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THE VISCOSITY AND DENSITY OF MOLTEN LANTHANUM, CERIUM AND PRASEODYMIUM METALS

by

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The recent appearance of high purity rare earth metals in large quantities has made available a series of elements whose unique, closely related physical properties have been comparatively well documented in the solid state[1,2] in contrast to the dearth of information on their liquid properties. Several general theories have been proposed that furnish means of predicting certain atomic mobility parameters in liquid metals. The theories include simple curve fitting as well as more fundamental considerations of the problem. Since the final arbiter for any theory is how well it is substantiated by experimental data, the lanthanide metals provide uniquely attractive materials for a test of the theories once the experimental measurements are accomplished.

As part of the Reactor Fuels Program at Mound Laboratory, apparatus exist for measuring viscosity coefficients and liquid densities for high melting point, highly reactive metals. The density and viscosity of molten lanthanum, cerium and praseodymium metals are reported from their melting points to 1000°C.

Apparatus

The viscosities of the liquid metals lanthanum, cerium and praseodymium are measured in an oscillating cup viscosimeter by an absolute method. In this apparatus the liquid is sealed in a right-circular cylindrical cup which is attached to a torsion fiber so that the system forms a torsion pendulum (see Figure 1). The damping effect of the molten metal upon the normal oscillations of the torsion pendulum is a function of the viscosity of the liquid. This method is advantageous because of the small amount of sample required (8 to 10 cc), the ease of remote operation of the apparatus in a vacuum system, and the containment of the liquid metal in a

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hermetically sealed, non-reactive, tantalum crucible. With the liquid metal sealed in the crucible, loss of liquid by vaporization and chemical reactions with atmospheric gases are prevented.

The oscillating cup viscosimeter was first described by Meyer in 1891.[3] The first complete mathematical treatment offering an absolute measurement of viscosity with this type of apparatus was proposed by Andrade and Chiong[4] in 1936 for an oscillating sphere filled with a liquid. This treatment was modified to include a right circular cylinder by Hopkins and Toye[5] in 1950, and R. Roscoe[6] in 1958. Roscoe's treatment for the calculation of the viscosity of a liquid at a particular temperature from the logarithmic decrement and period of oscillation of the torsion pendulum was used in this investigation. A detailed description of the method of calculating the viscosity from these measurements has been reported earlier.[7,8]

A pycnometric method developed for the measurement of the liquid density of active metals was described earlier.[9,10] The density apparatus (see Figure 2) consisted of a closed system in which a calibrated tantalum pycnometer (1) was suspended directly above the molten metal (8) in vacuo. The pycnometer became filled when the tip of the pycnometer was lowered into the melt and the system was returned to atmospheric pressure with dry helium. The pycnometer was subsequently withdrawn from the melt and quenched to room temperature. The density of the liquid at the testing temperature was calculated after the weight of the metal filling the known volume of the pycnometer was determined.

Metal Purity

Analyses of the metals used in the viscosity determinations are shown in Table 1. The lanthanum and praseodymium were described as nuclear grade materials by the manufacturer (Lunex Company). The cerium metal used did not have a manufacturer's analysis accompanying it, and was subjected, therefore, to spectrographic and differential thermal analyses. The results of the spectrographic analysis are likewise included in Table 1. Additionally, differential thermal analysis of the gamma to delta and delta to liquid transformations showed the S.E.C. #2 cerium metal to be of adequate purity for the viscosity measurements.[11]

Experimental Procedure

A rod of the metal under investigation was vacuum cast in a fused, high purity magnesia crucible. The crucible was broken away from the rod and the rod was mechanically cleaned to remove the oxide coat resulting from the casting operation. After the clean metal rod was inserted into a 0.585 inch I.D. tantalum cup, a bottom closure plug was inert gas arc welded in place. The capsule was suspended on either a 0.005 inch diameter platinum-tungsten alloy torsion fiber or a 0.010 inch diameter molybdenum torsion fiber resulting in periods of oscillation of 13 or 2.7 seconds, respectively. The system was held at each temperature for at least three hours prior to the viscosity measurement to be sure the sample was at the desired temperature. The period of the oscillating torsion pendulum was determined by a photo-electrically operated clock to 0.001 second. The amplitudes of the oscillations, which were used to calculate the logarithmic decrement, were measured by a photographic technique with a precision of one part in ten thousand. The detailed operation of the apparatus has been described previously.[7]

Density Results

The liquid densities of lanthanum, cerium, and praseodymium were measured at: four different temperatures between 951 and 1004°C, nine different temperatures between 824 and 990°C, and four different temperatures from 937 and 1004°C, respectively. Figure 3 is a composite graph of the results of these three sets of determinations. The lines drawn through the data points represent least squares fits of the data. The equations of the lines representing the change in density as a function of temperature are:

$$\text{La } \rho = (6.24 - 2.37 \times 10^{-4} T) \pm 0.01$$

$$\text{Ce } \rho = (6.94 - 2.37 \times 10^{-4} T) \pm 0.02$$

$$\text{Pr } \rho = (6.91 - 2.48 \times 10^{-4} T) \pm 0.01$$

where: ρ = Density in g/cc

T = Temperature in °K.

The thermal coefficients of cubical expansion for these three liquids are very small for liquid metals and only slightly larger than the coefficients of cubical expansion of their respective solid phases.

During the measurement of the density of cerium, an indication was obtained that cerium expanded when it changed from liquid to solid at the melting point since two of the pycnometers filled at temperatures near the melting point exhibited extrusions of cerium metal from their orifices. Similar extrusions were noted for the metals bismuth and plutonium[9] both of which expanded during solidification. Although the density of solid cerium has never been measured at the melting point, a prediction of the volume change during melting was made when the density of the liquid at the melting point, 6.68 g/cc, obtained from Figure 3, was compared with an estimated value for the solid. The estimate for the solid density was made when the density of delta (BCC) cerium at the melting point was calculated from crystallographic data and an assumed coefficient of cubical expansion, 20.4 cc/cc/°C obtained from lower temperature measurements of Trombe and Foex.[12] This calculation showed that 0.6 per cent expansion occurred during solidification. Interestingly, both cerium and plutonium, which exhibit volume expansion during solidification, have a body-centered cubic high temperature structure and a lower temperature face-centered cubic structure.

The liquid densities of lanthanum and cerium have been estimated earlier by Pulliam and Fitzsimmons of Ames[13] from the shape of a drop on various ceramic substrate surfaces. The densities measured by both methods show good agreement near the melting points (see Figures 4 and 5 wherein the lines representing the present work are compared with the points measured by Pulliam and Fitzsimmons). The Ames data, however, exhibit much greater changes with temperature than do the present work so that agreement between the two experimental results decreases rapidly with temperature.

The more rapid change in density as a function of temperature for lanthanum and cerium shown by the data of Pulliam and Fitzsimmons is questionable for two reasons. Firstly, the experimental difficulties associated with the

drop shape method become more pronounced as temperature increases since reactions between the liquid metal and ceramic substrate accelerate with temperature. Secondly, two metals having such a long liquid range (as indicated by their low vapor pressures[14,15]) would not be expected to exhibit such a rapid change in density with temperature.

A means of theoretically predicting the change in density of a liquid metal with temperature based on the law of corresponding states has been proposed by A. V. Grosse.[16] Experimental liquid density data for 27 liquid metals exhibit the change with temperature predicted by this application of the law of rectilinear diameters.[17] Good agreement is shown in the comparisons between the theoretical (designated Grosse) and experimental (designated Mound) slopes of the density versus temperature curves for cerium, lanthanum and praseodymium (Figures 4 and 5). These data lend support to the use of this method for the prediction of liquid density of other metals which, for one reason or another, have not been experimentally measured.

Pulliam and Fitzsimmons also measured the surface tension of lanthanum and cerium in their drop shape experiments. Recognizing the sources of error inherent in the density measurements by the drop shape method, they reported sufficient experimental data so that the reported surface tension data could be recalculated at a later date when better liquid density information were available. This commendable foresight made it possible to make the calculations of surface tension as shown in Table 2. Here again, since experimental errors loom larger with increasing temperature, the data near the melting points are probably the most accurate of the reported information.

Viscosity Results

The viscosities of molten lanthanum measured at 13 different temperatures between 931 and 1006°C, the viscosities of liquid cerium measured at 16 temperatures between 812 and 1011°C, and the viscosities of molten praseodymium metal measured at 15 temperatures between 936 and 1009°C are listed in Table 3 and plotted in Figures 6, 7, and 8, respectively. The viscosities of lanthanum, cerium and praseodymium are plotted as the logarithm η/T versus

1/T instead of the more conventional Log η versus 1/T since Saxton and Sherby[18] recently showed that the change in viscosity with temperature exhibits a first order dependency when Log η/T versus 1/T is used. This proposal is substantiated by earlier work done at Mound Laboratory on molten bismuth, lead and zinc metals,[8] and on liquid plutonium and plutonium alloys.[19] The straight lines drawn through the data points in Figures 6, 7, and 8 represent least squares fits of the data and have the following equations:

$$\text{La Log } (\eta/T \times 10^5) = 0.84828 \left(\frac{1000}{T}\right) - 0.39562$$

$$\text{Ce Log } (\eta/T \times 10^5) = 0.88379 \left(\frac{1000}{T}\right) - 0.39272$$

$$\text{Pr Log } (\eta/T \times 10^5) = 0.79714 \left(\frac{1000}{T}\right) - 0.29517$$

where η = Viscosity Coefficient (poise)

T = Temperature (°K).

The viscosities of lanthanum, cerium and praseodymium metals at various temperatures as calculated from these straight lines are reported in Table 4 together with the standard deviations.

Discussion

The change in viscosity with temperature has often been expressed as:[20,21]

$$\eta = A \exp \frac{E}{RT}$$

where: A = a constant

E = Activation energy for viscous flow (cal/g-atom)

R = Gas Constant.

A. V. Grosse has proposed an empirical relationship[22] between the activation energy for viscous flow (E in Equation 1) and melting point ($T_{m.p.}$). In this proposal Log E plotted as a function of Log $T_{m.p.}$ for 17 metals results in a smooth curve. Accordingly, once the melting point is known, the

temperature dependence of the viscosity coefficient of a liquid metal could, theoretically, be predicted.

Andrade's relationship[23] for the prediction of the viscosity of a metal at its melting point which has proved remarkably accurate[24] for many liquid metals has been used for the calculation of the viscosity of molten lanthanum cerium and praseodymium metals at their melting points with the good agreement shown in Table 5. When Andrade's predicted $\eta_{m.p.}$ is combined with the E anticipated by Grosse's curve, equations expressing the change in viscosity with temperature can be determined. A comparison of the theoretical and experimental equations are shown below:

	Theoretical Grosse[22] and Andrade[23]	Experimental
La	$\eta = 5.714 \times 10^{-3} \exp \frac{3850}{RT}$	$\eta = 1.464 \times 10^{-2} \exp \frac{1231}{RT}$
Ce	$\eta = 5.560 \times 10^{-3} \exp \frac{3550}{RT}$	$\eta = 1.310 \times 10^{-2} \exp \frac{1679}{RT}$
Pr	$\eta = 6.243 \times 10^{-3} \exp \frac{3800}{RT}$	$\eta = 1.758 \times 10^{-2} \exp \frac{1118}{RT}$

A comparison of the two sets of equations indicates that the predicted value for E is about two to three times larger than the experimental values for lanthanum, cerium and praseodymium.

The coefficient for self diffusion in a liquid metal changes with temperature according to the relationship:[25]

$$D_L = D_0 \exp - \frac{Q}{RT} \qquad (2)$$

where: D_L = Self diffusion coefficient (cm^2/sec)

D_0 = A constant

Q = Activation energy for self diffusion (cal/g-atom).

Saxton and Sherby[18] have shown that self diffusion and viscosity in liquid metals exhibit an interdependence best approximated by the Sutherland[26] relationship:

$$D_L = \frac{k}{(\eta/T)(di)} \qquad (3)$$

where: di = Pauling univalent ionic diameter

k = A constant (2×10^{-17} ergs/°K).

A comparison of experimental self diffusion data with calculated self diffusion values (from viscosity data) for molten lead and zinc[8] showed good agreement using this relationship. Saxton and Sherby proposed[18] further, a method for predicting self diffusion in liquid metals. The atomic mobility of a liquid metal was assumed to be a function of previous crystal history, melting point and atomic mass.

Six metals were considered for comparison between the theoretical predictions for the self diffusion equations and the equations calculated from experimental viscosity data using the Sutherland relationship (equation 3). The calculated self diffusion data (from viscosity data) were plotted as Log D versus 1/T. The equations for these straight lines (obtained by a least squares fit of the data) are compared in Table 6 with the theoretically predicted equations. The good agreement for bismuth, lead and zinc between the two sets of equations contrasts with the lack of agreement exhibited by lanthanum, cerium and praseodymium.

Both the theories by Grosse, and Saxton and Sherby predict much higher activation energies [E in equation (1) and Q in equation (2)] for the atomic mobility parameters of viscosity and liquid self diffusion for lanthanum, cerium and praseodymium than were observed experimentally. One shortcoming of the experimental data is that the temperature ranges of the present investigations are small. Nevertheless, since the viscosities of these molten metals at their melting points are low (approximately three centipoise) and all three have long liquid ranges as shown by their low vapor pressures, high activation energies for the atomic mobility parameters would not be anticipated. The vapor pressure term may well be a factor for inclusion in an empirical expression for predicting these activation energies.

In conclusion, the measurements of the liquid densities and viscosities of the first three lanthanide metals have provided a unique opportunity for a test of some of the predicted properties of these liquids. The low viscosities, moderate densities and long liquid range of these metals make them candidate liquids for liquid flow studies at very high temperatures. The completion of these liquid properties measurements for the remaining lanthanide metals should be an interesting and challenging study.

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Table 1

SPECTOGRAPHIC ANALYSIS

Element	La[a] (ppm)	Ce[b] (ppm)	Pr[a] (ppm)
Silicon	ND	<100	—
Iron	ND	200	ND
Manganese	ND	ND	—
Magnesium	10	500	—
Aluminum	ND	—	20
Tantalum	ND	300	20
Lead	ND	ND	ND
Nickel	ND	100	—
Copper	20	<100	100
Calcium	—	500	—
Chromium	—	<100	—
Vanadium	ND	—	10
Beryllium	ND	—	20
Boron	—	100	—
Niobium	ND	—	—
Sodium	ND	—	ND
Lithium	ND	—	ND
Barium	ND	—	ND
Potassium	ND	—	—
Titanium	10	100	ND
Cobalt	ND	—	ND
Zinc	ND	—	ND
Tin	—	—	—
Zirconium	10	—	ND
Cadmium	—	—	—
O_2	100 - 500	1700	100 - 500
N_2	0 - 10	1400	0 - 10

ND - Not Detectable

(a) Manufacturer's Analysis - Lunex Company

(b) Cerium SEC #2 - Mound Analysis

Table 2

COMPARISON OF LIQUID DENSITIES
AS DETERMINED BY THE PYCNOMETRIC AND DROP SHAPE METHODS

(Recalculation of Pulliam and Fitzsimmons[13] Surface Tension Data Using Mound Densities)

Temperature	Lanthanum				Cerium			
	Density (g/cc)		Surface Tension (dyne/cm)		Density (g/cc)		Surface Tension (dyne/cm)	
(°C)	Ames	Mound	Ames	Recalculated	Ames	Mound	Ames	Recalculated
810	———	———	—	—	6.696	6.680	695	701
900	———	———	—	—	6.612	6.659	680	686
950	5.876	5.950	710	719	———	———	—	—
1000	5.776	5.929	693	713	6.250	6.632	666	708
1100	5.630	5.910	648	681	———	———	—	—
1200	5.486	5.888	630	664	———	———	—	—

Table 3

MEASURED VISCOSITY COEFFICIENTS

Lanthanum		Cerium		Praseodymium	
Temperature °C	Viscosity Centipoise	Temperature °C	Viscosity Centipoise	Temperature °C	Viscosity Centipoise
1006	2.606	1011	2.827	1009	3.02
998	2.540	1004	2.542	1007	2.41
994	1.829	1001	2.478	1004	2.69
994	2.065	966	2.655	995	2.96
990	2.649	965	2.201	994	2.43
966	2.808	954	2.752	989	2.83
961	2.215	909	2.183	969	2.78
960	2.759	903	2.393	962	2.79
959	2.831	902	3.029	962	2.49
949	2.103	900	2.764	961	3.16
944	2.068	858	3.275	956	2.37
942	2.879	858	3.339	955	3.03
931	2.272	853	2.944	952	3.25
		853	2.616	944	2.79
		822	2.526	936	2.52
		812	2.707		

Table 4

SUMMARY OF CALCULATED[a] VISCOSITY DATA

Lanthanum		Cerium		Praseodymium	
Temperature °C	Viscosity Centipoise ± 0.341[b]	Temperature °C	Viscosity Centipoise ± 0.304[b]	Temperature °C	Viscosity Centipoise ± 0.270[b]
1006	2.369	1011	2.536	1009	2.719
990	2.384	965	2.594	995	2.730
960	2.417	900	2.692	955	2.774
942	2.438	853	2.778	944	2.787
931	2.451	812	2.865	936	2.795
930	2.452	804	2.882	935	2.796

(a) Viscosities are calculated from a line describing the least squares fit of the experimental data on a plot of Log η/T versus $1/T$.

(b) Obtained by standard deviation analyses of experimental data.

Table 5

PHYSICAL PROPERTIES OF MOLTEN LANTHANUM, CERIUM AND PRASEODYMIUM AT THEIR MELTING POINTS

	Melting Point (°C)	Density (g/cc)	Viscosity (Centipoise)	Self Diffusion ($cm^2/sec \times 10^5$)	Andrade's[22] Viscosity (Centipoise)
Lanthanum	930	5.95	2.45	3.53	2.86
Cerium	804	6.68	2.89	2.97	2.91
Praseodymium	935	6.61	2.80	3.66	3.04

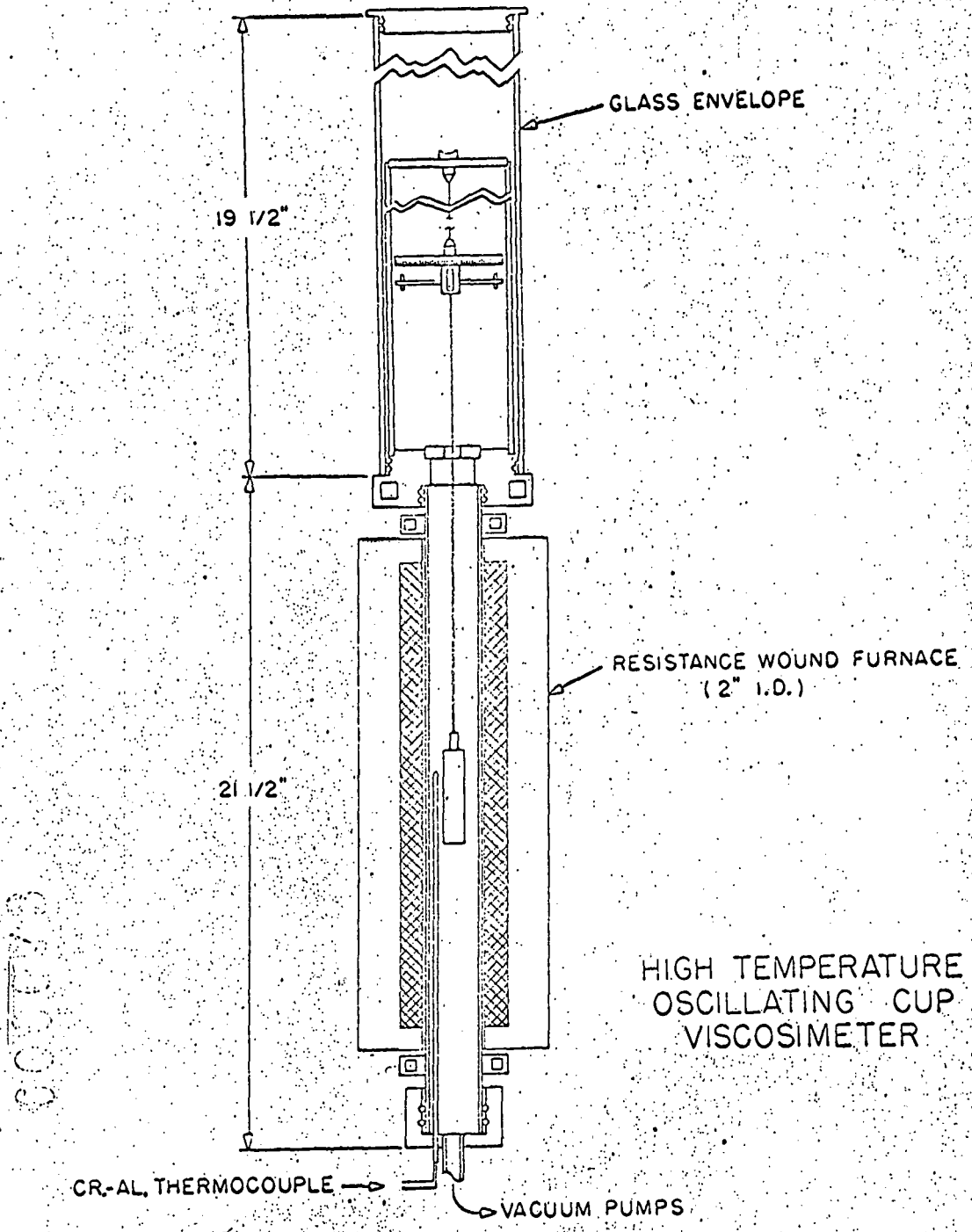
Table 6

EXPERIMENTAL (FROM VISCOSITY DATA) SELF DIFFUSION EXPRESSIONS COMPARED WITH THEORETICAL PREDICTIONS

Metal	Pauling Univalent Ionic Diameter cm x 10^8	Solid Crystal Structure	Self Diffusion Expression, D_L Experimental[a]	Self Diffusion Expression, D_L Theoretical[b]
Bi	1.96	Rhombohedral	$4.881 \times 10^{-4} \exp \frac{-3030}{RT}$	$9.34 \times 10^{-4} \exp \frac{-2974}{RT}$
Pb	2.12	FCC	$4.34 \times 10^{-4} \exp \frac{-3594}{RT}$	$2.43 \times 10^{-4} \exp \frac{-3579}{RT}$
Zn	1.76	HCP	$6.53 \times 10^{-4} \exp \frac{-4740}{RT}$	$4.33 \times 10^{-4} \exp \frac{-4128}{RT}$
La	2.78	BCC	$1.789 \times 10^{-4} \exp \frac{-3880}{RT}$	$5.94 \times 10^{-4} \exp \frac{-7171}{RT}$
Ce	2.54	BCC	$1.945 \times 10^{-4} \exp \frac{-4044}{RT}$	$5.91 \times 10^{-4} \exp \frac{-6420}{RT}$
Pr	2.36	BCC	$1.673 \times 10^{-4} \exp \frac{-3648}{RT}$	$5.90 \times 10^{-4} \exp \frac{-7201}{RT}$

(a) From Viscosity Data

(b) Saxton and Sherby[17] Theoretical Predictions



HIGH TEMPERATURE
OSCILLATING CUP
VISCOSIMETER

Figure 1

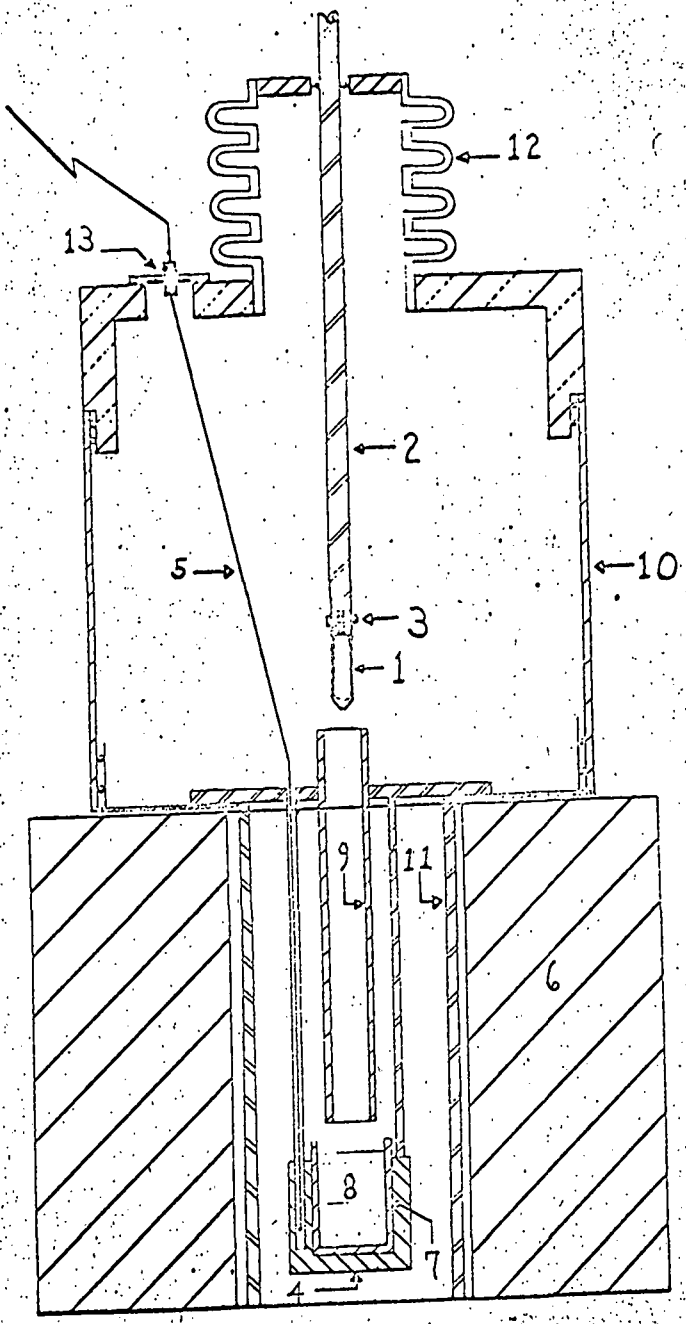


Figure 2

High-Temperature Density Apparatus

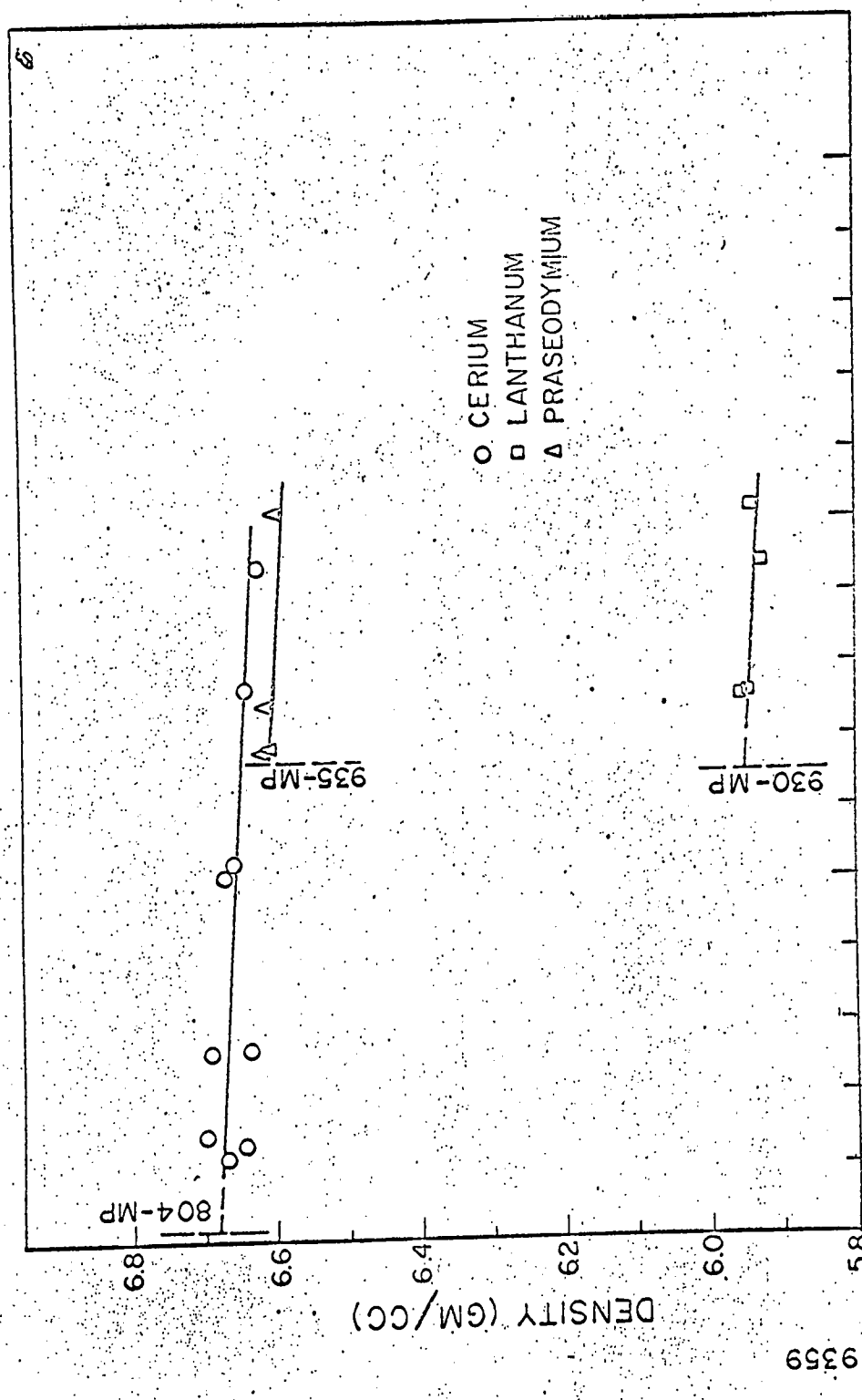


Figure 3
Density as a Function of Temperature (Lines represent least squares fit of data)

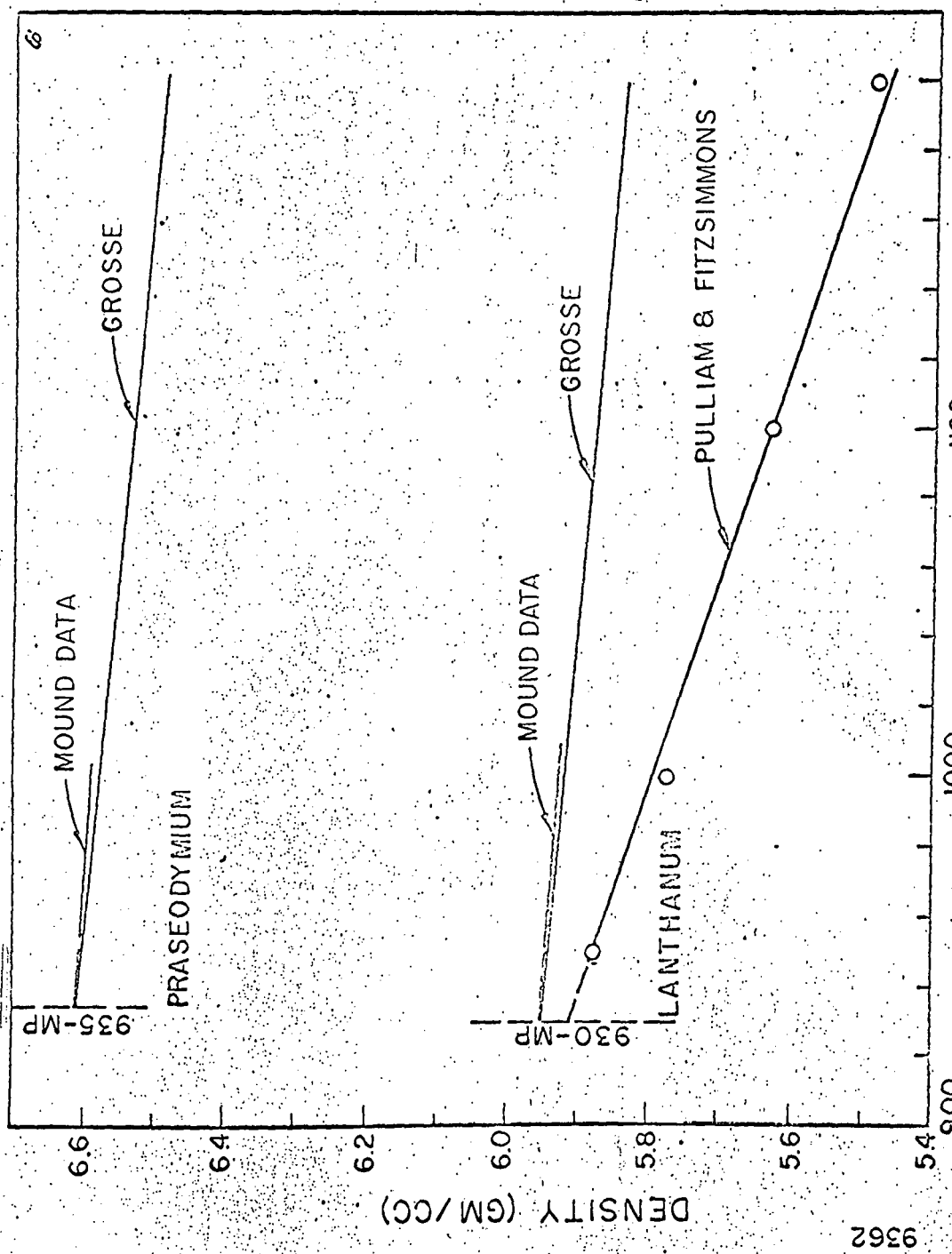


Figure 4
Comparison of Experimental and Theoretical Densities
of Praseodymium and Lanthanum

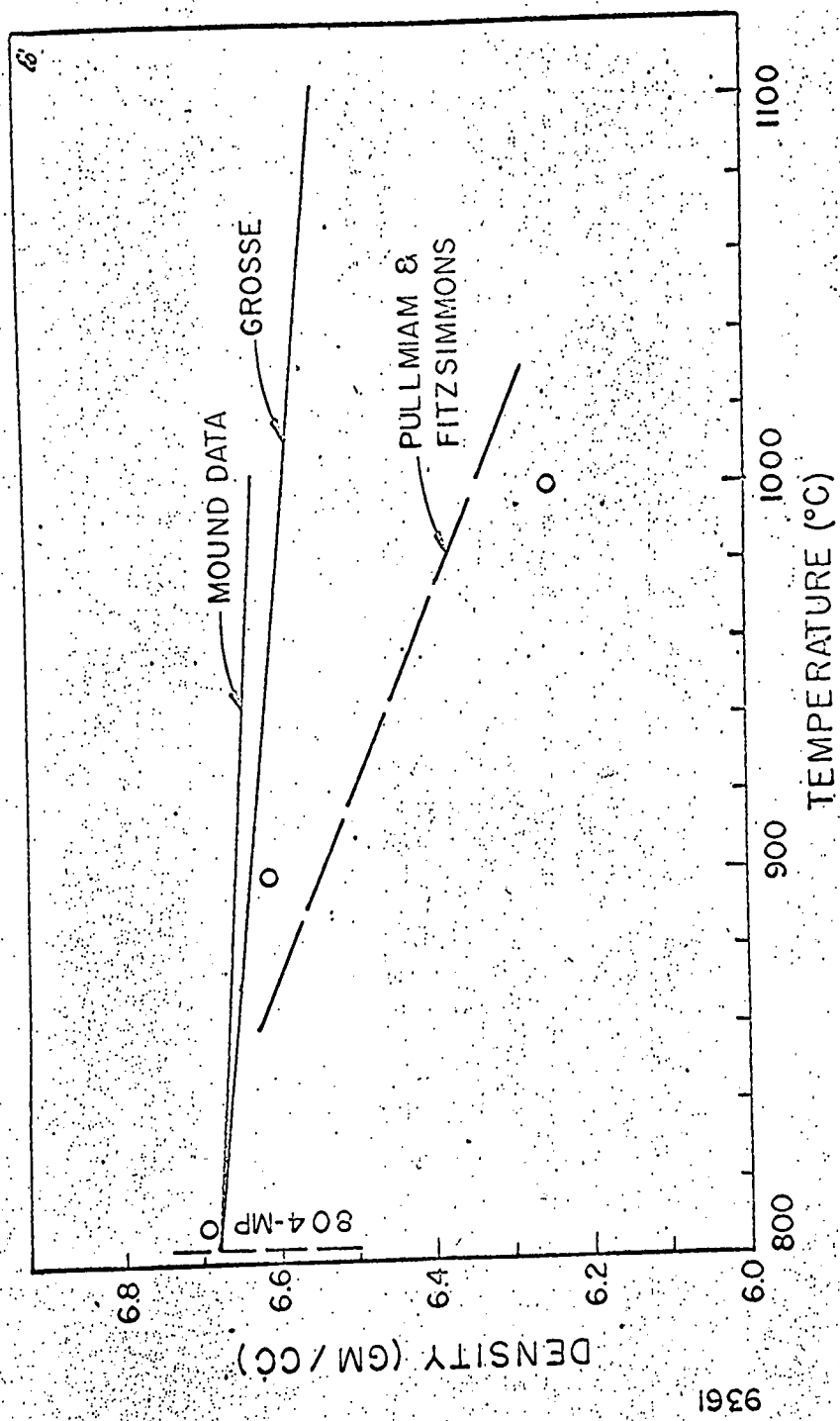


Figure 5
Comparison of Experimental and Theoretical Density of Cerium

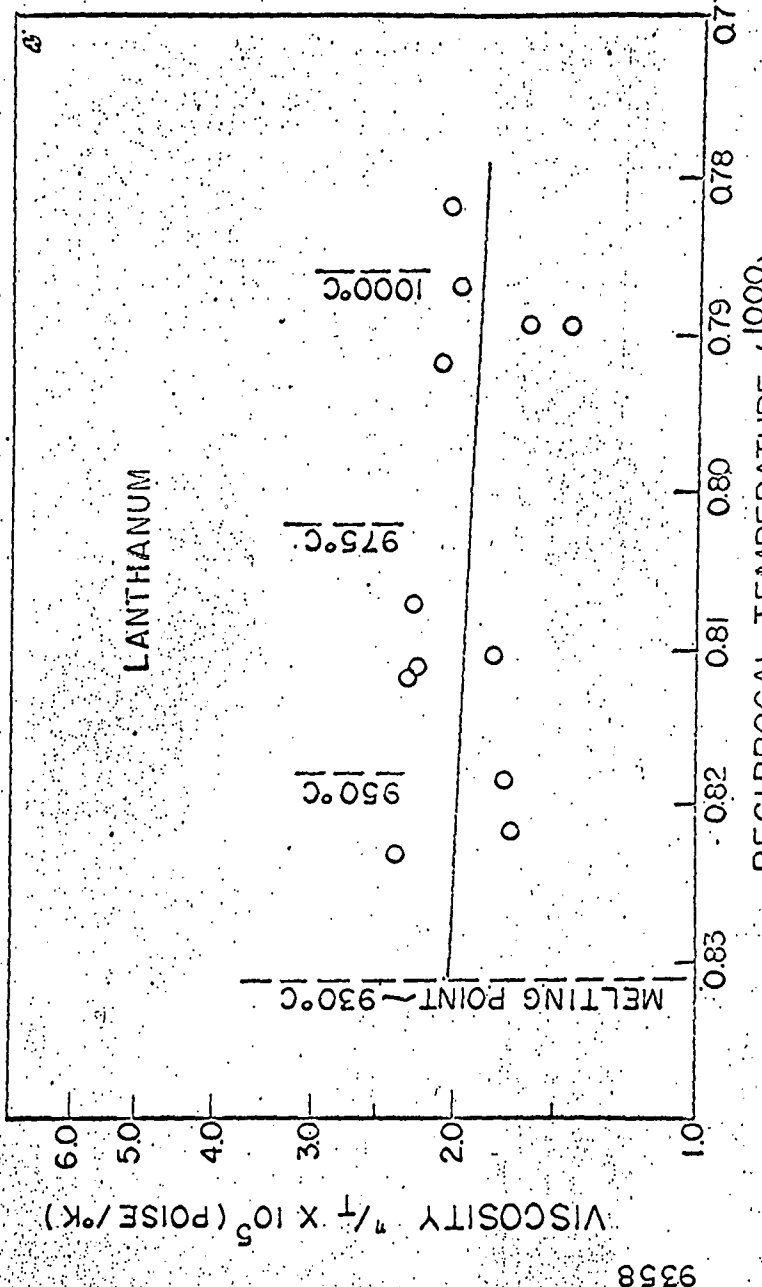


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Figure 6
Viscosity (Expressed as Log η/T) of Lanthanum as a Function of Absolute Temperature (Lines represent least squares fit of data)

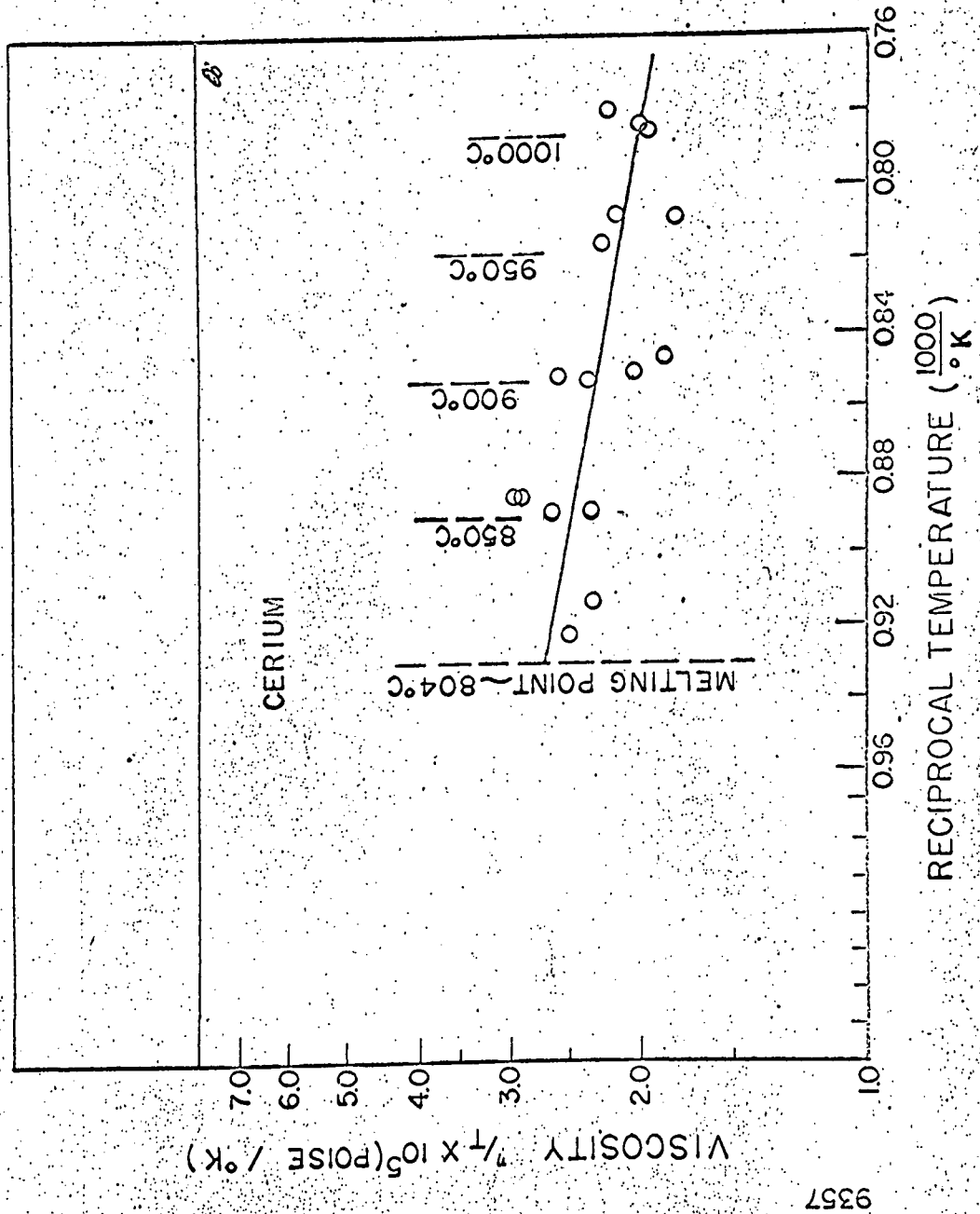

Figure 7
Viscosity (Expressed as Log η/T) of Cerium as a Function of Absolute Temperature (lines represent least squares fit of data)

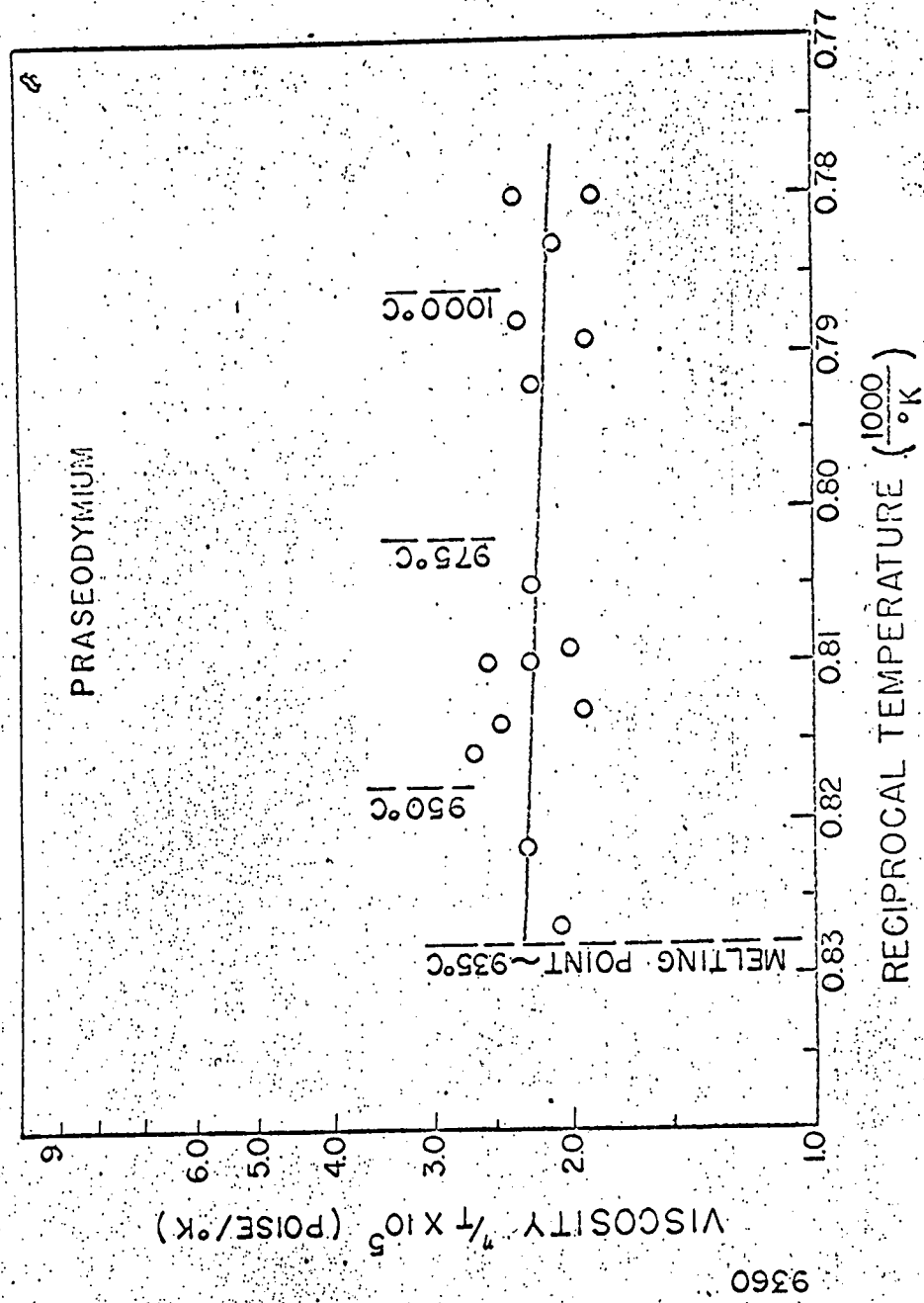

Figure 8
Viscosity (Expressed as Log η/T) of Praseodymium as a Function of Absolute Temperature (Lines represent least squares fit of data)