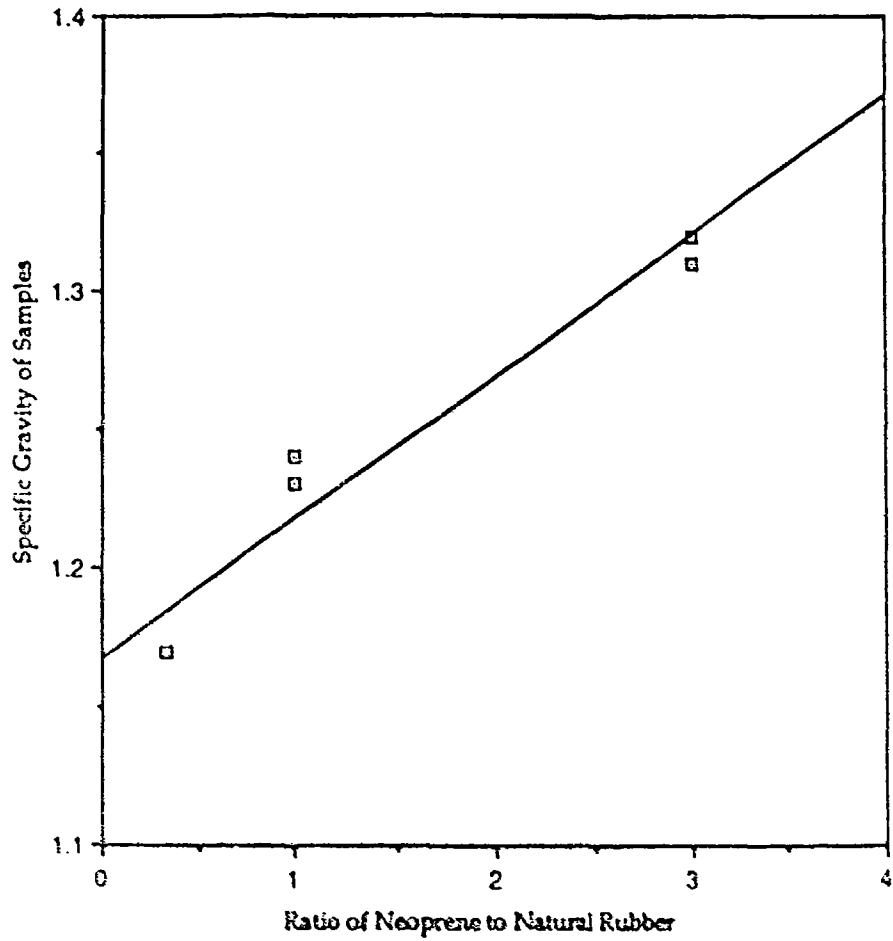


FIGURE 1 SAMPLE SPECIFIC VERSUS NEOPRENE-NATURAL RUBBER RATIO

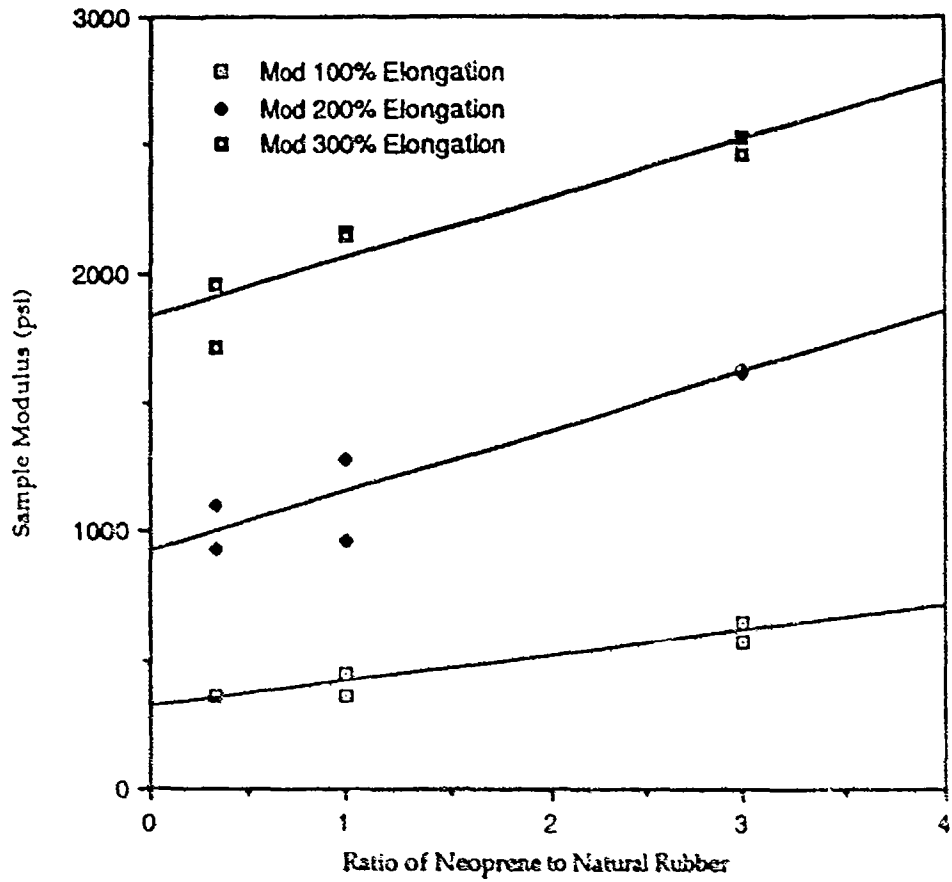


FIGURE 2 SAMPLE MODULUS VERSUS NEOPRENE-NATURAL RUBBER RATIO FOR DIFFERENT ELONGATIONS

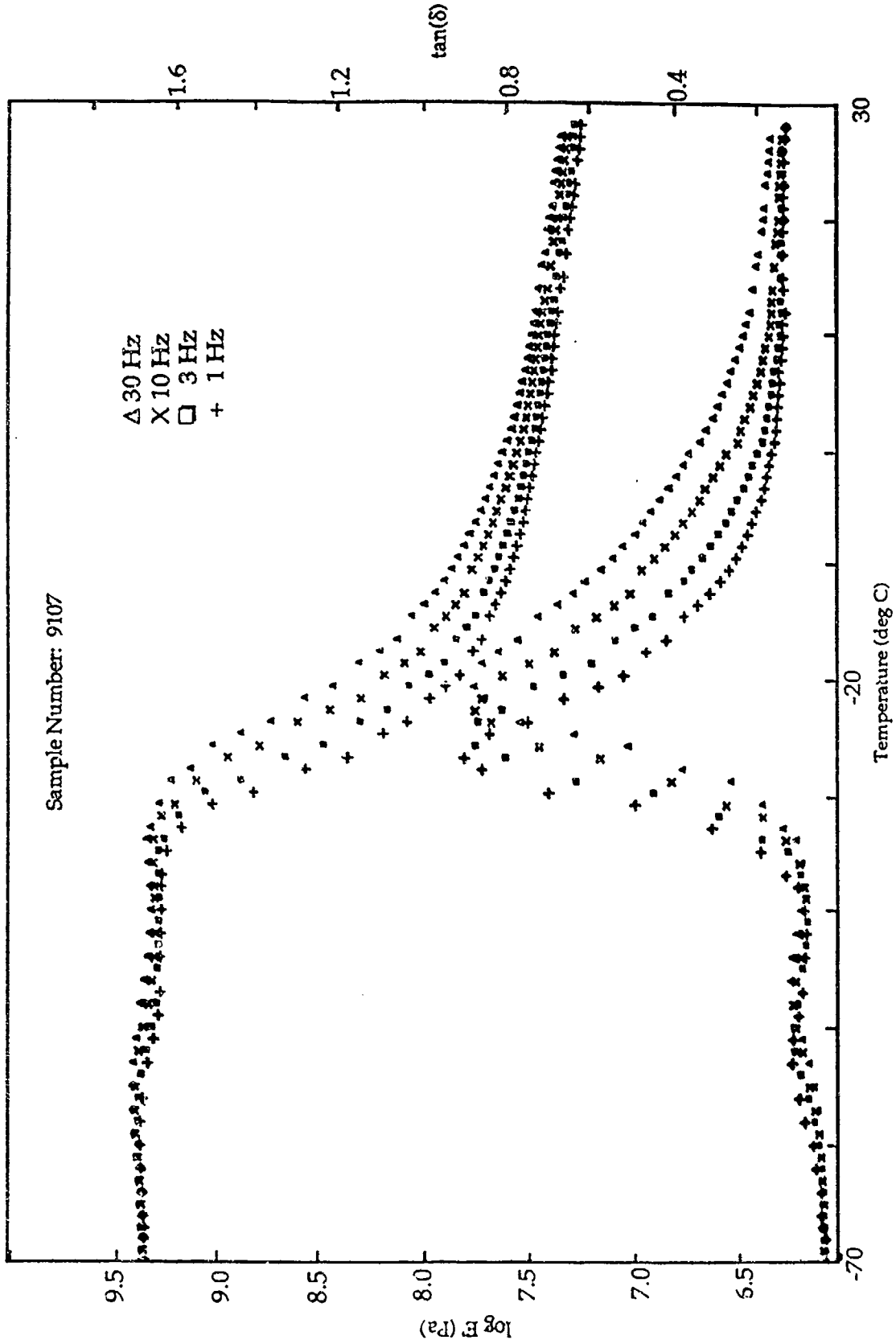


FIGURE 3. LOG E' AND TAN (δ) VERSUS TEMPERATURE AT VARIOUS FREQUENCIES FOR SAMPLE 9107

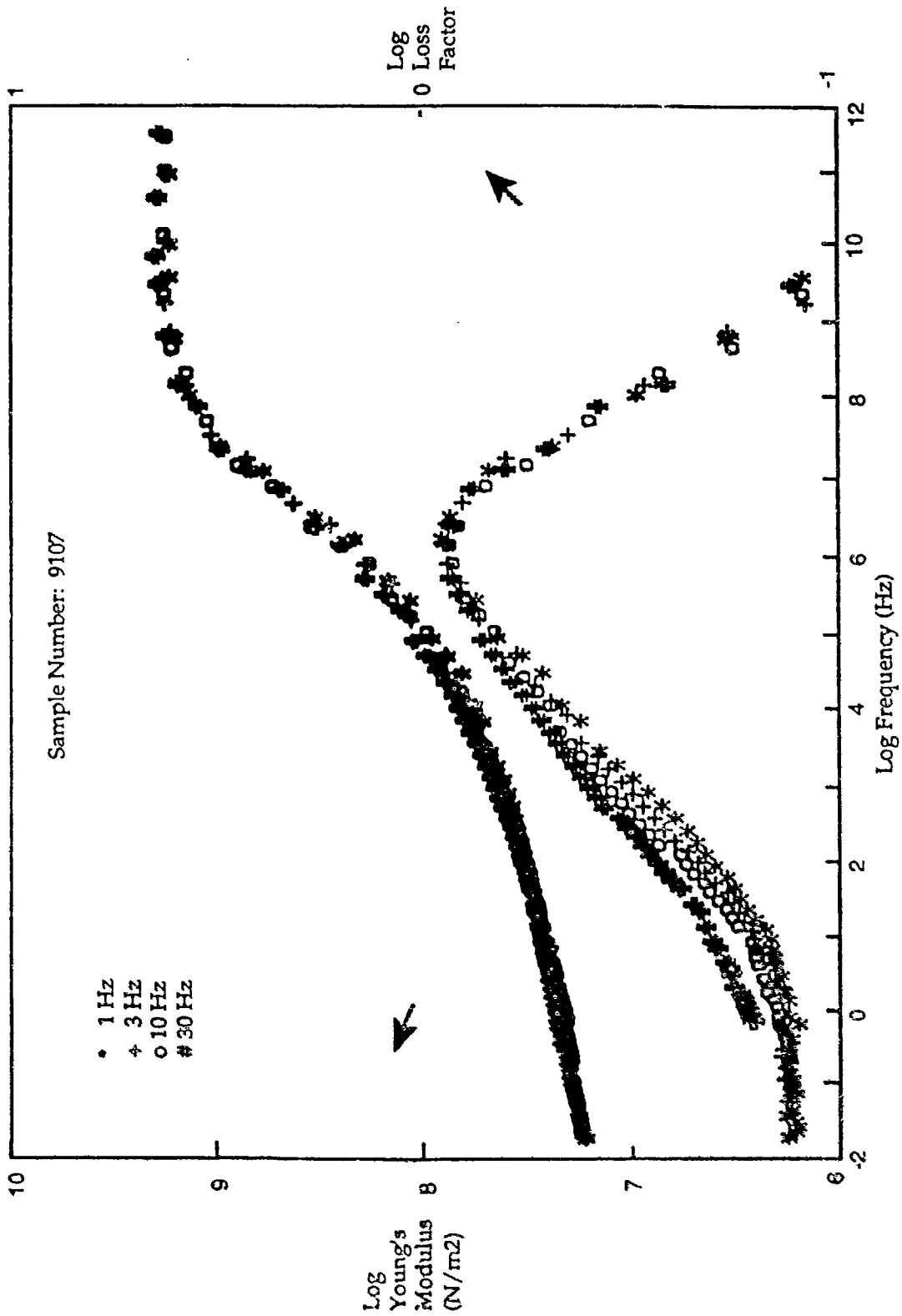


FIGURE 4. LOG YOUNG'S MODULUS AND LOG LOSS FACTOR VERSUS FREQUENCY FOR SAMPLE 9107

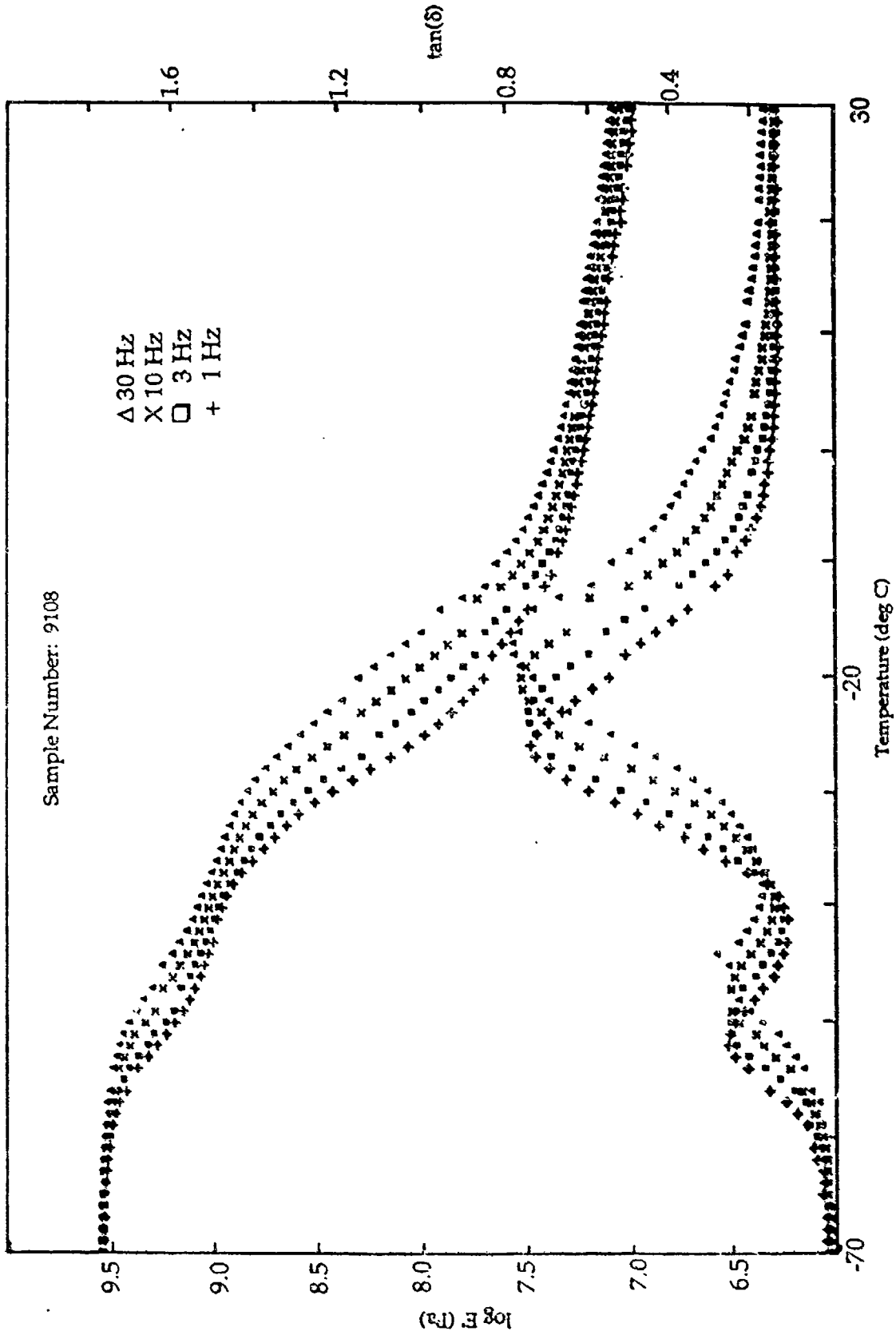


FIGURE 5. LOG E' AND TAN (δ) VERSUS TEMPERATURE AT VARIOUS FREQUENCIES FOR SAMPLE 9108

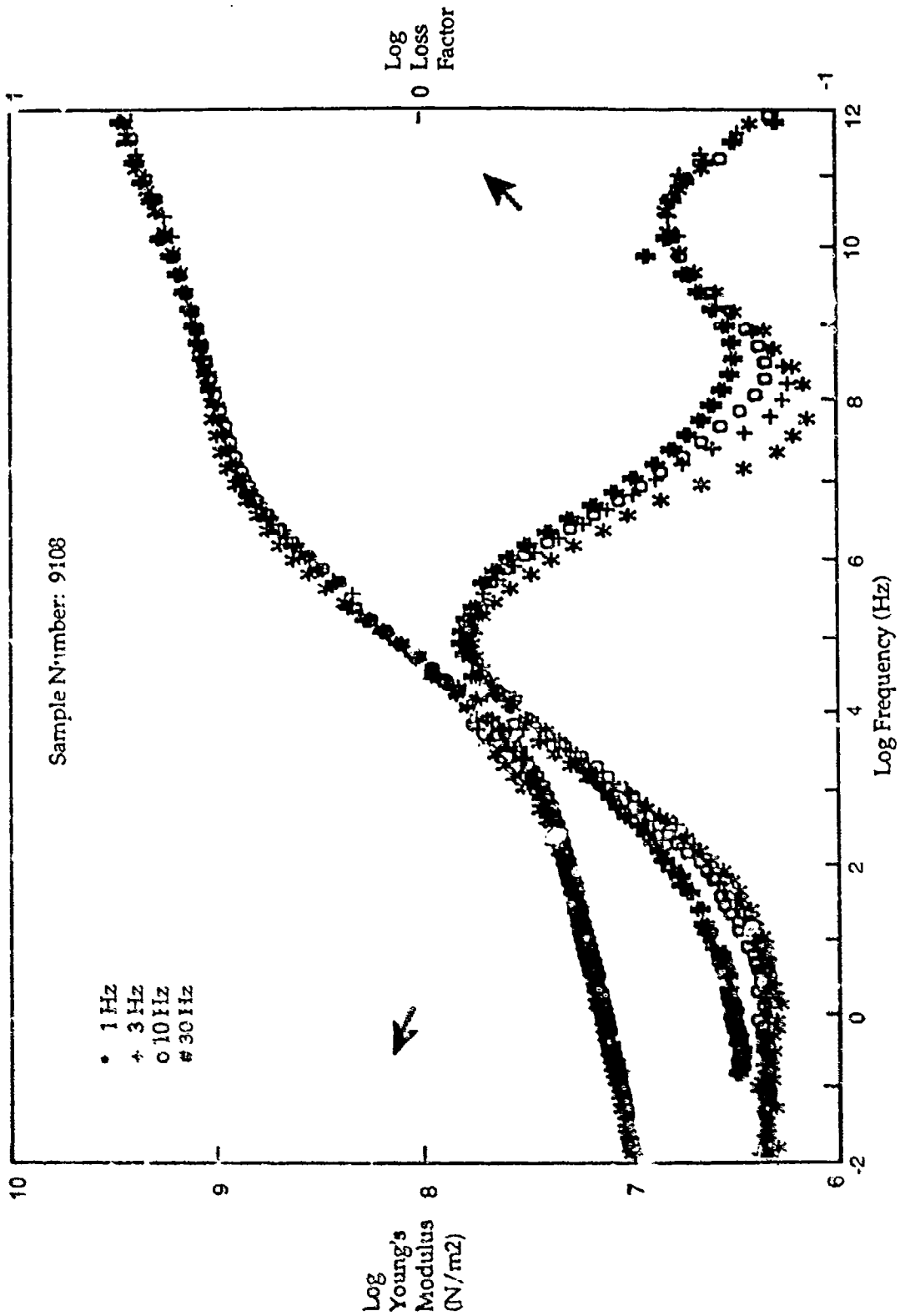


FIGURE 6. LOG YOUNG'S MODULUS AND LOG LOSS FACTOR VERSUS FREQUENCY FOR SAMPLE 9108

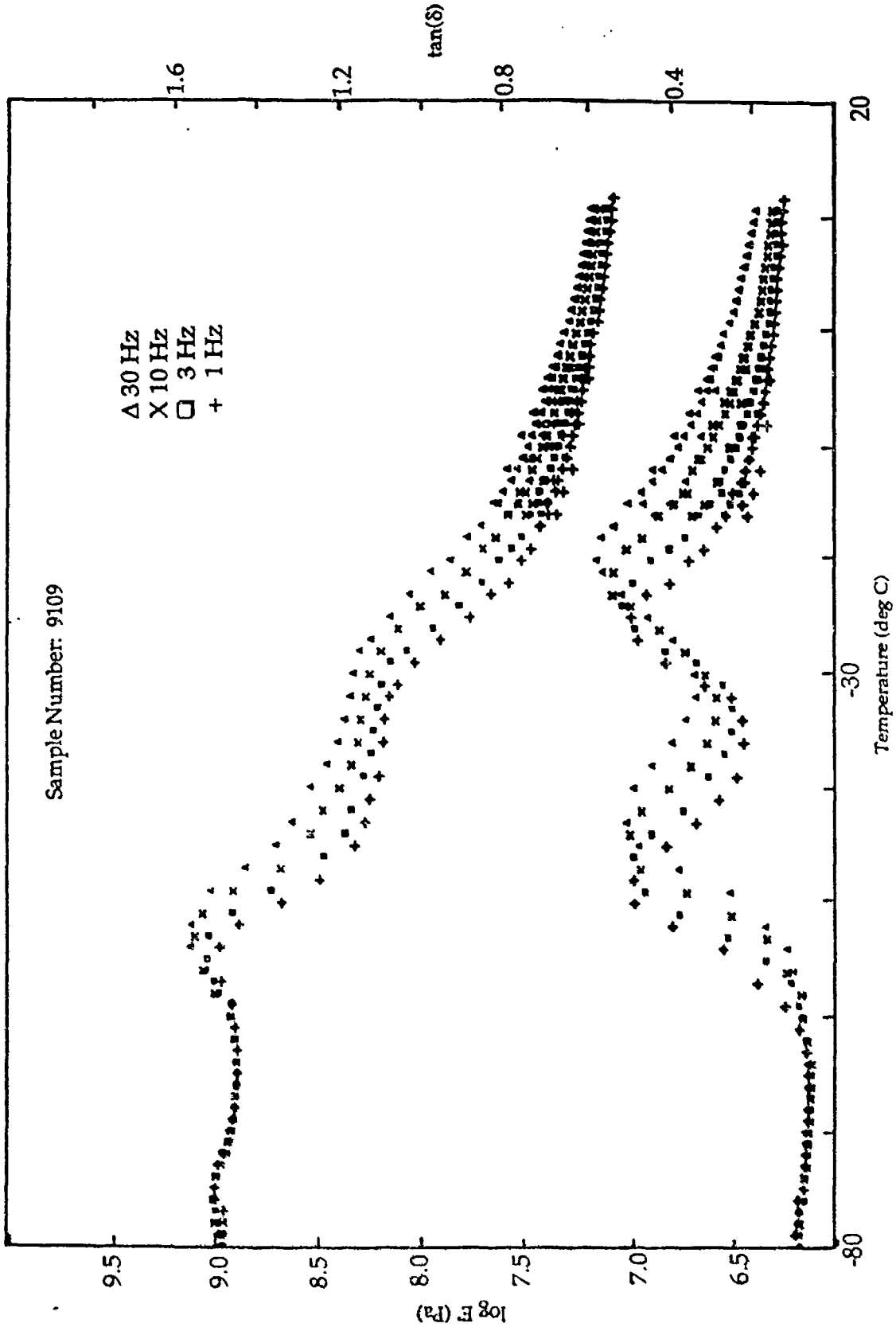


FIGURE 7. LOG E' AND TAN (δ) VERSUS TEMPERATURE AT VARIOUS FREQUENCIES FOR SAMPLE 9109

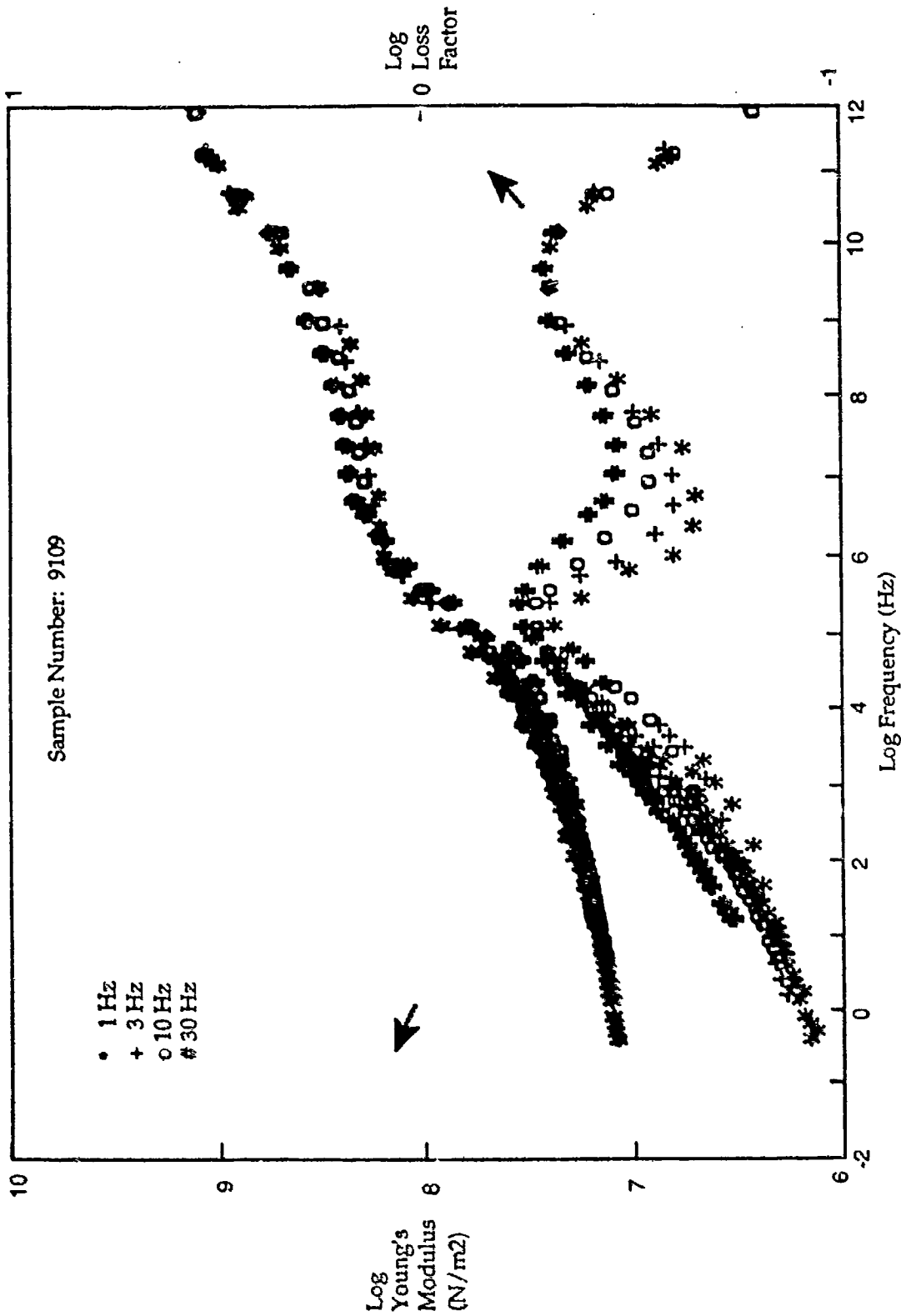


FIGURE 8. LOG YOUNG'S MODULUS AND LOG LOSS FACTOR VERSUS FREQUENCY FOR SAMPLE 9109

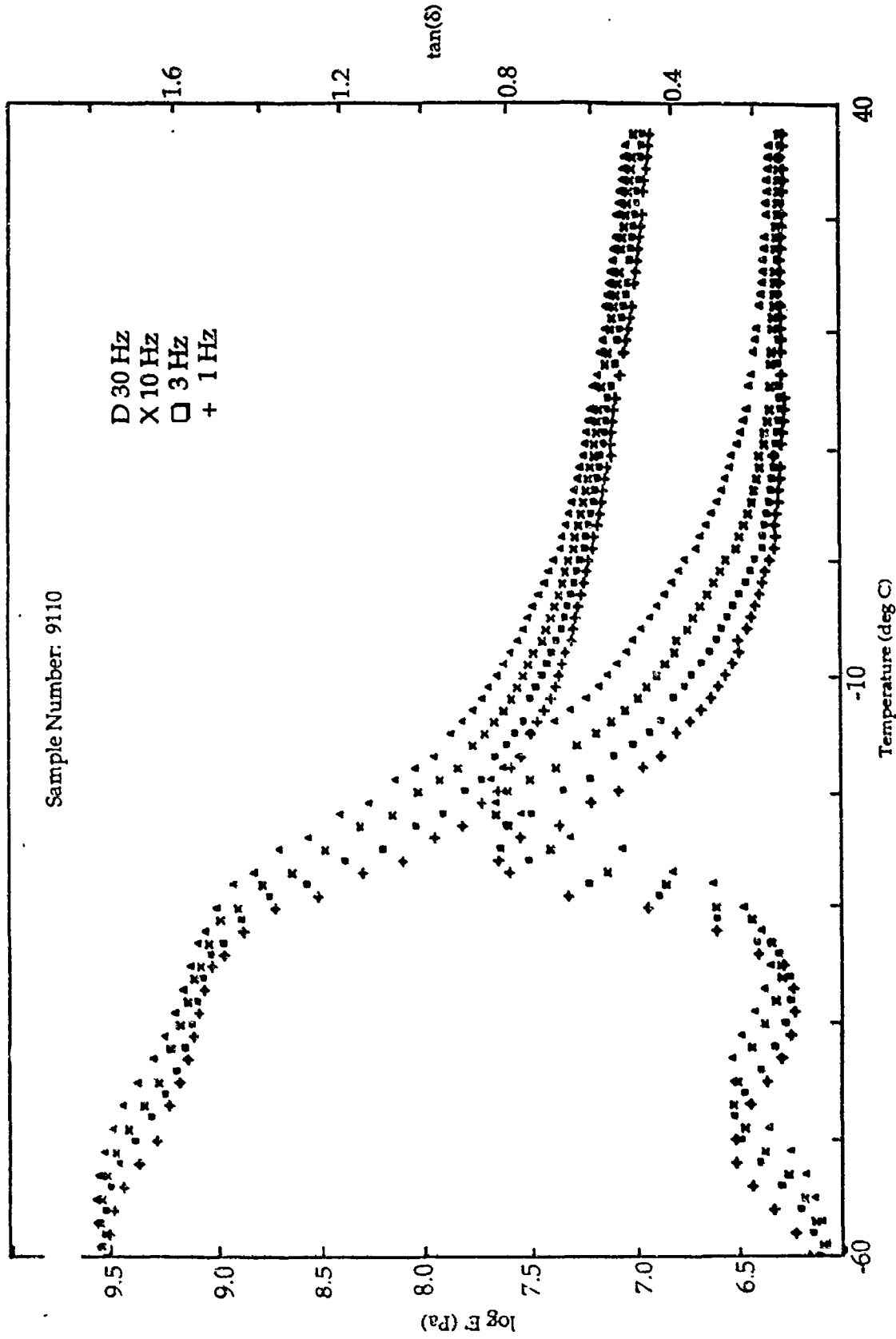


FIGURE 9. LOG E AND TAN (δ) VERSUS TEMPERATURE AT VARIOUS FREQUENCIES
 VARIOUS FREQUENCIES FOR SAMPLE 9110

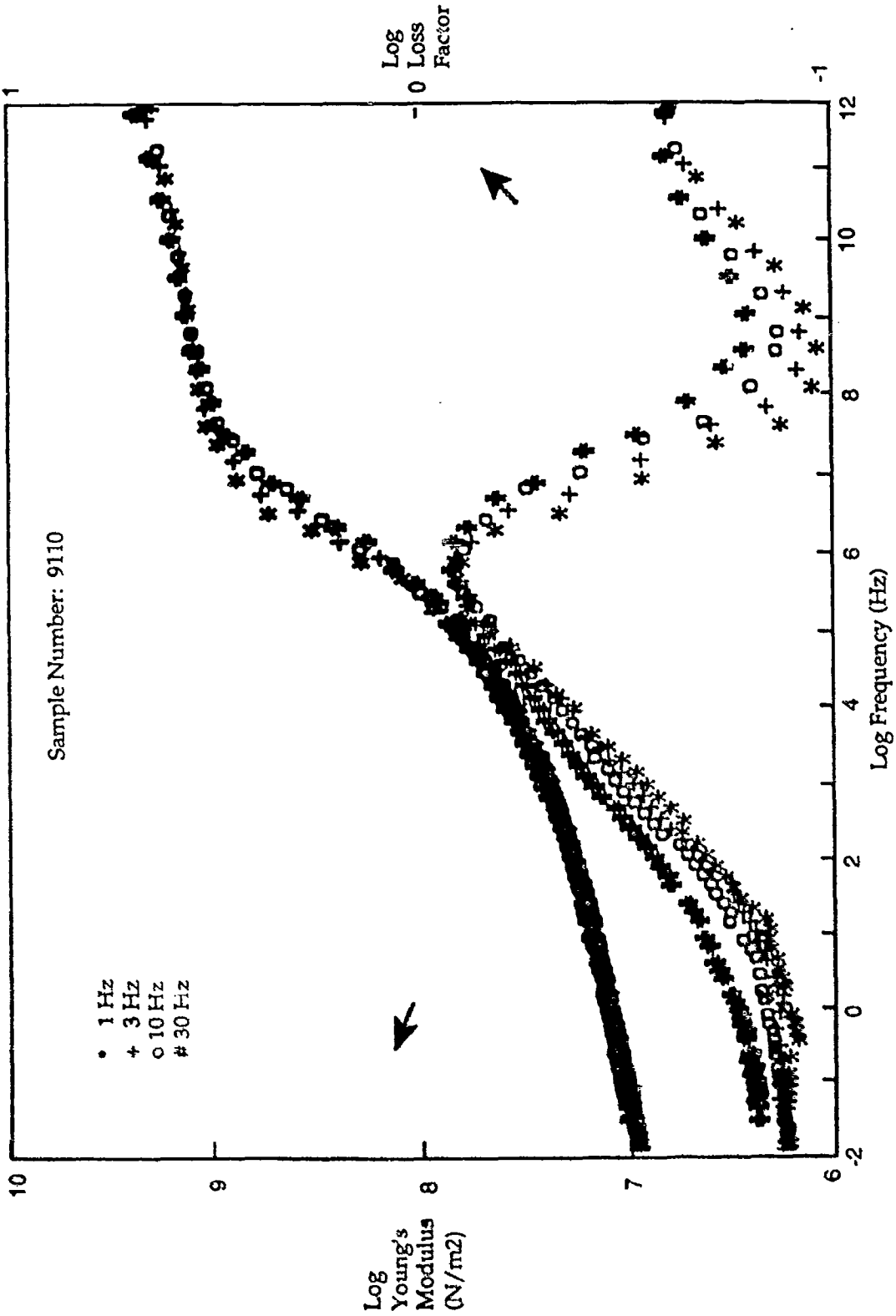


FIGURE 10. LOG YOUNG'S MODULUS AND LOG LOSS FACTOR VERSUS FREQUENCY FOR SAMPLE 9110

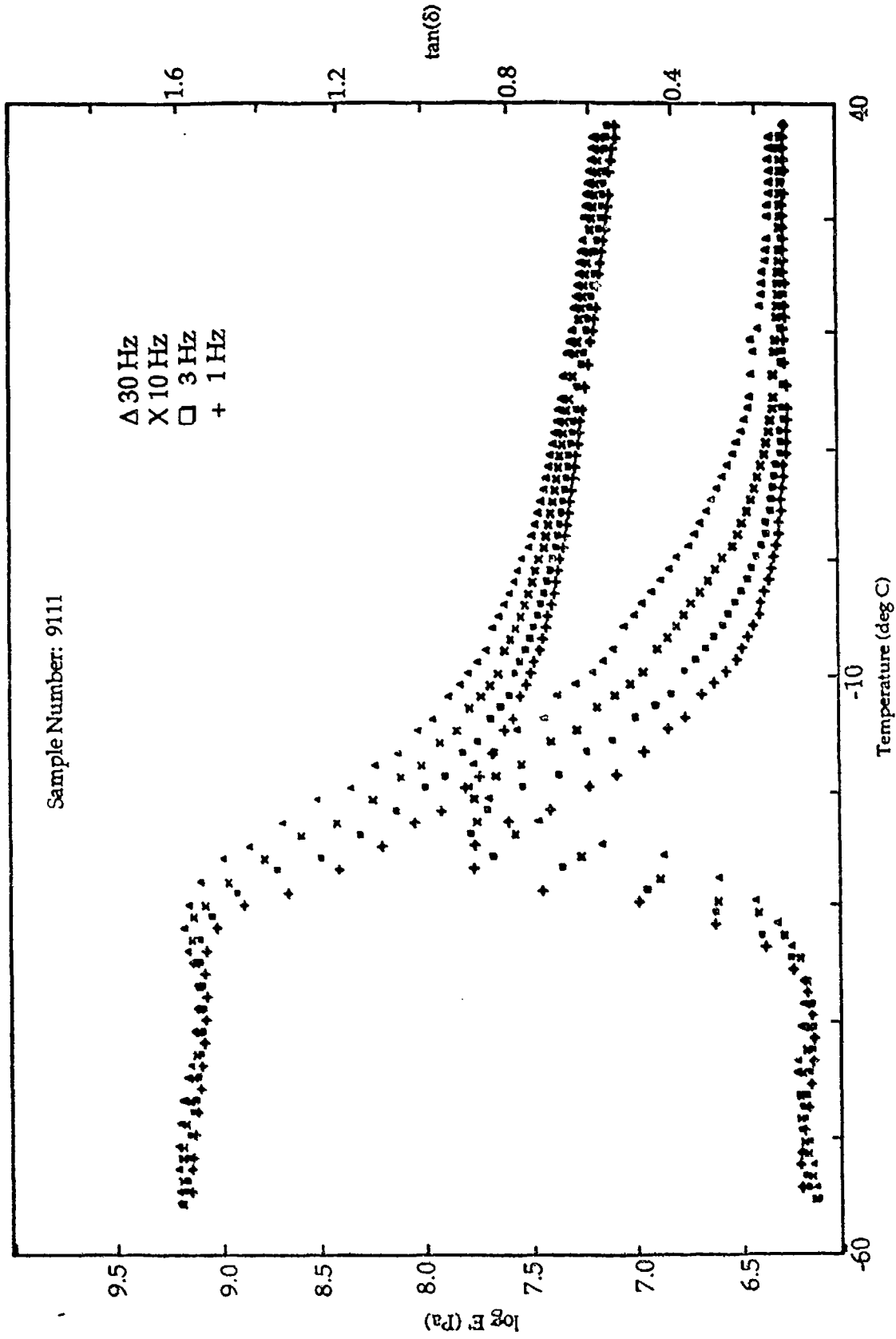


FIGURE 11. LOG E' AND TAN (δ) VERSUS TEMPERATURE AT VARIOUS FREQUENCIES FOR SAMPLE 9111

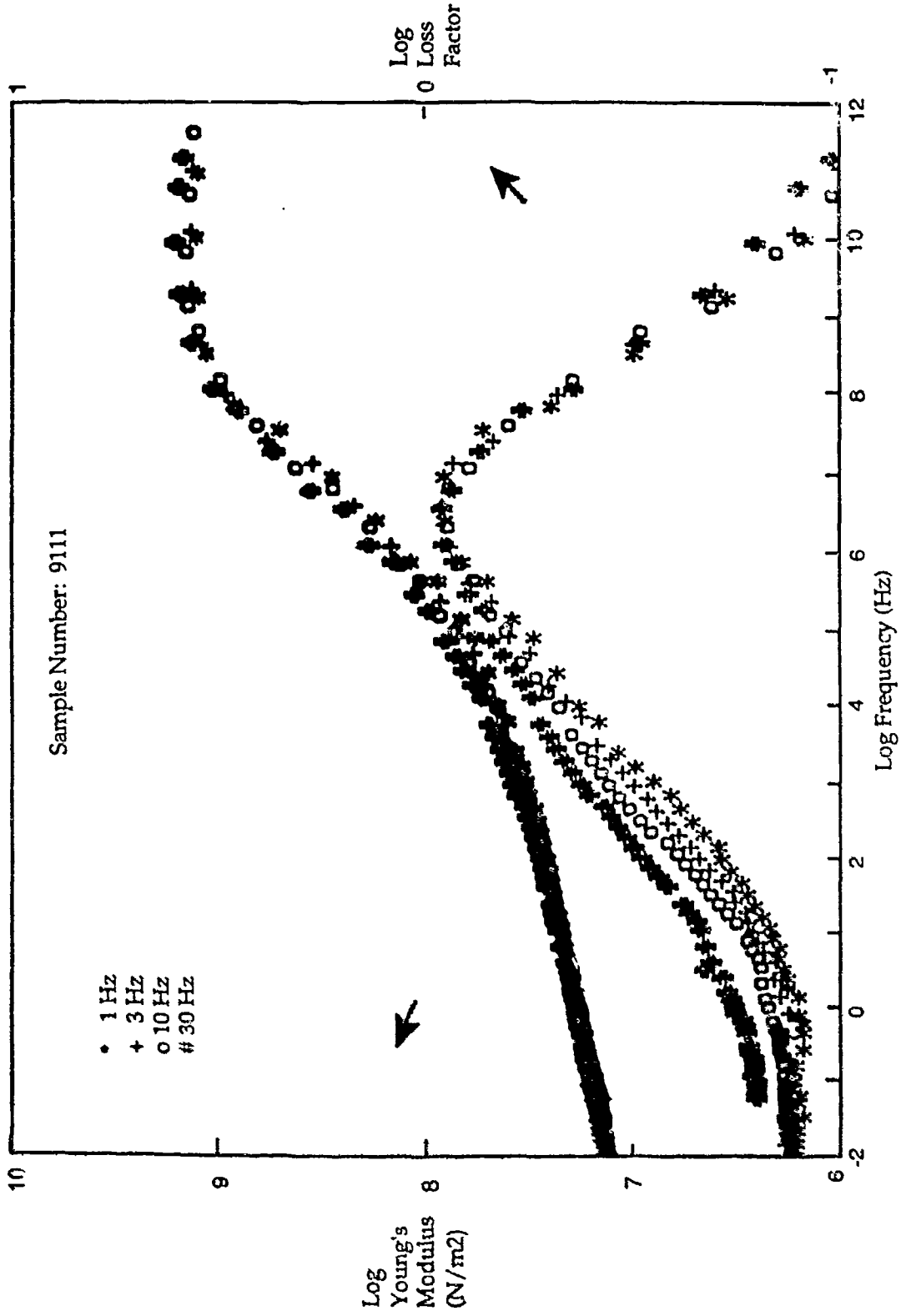


FIGURE 12. LOG YOUNG'S MODULUS AND LOG LOSS FACTOR VERSUS FREQUENCY FOR SAMPLE 9111

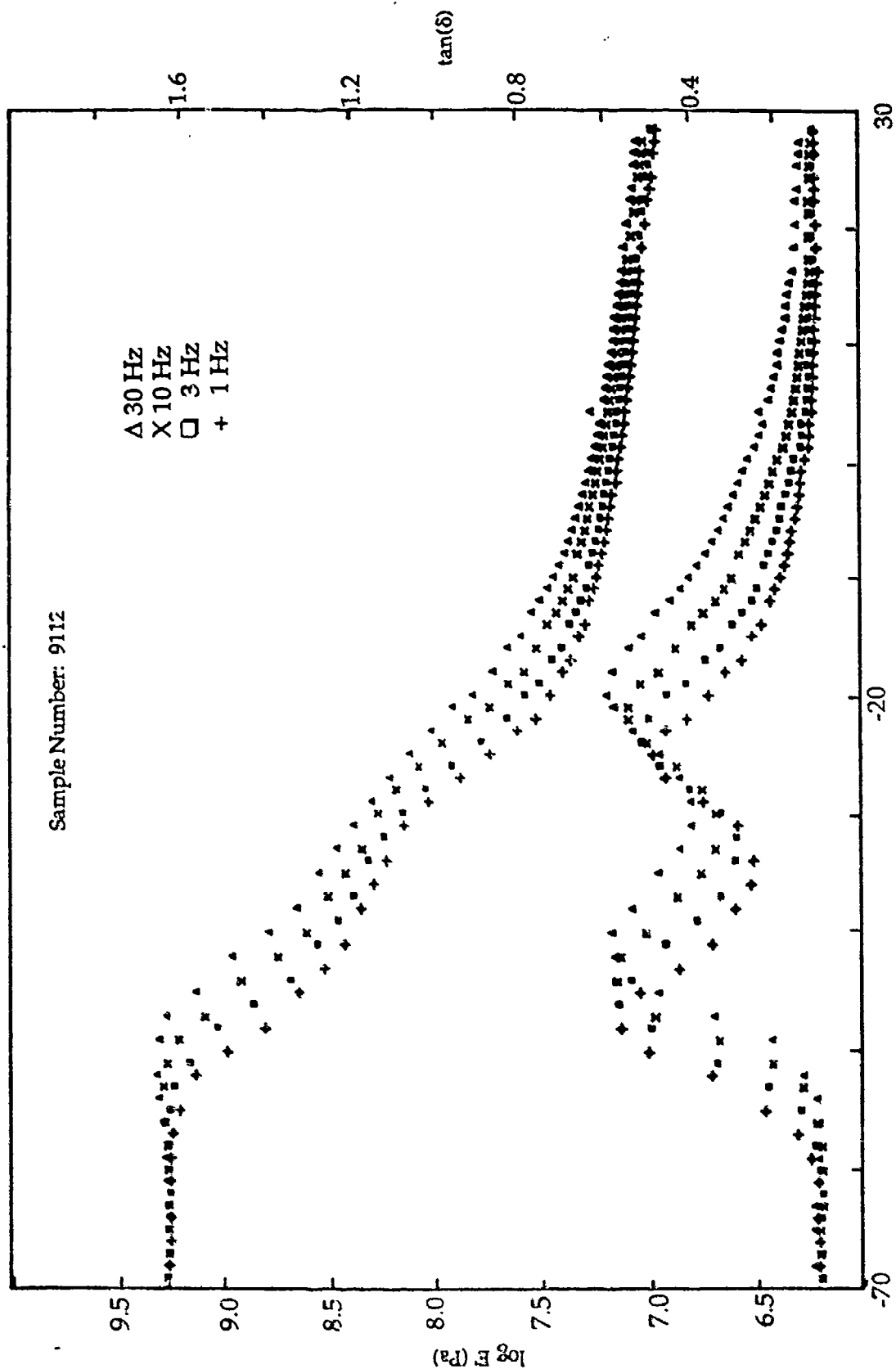


FIGURE 13. LOG E' AND TAN (δ) VERSUS TEMPERATURE AT VARIOUS FREQUENCIES FOR SAMPLE 9112

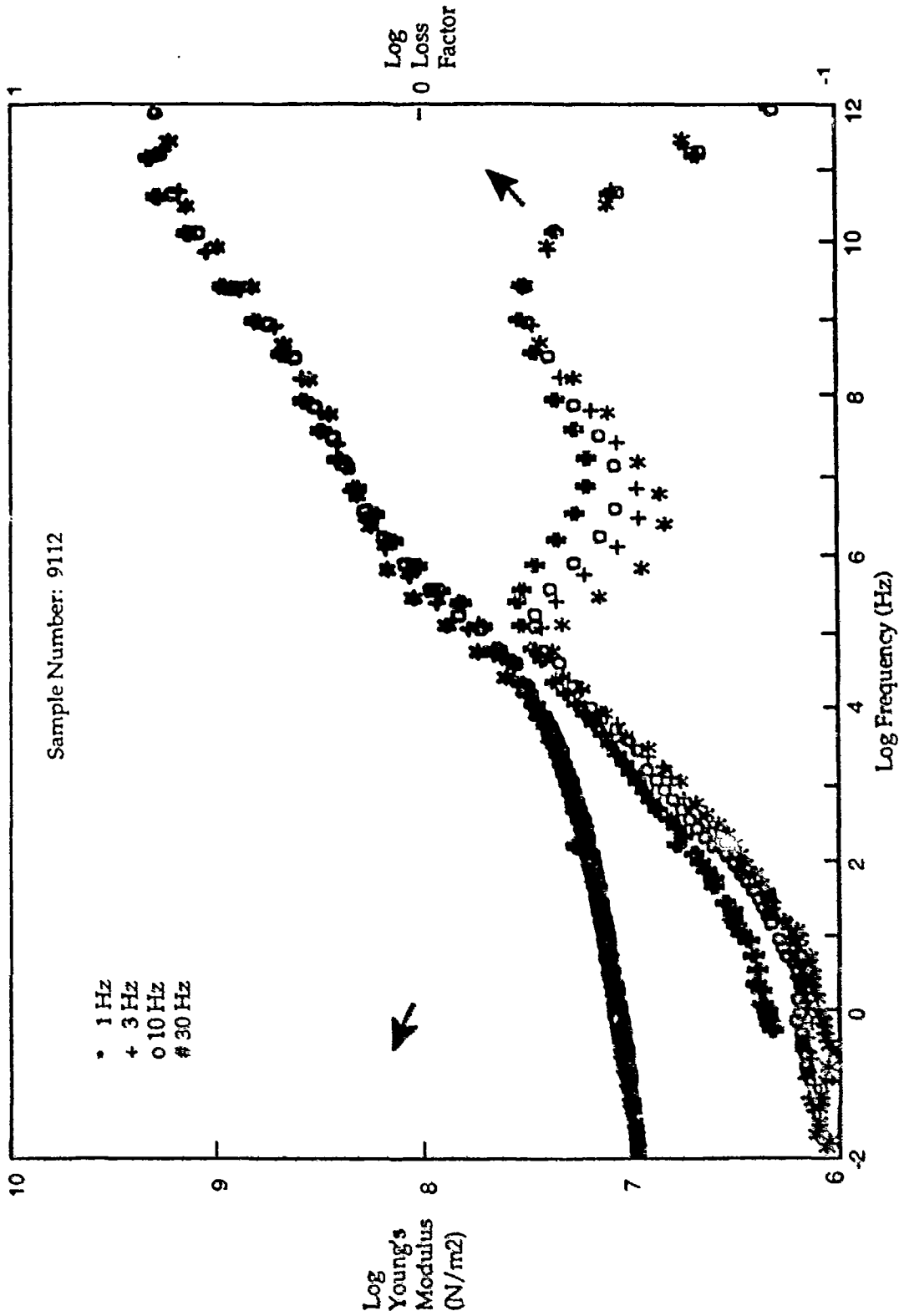


FIGURE 14. LOG YOUNG'S MODULUS AND LOG LOSS FACTOR VERSUS FREQUENCY FOR SAMPLE 9112

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TABLE 1. FRACTIONAL COMPOSITION OF SAMPLES
(parts by weight)

Ingredients	Sample Identification Number					
	9107	9108	9109	9110	9111	9112
NPX6398	75	50	25	50	75	25
SMR-20	25	50	75	50	25	75
Structol 40ms	1.923	1.929	1.939	1.929	1.923	1.942
Scorchguard O	1.442	1.447	1.454	1.447	1.442	1.456
Santoflex O	1.923	1.929	1.939	1.929	1.923	1.942
Flectol Flakes	0.962	0.965	0.969	0.965	0.962	0.971
FEF Black	50	50	50	50	50	50
Zinc Oxide	3.045	3.055	3.069	3.055	3.045	3.074
Santocure NS	0.481	0.965	1.454	0.965	0.481	1.456
Sulfur	0.321	0.643	0.969	0.643	0.321	0.971

TABLE 2. INTERMEDIATE BATCH COMPOSITION FOR
SAMPLES 9107-9109 (parts by weight)

Ingredients	Intermediate Batch Composition		
	9107-M	9108-M	9109-M
NPX6398	75	50	25
SMR-20	25	50	75
Structol 40ms	2	2	2
Scorchguard O	1.4	1.4	1.4
Santoflex O	2	2	2
Flectol Flakes	1	1	1
FEF Black	50	50	50
Total	156.4	156.4	156.4

TABLE 3. INTERMEDIATE BATCH COMPOSITION FOR SAMPLES 911-112
(parts by weight)

Ingredients	Intermediate Batch Number					
	9110-A	9110-B	9111-A	9111-B	9112-A	9112-B
NPX6398	100	0	100	0	100	0
SMR-20	0	100	0	100	1	100
Structol 40ms	2	2	2	2	2	2
Scorchguard O	1.4	1.4	1.4	1.4	1.4	1.4
Santoflex O	2	2	2	2	2	2
Flectol Flakes	1	1	1	1	1	1
FEF Black	50	50	50	50	50	50
Total	156.4	156.4	156.4	156.4	156.4	156.4

TABLE 4. RHEOLOGIC PROPERTIES OF SAMPLES
(mean values from three specimens of each blend)

Property	Sample Identification Number					
	9107	9108	9109	9110	9111	9112
Specific Gravity	1.31	1.24	1.17	1.23	1.32	1.17
Specimen Thickness (mils)	96	100	90	89	82	89
Modulus 100% (psi)	636	485	342	450	591	350
Modulus 200% (psi)	1590	1278	930	931	1633	1089
Modulus 300% (psi)	2480	2160	1714	2130	2503	1981
Tensile Strength (psi)	3040	3174	3037	3071	3102	3343
Elongation at Break	370	470	460	430	400	470
Durometer Hardness, Shore	71	69	66	65	72	65

TABLE 5. DIMENSIONS OF SAMPLES USED IN DMTA APPARATUS

Dimension	Sample Identification Number					
	9107	9108	9109	9110	9111	9112
Width (m)	0.01281	0.01323	0.01328	0.01234	0.01284	0.01219
Length (m)	0.01	0.01	0.01	0.01	0.01	0.01
Thickness (m)	0.0235	0.0248	0.0242	0.0236	0.0207	0.0236

TABLE 6. ESTIMATED GLASS TRANSITION TEMPERATURES AND WLF COEFFICIENTS FOR SIX NEOPRENE-NATURAL RUBBER BLENDS

Property	Sample Identification Number					
	9107	9108	9109	9110	9111	9112
Ratio of Neoprene-Natural Rubber	3	1	0.33	1	3	0.33
Glass transition temperature (degrees centigrade)	-55	-60	-60	-60	-60	-60
Williams-Landel-Ferry Coefficient C1	17	30	30	30	20	30
Williams-Landel-Ferry Coefficient C2	51	90	90	90	60	90

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13. ABSTRACT (Maximum 200 words) This report provides the results of measuring the various viscoelastic properties of specially formulated natural-neoprene rubber samples. Each sample is described in terms of its unique formulation, resulting specific gravity, Young's modulus, tensile strength, hardness, glass transition temperature, and Williams-Landel-Ferry coefficients.				
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