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(DDV-III-C)(U) LEHIGH UNIV BETHLEHEM PA COXE LAB
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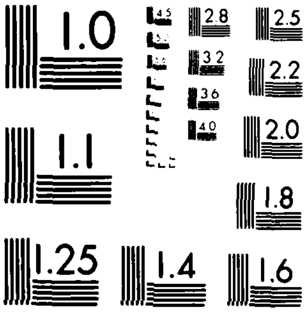

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Abstract

The Autovibron DDV-IIIC (IMASS, Inc.) is a forced vibration unit capable of operating at several constant frequencies for the determination of the dynamic mechanical response of a system. The automation provides a programmed heating rate, continuous sample tensioning, and acquisition and reduction of data. Problems have been encountered with sample alignment, tension adjustment and the measurement at low $\tan \delta$ values. Results obtained at several frequencies using the Autovibron for MBS-modified and unmodified PVC samples and a commercially available mineral-reinforced nylon composite (Minlon 12T, Dupont) are reported. It is concluded that this instrument has good potential for the convienent determination of dynamic spectra of polymers and their composites.

Dynamic Mechanical Spectroscopy Using the Autovibron (DDV-III-C)

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In determining the dynamic mechanical response of a system, it is often desirable to work with a forced-vibration instrument at a constant frequency. One of the instruments most commonly used for this purpose has been the direct-reading viscoelastometer originally developed by Takayanagi (1,2) —— the Rheovibron. A common model has been the model DDV-II (load capacity, 0.1-kgf); in recent years a 5-kgf-capacity model, the DDV-III-C, has been introduced (3,4).

While much valuable research has been based on results obtained using such units, several problems have been recognized. Thus the operator must give constant attention to the instrument over a period of 4 hr or more; also, maintenance of proper tension on the specimen is often far from easy. Other limitations include difficulty in working at $T > Tg$, and undesirably low ranges in tan δ and frequency. More detailed discussions are given by Yee and Takemori (5) and by Kenyon et al. (6).

To remedy or alleviate some of these problems, Yee and Takemori (5) modified and improved the Rheovibron model DDV-II-B by providing closed-loop control, and by improving and simplifying the technique used to determine the loss tangent and the storage modulus. Gains in accuracy, simplicity of operation, and adaptability to digital processing of the data were reported. The Rheovibron itself has been automated by the manufacturer (3); Kenyon et al. (6) also automated the Rheovibron DDV-II to provide automatic control of tension, increased sensitivity, and calculation and printout of E', E", and tan δ. The latter unit has been commercialized by Imass, Inc. (4) as the Autovibron, model DDV-II-C. Recently a generally similar adaptation has been introduced, based on the hydraulically operated Rheovibron DDV-III-C. Automation of a resonance-type (7) and a different constant frequency instrument(8) have also been described.

While a full critical analysis of the operation of the Autovibron DDV-III-C has not yet been possible, it is appropriate to describe our experience with this new instrument, and to make preliminary recommendations with respect to operation and future improvement. Since the instrument is the first of its type, the observations reported should be helpful to other investigators. Results obtained in our laboratory using an automated DDV-II are also described for comparison.

INSTRUMENTATION

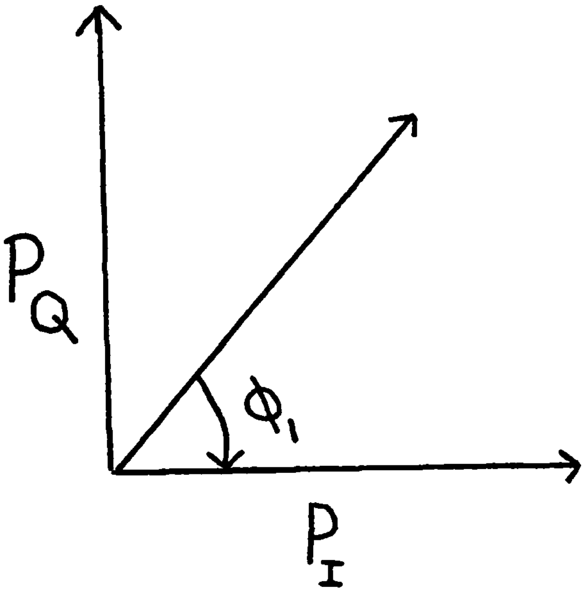
As mentioned above, the model DDV-III-C Rheovibron (Toyo Baldwin Co.) has been combined with an automation package supplied by Imass, Inc. The instrument maintains the essential characteristics of the Rheovibron DDV-III-C, utilizing the original sample bench, hydraulic system, load cell, and basic electronics. Four fixed frequencies — 3.5, 11, 35 and 110 Hz — are available. Sample sizes up to 7cm x 1cm x 5mm can be handled, with a claimed range for complex Young's modulus between 1 MPa and 100GPa (1GPa= $1GN/m^2=10^{10}$ $dynes/cm^2$). A low-temperature chamber allows measurements to be taken from -140°C to ∿175°C. with a programmed rate of temperature increase of ∿1°C/min. A second chamber is provided for temperatures up to ∿300°C. The automation package is responsible for sample tensioning, phase angle measurements, temperature control, data acquisition and data reduction. The key components of this package are a lock-in analyzer (Princeton Applied Research, Model 5204), a programmable calculator (Hewlett Packard Model 9825A), a multiprogrammer (Hewlett Packard Model 6940B), and an optional plotter (Hewlett Packard Model 9872B). The automation package can also be readily interfaced with a Rheovibron Model DDV-II. The essential differences in the two units are the driving unit and the load capacity. The hydraulic driving system of the DDV-III is replaced by an electro-mechanical driver in the smaller unit. The DDV-II is capable of handling sample sizes up to 5cm x 0.05cm x 0.4cm (Fig. 1 shows a comparison of sample sizes for the two models) with a maximum load capacity of 0.1kgf and modulus range of 100KPa to 100GPa. A schema is given in Figure 2.

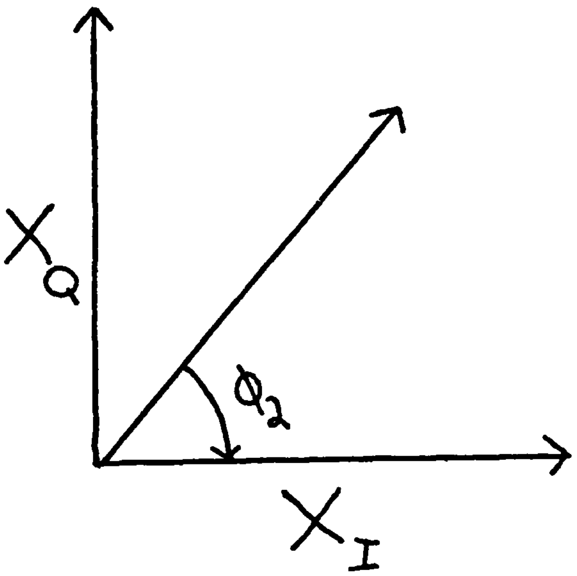
Temperature programming is effected through the calculator in conjunction with a platinum resistance thermometer. From -140°C to -45°C the temperature is allowed to increase without regulation at a rate of $\sim$1°C/min. At -45°C, power is supplied to the heaters and the temperature is controlled by programming the application of power. The temperature use can also be controlled at rates other than 1°C/min. through changes in the operating program. For temperatures above 175°C, the high-temperature chamber must be used.

Phase-angle measurements using the lock-in analyzer were incorporated to simplify automation of the measurements, improve resolution of small angles, and increase the range of tan δ measurements (4). The calculator alternately switches the load (P) and displacement (X) signal through the multiprogrammer to the lock-in analyzer. After a programmed delay for setting of the signal, the in-phase and quadrature components of the respective signals are measured with respect to a reference signal from the Autovibron. The complex Young's modulus, E*, is calculated using Equation 1.

$$E^* = R \frac{(P_I^2 + P_Q^2)^{1/2} \quad L^2}{(X_I^2 + X_Q^2)^{1/2} \quad V} \qquad (1)$$

The subscripts I and Q designate the in-phase and quadrature components of the respective signals, R is a ranging and scaling factor , L is the sample length and V is the original sample volume. The phase difference, δ is calculated using Equation 2.





$$\delta = \phi_1 - \phi_2 = \arctan \frac{P_Q}{P_I} - \arctan \frac{X_Q}{X_I} \qquad (2)$$

With E* and δ from Equations 1 and 2, respectively, the storage modulus, E', and loss modulus, E", can be calculated.

$$E' = /E^*/\cos \delta \qquad (3)$$

$$E'' = /E^*/\sin \delta \qquad (4)$$

$$\tan\delta = E''/E' \qquad (5)$$

The data acquired are displayed while the program is running and stored on magnetic-tape cartridges for further reduction. Results can be printed or plotted during the run with appropriate programming; programs for data reduction and plotting from tape are available.

EXPERIMENTAL

Problems and their correction. During start-up and subsequent trials using the DDV-III-C, several significant problems have been encountered. Much of our recent work has been conducted using an automated DDV-II because of problems with the DDV-III. Preliminary work with the large load capacity unit has shown problems in sample tensioning, load control, measurement of small phase angles and programming. These deficiencies are discussed below.

Sample tension is controlled by the calculator through the multiprogrammer and a stepping-motor that moves the load arm. The original stepping-motor assembly used springs to control tension and resulted in enough lateral motion to preclude maintenance of alignment. Such alignment is of critical importance, for otherwise serious errors in modulus and damping can result. A combination of shims to align the center of the load-arm with the center of the driver and a new screw-driven stepping-motor assembly (redesigned by Imass, Inc.) have minimized lateral motion. However, even with the modification mentioned, great care must still be taken with clamping and alignment of the specimen. Also, bowing in the sample can be introduced by non-uniform tightening of the grips. Some scatter may also be due to an inherent design problem; the manufactures is currently revising the signal reading section. As was the case with the Autovibron DDV-II(6), sample mounting and alignment are two major flaws of the instrument. Reproducible methods for sample mounting have been reported recently for the DDV-II(9); similar modifications should be included in further redesign of the DDV-III system.

The original software calculated the DC voltage of the load signal by sampling the sine wave, calculating the amplitude, and dividing by two to obtain the DC bias. Because of problems in measuring the load, the program was modified to measure the DC voltage bias directly by momentarily switching off the sine wave. Load corrections are then made by the stepping-motor to maintain a preset limit. This new load-control program functions acceptably through a programmed temperature run except in the region around the glass transition (T_g). Immediately after the transition, the sample is often put into compression. So far, reliable measurements of rubbery moduli on the order of 10MPa have been obtained only occasionally.

While monitoring the load signal (P) with an oscilliscope, a problem was evident in the switching of the signal. The value of the load was intermittently recorded as zero. Since tension is a function of load, when zero loads are recorded the rheovibron reacts by making drastic changes, in sample length resulting in incorrect modulus and phase angle measurement This problem appears to have been corrected by the replacement of a relay-readback board in the multiprogrammer.

Another problem has been the determination of optimum settings of the phase controls on the lock-in analyzer. After some experimentation it was determined that the in-phase and quadrature readings should be set approximately equal in magnitude and with the same sign (positive or negative) using the reference angle potentiometer and quadrant selector of the lock-in analyzer. Use of these settings appears to reduce the time required for the signal to stabilize and facilitates ranging of the signals. The ranging subroutine has also been rewritten by IMASS to alleviate a problem with signal saturation that was occurring when the in-phase and quadrature components of the signal became unbalanced.

Performance. Figures 3 and 4 compare data from two different runs at 110 Hz on a methacrylate-butadiene-styrene (MBS) rubber-modified PVC in the as-received condition (PVC M_w=0.95x10^{-5}; MBS, 10 phr). A previous paper by this group contains detailed characterization of the PVC (10). The sampleswere of similiar cross-sectional area and length (see Table 1); the test run using an oscillating displacement, Δl, of 2.5x10^{-3}cm (i.e., at $\approx$0.05%strain). Great care was taken with sample mounting and alignment. Over the temperature range from -115°C to +100°C, values of the storage modulus (E') obtained in the two tests agreed within less than 5%. Below -75°C (corresponding to tan $\delta \leq 0.02$) significant scatter is evident in the loss modulus (E") and tan δ, and the slopes differed considerably, so that the value of tan δ at -100°C is $\sim$40% less in Figure 4 than in Figure 3.(The shapes of the E" and tan δ curves in Figure 4 are in fact atypical.) Considerable scatter has also been seen at low values of tan δ with the Autovibron DDV-II-C (11). Note, however, that the peak for the MBS phase is clearly evident at $\sim$-60^{0}C. While data for specimen A (Fig. 3) could be taken up to $\sim$140°C, it was not possible to exceed

∿100°C with specimen B, due to excessive specimen deformation. Values of the glass transition temperature (T_g) and complex modulus (E*) are reported in Table 1.

Figures 5,6 compare data obtained by Matsuo (12) and by us using manually operated model DDV-II Rheovibrons with data obtained on an automated DDV-III Rheovibron. Values obtained by Matsuo (12) and by us using the manual DDV-II appear comparable. The shapes of the curves are similar and differences in E' and T_g are probably the result of different samples and operators. Figure 6 shows data obtained in our laboratory using an Autovibron DDV-III-C and a manual DDV-II. The E' values from the DDV-II have been corrected using the K-correction factor outlined in the Rheovibron instruction manual (2). The data from the DDV-III-C are uncorrected for instrument compliance. The values obtained agree well except in the T_g region where the temperature control of the manual DDV-II is suspect. The values of E* at 0°C (∿2.9 GPa) using the DDV-III-C is very close to the corresponding value found by Kenyon et. al. using their automated Rheovibron model DDV-II(6).

Figure 7 illustrates data for two different specimens of the same material obtained with an automated Rheovibron DDV-II. The two samples have almost identical length, width and thickness (see Table 1). Both samples were machined in the same manner and run by the same operator with the same operating conditions. The initial oscillating displacement, Δl, was 7.9×10^{-4}cm (i.e.≃0.01% strain) in both cases. E* values agree to within 9% at -100°C and are less than 6% different at 40°C. In the T_g region E', E" and Tan δ are virtually identical. The two samples do not, however have the same E" and tan δ in the region from -100°C to

+50°C. For example, tan delta readings of 0.033 and 0.056 were taken at -25°C for the two samples. These types of differences are as yet unexplained and suggest caution should be employed when analyzing data.

A comparison of data from the two automated units (Table 1 and Figures 3,4 and 7) reveals that the modulus of similar PVC samples is almost two times higher when measured on the automated DDV-III than on the automated DDV-II. A recent publication (9) addresses error analysis for the manual DDV-II and the analysis should be extended to the automated unit. Errors of up to 50% have been reported (9) and have been attributed to the instruments compliance, sample yielding and slipping in the clamps, sample alignment, the instrument's inertia, variable sample sizes, and structural changes in the sample during testing. Each of these problems should be addressed using the automated unit before a thorough understanding of the unit will be possible and true material properties can be measured with full confidence.

Studies of the effects of thermal history and frequency on PVC and MBS-modified PVC are under way. Preliminary results obtained on a quenched PVC ($M_w = 2 \times 10^5$; 14 phr MBS; quenched from 110°C in ice) are shown in Figure 8 with respect to an as-received sample. The results show an increase in damping between T_B and T_g in the quenched sample similar to the results of Struick (13); Figure 9 shows the same PVC run at 35 Hz. Note that the MBS T_g is clearly shown in both but the damping between -50°C and +50°C is slightly lower for the 35 Hz sample. The glass-transition temperature (T_g) of the PVC has shifted approximately 3°C. (See Table 1 for data).

Figures 10 and 11 show frequency data for a lower molecular weight PVC ($M_w = 7 \times 10^4$; 14 phr MBS) that has been quenched. An effect of frequency is not evident in the damping behavior between -50°C and +50°C of these two samples. Data in Figure 11 also shows the effects of vibrations and resonances on the data. At T = -10°C, the scatter in E" and tan δ was caused by lateral vibration of the sample, clamps and rods. This problem has also been seen by others and is discussed in a recent publication (9).

Figure 12 displays data for two samples that were quenched in ice from 110°C and then annealed at 65°C for a period of 7 days or 35 days. The damping between 0°C and 50°C had clearly been effected by the aging process in a manner similar to data presented by Struick (13). (The general behavior of a non-aged quenched sample can be seen in Figure 8 and used for a qualitative comparison.) The difference in the magnitude of damping from -150°C to 75°C is at this point unexplained.

Studies of the effects of frequency and water content on the dynamic spectra and fatigue of various reinforced polymers (14) are also in progress. Typical best fits for the dynamic spectra for specimens of a mineral-reinforced nylon are shown in Figures 13-16, inclusive. Three frequencies (3.5, 11, and 110 Hz) and five water contents (dry, 0.6, 1.3, 2.8, and 4.8%) were studied; the data show the trends expected with respect to frequency and water content. It may be noted that the non-automated Rheovibron is not easily used at frequencies <110 Hz. Apparent activation energies of the principal relaxation processes have also been estimated to be: 68kJ/mole (16 kcal/mole) for the β relaxation, and 160kJ/mole (39 kcal/mole) for the α transition. The curves of the frequencies of the maxima in E" are almost coincident with those presented by McCrum et al. (15) for unmodified nylon 66.

Conclusions and Recommendations

Several conclusions and recommendations are in order:

1. Although many problems have been encountered, the Autovibron (DDV-IIIC) does show promise for the convenient determination of dynamic spectra of a variety of standard and multi-phase polymer systems at frequencies from 3.5 to 110 Hz.

2. Further work is needed on methods for clamping and alignment and the adjustment of tension at $T > T_g$.

3. A thorough analysis of errors resulting from instrument compliance, sample yielding and slipping in the clamps, variable sample sizes, and instrument inertia is needed in light of the recent work with the manual Rheovibron(9).

4. Further documentation from the manufacturer is in order.

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Table I. Comparison of Dynamic Data on a Standard and Modified PVC using Autovibron and Rheovibron Model DDV-II.

Sample	Rheovibron Model	Frequency, Hz	Tg, °C	Complex Modulus, $E^* \times 10^{-9}$ Pa			L, cm[1]	L/W^2[2] cm^{-1}	L/A^3[3] cm^{-2}
				E* at -100°C	E* at 0°C	E* at 40°C			
131-4-Q[4]	II	110	78	2.363	1.694	1.320	6.25	28.8	720
131-4-Q	II	35	76	2.389	1.599	1.335	6.31	30.5	677
135-4	II	35	83	2.155	1.549	1.400	6.47	31.2	665
135-4	II	110	83	2.047	1.521	1.350	6.48	30.6	650
135-4	II	110	83	2.240	1.639	1.434	6.45	29.7	660
135-4-Q	II	110	83	2.20	1.571	1.307	6.37	28.1	624
132-3 A	III	110	94	4.240	2.867	2.501	4.942	12.7	59.5
132-3-B	III	110	95	4.125	2.790	2.464	4.956	13.0	62.1
135-1	manual II	110	116	4.51	2.51	2.24	1.648	18.0	708
135-1	III	110	93	3.794	2.889	2.625	3.766	9.0	38
135-1 35 Day[5]	II	110	82	2.086	1.675	1.495	6.53	26.1	580
135-1 7day[6]	II	110	83	2.178	1.735	1.533	6.43	25.7	571

1 - L = sample length
2 - L/w = length ÷ width
3 - L/A = length ÷ area
5 - annealed 35 days at 65°C
6 - annealed 7 days at 65°C

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Figure Captions

Figure 1. Comparison of the correct sample size versus modulus for the DDV-II(—) and DDV-III (---)Rheovibrons.

Figure 2. Block diagram of Autovibron

Figure 3. Dynamic mechanical spectra (110 Hz) of MBS-modified PVC using a DDV-III-C Rheovibron.

Figure 4. Dynamic mechanical spectra (110 Hz) of MBS-modified PVC using a DDV-III-C Rheovibron.

Figure 5. Dynamic mechanical spectra of PVC (110 Hz) using a manual DDV-II Rheovibron (ref. 12).

Figure 6. Comparison of dynamic mechanical spectra of PVC (M_w = 2x10^5) using model DDV-III-C (•••) and manual DDV-II.

Figure 7. Dynamic mechanical spectra of two replicate MBS-modified PVC samples using an automated DDV-II. (M_w = 2x10^5; 14 phr MBS).

Figure 8. Dynamic mechanical spectra using an automated DDV-II of MBS-modified PVC (Mw = 2x10^5; 14 phr MBS) as received (•••), quenched (---).

Figure 9. Dynamic mechanical spectra of an MBS-modified PVC (Mw = 2x10^5; 14 phr MBS) run a 35 Hz using an automated DDV-II.

Figure 10. Dynamic mechanical spectra of a quenched MBS-modified PVC (Mw = 7x10^4, 14 phr MBS) run at 110 Hz using an automated DDV-II.

Figure 11. Dynamic mechanical spectra of a quenched MBS-modified PVC (Mw = 7x10^4; 14 phr MBS) run a 35 Hz using an automated DDV-II.

Figure 12. Dynamic mechanical spectra of MBS-modified PVC (Mw = 2x10^5; 0phr MBS) after 7 day (---) and 35 day (•••) treatment at 65°C using an automated DDV-II at 110 Hz.

Figure 13. Effect of frequency on dynamic mechanical spectra (14) of Minlon 12T using autovibron DDV-III-C; 110 Hz (-*-) and 11 Hz (-o-).

Figure 14. Effect of frequency on dynamic mechanical spectra (14) of Minlon 12T using autovibron DDV-III-C; 110 Hz (-*-) and 3.5 Hz (-o-).

Figure 15. Effect of water on dynamic mechanical spectra (14) of Minlon 12T using an autovibron DDV-III-C; 0.6% Hzo (x) and 2.8% H_2O(0).

Figure 16. Effect of water on dynamic mechanical spectra (14) of Minlon 12T using an autovibron DDV-III-C; 1.3% H_2O(x) and 4.8% H_2O(o)

Figure. 1

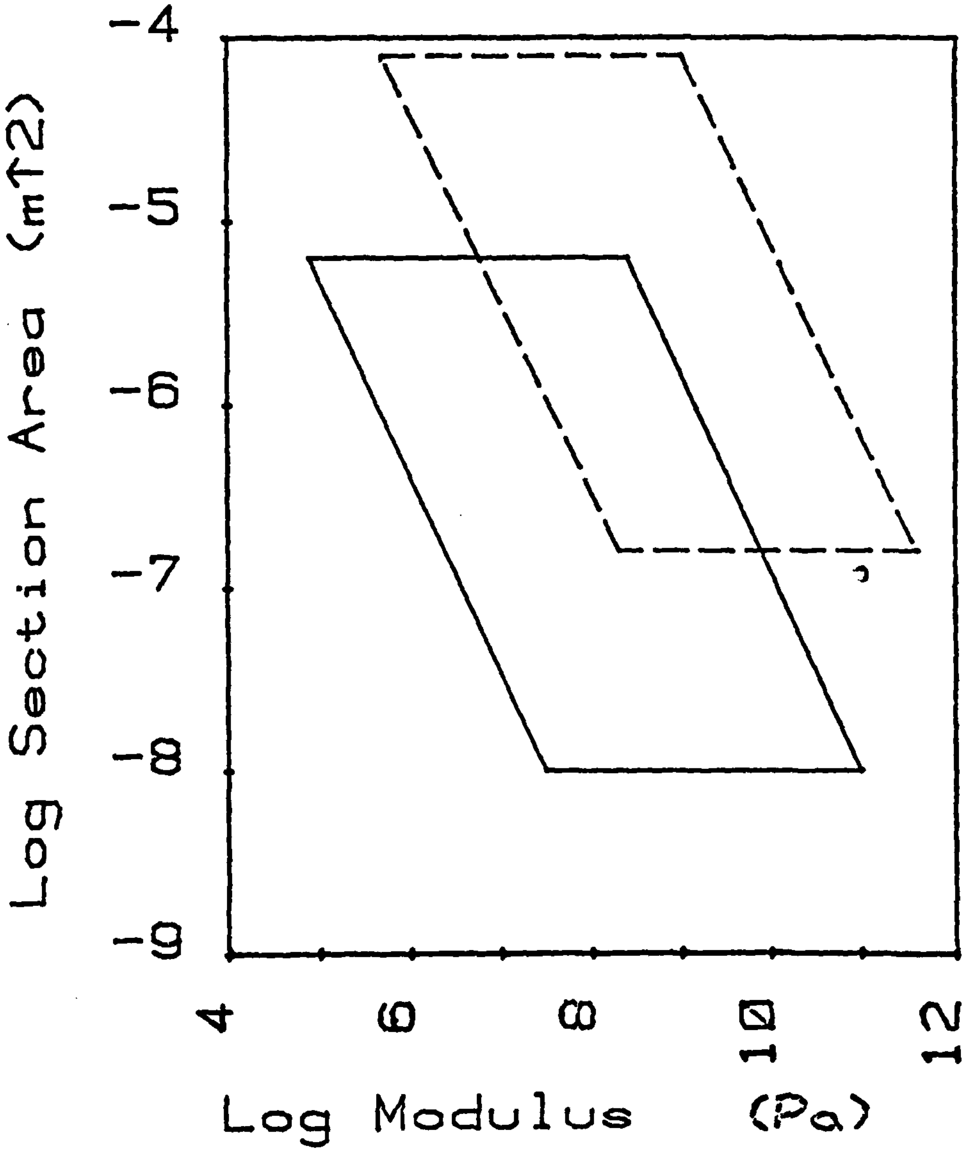
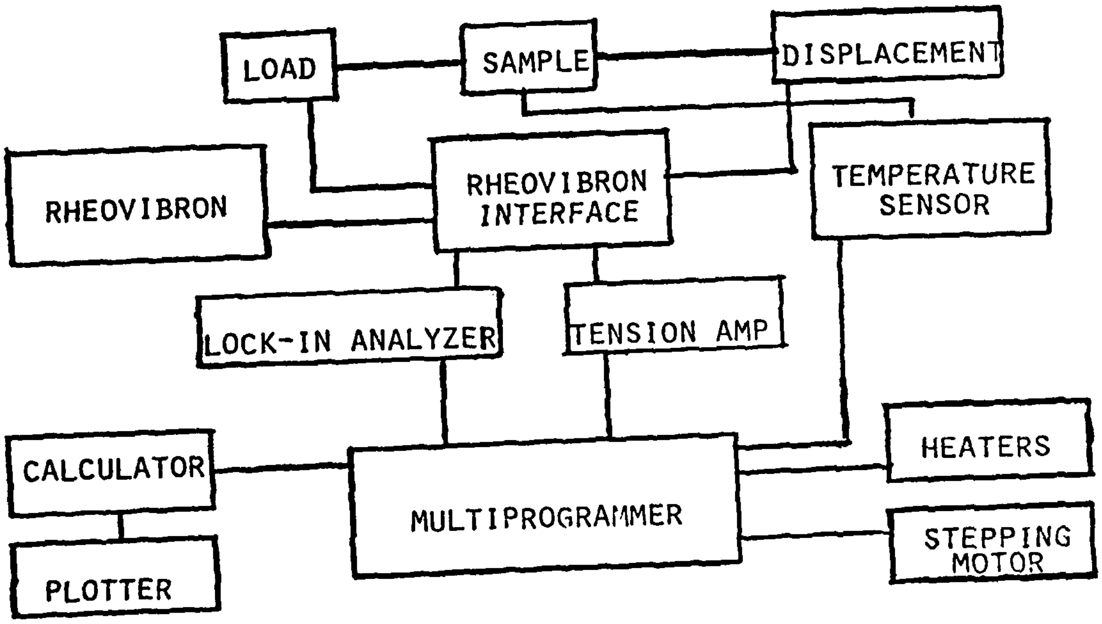
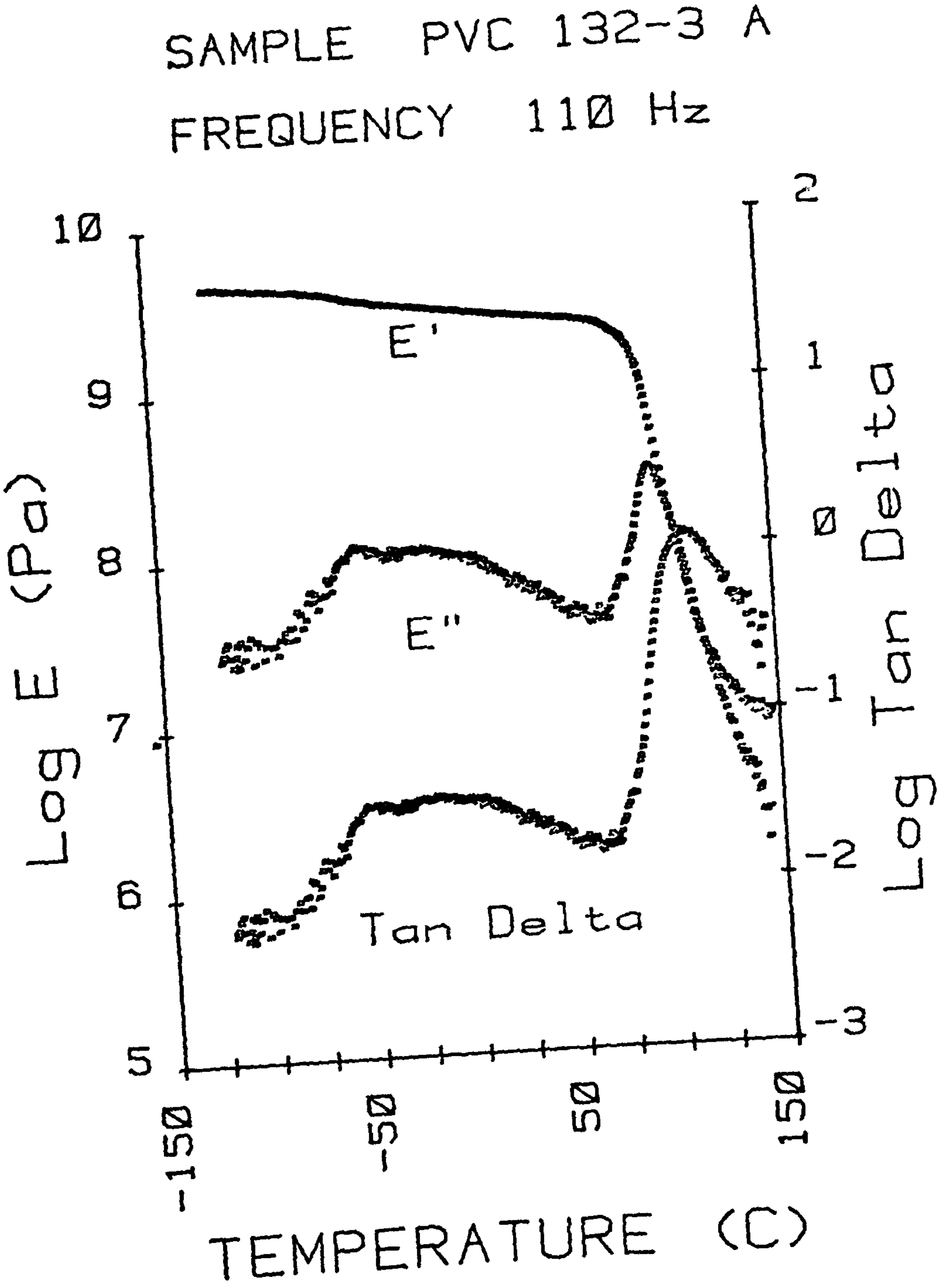


Figure. 2



BLOCK DIAGRAM OF AUTOVIBRON

Figure. 3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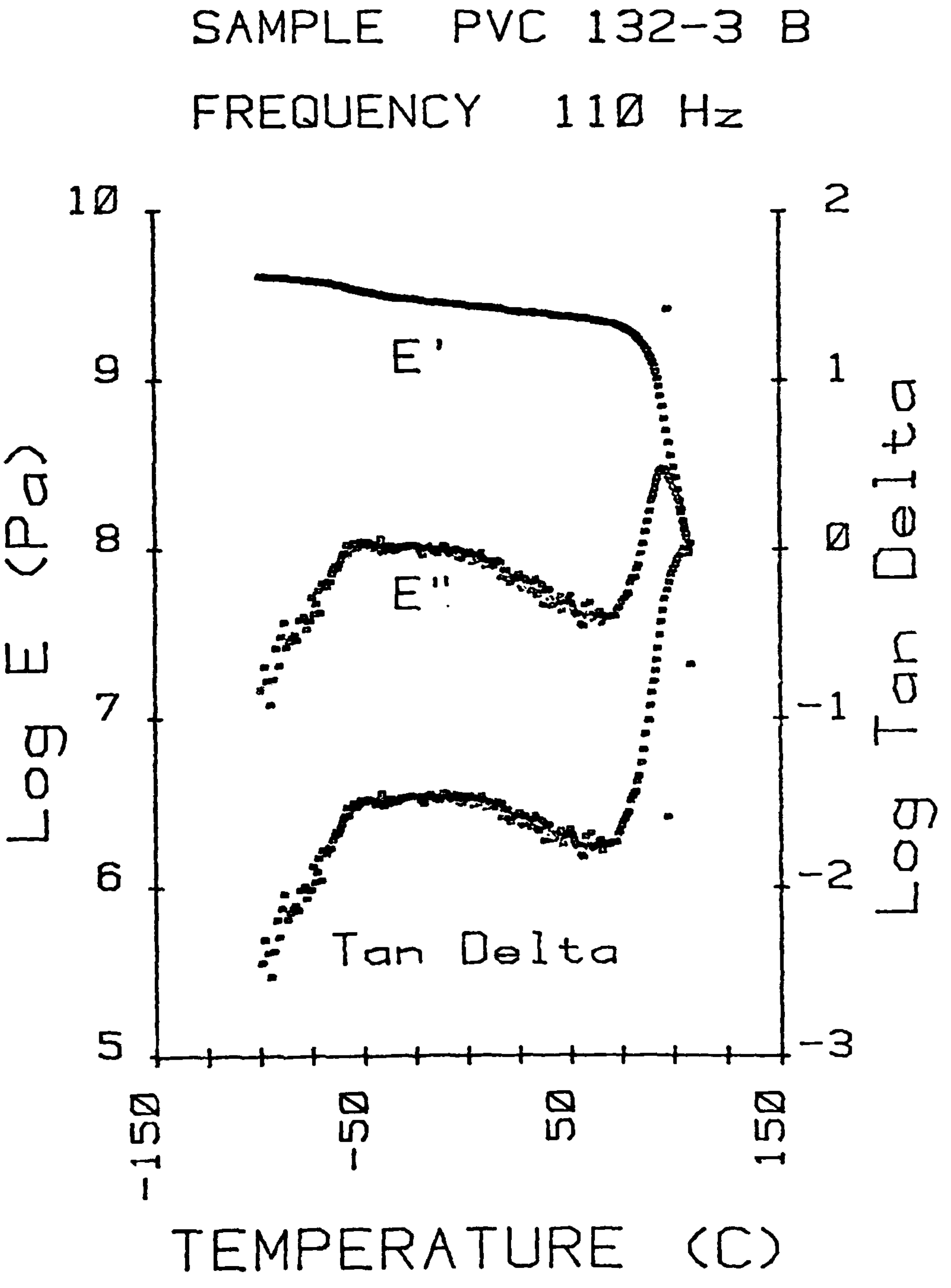
FREQUENCY 110 Hz
10
9
8
7
6
5
Log E (Pa)
2
1
0
-1
-2
-3
Log Tan Delta
E'
E"
Tan Delta
-150
-50
50
150
TEMPERATURE (C)

Figure. 4



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FREQUENCY 110 Hz
Log E (Pa)
10
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Log Tan Delta
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-3
E'
E"
Tan Delta
-150
-50
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150
TEMPERATURE (C)

Figure. 5

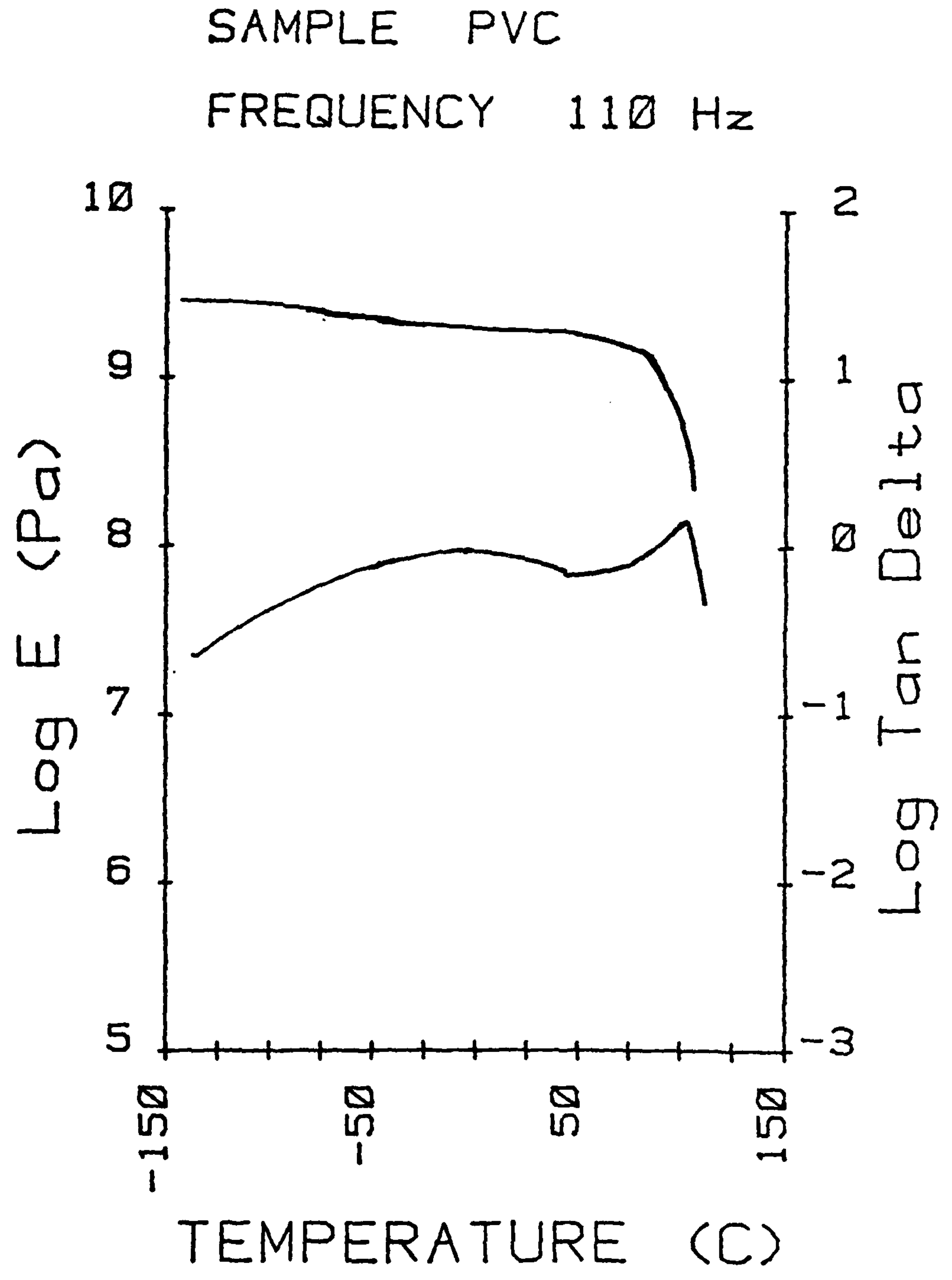

SAMPLE PVC
FREQUENCY 110 Hz
10
9
8
7
6
5
Log E (Pa)
2
1
0
-1
-2
-3
Log Tan Delta
-150
-50
50
150
TEMPERATURE (C)

Figure. 6

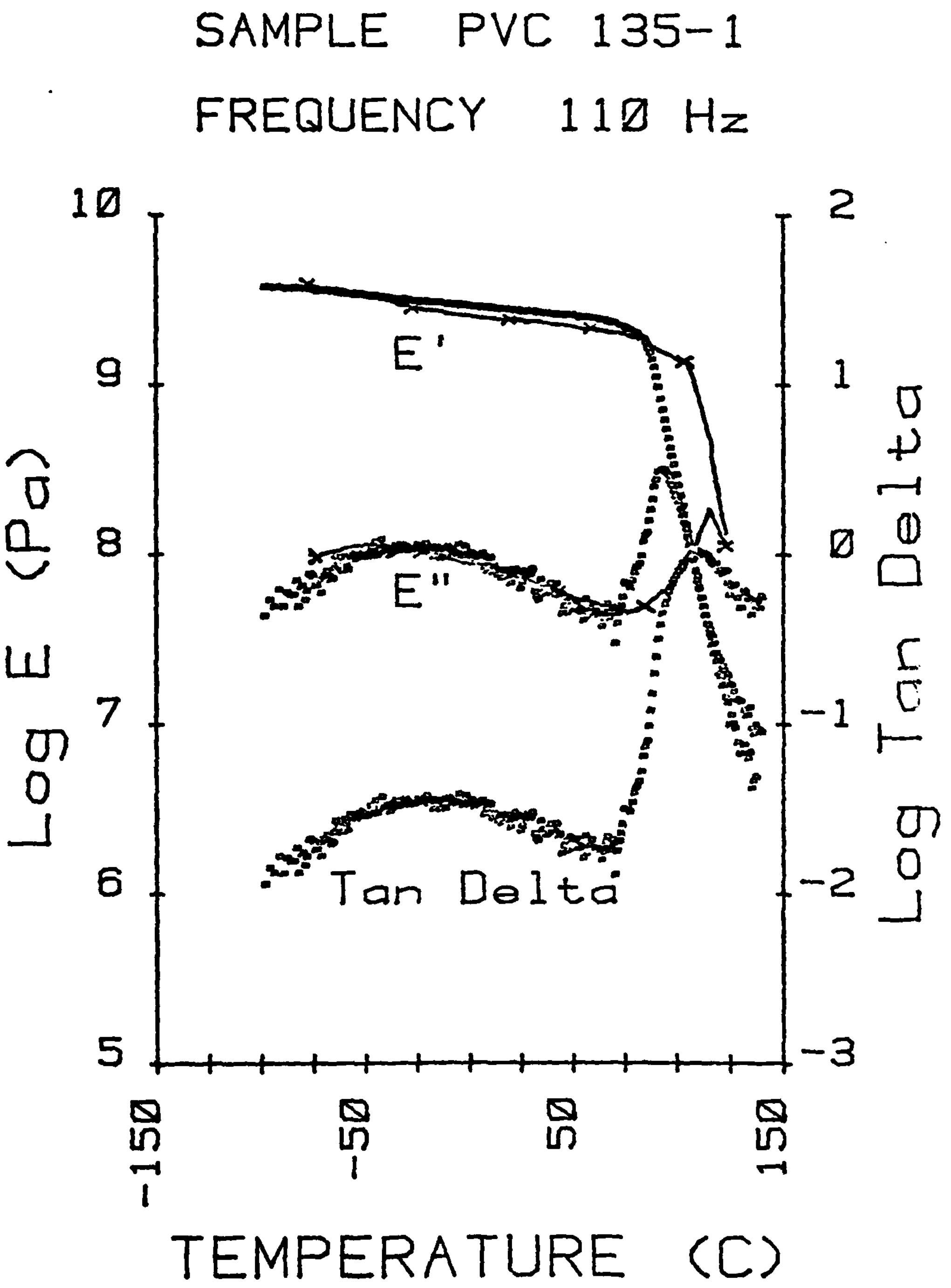

SAMPLE PVC 135-1
FREQUENCY 110 Hz
Log E (Pa)
Log Tan Delta
TEMPERATURE (C)
10
9
8
7
6
5
2
1
0
-1
-2
-3
-150
-50
50
150
E'
E"
Tan Delta

Figure. 7

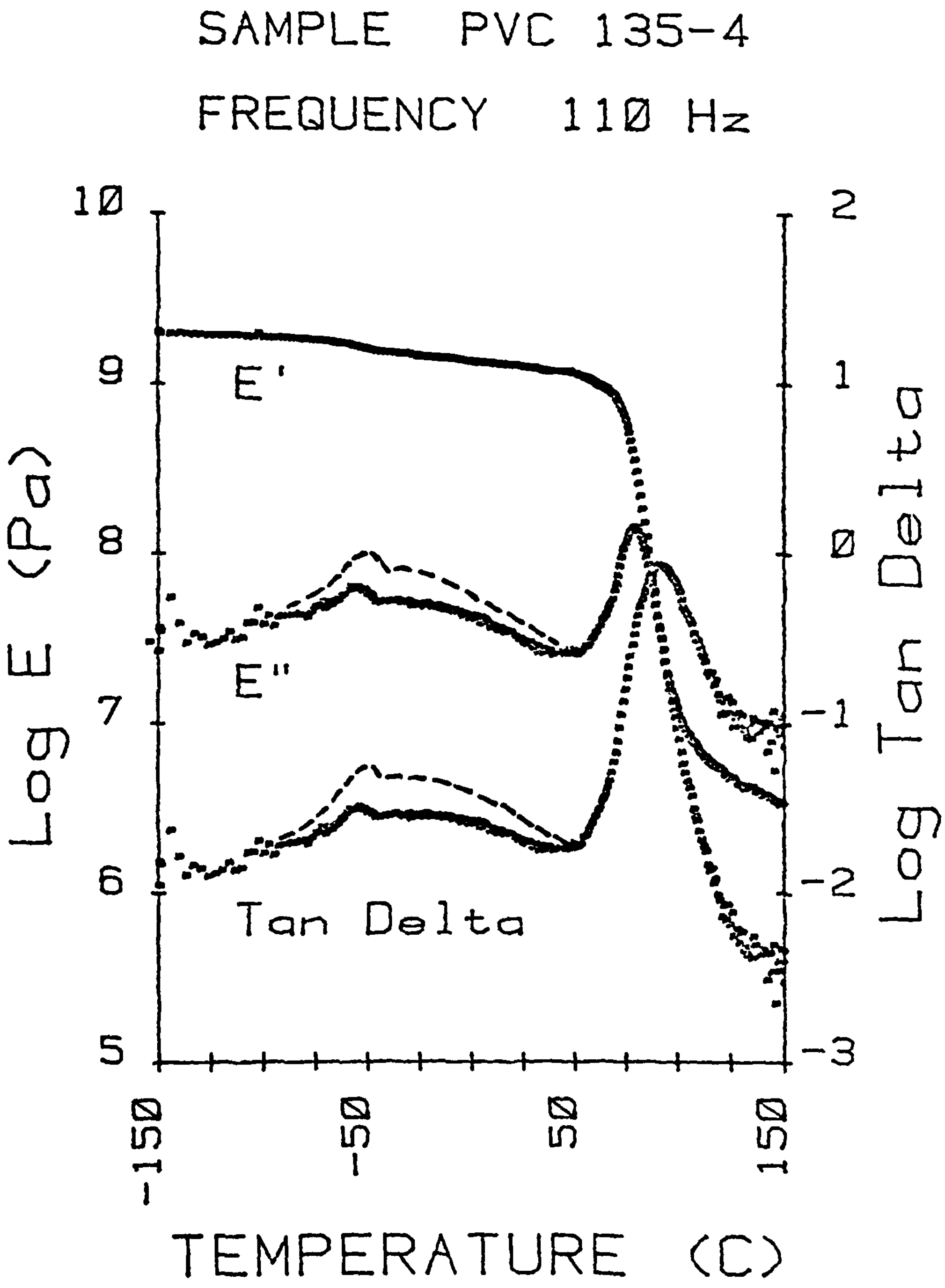

SAMPLE PVC 135-4
FREQUENCY 110 Hz
10
9
8
7
6
5
2
1
0
-1
-2
-3
E'
E"
Tan Delta
Log E (Pa)
Log Tan Delta
-150
-50
50
150
TEMPERATURE (C)

Figure. 8

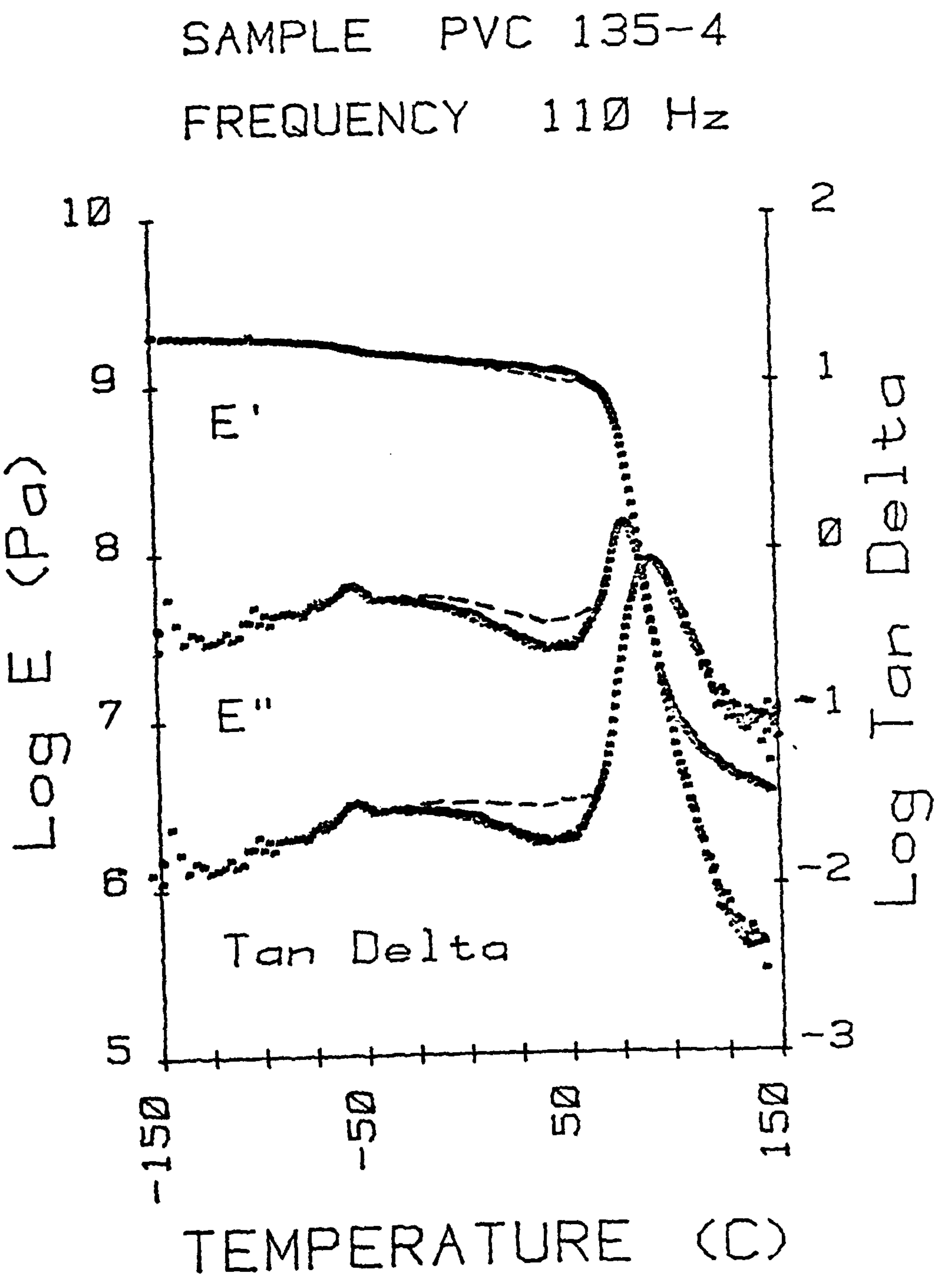

SAMPLE PVC 135-4
FREQUENCY 110 Hz
Log E (Pa)
10
9
8
7
6
5
Log Tan Delta
2
1
0
-1
-2
-3
E'
E"
Tan Delta
-150
-50
50
150
TEMPERATURE (C)

Figure. 9

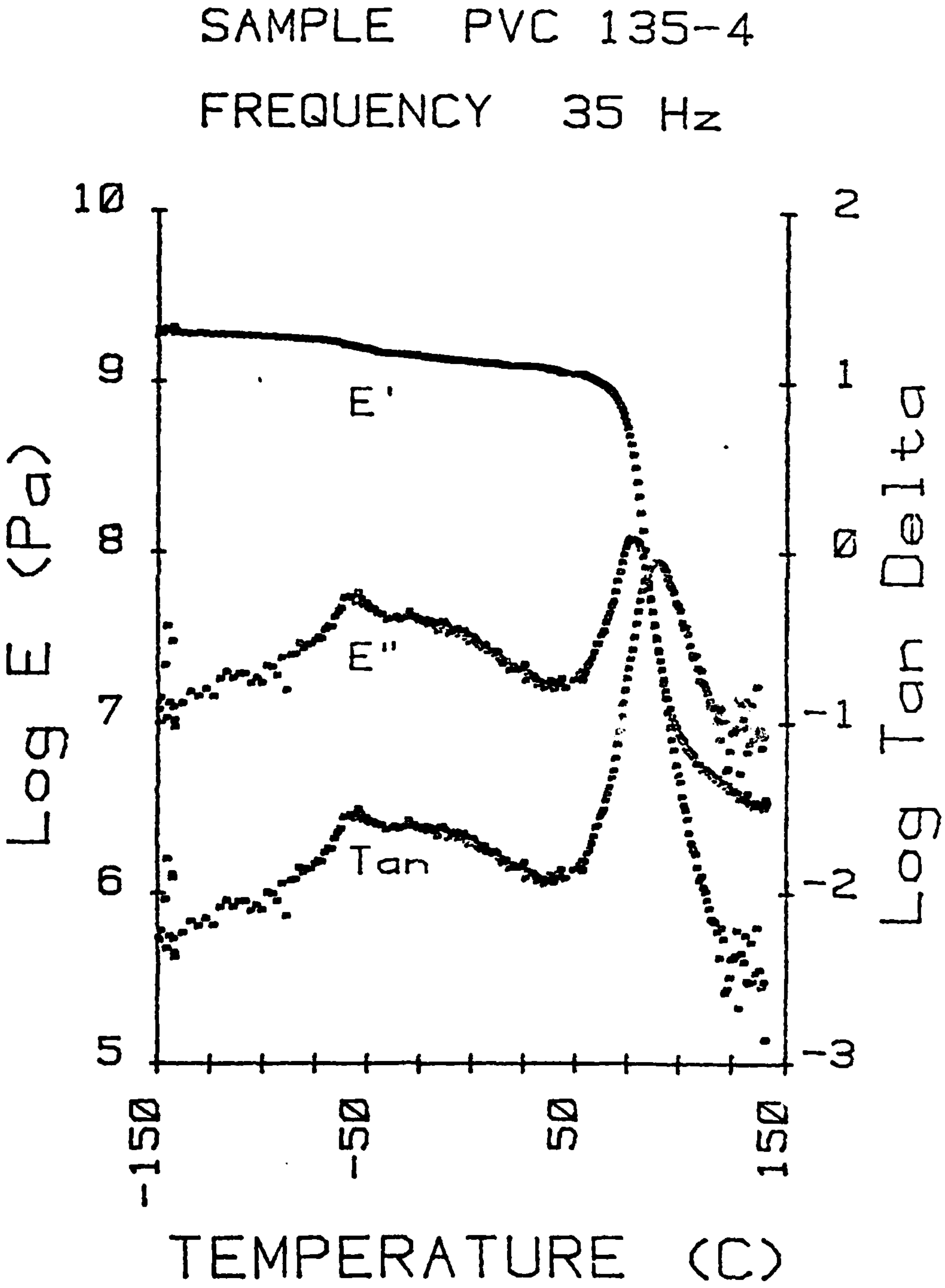

SAMPLE PVC 135-4
FREQUENCY 35 Hz
10
9
8
7
6
5
2
1
0
-1
-2
-3
E'
E"
Tan
Log E (Pa)
Log Tan Delta
-150
-50
50
150
TEMPERATURE (C)

Figure. 10

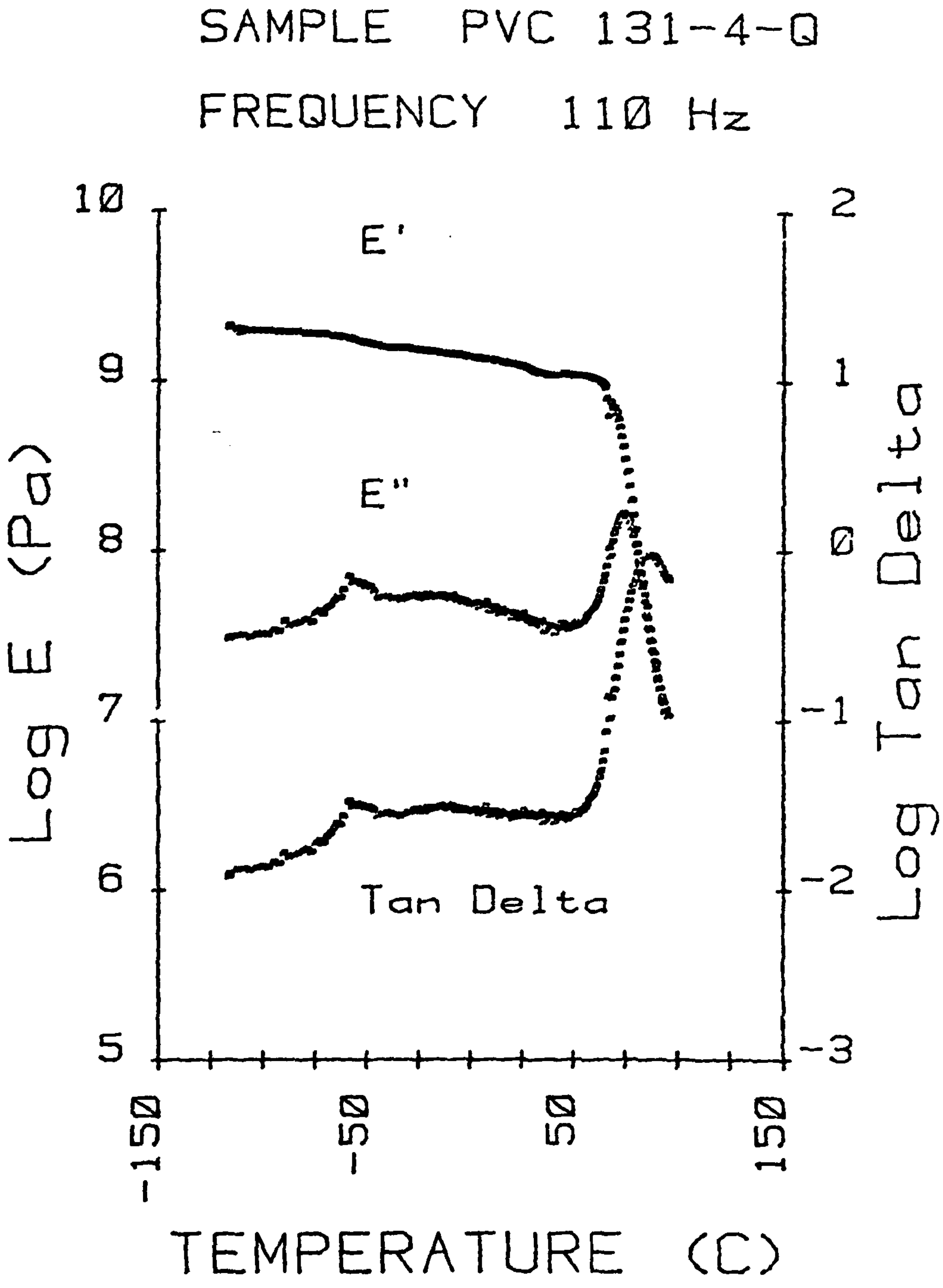

SAMPLE PVC 131-4-Q
FREQUENCY 110 Hz
E'
E"
Tan Delta
Log E (Pa)
Log Tan Delta
10
9
8
7
6
5
2
1
0
-1
-2
-3
-150
-50
50
150
TEMPERATURE (C)

Figure. 11

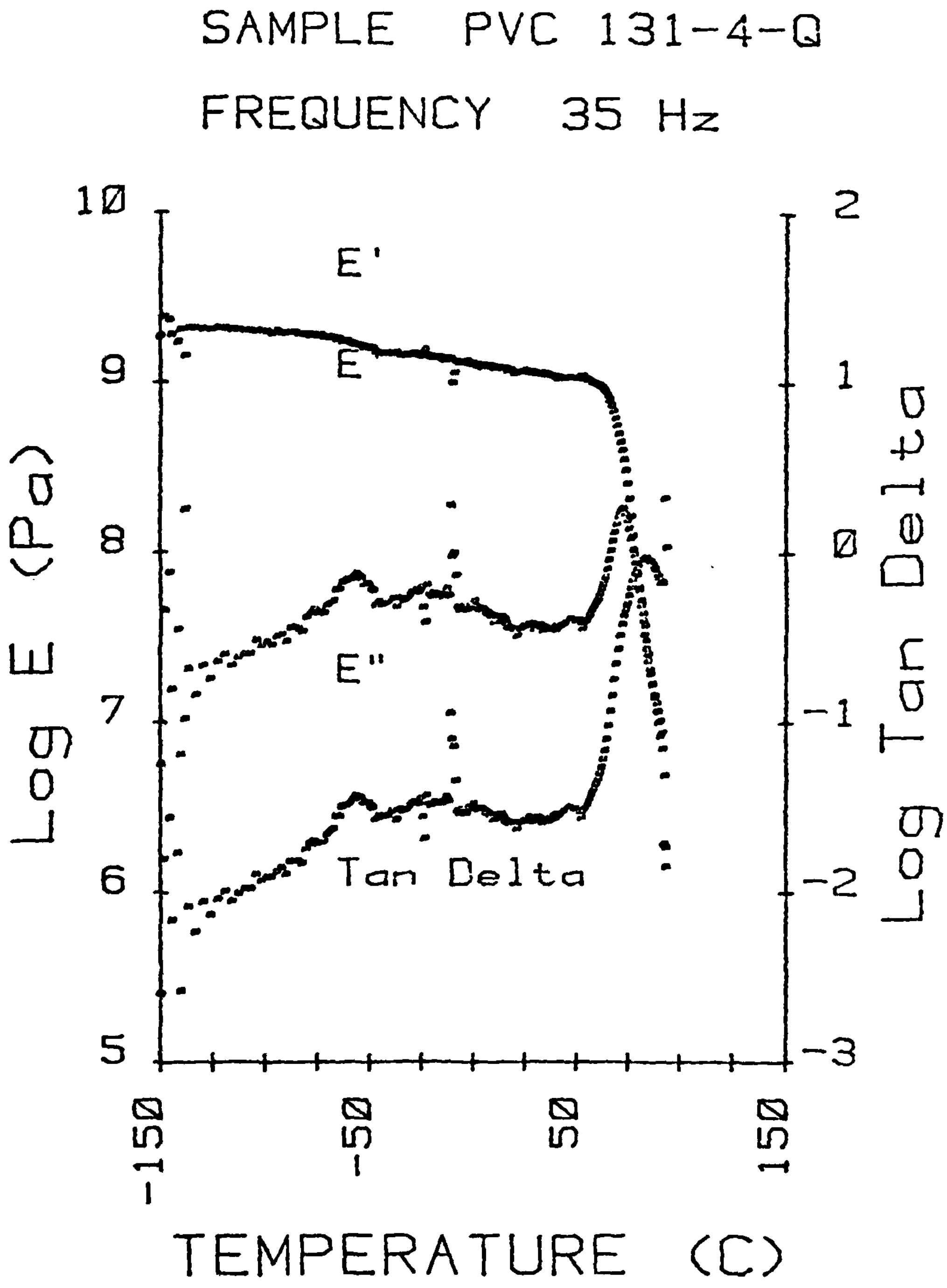

SAMPLE PVC 131-4-Q
FREQUENCY 35 Hz
Log E (Pa)
10
9
8
7
6
5
Log Tan Delta
2
1
0
-1
-2
-3
E'
E'
E"
Tan Delta
-150
-50
50
150
TEMPERATURE (C)

Figure. 12

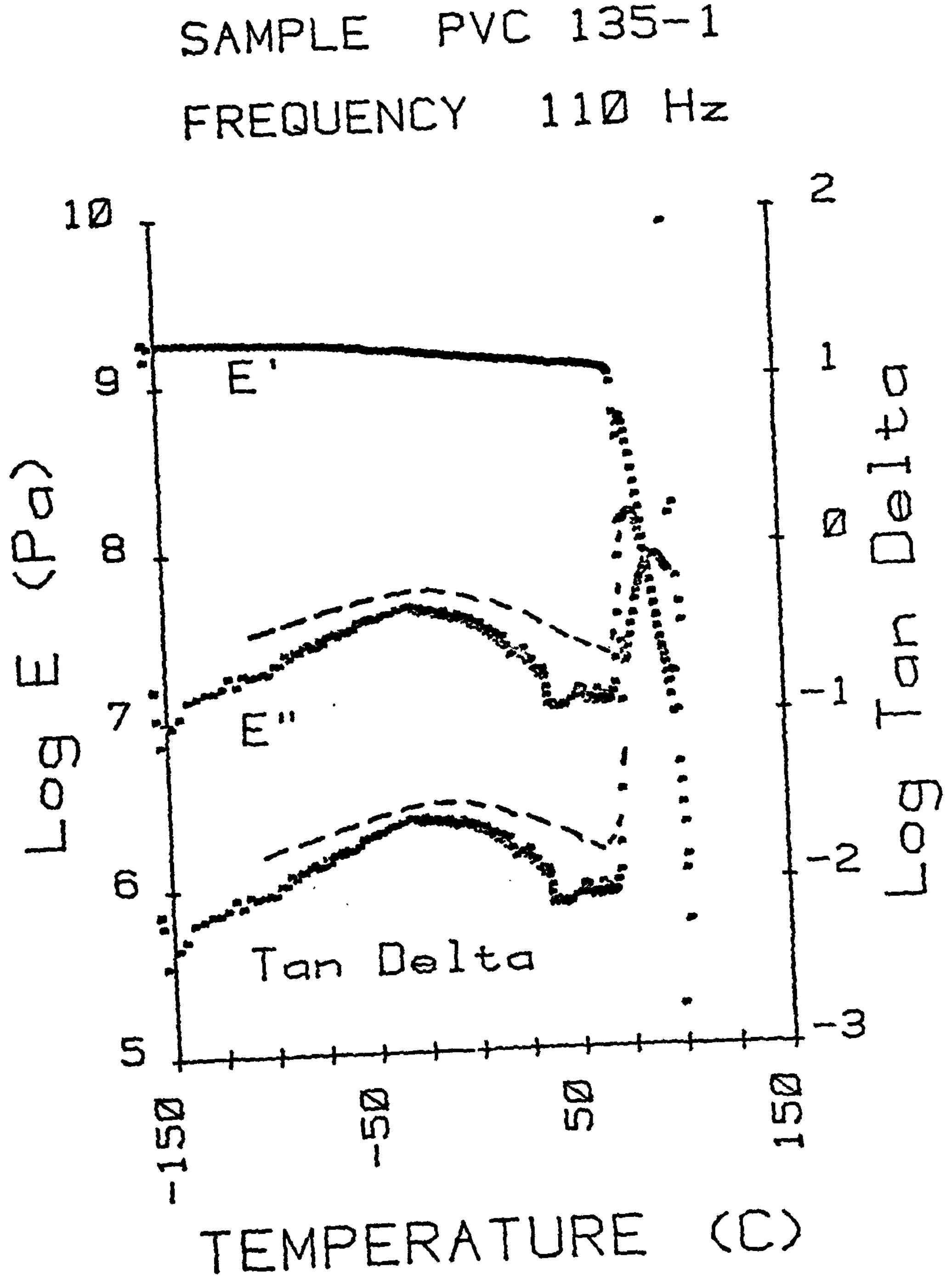

SAMPLE PVC 135-1
FREQUENCY 110 Hz
10
9
8
7
6
5
Log E (Pa)
2
1
0
-1
-2
-3
Log Tan Delta
E'
E"
Tan Delta
-150
-50
50
150
TEMPERATURE (C)

Figure. 13

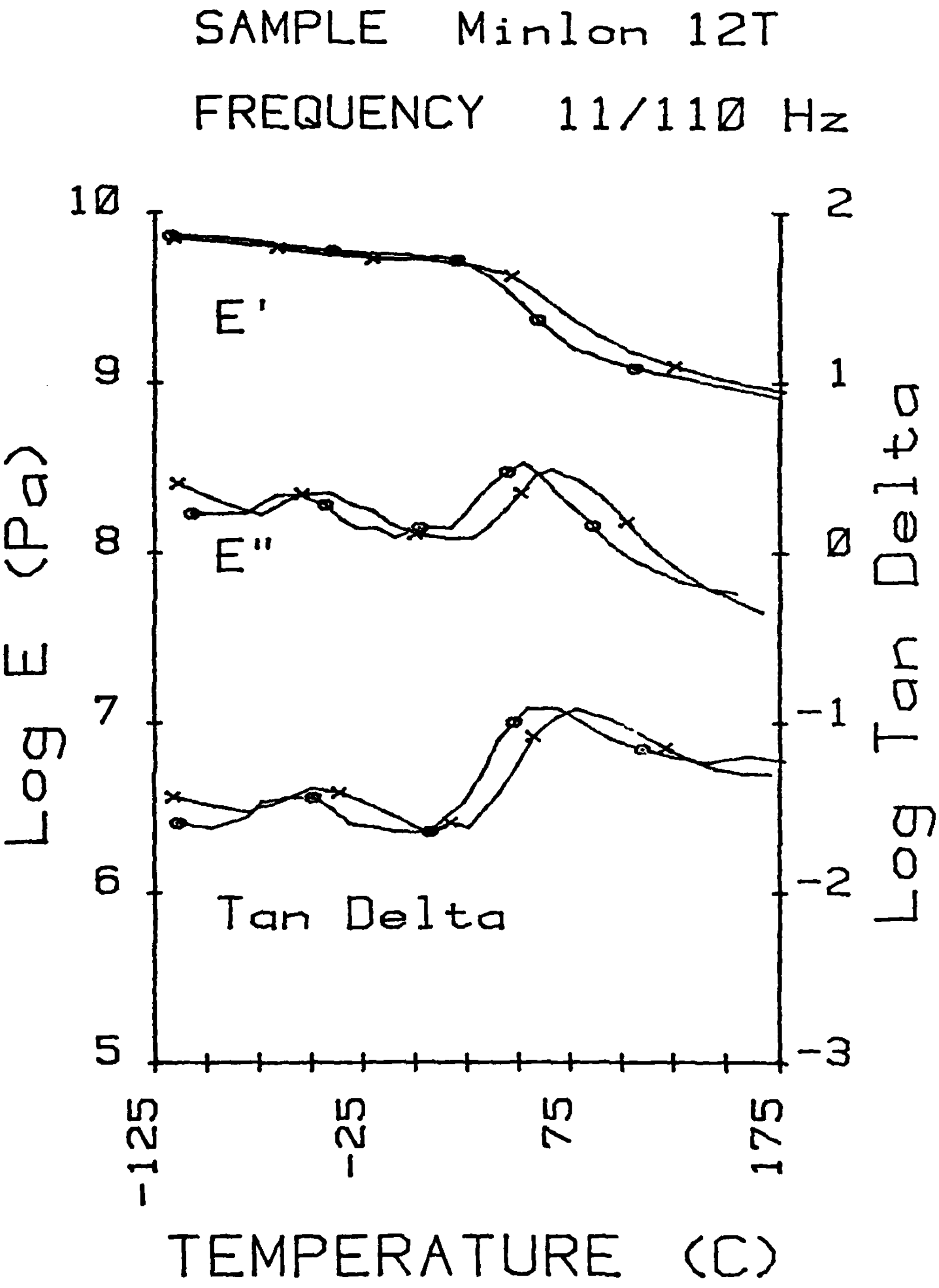

SAMPLE Minlon 12T
FREQUENCY 11/110 Hz
E'
E"
Tan Delta
Log E (Pa)
Log Tan Delta
10
9
8
7
6
5
2
1
0
-1
-2
-3
-125
-25
75
175
TEMPERATURE (C)

Figure. 14

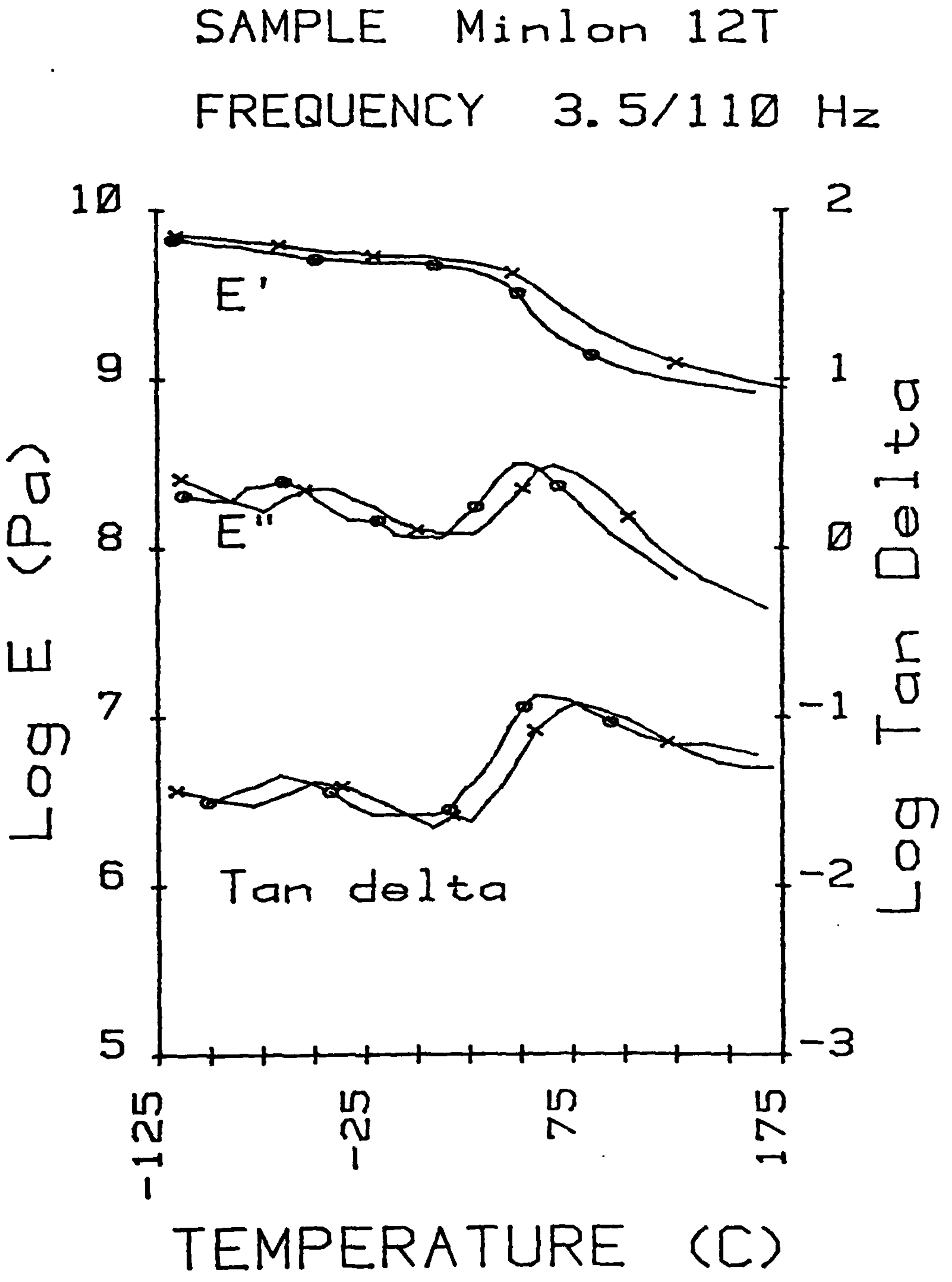

SAMPLE Minlon 12T
FREQUENCY 3.5/110 Hz
Log E (Pa)
10
9
8
7
6
5
Log Tan Delta
2
1
0
-1
-2
-3
E'
E"
Tan delta
-125
-25
75
175
TEMPERATURE (C)

Figure. 15

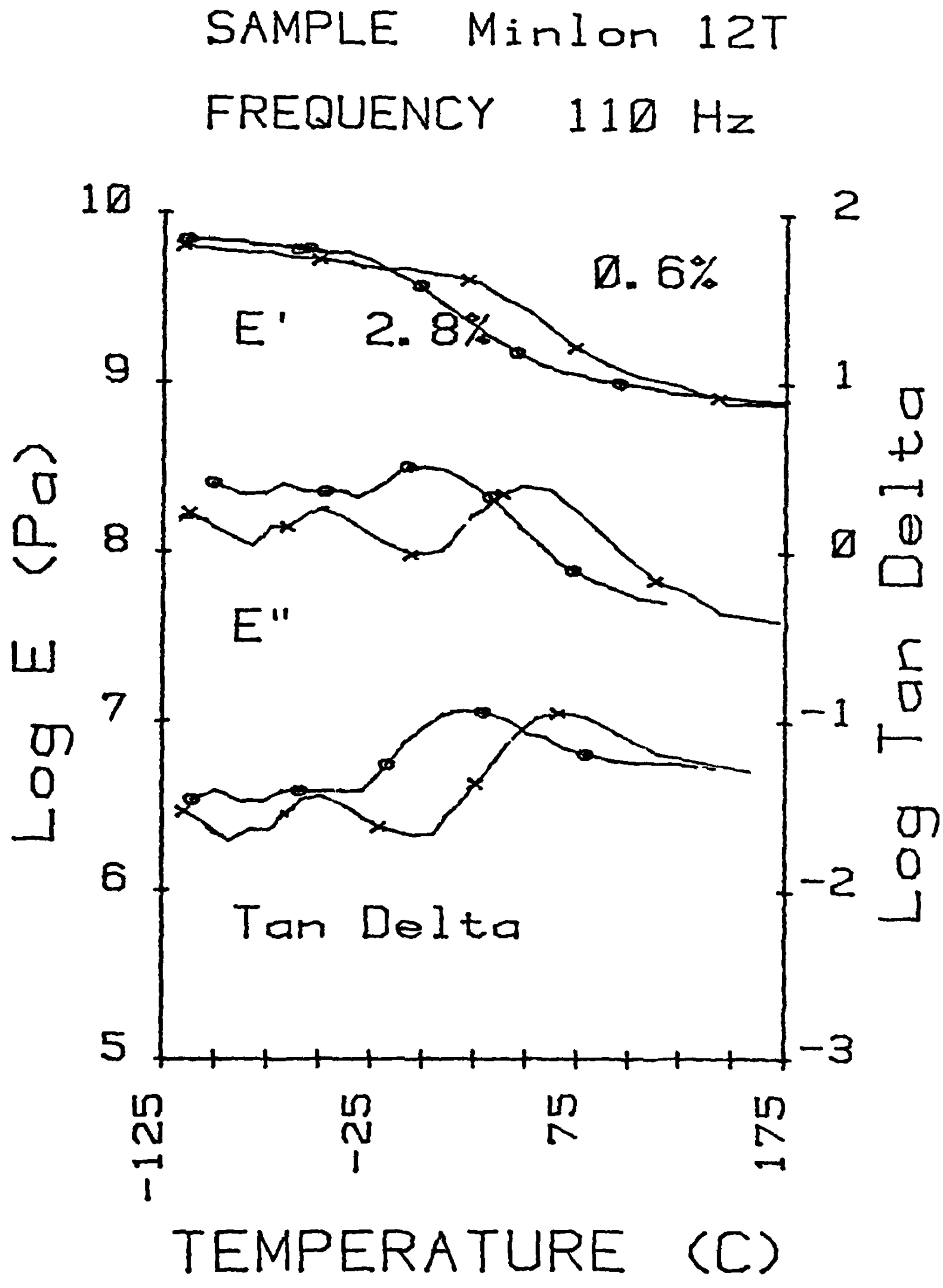
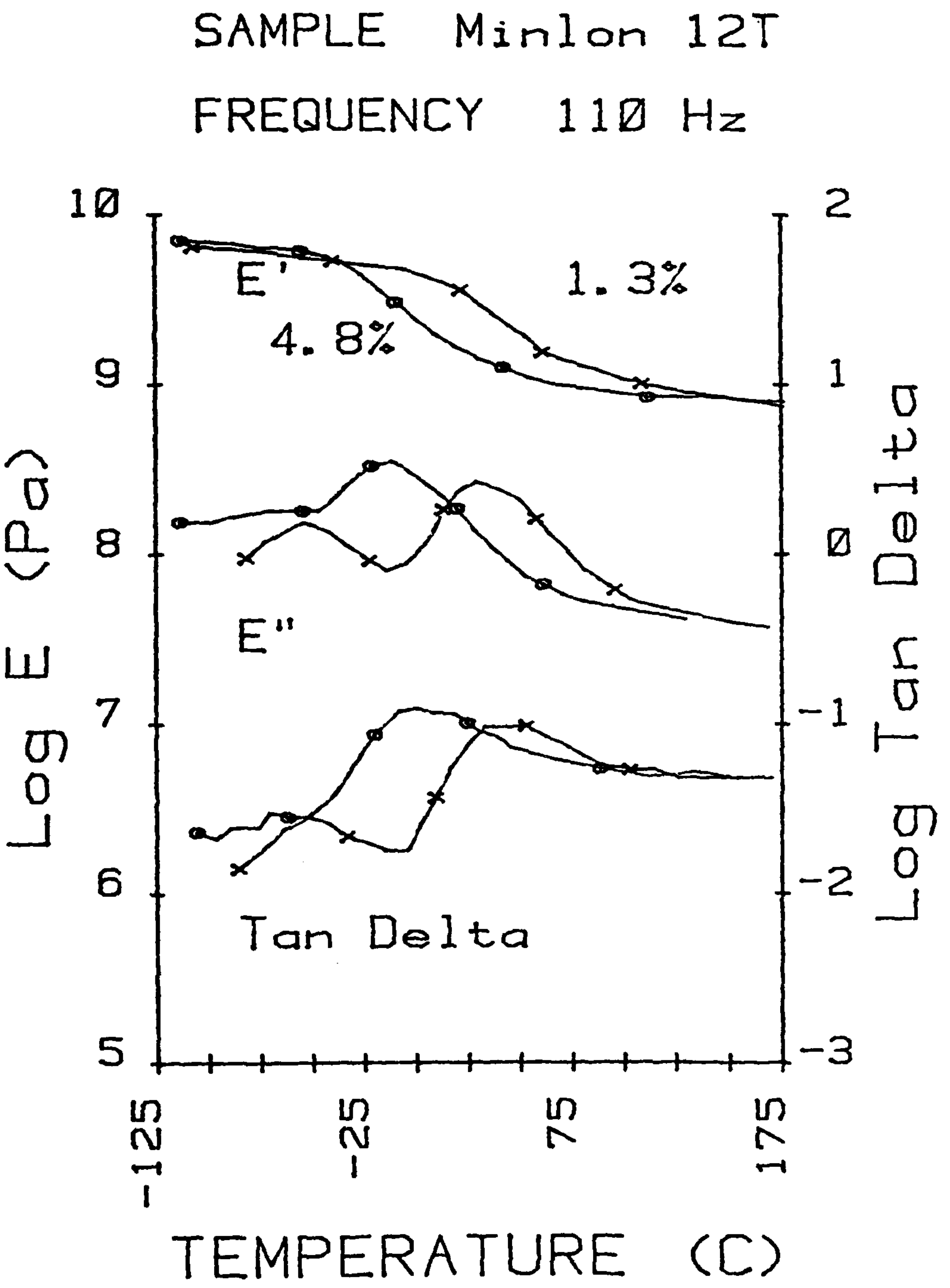

SAMPLE Minlon 12T
FREQUENCY 110 Hz
10
9
8
7
6
5
2
1
0
-1
-2
-3
0.6%
E' 2.8%
E"
Tan Delta
Log E (Pa)
Log Tan Delta
-125
-25
75
175
TEMPERATURE (C)

Figure. 16


SAMPLE Minlon 12T
FREQUENCY 110 Hz
10
9
8
7
6
5
2
1
0
-1
-2
-3
E'
1.3%
4.8%
E"
Tan Delta
Log E (Pa)
Log Tan Delta
-125
-25
75
175
TEMPERATURE (C)

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