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# Fusion Curve and Electrical Conductivity of Molten $HgCl_2$ and $HgI_2$ at Elevated Pressure*

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The fusion curves of $HgCl_2$ and $HgI_2$ were measured to a pressure of 22 kbar. The melting temperature can be represented as a function of pressure by $t_P = t_0 + b_1 P + b_2 P^2$, where $t_P$ is the melting temperature in degrees Centigrade at a pressure $P$ in kilobars; $b_1$ and $b_2$ are constants. Values for $t_0$ (deg), $b_1$ (deg kbar$^{-1}$), and $b_2$ (deg kbar$^{-2}$) are 276, 259; 19.8, 17.1, and −0.26, −0.41, respectively. The electrical conductivities ($K$) of molten $HgCl_2$ and $HgI_2$ were measured to 805 and 620°C, respectively, at a pressure of 5.4 kbar. The electrical conductivity of $HgCl_2$(l) was also measured from 557 to 634°C at $P = 20.5$ kbar. At these elevated pressures $K$ varies exponentially with $1/T$, i.e., $K \cong A \exp-(E_k/RT)$. At 5.4 kbar, $A = 2.13$ and 0.36 $(\Omega\cdot\mathrm{cm})^{-1}$; $E_k = 8.46 \pm 0.14$ and $2.53 \pm 0.12$ kcal/mole, respectively, for molten $HgCl_2$ and $HgI_2$. The behavior of $HgCl_2$(l) and $HgI_2$(l) at this elevated pressure is now typical for a strong electrolyte. This is in contrast to their behavior at ordinary pressures where $HgCl_2$ shows a maximum in its $K$ vs $T$ curve and $HgI_2$ has a negative temperature coefficient of $K$ from the onset of melting. The conductivities of molten $HgCl_2$ and $HgI_2$ were also examined from 3 to 20 kbar at constant $T$. At 600°K, $K_{P=20\ \mathrm{kbar}}/K_{P=3\ \mathrm{kbar}}$ for $HgCl_2 = 12.4$; at 555°K this ratio for $HgI_2$ is 1.66. Thus pressure increases the conductivity of these salts. This is attributed to a greater degree of ionic dissociation at elevated pressures.

## I. INTRODUCTION

The electrical conductivities of the molten mercuric halides suggest that they are predominantly covalent in character.[1–3] Hevesy[4] has observed that $HgI_2$(l) has a negative temperature coefficient from the onset of melting. Grantham and Yosim[5] found that molten $HgCl_2$ and $HgBr_2$ have a maximum in their specific electrical conductivity vs temperature ($K$ vs $T$) curves approximately 200° above their melting points. These measurements by Grantham and Yosim were carried out with the salt at its own vapor pressure. Similar conductivity maxima were found by them in other molten salts, i.e., in the bismuth trihalides[6,7] and in $ZnI_2$, $CdI_2$, $SnCl_2$, and in CuCl.[5] These authors attribute these maxima in $K$ to increased ionic association at higher temperatures. Darnell, McCollum, and Yosim[8] have shown that extreme pressure increases the electrical conductivity of $BiCl_3$, $BiBr_3$, and $BiI_3$ and furthermore removes this maximum in the $K$ vs $T$ curve.

The pressure–temperature phase diagrams of $HgCl_2$ and $HgI_2$ were examined to determine the extent of the liquid phase at pressures up to 22 kbar. The electrical conductivities of molten $HgCl_2$ and $HgI_2$ were then measured at elevated pressure, first as a function of temperature at constant but elevated pressure, then as a function of pressure at constant temperature.

## II. EXPERIMENTAL

### A. Materials

Reagent-grade $HgCl_2$ and $HgI_2$ were further purified by distillation under a partial pressure of oxygen[9] and then sublimed in vacuo. The purified compounds were loaded into the conductivity cells within an inert-atmosphere glove box.

### B. Apparatus and Procedure

The phase diagram studies and electrical conductivity measurements on $HgCl_2$ and $HgI_2$ were carried out in a piston-cylinder high-pressure apparatus using an internally heated furnace. The apparatus and procedure used here have been described in previous papers by Darnell *et al.*[8,10] The pressure–temperature phase diagrams of $HgCl_2$ and $HgI_2$ were determined using differential thermal analysis (DTA) and resistivity measurements. The solid$\rightleftarrows$liquid curve was examined both upon freezing and melting over the pressure range 2–22 kbar. Platinum and/or nickel cells were used as sample containers in these DTA measurements. In all cases the freezing point of the salt was redetermined at low temperature (and at low pressure) after measurements had been made at higher temperatures. This was done in order to ascertain that the freezing point and thus the purity had not changed as a result of exposure to higher temperatures.

The conductivity measurements upon molten $HgCl_2$ and $HgI_2$ were made following the technique used by Darnell *et al.*[8] for the bismuth trihalides. For $HgCl_2$, a quartz cell with platinum electrodes was used; for $HgI_2$ both Pt and W electrodes were used. Use of either electrode material yielded essentially the same value for the specific conductivity for $HgI_2$.

A checking procedure was also used here in order to ascertain that exposure of these compounds to high temperature and/or high pressure did not irreversibly change the cell constant or cause contamination of the compound. In this procedure the conductivity was first determined at a temperature just above the melting point at a pressure of 5 kbar. The conductivity was then redetermined at this same pressure and temperature after the sample had been exposed to higher temperatures and/or higher pressures. In general we found that temperatures above 850°C and pressures above 25 kbar resulted in significant irreversible changes in the conductivity when remeasured at the low-temperature check point at 5 kbar.

The electrical conductivities reported here were determined at a frequency of 1000 Hz. The conductivity, however, was examined as a function of frequency (500–5000 Hz) at one pressure (5.4 kbar) and temperature (500°C) for $HgCl_2$ and $HgI_2$. No dependency of $K$ upon frequency was noted within the limit of precision of these measurements.

Stainless-steel-clad chromel–alumel junctions were placed within the high-pressure chamber to measure the temperature of the compound. No corrections have been applied to account for the effect of pressure upon the emf of these couples; however, this effect is reported to be small (i.e., less than 1%) at pressures below 20 kbar.[11]

## III. RESULTS

### A. Phase Studies

The pressure–temperature phase diagrams for the condensed phases of $HgCl_2$ and $HgI_2$ are shown in Figs. 1 and 2, respectively. In each case the solid⇄liquid transition curve was determined to a pressure of 22 kbar. Both melting point and freezing point determinations were made upon these compounds using DTA and resistance measurement techniques. No polymorphic transition was observed in $HgCl_2$ at pressures up to 45 kbar and at temperatures up to 500°C. The red⇄yellow polymorphic transition in $HgI_2$ was detected by its resistance change. The results obtained here for this polymorphic transition are compared with Bridgman's[12] data in Fig. 2. Brasch *et al.*[13] have shown that the polymorphic transition which occurs at a pressure of 13 kbar at room temperature is the same as the red⇄yellow transition which is observed at 127°C at atmospheric pressure. This solid–solid transition is somewhat unusual since a maximum is exhibited in its $P$–$T$ curve.

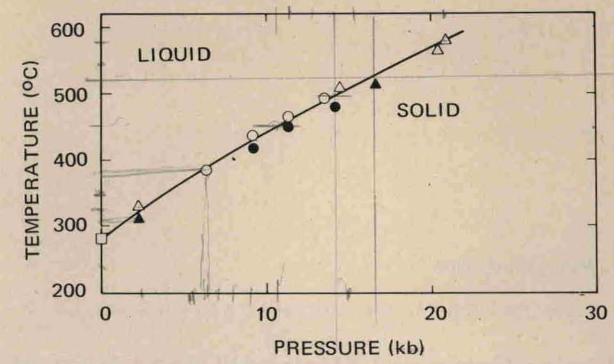

FIG. 1. Pressure–temperature phase diagram of $HgCl_2$. (△) melting; (▲) freezing, by differential thermal analysis. (○) melting; (●) freezing, by resistance measurements. (□) melting point at 1 atm. Pressure in kilobars.

Equations for the melting temperature vs pressure for $HgCl_2$ and $HgI_2$ were computed by method of least squares of the experimental data and are given in the polynomial form,

$$t_P = t_0 + b_1 P + b_2 P^2, \qquad (1)$$

where $t_P$ is the melting point (degrees centigrade) at pressure $P$ (kilobars), $t_0$ is the melting temperature at 1 atm, $b_1$ and $b_2$ are constants. Values for the constants of this melting curve equation obtained for $HgCl_2$ and $HgI_2$ are given in Table I.

The constant $b_1$ in Eq. (1) gives the initial (atmospheric pressure) slope of the melting curve. For $HgCl_2$, $b_1$ is 19.8 deg/kbar; for $HgI_2$ it is 17.1 deg/kbar. As a comparison the initial slope of the melting curve was calculated by use of the Clausius–Clapeyron equation, $dT/dP = k\Delta V/\Delta S$, where $k$ is a proportionality constant, $\Delta V$ and $\Delta S$ are the volume and entropy of fusion. Taking Janz's[14] values of 11.4 cm$^3$/mole for $\Delta V_f$ and 7.5 cal/mole deg for $\Delta S_f$ of $HgCl_2$ yields 15.7 deg/kbar for the initial slope of the melting curve for $HgCl_2$. Using Topol's[15] entropy of fusion of 8.40 cal/mole/deg gives 17.6 deg/kbar for the slope, $dT/dP$ at atmospheric pressure. Likewise using 14.5 cm$^3$/mole and 8.6 cal/mole/deg for $\Delta V_f$ and $\Delta S_f$ of $HgI_2$[14] gives 14.2 deg/kbar for the slope of the liquid⇄solid $P$–$T$ curve at atmospheric pressure. The molar volume of the yellow (high temperature) form of $HgI_2$ has been used to calculate the volume change during fusion. For both compounds the calculated value of $dT/dP$ from the Clausius–Clapeyron equation is in relatively good agreement with the measured value of the slope of the melting curve, i.e., for $HgCl_2$ $dT/dP$ (calc) is 15.7 (Janz's $\Delta S_f$) or 17.6 (Topol's $\Delta S_f$) the measured value is 19.8 deg/kbar; for $HgI_2$ $dT/dP$ (calc) is 14.2, measured 17.1 deg/kbar.

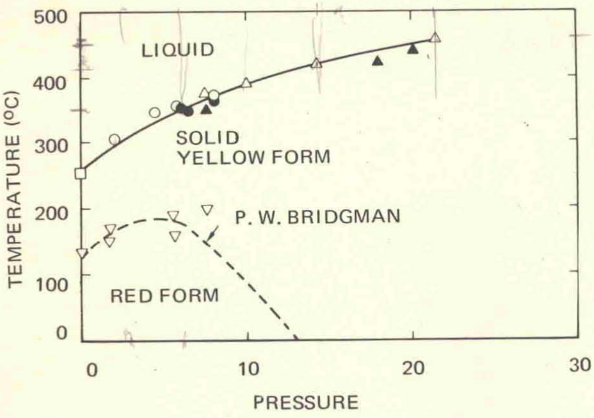

FIG. 2. Pressure-temperature phase diagram of $HgI_2$. (△) melting; (▲) freezing, Sample No. 4 (○) melting; (●) freezing, Sample No. 5. (□) melting point at 1 atm. (▽) red–yellow transition. Pressure in kilobars.

### B. Electrical Conductivity

The electrical conductivities of molten $HgCl_2$ and $HgI_2$ were examined (a) as a function of temperature at a constant but elevated presure and (b) as a function of pressure at constant temperature.

#### *1. $HgCl_2$*

The conductivity of molten $HgCl_2$ was measured over the temperature range 327 to 805°C at a pressure of 5.4 kbar. The results are shown in Fig. 3. It should be noted that the melting point at this pressure of 5.4 kbar is 368°C (Fig. 1); thus the conductivity measurements between 327° and 368° were made upon the supercooled liquid.

The conductivity of $HgCl_2$ was also measured at a pressure of 20.5 kbar from 557°C (the melting temperature at this pressure) to 634°C. Two separate attempts which were made to measure the conductivity to higher temperatures at this pressure resulted in failure of the conductivity cell. The conductivity data over this rather limited temperature range at 20.5 kbar suggest that the slope of $\log K$ vs $1/T$, is not greatly different from the slope obtained at 5.4 kbar.

Conductivity data for molten $HgCl_2$ reported by Grantham and Yosim[5] at a pressure approximately equal to the salts own vapor pressure ($P<0.1$ kb) are also shown in Fig. 3 for comparison with our data obtained at a much higher (but constant) pressure. Two differences are immediately apparent from comparison of $K$ at 5.4 kbar and at the lower pressure. First of all, it can be seen that an increase in pressure brings about a large increase in the specific conductivity of molten $HgCl_2$. For example, at 600°C the specific conductivity increases by more than three orders of magnitude as the pressure is increased from near atmospheric pressure up to a pressure of 20.5 kbar. Second, the behavior of the conductivity with temperature variation is markedly altered at these elevated pressures. At temperatures up to ~150°C above the melting point the slopes of $\log K$ vs $1/T$ curves for the two widely different pressures (near atmospheric and 5.4 kbar) are similar (Fig. 3). However, at higher temperatures this similarity in slopes no longer exists. Grantham's and Yosim's conductivity data[5] show a maximum at 480°C and consequently exhibit a decrease in specific conductivity with increasing temperature above 480°C.

The effect of pressure upon the conductivity of molten $HgCl_2$ at constant temperature was also examined. The results from measurements carried out at 600° are shown in Fig. 4. Here, the variation in conductivity with pressure over the interval 3 to 20 kbar is shown. (The specific conductivity is only approximate in this case since the compressibility of the molten salt is not known. The cell constant has been adjusted, as an approximation, taking into account the compression experienced by the entire high-pressure chamber assembly.) A temperature of 600° was chosen because here, $HgCl_2$ remains a liquid at all pressures below 20 kbar. The data shown in Fig. 4 were obtained after compressing solid $HgCl_2$ to a pressure of 20 kbar at 25°C and then raising the temperature to 600°C. Conductivity data were then taken as the pressure was decreased from 20 to 3 kbar (upper curve in Fig. 4). Following this, conductivity measurements were made as the pressure was increased to 17.5 kbar. The temperature and pressure were then returned to the original reference condition of 350°C and 5.4 kbar and the conductivity was again remeasured at this pressure and temperature and was found to be almost identical to its original value at this same temperature and pressure. It can be seen in Fig. 4 that hysteresis is exhibited in the $K$ vs $P$ curves for the decompression and compression stages. However, this is to be expected in this kind of experimental procedure[16] where the pressure within the high-pressure chamber is calculated from the force applied to the pressurizing piston.

In order to ascertain if these large conductivity changes brought about by pressure were reversible, particularly between atomspheric pressure and the pressure at which these conductivity experiments were performed, the following additional experiment was carried out. This experiment consisted of comparing the

TABLE I. Parameters for the constants in the polynomial, $t_P=t_0+b_1P+b_2P^2$, representing the fusion curves of $HgCl_2$ and $HgI_2$.

| Compound | $t_0$ (deg) | $b_1$ (deg/kbar) | $b_2$ (deg/kbar$^2$) |
|---|---|---|---|
| $HgCl_2$ | 276 | 19.8 | −0.26 |
| $HgI_2$ | 259 | 17.1 | −0.41 |

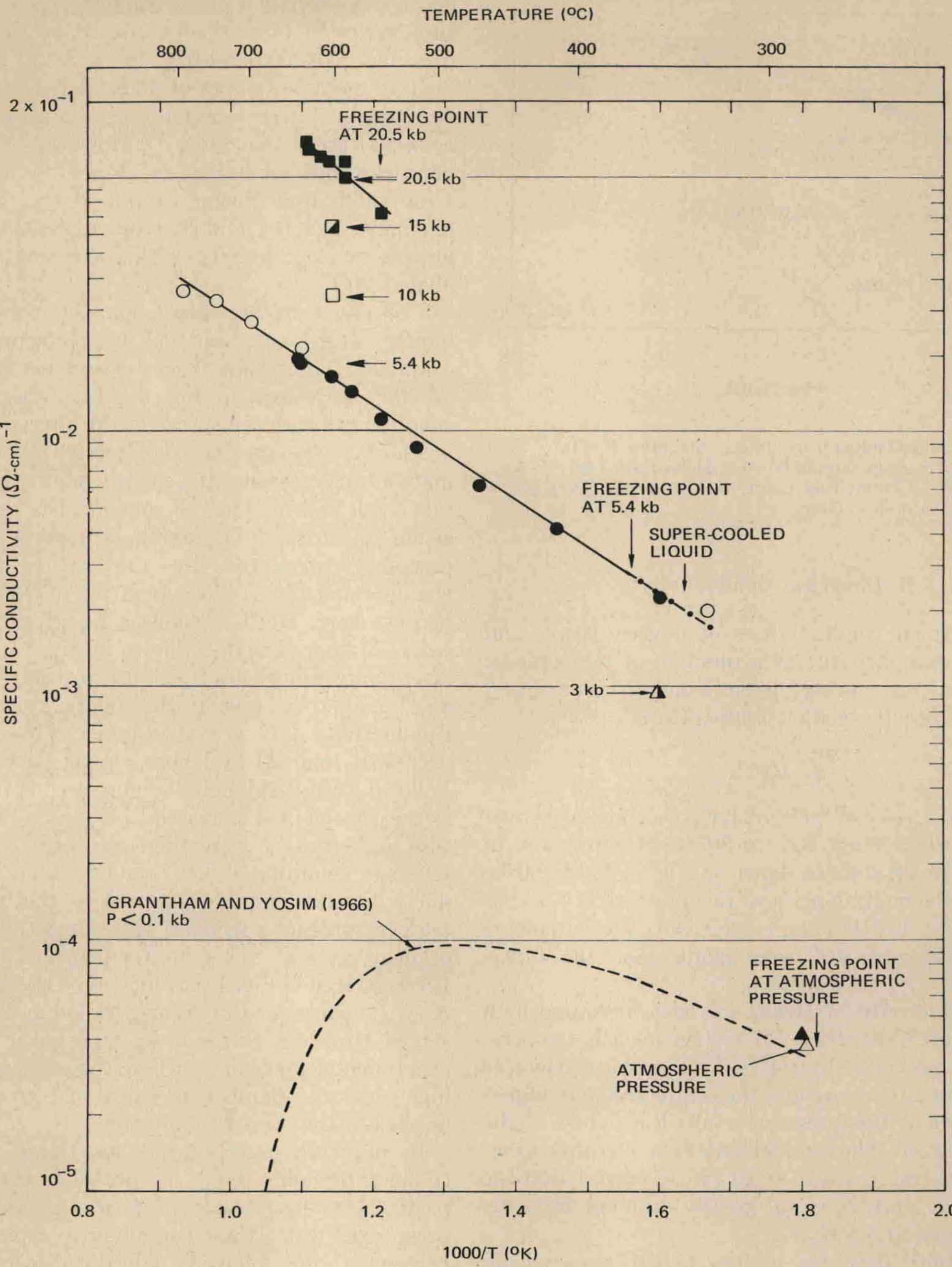

FIG. 3. Log$K(\Omega\cdot cm)^{-1}$ vs $1/T$(°K) for $HgCl_2$ (△) $P=1$ atm, before pressurization; (▲) $P=1$ atm, after pressurization; (◭) $P=3$ kbar; (○) $P=5.4$ kbar, Sample No. 1; (●) $P=5.4$ kbar, Sample No. 4; (□) $P=10$ kbar; (◪) $P=15$ kbar; (■) $P=20.5$ kbar.

electrical conductivity of molten $HgCl_2$ at atmospheric pressure before and after it had been subjected to a pressure of 3 kbar. In order to do this, however, it was necessary to employ a different type of conductivity cell since the quartz cell after once having been subjected to high pressure cannot be returned to atmospheric pressure and still retain the liquid sample. A Teflon conductivity cell, similar to the device described by Jayaraman *et al.*[17] was used to carry out this reversibility test. The conductivity of $HgCl_2(l)$ was first measured at a pressure of one atmosphere at a temperature 5°C above the mormal melting point. This sample was then pressurized to 3 kbar and its conductivity measured at 350°C. (350°C is the upper tem-

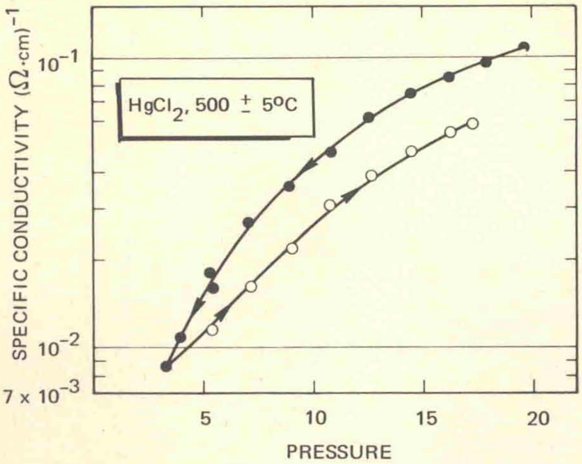

FIG. 4. Isothermal specific conductance ($K$) of $HgCl_2$ as a function of pressure (kbar).

perature limit to which the Teflon cell can be heated without contamination of $HgCl_2$.) It was then cooled, removed from the high-pressure apparatus (as a further precaution the weight and the dimensions of the cell were checked). The conductivity was again measured at 281°C at pressure of 1 atm. The data from this test are shown in Fig. 3. The conductivities before and after pressurization are seen to be in good agreement. These results as well as the results shown in Fig. 4 demonstrate that this effect of pressure upon the conductivity of molten $HgCl_2$ which we observe is indeed reversible.

The temperature dependence of the isobaric specific conductance of liquid $HgCl_2$ at a pressure of 5.4 kbar can, to a first approximation, be represented by the Arrhenius equation

$$K = A \exp(-E_k/RT). \qquad (2)$$

Values for the constant $A$ and of the activation energy for conduction ($E_k$) were determined empirically by the method of least squares from the $\log K$ vs $1/T$ data. $A$ and $E_k$ are $2.13 \pm 0.09$ $(\Omega\cdot\text{cm})^{-1}$ and $8.46 \pm 0.14$ kcal/mole, respectively.

### 2. $HgI_2$

The conductivity of molten $HgI_2$ was measured over the temperature range 292–620°C at a constant pressure of 5.4 kbar. The results from these measurements are shown in Fig. 5. Conductivity measurements were attempted at higher temperatures (i.e., up to 800°C). However, an irreversible change in the conductivity was found to occur at temperatures above 650°C. The electrodes were changed from Pt to W—however, without success. Below 650°C use of both types of electrodes gives essentially identical results (see Fig. 5; with sample No. 3 Pt electrodes were used; with No. 4 W electrodes were employed). Note that conductivity measurements made from 322 down to 292°C were upon a supercooled liquid phase.

Conductivity data for molten $HgI_2$ reported by Grantham and Yosim[5] at a pressure approximately equal to the salts own vapor pressure (i.e., $P < 0.1$ kbar) are also shown in Fig. 5 for comparison with these conductivity data taken at a pressure of 5.4 kbar. Here, as in the previous case with $HgCl_2$, an increase in pressure causes an increase in the conductivity of $HgI_2$ (at a given temperature). However, the ratio for the change in conductivity for a pressure change from 0.1 to 5.4 kbar is considerably less than was found in the case of $HgCl_2$. On the other hand, the conductivity of $HgI_2$ is some three orders of magnitude greater than $K$ for $HgCl_2$ at the respective melting points of these two salts at atmospheric pressure.[1–4]

An unusual result is noted in the temperature coefficient of conductivity of molten $HgI_2$ when the pressure of the salt is increased from a pressure equal to its own vapor pressure to a pressure of 5.4 kbar. At ordinary pressure the temperature coefficient of conductivity is negative.[4] At 5.4 kbar the temperature coefficient is positive and the $\log K$ vs $1/T$ (degree Kelvin) plot is linear. As in the previous case with $HgCl_2$, we have subjected these data to a least squares analysis in order to obtain the best linear fit in terms of two constants, $A$ and $E_k$, The value for $A$ is $0.36 \pm 0.04$ $(\Omega\cdot\text{cm})^{-1}$; for $E_k$ it is $2.53 \pm 0.12$ kcal/mole.

The effect of pressure upon the conductivity of molten $HgI_2$ was also examined at constant temperature over the pressure range 3 to 20 kbar (Fig. 6). These measurements were carried out at a temperature of 555±5°C. It can be seen from Fig. 2 that $HgI_2$ remains a liquid at this temperature at the highest pressure (20 kbar) used in this test. The same experimental techniques which were employed with $HgCl_2$ were also used here; however, the conductivity cell ruptured in an attempt to repressurize the test cell to 20 kbar. The value for the specific conductivity upon decompression at an indicated pressure of 5.4 kbar is $9 \times 10^{-2}$ $(\Omega\cdot\text{cm})^{-1}$. This is in fair agreement with the conductivity obtained in the isobaric measurement at this same temperature of 555°C [from Fig. 3, $K = 8 \times 10^{-2}$ $(\Omega\cdot\text{cm})^{-1}$ at this temperature]. The conductivity at 5.4 kbar upon this decompression cycle agrees with the initial value obtained for the conductivity of this same sample made at a pressure of 5.4 kbar before this sample was carried to a pressure of 20 kbar. On this basis it appears that the effect upon the conductivity of molten $HgI_2$ is reversible over the pressure interval 3 to 20 kbar.

## IV. DISCUSSION

The high vapor pressure, low viscosity, and low specific conductance indicate that the molten mercuric halides $HgCl_2$, $HgBr_2$, and $HgI_2$ are largely molecular or

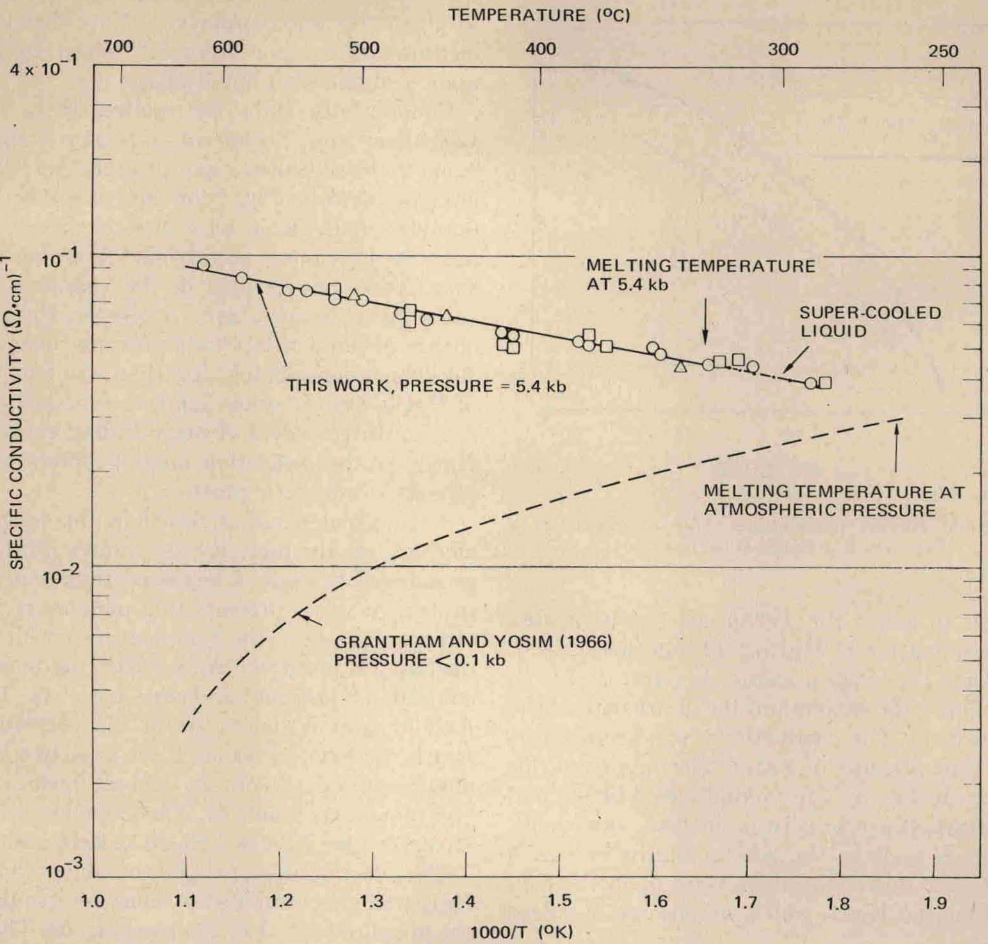

FIG. 5. Log$K(\Omega\cdot\text{cm})^{-1}$ vs $1/T$ (°K) for $HgI_2$. (○) Sample No. 3, Run No. 3; (□) Sample No. 3, Run No. 4; (△) Sample No. 4, Run No. 1. Pressures given in kbars.

associated at ordinary pressures.[18] Janz and McIntyre[14] have suggested that the species in the molten state of these compounds may involve an equilibrium between molecular and ionic species such as

$$2HgX_2 \rightleftarrows HgX^+ + HgX_3^-. \qquad (3)$$

The low electrical conductivity of these molten salts indicates that they are only slightly dissociated; the order of decreasing conductance, iodide>bromide>chloride, reflects the relative degree of dissociation.[3]

The temperature coefficient of conductivity of a liquid in which an equilibrium such as (3) is neither far to the left (molecular) nor far to the right (strong electrolyte) might be expected to exhibit a complicated behavior since a change in temperature might also shift this equilibrium and thus the number of the ionic species available for the conduction process in addition to the usual effect of temperature upon the mobility of the conducting species.

Grantham and Yosim have indeed observed a maximum in the temperature coefficient of electrical conductivity of several salts.[5–7] In such a case, this maximum has been observed in the $K$ vs $T$ curve when the electrical conductivity was measured under the conditions where the molten salt was allowed to expand freely as the temperature was increased.[19] (The salt was under a pressure closely approximating its own vapor pressure in these measurements carried out by Grantham and Yosim.[5,7,9]) Yosim[20] has pointed out that it would be advantageous to measure the electrical conductivity of such systems wherein the liquid kept at constant volume. However, neither the experimental apparatus nor the necessary $PVT$ data are currently available with which to carry out such constant volume conductivity measurements over an extended temperature range, i.e., as much as 500°C above the normal melting point. As an alternative, the electrical conductivity of the molten state of these relatively low

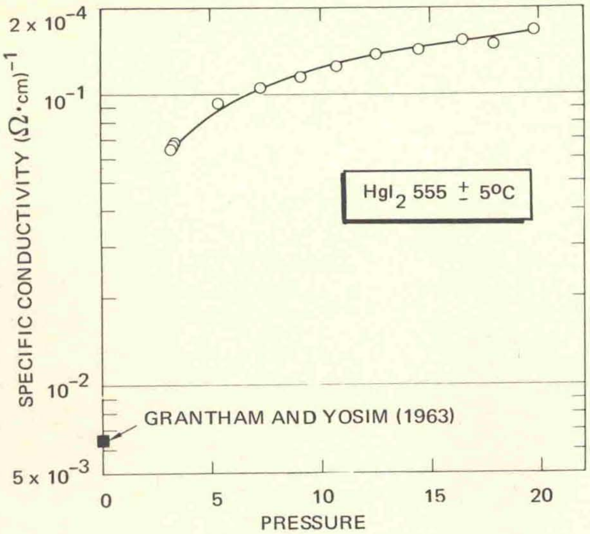

FIG. 6. Isothermal specific conductance ($K$) of $HgI_2$ as a function of pressure (kbar).

conducting salts has been measured over a large temperature interval (~500°C) at a constant, but high, pressure of 5.4 kbar.

The conductivity data at these elevated pressures obtained by Darnell *et al.* on the bismuch trihalides[8] and upon the molten compounds of $HgCl_2$ and $HgI_2$ reported here would tend to support the suggestion made by Grantham and Yosim[5] that these systems become more molecular with decreased density when heated at ordinary pressures. The increase in conductivity with pressure at constant temperature observed for these molten salts makes this idea attractive. Schlichtärle, Töheide, and Franck[21] have shown on the other hand, that the electrical conductivity of the "strong" electrolyte $NaNO_3$ decreases with an increase in pressure. One might expect that this decrease in conductivity with increasing pressure is due to a decreased mobility of the ions of this strong electrolyte and not due to a decrease in the number of ionic carriers. In the case of $BiX_3$ and $HgX_2$ however, such a decrease in ionic mobility with an increase in pressure could easily be overcome by an increase in the number of ionic species. For example, in $NaNO_3$ an increase in pressure by 10 kbar decreases the conductivity to 0.62 of the conductivity at zero pressure. For $HgCl_2$ such an increase in pressure increases the conductivity of the liquid by a factor of 5. For $HgI_2$ this ratio is 2.5. Thus the enhancement in electrical conductivity with an increase in pressure appears to be greater for salts that are more molecular at ordinary pressures, i.e., $K_{P=10\text{kbar}}/K_{P=0}$ decreases in the sequence $HgCl_2 > HgI_2 > NaNO_3$. These data suggest that the $K$ vs $P$ curve for a weak electrolyte may perhaps exhibit a maximum at sufficiently high pressures where the two opposing pressure coefficients (increased ionization and decreased mobility) become comparable. The $K$ vs $P$ curve for $BiCl_3$ [8] suggests that such a maximum may exist.

Thus these results show that pressure brings about a large change in the electrical conductivity of the mercuric halides, $HgCl_2$ and $HgI_2$. For example, in the former case an increase in pressure from near atmospheric to 20 kbar brings about an increase in conductivity of over three orders of magnitude. In the case of $HgI_2$, pressurization to 5 kbar changes the sign of the temperature coefficient of conductivity from its anomalous negative value at zero pressure to a positive temperature coefficient whose Arrhenius activation energy for conduction is comparable to that of a strong electrolyte, i.e., 2.5 kcal/mole.

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