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PRESSURE AND ELECTRONIC STRUCTURE*

by

H. G. Drickamer

Department of Chemistry and Chemical Engineering

and

Materials Research Laboratory

University of Illinois

Urbana, Illinois

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Abstract

Pressure is a variable which permits the change of interatomic distance at constant temperature. It is therefore of prime importance in obtaining an understanding of the electronic structure of solids. Three examples of its use are discussed in this paper: (1) The elucidation of the mechanism of light absorption in alkali halide phosphors, (2) The approach to the metallic state in insulators and semiconductors, and (3) Electronic transitions in metals involving the promotion of electrons between bands and the modification of the chemical valence of elements.

Two of the basic properties which help determine the electronic structure of solids are the arrangement of the atoms (or ions) and the equilibrium interatomic distance. Pressure is a variable of prime importance for determining both these parameters. High pressure experimentation is, then, significant because it permits one to test theories where the variation of a property with interatomic distance is critical and because, on occasion, new polymorphic forms appear which then permit the study of the relationship between atomic arrangement and electronic properties.

The range of pressure to be used depends on the type of measurement to be made, the degree of accuracy to which the data (and the pressure) must be known, and the degree of hydrostaticity required. The recent book "Solids Under Pressure" (1) gives a general review of experiments in the field. Techniques are discussed in a review article by Swenson (2) and in the book by Wentorf (3).

In this paper we shall review some exploratory experiments on optical absorption and electrical resistance at relatively high pressures, the optical work to 160 kilobars (one kilobar = 0.986 atmospheres), the electrical measurements to over 500 kilobars.

The pressures, especially in the electrical work, are rather imprecisely known. A complete understanding of the electronic structure would involve a much wider variety of experiments. In particular, accurate knowledge of the interatomic distance as a function of pressure, and of the structure and lattice parameters of the new phases which appear at high pressure would be desirable. Nevertheless these experiments have contributed to the understanding of a variety of problems and have unearthed a large number of new and interesting phenomena. From these we select a few illustrations which give some notion of the power and versatility of the techniques.

The energy states of an electron on a free atom or ion can be described in terms of its four quantum numbers. The lowest of these states is the ground state. These states are determined experimentally either by measuring the absorption of (usually electromagnetic) energy or by the emission of energy as an excited electron returns to the ground state. In this discussion we shall be concerned only with outer electrons generally called valence electrons because they are involved when chemical reaction takes place. Only the ground state and the excited states which be nearest above them will be important in the phenomena included here.

When an atom or ion appears in a crystal the electronic energy states are affected to a degree which depends on the interaction between the wave functions which describe the state in question with the wave functions of the other electrons in the crystal.

Both the ground state and the excited state may remain closely associated to the original atom. In this case, the energy states may be perturbed by the potential of the surrounding atoms and affected because this potential has less than spherical symmetry. The optical absorption which represents transitions from the ground state to the first excited state still gives discrete peaks, perhaps somewhat broadened by the interaction described above. In some cases, a single peak in a free ion may be split into two or more peaks in the crystal. This sort of effect can be observed in transition metal ions in crystals, or in the spectra of some crystals of fused ring aromatic compounds. An impurity in an insulating crystal may have highly localized states. The phosphor made by dissolving Tl^+ ion in alkali halides is an example. Since in both the ground state and excited state the electron is highly localized, one obtains no electrical conductivity in such systems.

As the wave function becomes a little more delocalized it is no longer practical to think of slightly perturbed atomic levels. The solution of the Schroedinger equation in the periodic potential of the lattice is known as a Bloch function. It is of the form

$$\psi = u(r) \exp (i \vec{k} \cdot \vec{r})$$

where $u(r)$ has the symmetry of the lattice and the exponential represents a wave motion. $\vec{k}$ is a reciprocal lattice vector which is called the crystal momentum since $\hbar \vec{k}$ represents the momentum of the wave. This solution of the wave equation gives rise to bands of closely spaced allowed states separated by relatively large energy gaps where, in a perfect crystal, there are no allowed states. Each of the states in the band can accommodate two electrons of opposite spin.

If one now considers a crystal made of atoms or ions having filled electronic shells (e.g. Na^+ and Cl^- or I_2), each state in the band is filled. Since the next available level is far out of reach, when an electrical potential is applied there are no available empty states and therefore there can be no net displacement of electrons. Thus one has an electrical insulator. One can get significant conductivity by supplying energy to excite electrons to the next empty (conduction) band, or by adding impurities which will supply conduction electrons or trap electrons from the valence band, leaving it a partially filled band. In the absence of these effects the only conducting electrons will be those which can escape by fluctuations in thermal energy. It can be shown that the number of these is proportional to $\exp (\frac{\Delta E}{2kT})$ where

ΔE is the energy difference between the top of the valence band and the bottom of the conduction band. Thus the temperature coefficient of electrical conductivity gives a measure of this energy gap. As mentioned above, it is possible to excite electrons across the gap by absorption of electromagnetic radiation of appropriate energy. By measuring the wavelength at which the very sharp increase in absorption due to this allowed transition begins, one has a second measure of the energy gap. In many practical cases there are complications in these measurements, due to impurities, due to complex band structure, and due to bound excited states below the conduction band, but the general picture still has definite experimental validity.

At room temperature these gaps can be as large as 5 to 6 electron volts (one electron volt equals 23 kilocalories) as in NaCl or in diamond. On the other hand, the gap can be vanishingly small. When the gap is small enough so that the resistivity is of the order of 10^4 ohm cm or less, the material is called a semiconductor. Both insulators and semiconductors are characterized by an exponential decrease in resistivity with increasing temperature (provided impurity concentrations are not too high).

Let us return now to our picture of the energy levels in the valence band. Consider a crystal made from atoms which do not have filled shells. (Typical examples would be copper, silver, gold, or the alkali metals.) The valence band would, in these cases, be only half full. Since there are now states to which an electron can easily move, an electrical potential results in a flow of electrons, and one thus obtains a typical meta. The electrons can no longer be associated at all with individual atoms but belong to the high molecule which is the piece of metal, much as the so called "pi electrons" in benzene belong to the molecule as a whole. For the noble metals the electrons have almost the characteristics of free electrons. It can be shown that a perfectly periodic array of atoms would offer no resistance to electronic motion. In a real lattice the atoms are vibrating about an equilibrium position and these displacements tend to scatter electrons. Under these circumstances, the resistivity should increase linearly with temperature, and this actually obtains for many metals. Impurities give additional scattering, and complications in band structure can grossly modify this "almost free electron" picture. Even the sign of the temperature coefficient of resistance may be changed. One can speak of "typically metallic behavior", but one cannot simply characterize all metals from their resistivity behavior.

Finally, it is not necessary that a gap exist between the highest filled band and the next empty band at the equilibrium interatomic distance. These two bands may overlap, and incur typically metallic behavior for a solid of closed shell atoms. The crystals of the alkaline earths provide an example of such behavior.

In general, the effect of pressure is to reduce the interatomic distance. In a pressure range of several hundred kilobars, salts such as the alkali halides may decrease in volume as much as 40-50%, while even such metals as silver compress by 20% or more, so the changes in interatomic distance are significant.

In the case of internal transitions, the effect of increasing pressure is to increase the perturbing effect of the field provided by the lattice. Where a theory predicts the effect of the field, this can provide a quantitative test of the theory. Where two theories predict different effects of increasing field intensity, pressure experiments provide a choice between theories.

For typical semiconductors of simple hand structure the effect of pressure should be to reduce, and ultimately to eliminate the gap between the conduction and valence band, converting an insulator or semiconductor into a metal. This may take place with no change in crystal structure or through a polymorphic transition to a new phase, in which case the change in gap is discontinuous. For semiconductors of complex band structure, such as germanium or gallium arsenide, the changes in gap with pressure can help elucidate the structure.

In the simplest picture, the effect of pressure on the resistance of a metal is straightforward to analyze qualitatively. The decrease in interatomic distance should decrease the amplitude of vibration and "stiffen" the structure. Thus increasing the pressure acts in the opposite direction from increasing temperature, and should result in a decrease in resistivity. This indeed occurs for such metals as gold, silver, copper, and many others. There exist a significant number of cases where there are more complex,and more interesting, effects of interatomic distance on band structure. Here pressure experiments can add much to our understanding. In addition, there exist possibilities of polymorphic transitions with a variety of possible effects on electronic structure.

Alkali Halide Phosphors

One of the most useful and most thoroughly studied types of phosphors consists of an alkali halide crystal with a fraction of a percent Tl^+ ion dissolved in it. The Tl^+ ion substitutes for an alkali ion at a lattice site. There is an optical absorption band in the near ultraviolet and an emission peak in the visible. We shall be here concerned with the nature of the absorption band.

The two extreme viewpoints concerning this absorption are: (1) that it is an electron transfer or sharing between the Tl^+ and neighboring halide ions, and (2) that it is an internal transition localized on the Tl^+ ion and perturbed by the crystal field imposed by its nearest neighbors. Actually, there are limitations on either viewpoint as discussed by Knox and Dexter (4).

One can say that if the first viewpoint were essentially correct, the nature of the state, and the shift of the absorption peak with pressure should depend strongly on the halide ion (Cl^-, Br^- or I^-) and only in second order on the symmetry of the lattice. On the other hand, the second approach would predict a pressure shift which would be essentially independent of the halide ion, but might depend strongly on the lattice involved. Williams and his co-workers (5) have developed a semi-quantitative formulation of the second viewpoint as applied to KCl in particular. To summarize his results, the theory predicts a shift to lower energies with increasing pressure. This should be true for all alkali halides with the face centered cubic structure, but not necessarily for the simple cubic (cesium chloride) structure. Figure 1 (6) shows the low pressure shift for a series of these compounds. Energy shifts are shown in wave numbers (1000 cm^{-1} equal 2.88 kilocalories). Indeed, it can be seen that the predicted red shift prevails for all the fcc structures quite independent of the halide ion involved. Williams calculations tell nothing explicit about the simple cubic structure, but one can see from the initial blue shift that there is a marked effect of crystal symmetry. KCl has a transition at 19 kilobars to the simple cubic structure. Based on the low pressure results and Williams theory one would expect a discontinuous shift to higher energy for the absorption peak at the transition. Figure 2 shows that one indeed obtains just such a shift, which again confirms the basic features of the picture.

Finally in Figure 3 is shown the shift of the absorption peak at high pressure for a series of alkali halides having both the NaCl and CsCl structure. One can see that there is a remarkable similarity in the high pressure slopes, all showing a large shift to lower energy. While there is as yet no theory which predicts the behavior at very small interatomic distance, it is clear that any such theory must predict that the ground state and the first excited state get closer together in energy under these conditions, quite independent of the crystal lattice.

We see, then, that the high pressure results allow us to choose between available simple theories although we must understand that these may be oversimplified. Secondly, the results present a test for any theory attempting to describe the potential energy surfaces at small interatomic distances.

The Approach to the Metallic State

A second type of problem where high pressure optical and electrical measurements make a unique contribution involves the approach to the metallic state at high pressure. As discussed earlier, an insulator or semiconductor involves a filled valence band, separated by an energy gap large compared with the thermal fluctuations in energy of the electrons, from an empty band available for electrical conduction. In the absence of impurities the process of supplying electrons capable of moving through the lattice involves exciting electrons across this gap. The smaller the gap the more likely that this can occur by thermal fluctuation and the higher the conductivity. This excitation is an activated process. Theory shows that the activation energy is one half the energy of the gap. Thus one can obtain a measure of the gap width from the temperature coefficient of the electrical resistance at constant pressure. In a solid of simple band structure one would expect the bands to broaden with increasing pressure, and the gap to narrow accordingly. Thus one would expect a rapid decrease in electrical resistance with increasing pressure, accompanied by a decrease in the temperature coefficient of resistance.

In addition to exciting electrons by thermal fluctuations, it is possible to excite electrons across the energy gap by means of electromagnetic radiation of the appropriate energy. The conductivity obtained thus in the presence of light of the right wavelength is known as photoconductivity. The very intense absorption of light of the energy of the gap is called the absorption edge of the crystal. It provides an independent measure of the size of the gap.

In recent years we have made both optical and electrical measurements as a function of pressure on iodine crystals (7). Iodine forms a base centered orthorhombic crystal with the I_2 molecules lying in the ac plane. It is quite practical to grow single crystals of usable size from the vapor phase. The measurements we have made include: (1) optical absorption measurements (location of the absorption edge) as a function of pressure to about 90 kilobars, (2) measurements of electrical resistance both parallel and perpendicular to the molecular plane to over 400 kilobars, (3) measurements of the temperature coefficient of resistance between $77^{o}K$ and $296^{o}K$ from 60 to 400 kilobars.

Figure 4 shows resistance vs pressure measured both perpendicular and parallel to the molecular plane. In our apparatus it is not possible to correct for contact resistance, so that the curves have been placed relative to each other by correcting for sample geometry only. Below 50 kilobars the resistances are too large to be measured in our apparatus, but must be decreasing by many orders of magnitude. For measurements made in the ac plane, the rapid drop continues to about 230-240 kilobars, where there is a relatively sharp break. Beyond this pressure the resistance decreases at a rate which would be anticipated for a relatively compressible metal.

The broken curve in Figure 4 represents measurements made perpendicular to the ac plane. The curve is qualitatively like the one discussed above, but the break comes at 160 kilobars. In the high pressure region the resistance perpendicular to the ac plane is apparently 5-7 times greater than in the other direction, although corrections for contact resistance could alter this number.

Figure 5 shows the measured optical absorption edge as a function of pressure (black triangles). Compared with this is shown twice the activation energy for electrical conductivity measured in the ac plane (open circles) and perpendicular to the ac plane (black circles). In the pressure region where both optical and electrical measurements could be made the agreement is excellent, confirming that the simple band picture is a reasonable description for iodine. The activation energy measured perpendicular to the ac plane vanishes at 160 kilobars where the break in the resistance-pressure curve occurs. As one would expect, below 160 kilobars the activation energy is independent of direction. Above 160 kilobars the activation energy in the ac plane tails off to zero by about 220 kilobars.

Figure 6 shows a resistance-temperature plot obtained at 240 kilobars and measured perpendicular to the ac plane. It shows the linear increase of resistance with temperature which one would expect for a typical metal. Essentially identical curves were obtained from 170 kilobars to 400 kilobars, indicating that the transition to the metallic state occurs in a very small pressure range. Above 240 kilobars measurements in the ac plane also revealed typical metallic behavior. In the region between 160-220 kilobars the electronic properties are very highly directional, in a general way analogous to the behavior of single crystal graphite.

At room temperature and atmospheric pressure an array of atoms assumes the crystalline configuration which balances cohesive and repulsive energy. Often different structures differ in energy by relatively small amounts. Because their valence electrons form strongly directionalized orbitals, silicon and germanium crystallize in the diamond lattice having four neighbors located tetrahedrally, with rather tight binding. ZnS, ZnSe, and ZnTe have much more ionic character than Si and Ge, but they crystallize in the zinc blende lattice which differs from diamond only in that alternate neighbors are anion and cation.

These are relatively open structures with only four nearest neighbors, so that one might expect that, at sufficiently high pressure, one might obtain a first order phase transition to a more efficient packing.

In Figure 7 are shown the resistance of Ge and Si as a function of pressure (8). Ge exhibits a maximum at 30 kilobars which is consistent with optical observations (9). The explanation is well understood but too complex for the present purposes. At 120 kilobars there is a precipitous drop in resistance by many orders of magnitude. Beyond this pressure the resistance drops slowly as one might expect of a metal. The high pressure phase indeed exhibits metallic behavior (10).

Jamieson (11) has shown that the structure is the same as white tin, so that the transition is quite analogous to the well known grey tin-white tin transformation.

Silicon shows a small continuous drop in resistance with pressure in the low pressure phase. Again this is consistent with measurements on the shift of the absorption edge (9). At 190-195 kilobars there is a very large drop in resistance. The high pressure phase is metallic and has the white tin structure.

Figure 8 shows resistance-pressure curves for ZnS, ZnSe and ZnTe (12). The sulfide and selenide have very high resistances in the low pressure phase, so that the dotted lines show merely the resistance of the cell. The ZnTe curve is, again, consistent with optical measurements (13). For each of these there is a transition accompanied by a very large drop in resistance. These transitions occur at 135, 165 and 240 kilobars respectively. In each case the high pressure phase behaves as a metal (10). Recent unpublished X-ray measurements indicate that ZnTe, ZnSe and possibly ZnS have the simple cubic CsCl structure at high pressure. Thus we see that it is possible for a single material to behave as an insulator, a semiconductor, or a metal, depending on the interatomic spacing. The transformation may take place continuously, or accompanied by a first order phase transition.

Electronic Transitions in Metals

As discussed in the introduction to this paper, the normal effect of pressure on the electrical resistance of a metal is to cause a modest decrease in resistance due to the stiffening of the lattice. There are a variety of exceptions to this rule. It is of interest to discuss one class of exceptions which illustrate again a case where pressure is a unique tool for investigating electronic structure.

The periodic table of the elements and the corresponding periodic properties are built up by adding electrons according to the rules of quantum mechanics. The electrons are classified according to their four quantum numbers. Each electronic shell, characterized by a principal quantum number (17), contains subshells classified according to their angular momentum quantum numbers, $\ell = 0,1,2,3 \cdots n-1$. These latter states are generally labeled $S(\ell = 0)$, $p(\ell = 1)$, $d(\ell = 2)$, $f(\ell = 3)$, etc. The electrons build up regularly from element to element, filling first the 1s shell (2 electrons) then the 2s, 2p (6 electrons)etc., up to potassium. In potassium, however, the last electron enters the 4s state leaving the 3d shell empty; calcium has two 4s electrons and an empty 3d shell. Similarly the valence electrons on rubidium and strontium are in the 5s state while the 4d shell is empty, and cesium and barium have 6s valence electrons with an empty 5d shell. The transition metals are characterized by partially filled 3d, 4d, or 5d shells with two electrons in the s shell of next higher principal quantum number. In the rare earth elements there are 6s electrons with an empty 5d shell and a partially filled 4f shell.

Let us now look at the electronic structure of an alkali metal, say cesium. The lattice is body centered. There is a spherically symmetric band which is related to the 6s atomic level and contains one electron per atom. This is, of course, the conduction band. In the free atom the 5d shell is five fold degenerate, that is, it contains five substates of equal energy, each capable of containing two electrons. In the crystalline field of the lattice two bands appear connected with the 5d shell, one capable of containing six electrons per atom and one with a capacity of four electrons. Both these bands are higher in energy than the band arising from the 6s shell, and, of course, contain no electrons.

Bridgman (14) measured the volume and electrical resistance of cesium as a function of pressure. He found a volume discontinuity at 22 kilobars, doubtlessly a first order transition to a cubic close packed structure. There is a further large volume discontinuity at 41 kilobars accompanied by a very definite cusp in the electrical resistance. Sternheimer (15) has suggested that the bands arising from the 5d shell lower in energy with pressure vis-a-vis the 6s band, and that the events at 41 kilobars are associated with the transfer of the conduction electron from the 6s to the 5d band. Sternheimer assumed a single, spherically symmetric d band so that his calculations cannot be rigorously correct. Recently Ham (16) has shown that the situation must be somewhat more complicated. Nevertheless, the notion of this "electronic transition" is probably sound and very intriguing.

Very recently (17) we have measured the electrical resistance of rubidium and potassium to very high pressures. Figure 9 shows the results for rubidium at 77°K and 296°K. There is a sharp rise in resistance at 190-200 kilobars, and a maximum beyond 400 kilobars. These events show no lag at 77°K which is consistent with the notion that this is an electronic transition rather than a diffusion controlled rearrangement.

In rubidium the separation in energy between the 5s and 4d atomic states is much larger than the 6s-5d splitting in cesium, so that it is reasonable that it would take a higher pressure to effect the electronic promotion.

The 3d energy in potassium is even further above the 4s state so that one would expect any electron transfer to occur at very high pressure. Figure 10 shows the resistance-pressure curves. The resistance at room temperature rises by a factor of 50 in 600 kilobars. It seems very likely that there is a maximum at still higher pressures. At 77°K the features at low pressure are similar. The very sharp rise at 360 kilobars is the result of a martensitic transition, the details of which are not germane to this paper.

One would expect that there would be electronic transitions in the rare earths involving promotions of 4f electrons to bands arising from the empty 5d shell. Indeed one such transition in cesium has been studied in much detail (18). More recently evidence has come forth that a similar transition takes place in ytterbium (19).

It seems probable that this is not an uncommon event in the heavier elements where empty and filled bands do not differ too much in energy. These radical changes in the character of the valence electrons offer the possibility of new chemical valences, and conceivably radical changes in chemical reactivity.

Summary

In this paper we have presented a general discussion of pressure as a variable in understanding electronic structure, and have used three types of illustrations.

We have shown that high pressure optical absorption measurements can be used to establish a satisfactory explanation of the characteristics of alkali halide phosphors.

We have combined high pressure optical absorption and electrical resistance data to illustrate the mechanisms whereby an insulator or semiconductor can become a metal.

We have presented the notion of an electronic transition in metals wherein distortion of the bands at high pressure can result in electron transfer between atomic shells and new valence states for some elements.

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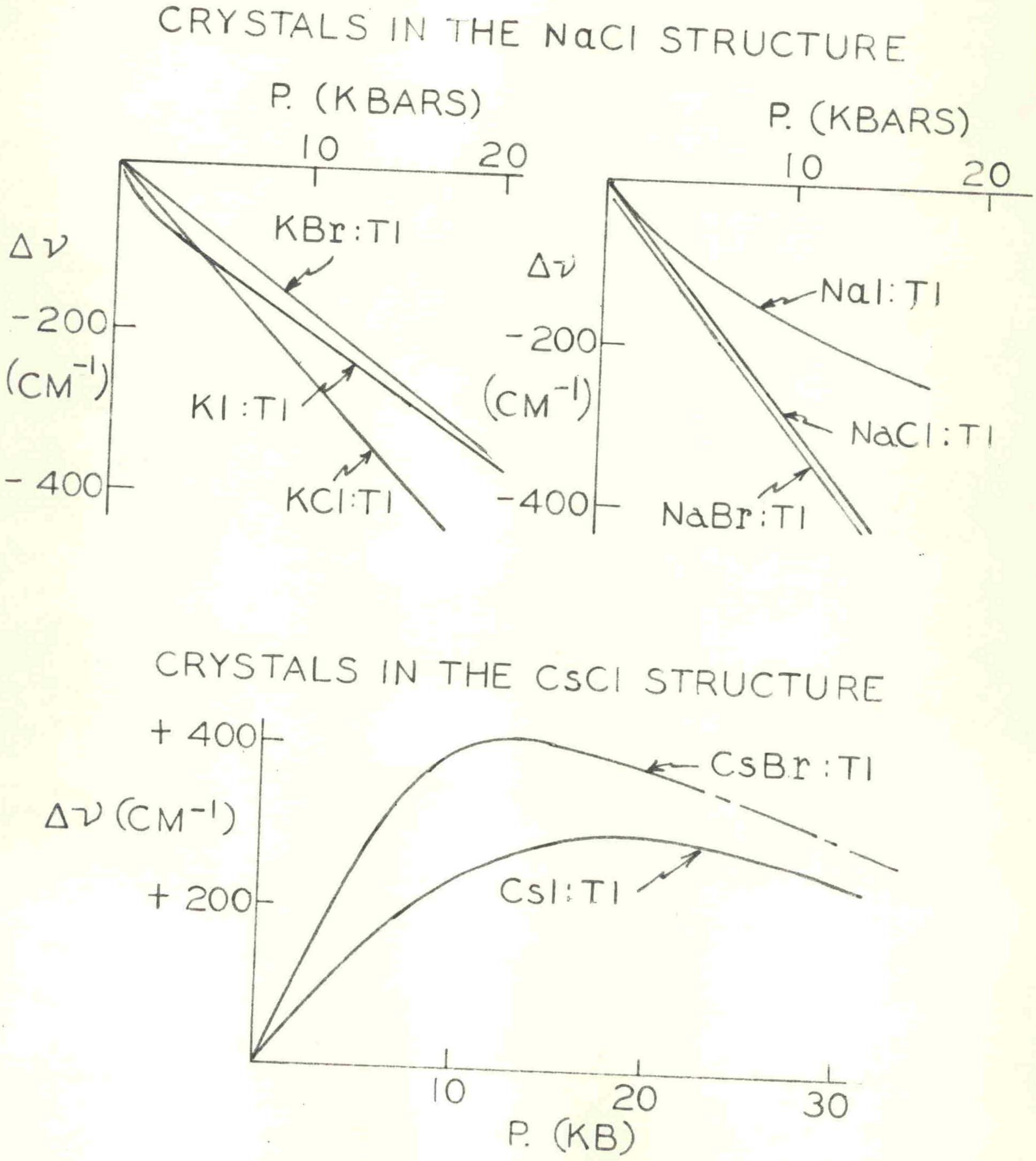

FIG. 1 - INITIAL FREQUENCY SHIFT VS. PRESSURE TEN ALKALI HALIDES ACTIVATED BY Tl

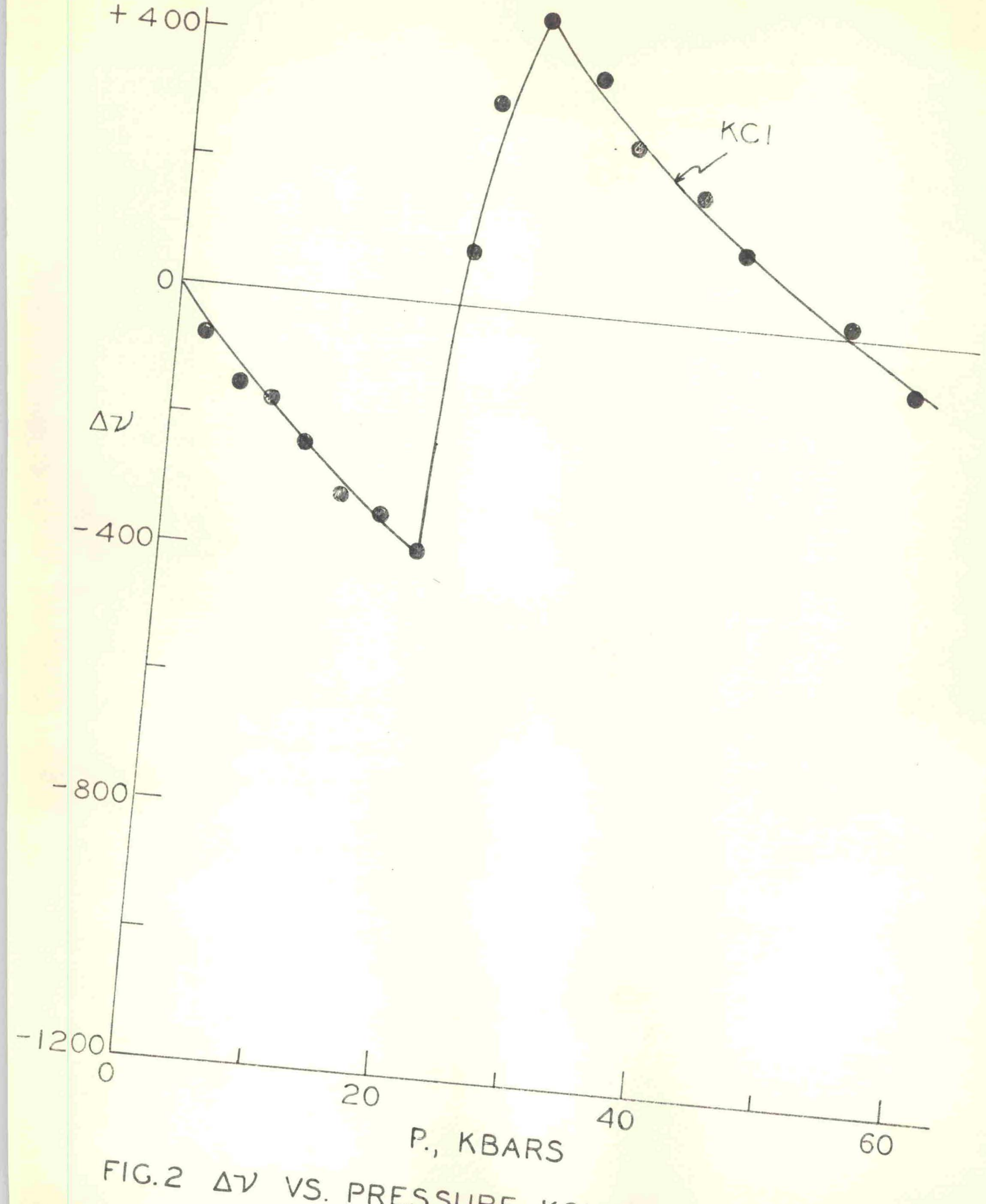

FIG. 2 Δν VS. PRESSURE KCl WITH Tl+

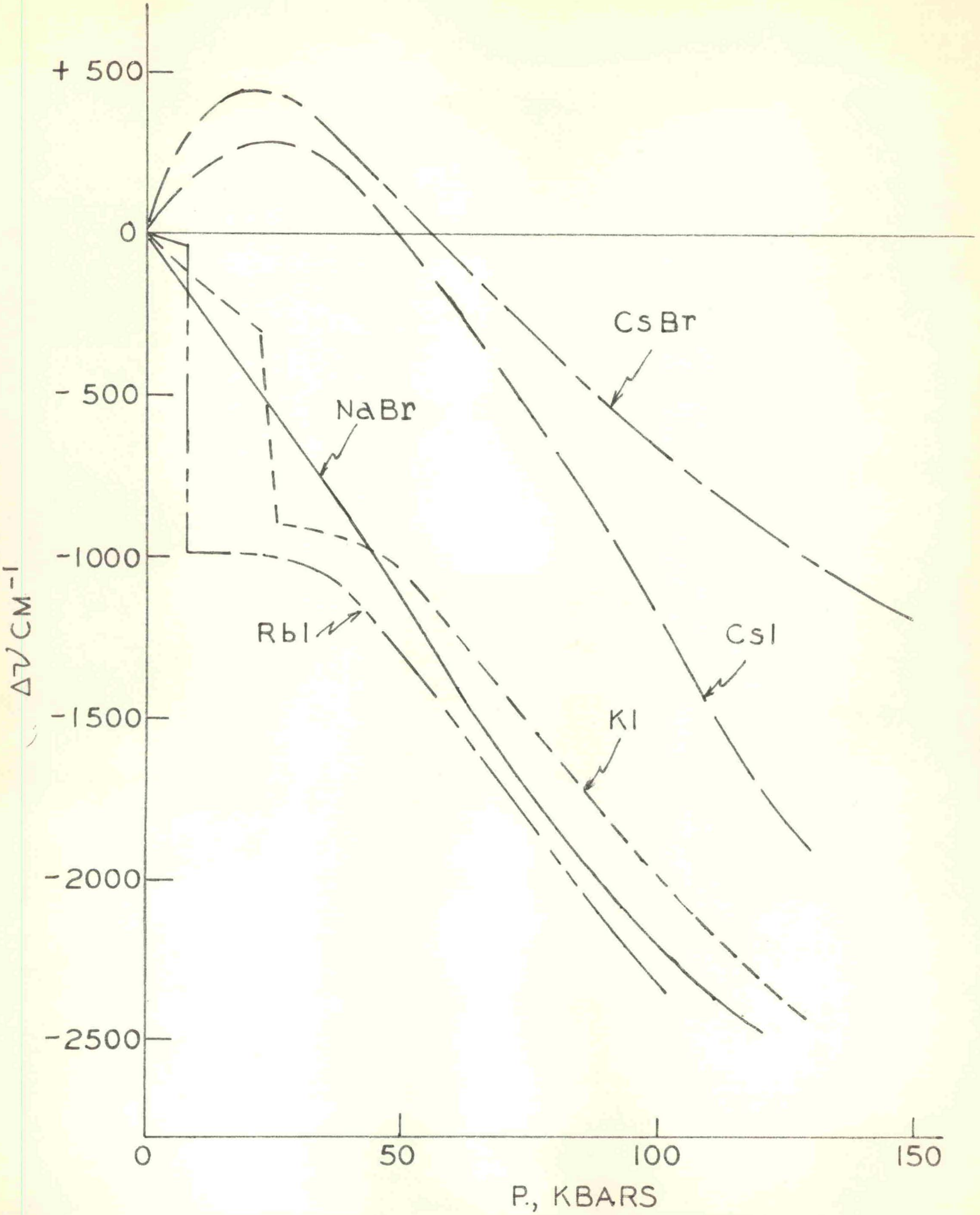

FIG. 3 $\Delta\nu$ VS. PRESSURE Tl$^+$ IN ALKALI HALIDES

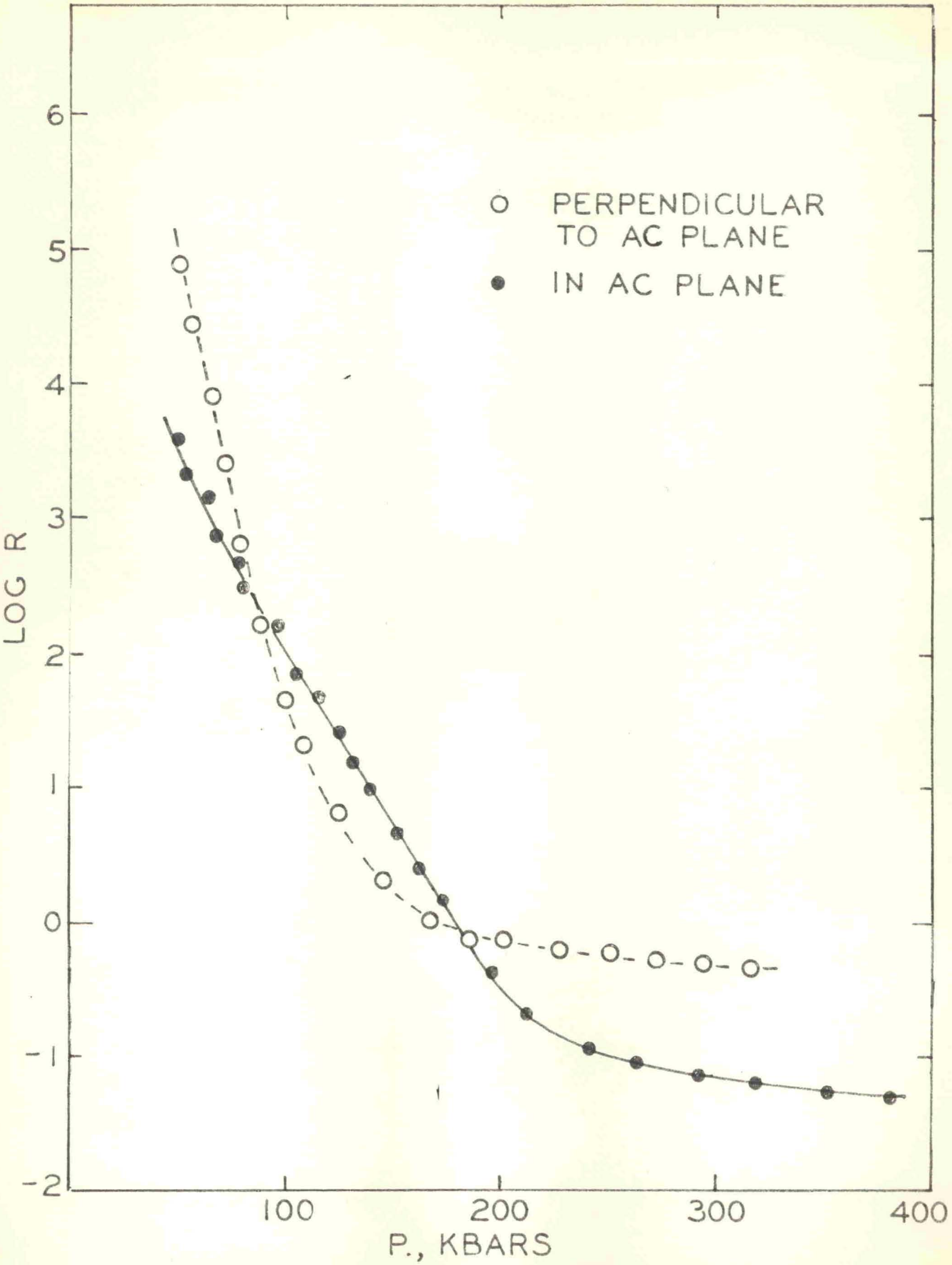

FIG. 4 LOG RESISTANCE VS. PRESSURE - IODINE

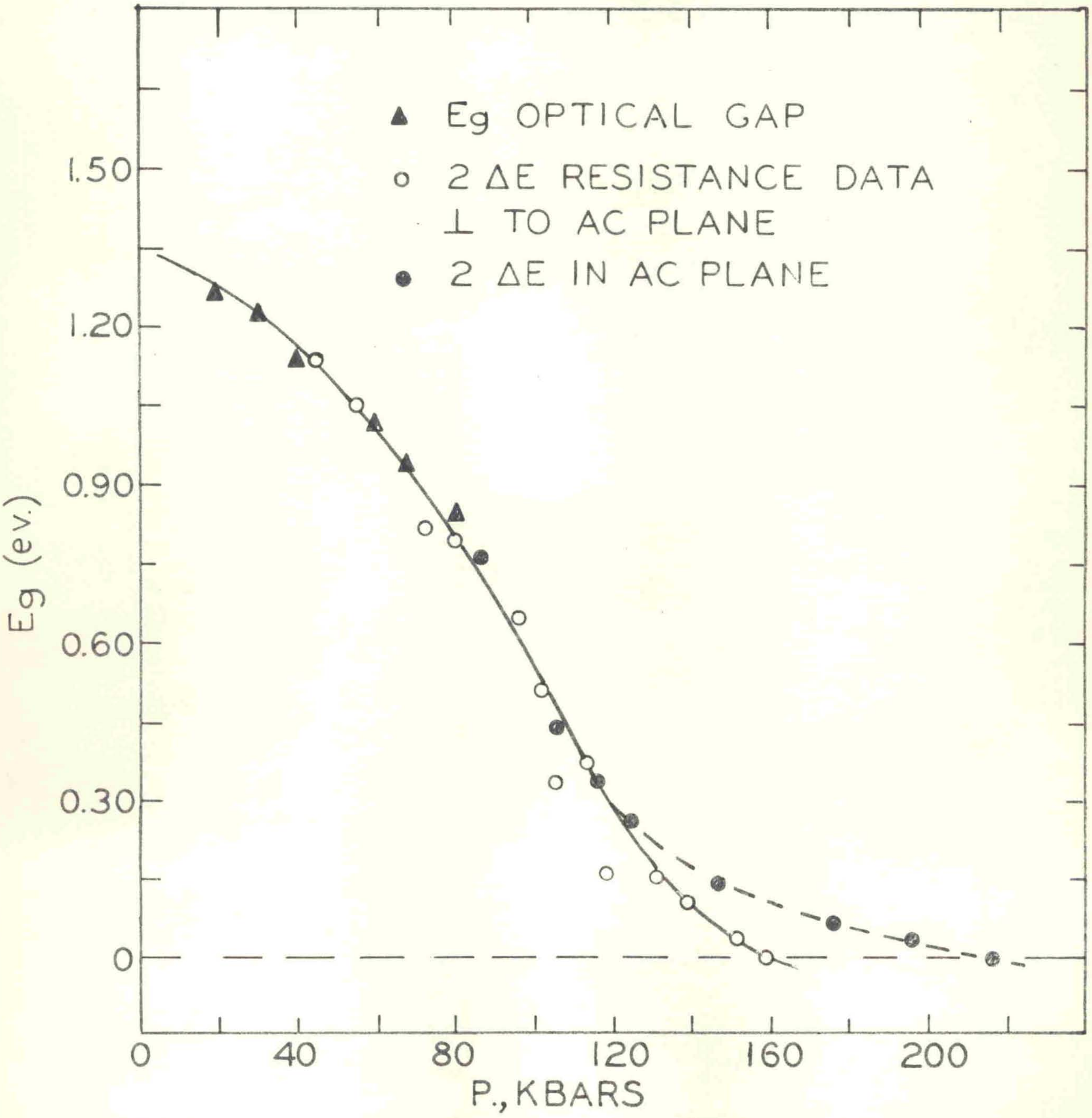

FIG. 5 ENERGY GAP VS. PRESSURE. IODINE

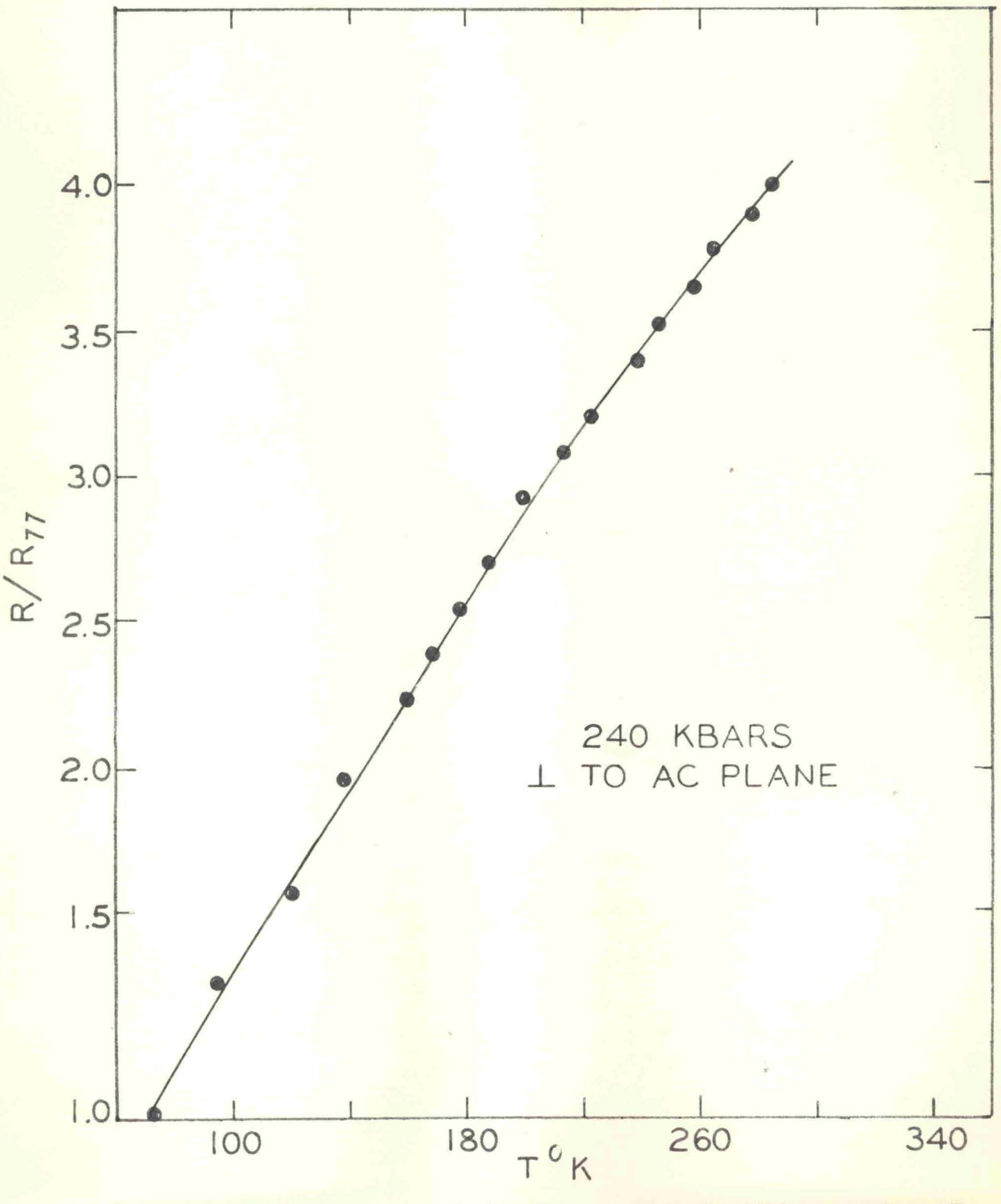

FIG. 6 RESISTANCE VS. TEMPERATURE - IODINE

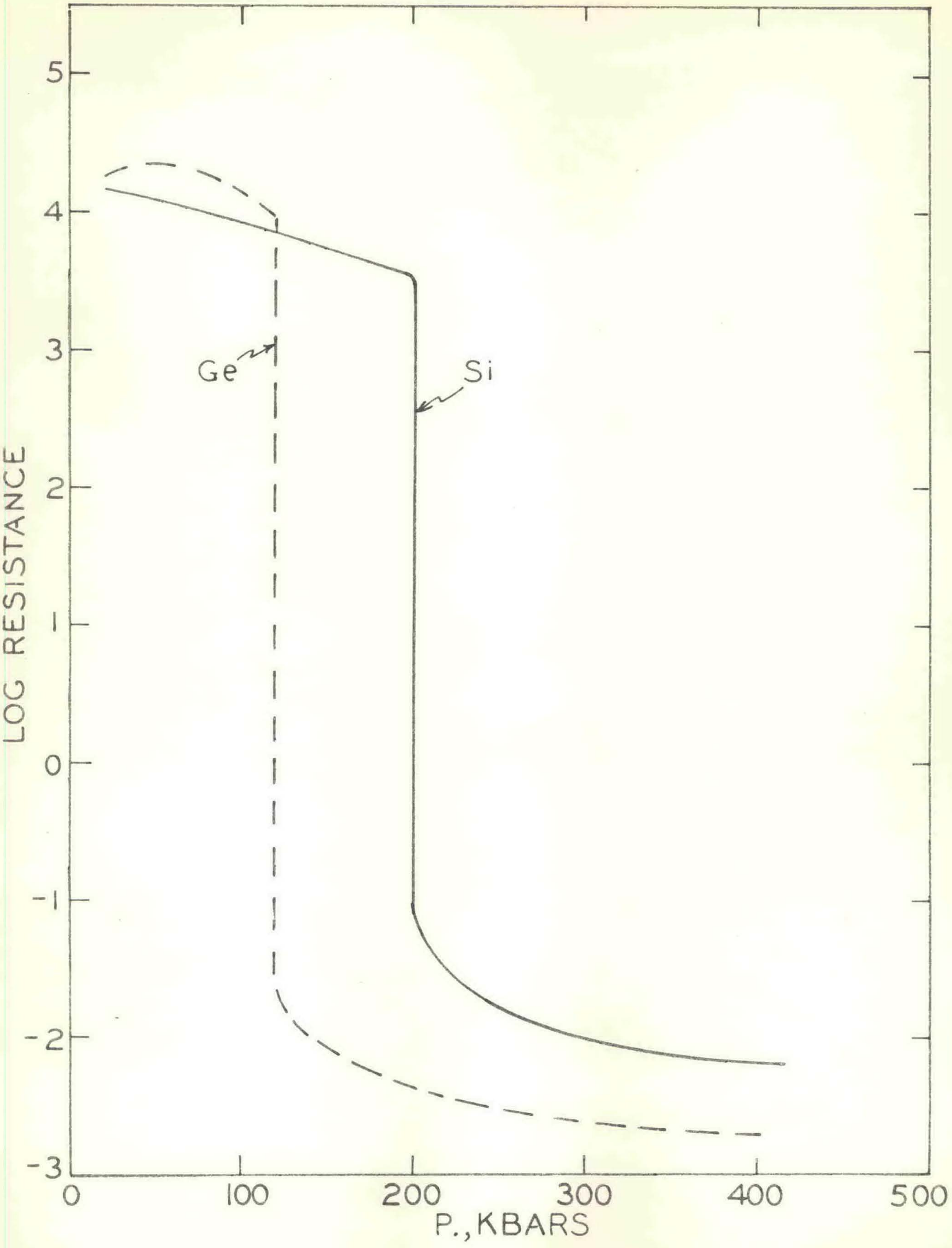

FIG. 7 RESISTANCE VS. PRESSURE, Ge AND Si

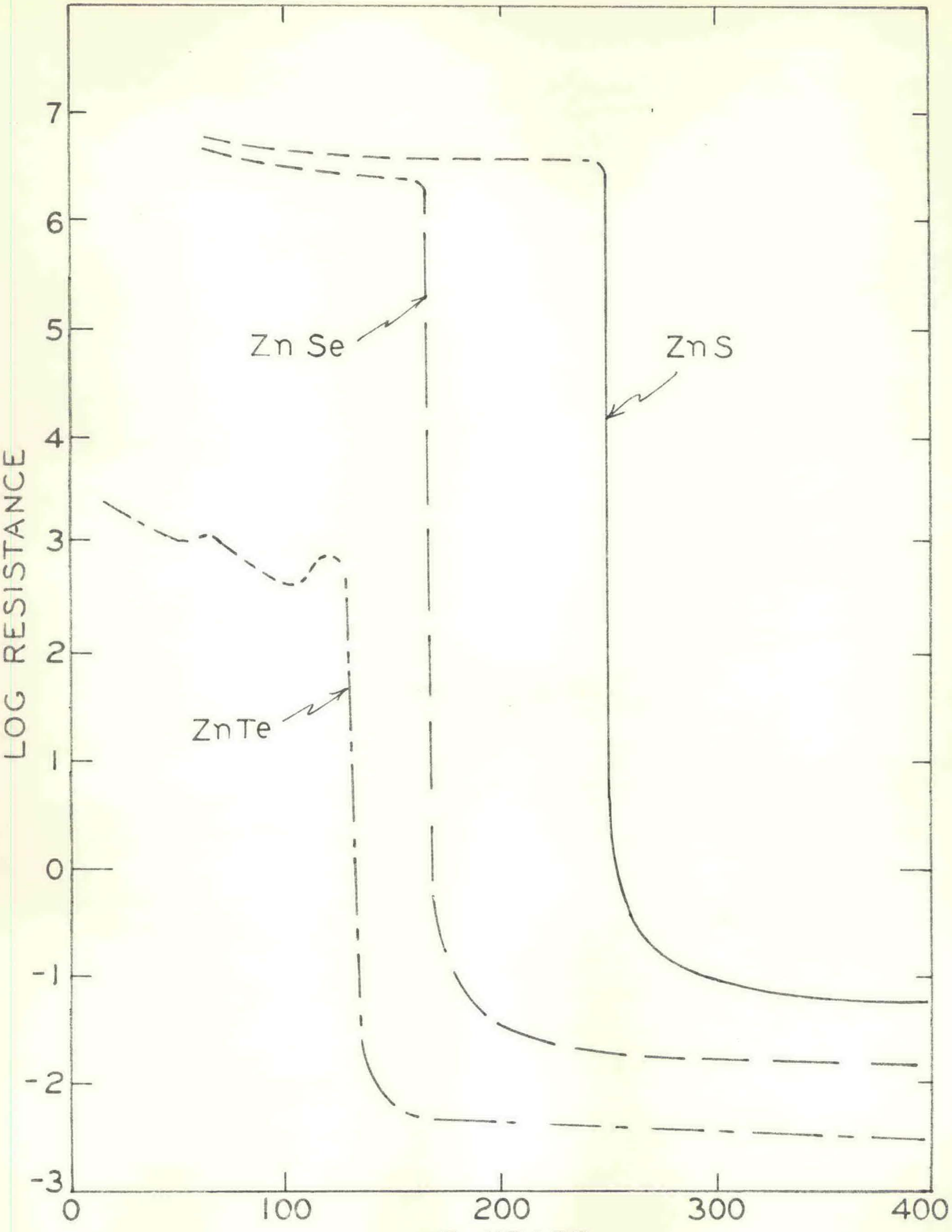

FIG. 8 RESISTANCE VS. PRESSURE ZnS, ZnSe, ZnTe

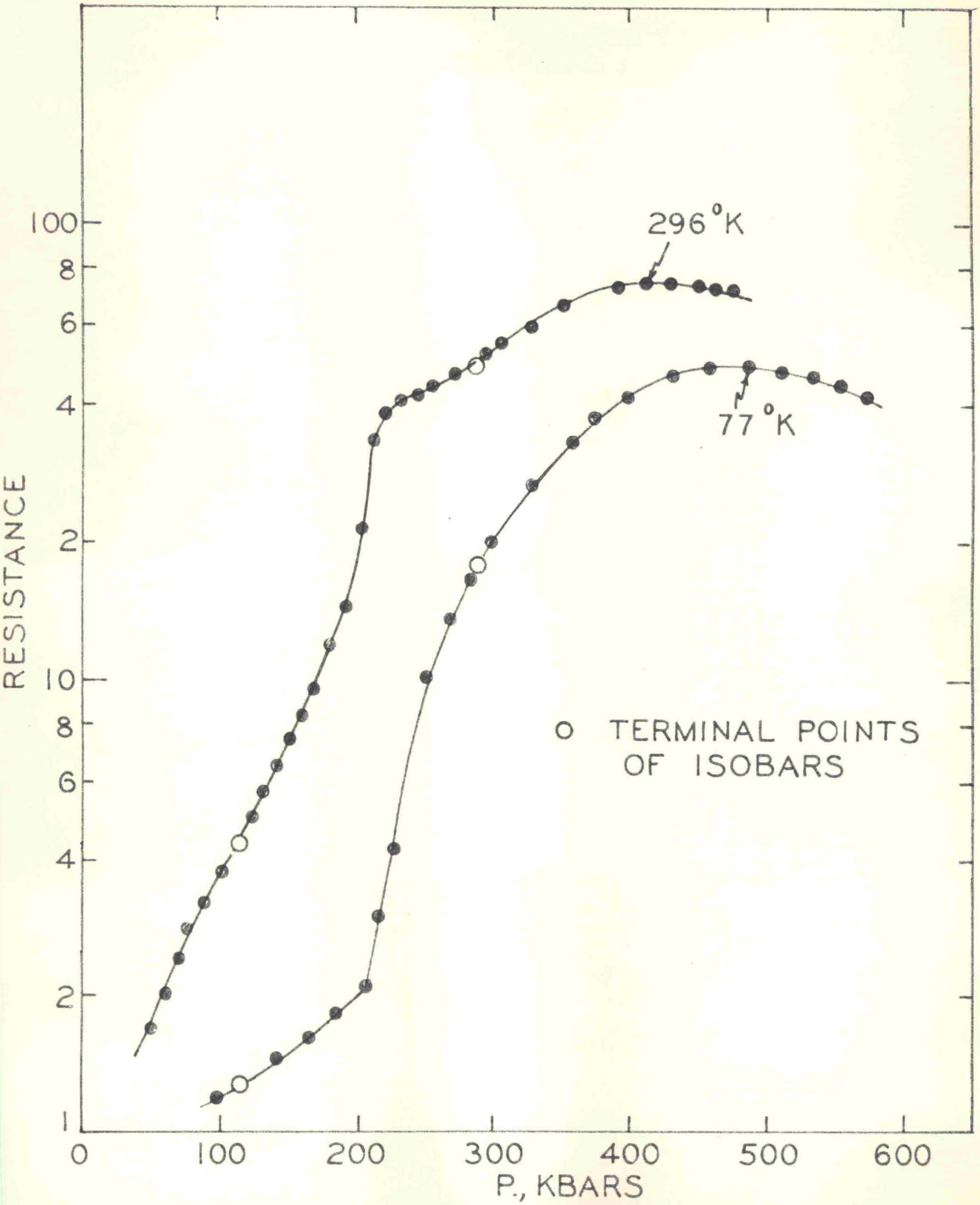

FIG. 9 RESISTANCE VS. PRESSURE FOR RUBIDIUM

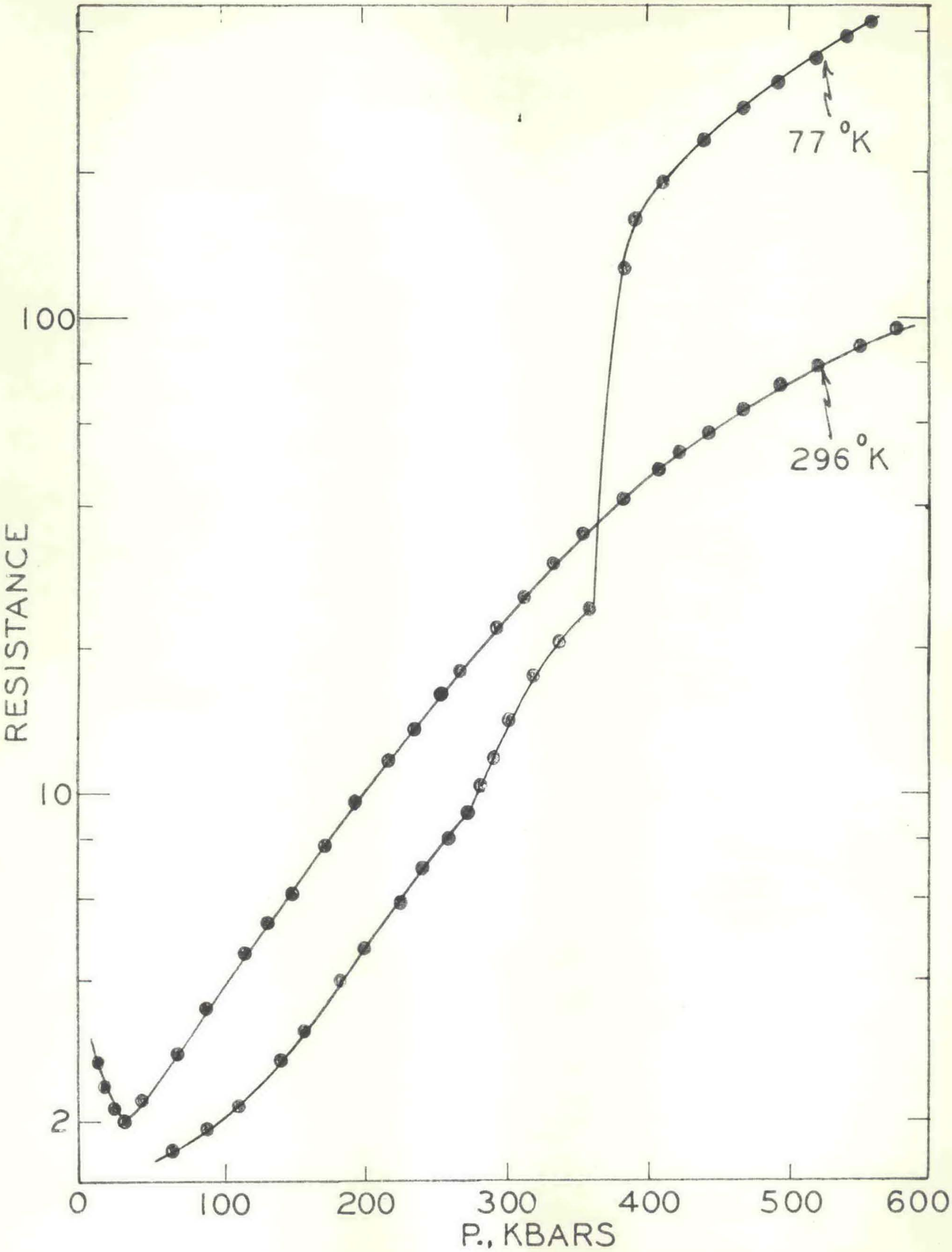

FIG.10 RESISTANCE VS. PRESSURE FOR POTASSIUM