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GALLIUM SESQUIOXIDE USING A CALCIA-STABILIZED ZIRCONIA ELECTROLYTE

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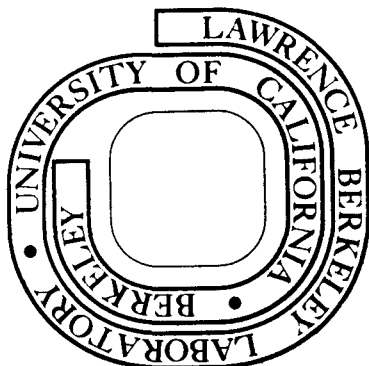
SOLID-STATE ELECTROCHEMICAL STUDY OF THE FREE
ENERGY OF FORMATION OF β -GALLIUM SESQUIOXIDE USING A
CALCIA-STABILIZED ZIRCONIA ELECTROLYTE

T. J. Anderson and L. F. Donaghey

August 1975

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Solid-State Electrochemical Study of the Free Energy of
Formation of β -Gallium Sesquioxide Using a
Calcium-Stabilized Zirconia Electrolyte

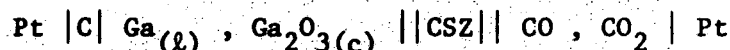
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August, 1975

Abstract

The free energy of formation of $\beta - \text{Ga}_2\text{O}_3(\text{c})$ was measured using a high-temperature, solid-state electrochemical cell utilizing calcium-stabilized zirconia as the solid electrolyte and a $\text{CO} - \text{CO}_2$ gaseous mixture as the reference electrode. The Nernst voltage of the cell,



was measured over the temperature range from 775 to 1100°K. The standard free energy of formation, $\Delta G_f^{\circ}(\beta - \text{Ga}_2\text{O}_3(\text{c}))$, was found to be $(-265,309 \pm 152) + (82.47 \pm 0.16)(T/\text{K}) \text{ cal}_{\text{th}} \text{ mole}^{-1}$. A third-law comparison to previous data is presented.

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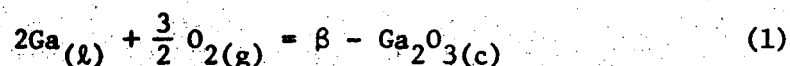
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Short Title: Free Energy of Formation of Gallium Sesquioxide

Introduction

Gallium has become important in the semiconductor microelectronics industries as a component of compounds for electroluminescent and microwave devices. Gallium is also an important solvent for liquid-phase epitaxial crystal growth of compound semiconductors where oxygen is an electrically active impurity. Gallium sesquioxide added to Ga - GaP solutions has been used to achieve high electroluminescence efficiency in Zn-doped GaP light-emitting diodes. (1-4) Effective design of semiconductor device processes involving gallium should be aided by accurate thermodynamic data for gallium oxide.

The Gibbs free energy of formation of $\beta - \text{Ga}_2\text{O}_3$ by the reaction



has been estimated by Caughlin (5) and measured using solid-state electrochemical methods by Klinedinst and Stevenson (6) and Smith and Chatterji. (7) Pankratz and Kelley determined the high temperature heat content of $\beta - \text{Ga}_2\text{O}_3$ by calorimetric methods. (8) More recently Mills measured the enthalpy of formation of $\beta - \text{Ga}_2\text{O}_3$ by drop calorimetry. Unfortunately, the Gibbs free energies of formation as determined in the above studies show marked discrepancies, especially in the temperature dependence, i.e., the standard entropy of formation. In this study the Gibbs free energy of formation of $\beta - \text{Ga}_2\text{O}_3$ was measured in a high-temperature, solid-state electrochemical cell under microcomputer control.

Experimental

The Experimental Cell

The basic, solid-state galvanic cell design is depicted in figure 1. The cell consisted of a 2-inch diameter, closed-end alumina tube 18 inch in length secured by a Viton o-ring to a water-cooled, brass cell-head. Three 1/8-inch cajon through-bore fittings were located on the cell head symmetrically around a 1/4 inch cajon through-bore fitting. The 1/8-inch thermocouples and lead wires in ceramic sheaths were passed through the 1/8-inch fittings. The larger, centered fitting was used to seal to a 1/4-inch calcia-stabilized (CSZ) electrolyte tube, closed at the bottom.

Included in the brass head were bores for gas entrance and exit. The exit placement was such that the gas had to flow downward to the experimental cell, then through an alumina pushrod. Three spring-loaded tungsten wires provided support for a graphite chamber containing liquid gallium and $\beta - \text{Ga}_2\text{O}_3$ in contact with the CSZ tube.

The electrode materials were contained in a 2-inch diameter closed graphite crucible fitted with a screw cap containing a closely fitting hole through which the CSZ tube was inserted into the gallium electrode. Powdered graphite was added to seal the CSZ tube to the graphite lid. Details of electrode compartments are shown in figure 2.

The cell was heated with a Marshall resistance-heated furnace 20 inch in length with a 2 1/2 inch bore. Excellent temperature control was furnished by an integrating, temperature-regulated, power supply designed and built at the Lawrence Berkeley Laboratory.

Temperature uniformity and stability was held to within $\pm 0.5^\circ\text{C}$ in the vicinity of the electrodes. Stray cell voltages induced by the temperature regulation circuit were eliminated by a metal ground shield placed around the 2-inch alumina tube. Type-k thermocouples (0.020 inch Pt - Pt 10%Rh) were calibrated in the cell under vacuum against a thermocouple calibrated at the National Bureau of Standards. The calibration was performed with the standard thermocouple occupying the position normally that of the electrode, while the unknown thermocouple was placed in their normal operating positions. Thermocouple emf's were amplified using a x100 thermocouple amplifier calibrated with a Leeds and Northrup Type K-3 potentiometer facility.

Cell emf's were measured with a Keithley 640 electrometer. High-isolation triaxial cable was used to connect the experimental cell to the input head of the electrometer. The inner conductor of the triaxial cable was connected to the positive lead wire of the cell. The outer conductor was driven by the unity-gain feedback from the electrometer, thus acting as a driven guard. Leakage sources at the input head were negligible since the cable was guarded up to the sapphire insulated electrometer contact.

Materials

Gallium metal used in the study was semiconductor grade, 99.9999% pure, obtained from Cominco American. Gallium oxide was obtained from Alfa Products, and had a purity of 99.99%. The oxide was confirmed to be $\beta - \text{Ga}_2\text{O}_3$ and to show no detectable second phases by X-ray diffraction analysis.

The calcia-stabilized zirconia tubes containing 7.5 weight per cent calcia were obtained from the Zirconia Corporation of America. The tubes were confirmed to be leak tight both before and after the experiments. Spectroscopic-grade graphite was employed in the electrode compartment containing the liquid metal and a small amount of the oxide. Platinum lead wires were attached to the graphite compartment and to a platinum electrode made from Englehard unfluxed platinum paste (No. 6926) inside the CSZ tube end.

The CO - CO₂ reference electrode gas mixture was obtained from Matheson Gas Products. Chemical analysis showed the mixture to contain (25.29 ± 0.02) per cent CO₂ and (2.45 ± 0.02) per cent CO in high-purity argon.

High-purity argon was prepared by passing it over Linde 4A molecular sieve at 195°K, then through a special, alumina-lined titanium-sponge purifier heated to 800°C. This gas was used as an inert purge gas for the metal-oxide electrode compartment, with the purge flow rate adjusted to 1 cm³/sec.

Reagent dibutyl phthalate was used in gas exhaust traps to prevent back-diffusion of oxygen into the experimental cell.

Microcomputer Control System

Operation of the electrochemical experiments and data acquisition were automated with a microcomputer system based on an Intel 8008 microprocessor. The facility is schematically shown in figure 3. The system had the capability of setting the cell operation temperature and monitoring cell emfs and thermocouple outputs on a predetermined

time base until equilibrium had been established, thus allowing continuous cell operation without operator assistance.

A 4 1/2 digit panel meter alternately displayed the cell voltage and temperature, while a permanent record was obtained on a teletype. The microcomputer was constructed from components stocked at the Lawrence Livermore Laboratory.

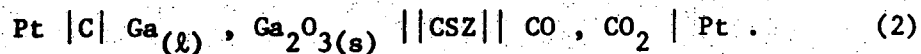
Procedure

In each of several experiments the cell compartments were leak-checked using a high vacuum system, then purged with high purity argon. The cell temperature was programmed to rise at 100°K/hour to the lowest measurement temperature, after which the reference gas flow was initiated. The cell temperature was then sequentially incremented and decremented throughout the experimental range, with the cell voltage measured and periodically recorded at each temperature until equilibrium was reached and confirmed. Reference gas and argon flow rates did not affect the cell voltage in the ranges selected. At the conclusion of each experiment the cell compartments were again checked for leaks and the metal electrode examined for evidence of oxidation. The electrode material was then analyzed by X-ray diffraction to confirm the absence of side-reaction products.

Results

The solid-electrolyte galvanic cell employed in measuring the free energy of formation of $\beta - \text{Ga}_2\text{O}_3$ can be represented schematically as

3 7 5 8 0 2 4 0 0 0 0



The equilibrium partial pressures of oxygen in the two electrodes are related to the cell potential E , by the Nernst equation:

$$E = (RT/4F) \ln \{P(\text{O}_2, \text{CO} + \text{CO}_2) / P(\text{O}_2, \text{Ga}_{(\ell)}, \beta - \text{Ga}_2\text{O}_3)\} \quad (3)$$

In this equation, $P(\text{O}_2, \text{CO} + \text{CO}_2)$ indicates the oxygen fugacity of the $\text{CO} - \text{CO}_2$ gas mixture, $P(\text{O}_2, \text{Ga}_{(\ell)}, \beta - \text{Ga}_2\text{O}_3(\text{s}))$ represents the $\text{Ga} + \text{Ga}_2\text{O}_3$ mixture, R is the gas constant, T is the thermodynamic temperature and F is the Faraday constant.

The measured cell emf differs from the equilibrium cell potential by the thermal emf generated by the $\text{Pt} - \text{C}$ couple. This thermal emf was measured by Chatterji and Smith⁽¹⁰⁾ and found to vary nearly linearly with temperature in the range investigated. The measured cell voltage $E_{(m)}$ was corrected by the following equation for the equilibrium cell voltage,

$$E = (E_{(m)}/\text{mV}) - 3.7 + .007(T/\text{K})\text{mV} \quad (4)$$

The Gibbs free energy of formation of $\beta - \text{Ga}_2\text{O}_3$ is related to the oxygen partial pressure over the $\text{Ga} + \text{Ga}_2\text{O}_3$ mixture by the equilibrium constant for the formation reaction:

$$K_f(\beta - \text{Ga}_2\text{O}_3) = a(\beta - \text{Ga}_2\text{O}_3) / a(\text{Ga})^2 P(\text{O}_2, \beta - \text{Ga}_2\text{O}_3, \text{Ga})^{3/2}$$

or

$$K_f(\beta - \text{Ga}_2\text{O}_3) = \exp \{-\Delta G_f^0(\beta - \text{Ga}_2\text{O}_3) / RT\} . \quad (5)$$

Equations 3 and 5 allows one to calculate $\Delta G_f^0(\beta - \text{Ga}_2\text{O}_3)$ from the measured temperature and experimental cell voltage:

$$\Delta G_f^0(\beta - \text{Ga}_2\text{O}_3) = 1.5 RT \ln P(\text{O}_2, \text{CO} + \text{CO}_2) - 6FE . \quad (6)$$

Here, unit activities for $\text{Ga}_{(l)}$ and $\beta - \text{Ga}_2\text{O}_{3(s)}$ in the mixture were assumed.

The reference oxygen partial pressure was established by the equilibrium reaction,



and is related to the Gibbs free energy change for this reaction, $\Delta G_{(7)}$, by:

$$P(\text{O}_2, \text{CO} + \text{CO}_2) = \left(\frac{P(\text{CO}_2)}{P(\text{CO})} \right)^2 \exp \{2\Delta G_{(7)} / RT\} \quad (8)$$

where $\Delta G_{(7)} = -68,270 - 0.18T \ln T - 0.34 \times 10^{-3} T^2 + 0.87 \times 10^5 / T + 23.28T$, $\text{cal}_{\text{th}} \text{mol}^{-1}$ was taken from the equations given by Wicks and Block⁽¹¹⁾

for the free energies of formation of CO and CO₂. The ratio of the partial pressure of CO₂ to the partial pressure of CO in the primary standard gas was $10.322 \pm .092$.

Table 1 shows the measured open-cell potentials, partial pressures of oxygen in the reference gas mixture, the calculated equilibrium

7 7 5 8 0 2 7 0 0 0 0

Table 1 Summary of Experimental and Derived Data

T/K	$E_{\text{measured}}, \text{mV}$	$\log_{10} \frac{P(\text{O}_2, \text{CO} + \text{CO}_2)^*}{\text{atm}}$	$\log_{10} \frac{P(\text{O}_2, \text{Ga} + \text{Ga}_2\text{O}_3)}{\text{atm}}$	$E_{\text{corrected}}, \text{mV}$	$\Delta G_f^\circ(\beta\text{-Ga}_2\text{O}_3), \text{cal}_{\text{th}} \text{mol}^{-1} \dagger$
776.9	421.1	-26.78	-37.75	422.8	-201,320.
815.1	418.4	-25.00	-35.40	420.4	-198,020.
855.4	416.5	-23.29	-33.16	418.8	-194,670.
902.3	415.4	-21.49	-30.83	418.0	-190,930.
933.4	414.0	-20.40	-29.40	416.8	-188,360.
967.9	412.4	-19.27	-27.92	415.5	-185,510.
1004.4	410.9	-18.16	-26.48	414.2	-182,520.
1039.9	409.1	-17.16	-25.16	412.7	-179,570.
1044.7	408.5	-17.03	-24.98	412.1	-179,110.
1048.8	408.5	-16.92	-24.84	412.1	-178,800.
1051.1	407.9	-16.86	-24.74	411.6	-178,550.
1059.5	408.0	-16.63	-24.47	411.7	-177,920.
1091.2	406.9	-15.82	-23.41	410.8	-175,350.

* atm = 101 325 Pa

† cal_{th} = 4.184 J

Table 2 Comparison of Reported Standard Enthalpy and Entropy of Formation of β - Ga_2O_3

ΔH° $\frac{\text{Kcal}}{\text{mol}^{-1}}$	ΔS° $\frac{\text{Kcal}}{\text{mol}^{-1}\text{K}^{-1}}$
-252.4	.0702 ^a
-252.5	.0711 ^b
-259.34	.07811 ^c
-265.31	.08247 ^d

a From reference 6, obtained by titration.

b From reference 6, phase equilibrium.

c From reference 7.

d From this work.

The $\Delta G_f^\circ(\beta - \text{Ga}_2\text{O}_3)$ values calculated in the present study from the experimental data showed excellent internal consistency with a standard error at 1000°K of only $\pm .312$ Kcal/mole . On comparing the results with those obtained from other emf measurements, however, one finds an apparent discrepancy in both the standard enthalpy and entropy. In particular, the standard entropy found in this work was 17.5% above that of Klinedinst and Stevenson's result (see table 2). On the other hand, the absolute values of $\Delta G_f^\circ(\beta - \text{Ga}_2\text{O}_3)$ show relatively good agreement but differ markedly in the temperature dependence as shown in figure 4.

Because of differences in the results of this study as compared to results of previous emf studies and also of the availability of good colorimetric data, a "third law" calculation was carried out. The results of this calculation are shown in figure 5. The values of $\Delta H_{f,298.15}^\circ$ for the $\beta - \text{Ga}_2\text{O}_3$ formation reaction was calculated from the following equation:

$$\begin{aligned} \Delta H_{f,298.15}^\circ &= \Delta G_{f,T}^\circ - \Delta(H_T^\circ - H_{298.15}^\circ) \\ &+ T(\Delta S_{f,298.15}^\circ + \Delta(S_T^\circ - S_{298.15}^\circ)) \end{aligned} \quad (10)$$

where $\Delta G_{f,T}^\circ$ is the experimental value of the Gibbs free energy of formation of $\beta - \text{Ga}_2\text{O}_3$. The values of $H_T^\circ - H_{298.15}^\circ$ and $S_T^\circ - S_{298.15}^\circ$ for $\text{O}_2(\text{g})$ were taken from Wicks and Block,⁽¹¹⁾ that for $\text{Ga}_{(l)}$ from Hultgren,⁽¹²⁾ and that for $\beta - \text{Ga}_2\text{O}_3(\text{c})$ from Mills.⁽⁹⁾ A value for $\Delta S_{f,298.15}^\circ$ of -72.87 cal/mole°K was determined

from the data on $S_{298.15}^{\circ}$ for O_2 taken from Kelley⁽¹³⁾ and on Ga and $\beta - Ga_2O_3$ from Adams, Johnston and Kerr.⁽¹⁴⁾ The calculated results for $\Delta H_{298.15}^{\circ}$ are compared to the data of Mah,⁽¹⁵⁾ determined by direct combustion calorimetry, and to the value of Shchukarev, Semerov and Rat'kovskii⁽¹⁶⁾ obtained by isothermal evaporation studies. The close agreement between the data derived from this study and the findings of the latter two different methods tend to support the experimental value for $\Delta H_{298.15}^{\circ}$ of $-261.9 \text{ kcal}_{th} \text{ mol}^{-1}$.

Assuming all calorimetric data to be correct, the emf results would be expected to give calculated $\Delta H_{298.15}^{\circ}$ values which fall on a horizontal line in figure 5 at the value of the calorimetrically determined result. As seen in figure 4, the results of Klinedinst and Steveson⁽⁶⁾ exhibit a large temperature dependence, with the average lying somewhat above the calorimetrically determined value. In their reported results, no mention was made of correcting for the thermal emf developed by the W - Pt junction on the metal-metal oxide electrode. Chatterji and Smith⁽¹⁰⁾ measured this potential and found it sizeable (i.e., 15.6 mV at 1000°K). Adding these corrections to the data of Klinedinst and Stevenson would have the effect of flattening the calculated $\Delta H_{298.15}^{\circ}$ values but would also raise them (i.e., 1.515 Kcal/mole at 873°K and 3.541 Kcal/mole at 1273°K for the two-phase mixture).

Also shown in figure 4 are the emf results of Smith and Chatterji.⁽⁷⁾ The actual data was not available for this calculation. Instead, the fitted linear equation was used. The use of this equation gave nearly

temperature-independent results, but shifted to approximately 1.75 Kcal above the experimental value. Smith and Chatterji reported an error range of 0.8 Kcal/mole with no apparent temperature dependence from a similar third-law calculation on their data.

The calculated results of this investigation gave the value of $\Delta H_{298.15}^{\circ}$ reported to within the experimental error for all data points, but produced a small temperature dependence. This could be accounted for by a systematic error in our experimental data or by inaccurate specific heat data. The specific heat data for O_2 should be very accurate and the more recent calorimetric data of Mills⁽⁹⁾ agreed to within 1% of that of Pankratz and Kelley.⁽⁸⁾ The most likely source of error is in the specific heat of gallium, for which a constant heat capacity was assumed above 700°K.

A potential source of error in the present study was electronic conduction in the CSZ electrolyte. Shown in figure 2 are conservative and liberal lower oxygen partial pressure limits to the electrolytic domain of CSZ as derived from the data of Schmalzreid⁽¹⁷⁾ and Patterson, Bogden and Rapp,⁽¹⁸⁾ along with the free energies of formation of Ga_2O_3 and other oxides.⁽¹⁹⁾ The conservative limit places the $Ga_2O_3 - Ga - O_2$ equilibrium outside the electrolytic domain. The more recent and liberal limit places the most stable equilibrium of interest well within the limits of operation. In this study the working temperature was maintained below 1100°K in order to prevent conditions allowing electronic conduction in the solid electrolyte. The fact that the EMF data did not show a change in the temperature dependence at high temperatures is evidence that the electrolyte was

maintained in the ionic conduction domain. The high ionic conductivity of CSZ as the solid electrolyte aids in preventing electrode-polarization errors which could more easily occur with YDT, used in the study by Klinedinst and Stevenson. (16)

Regardless of the error source, the direct measurement of $\Delta G_f^\circ(\beta - \text{Ga}_2\text{O}_3)$ performed in this study showed much better consistency with the calorimetric data than did the data from earlier emf studies, when compared with "third law" calculations.

Acknowledgment

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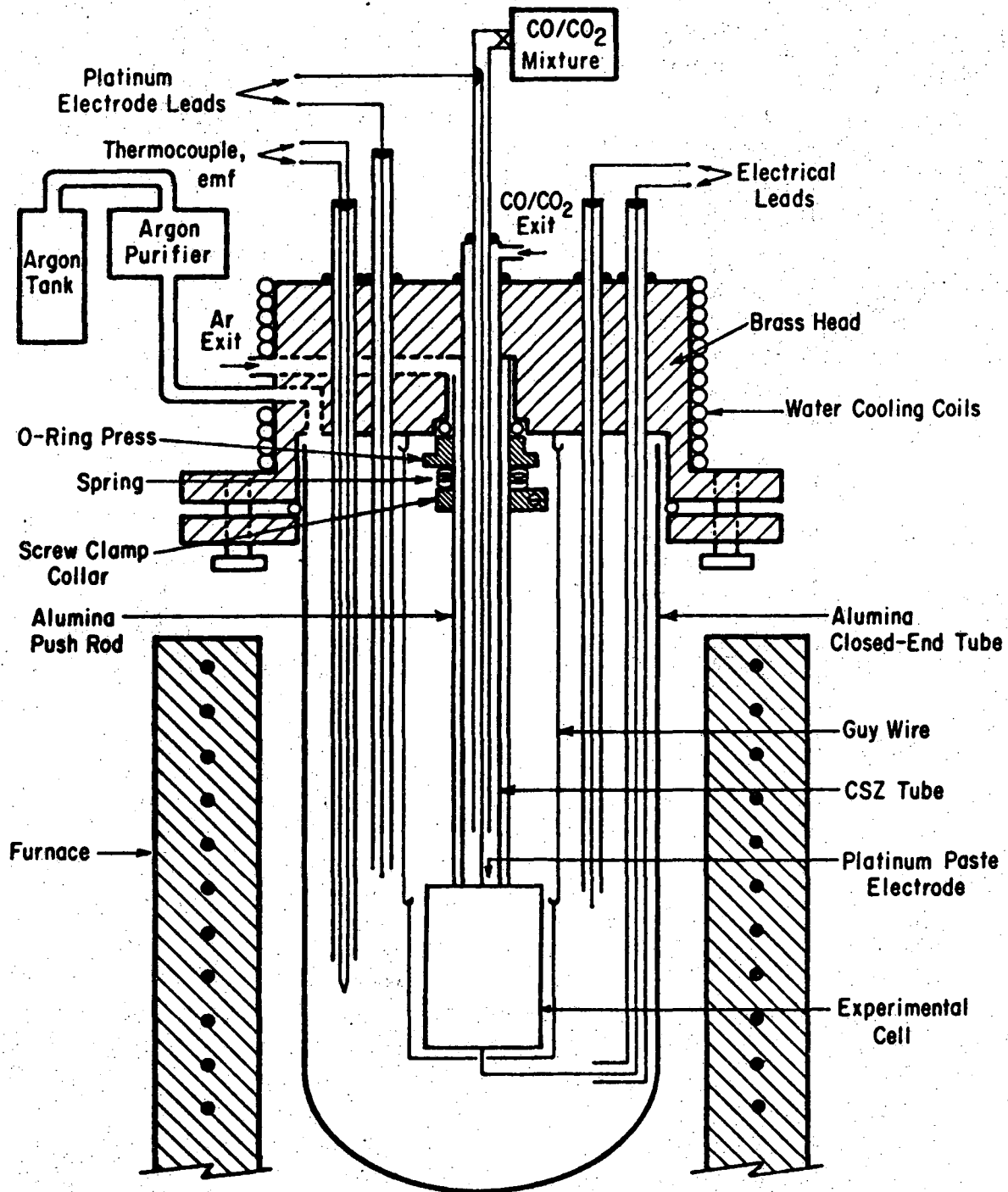
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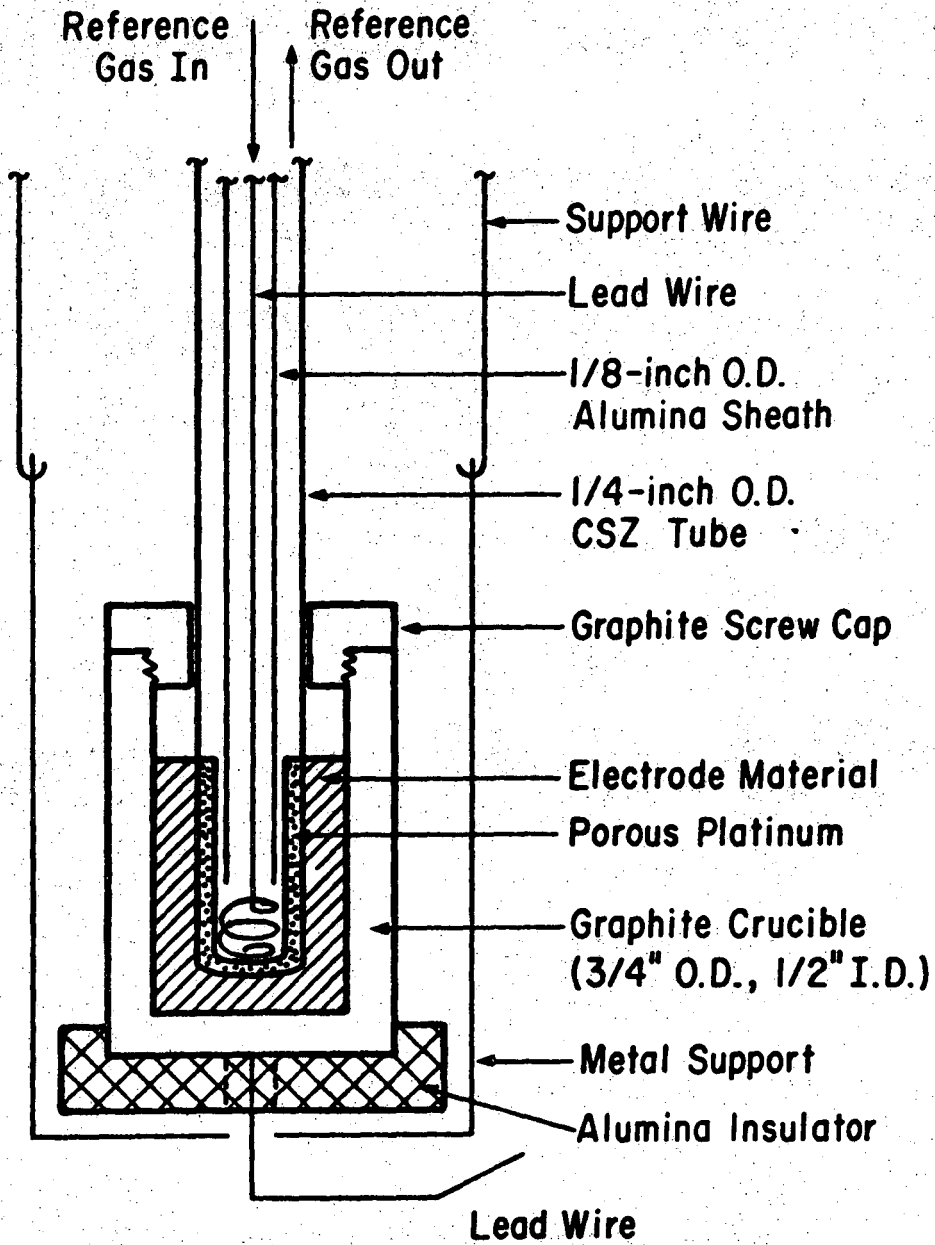
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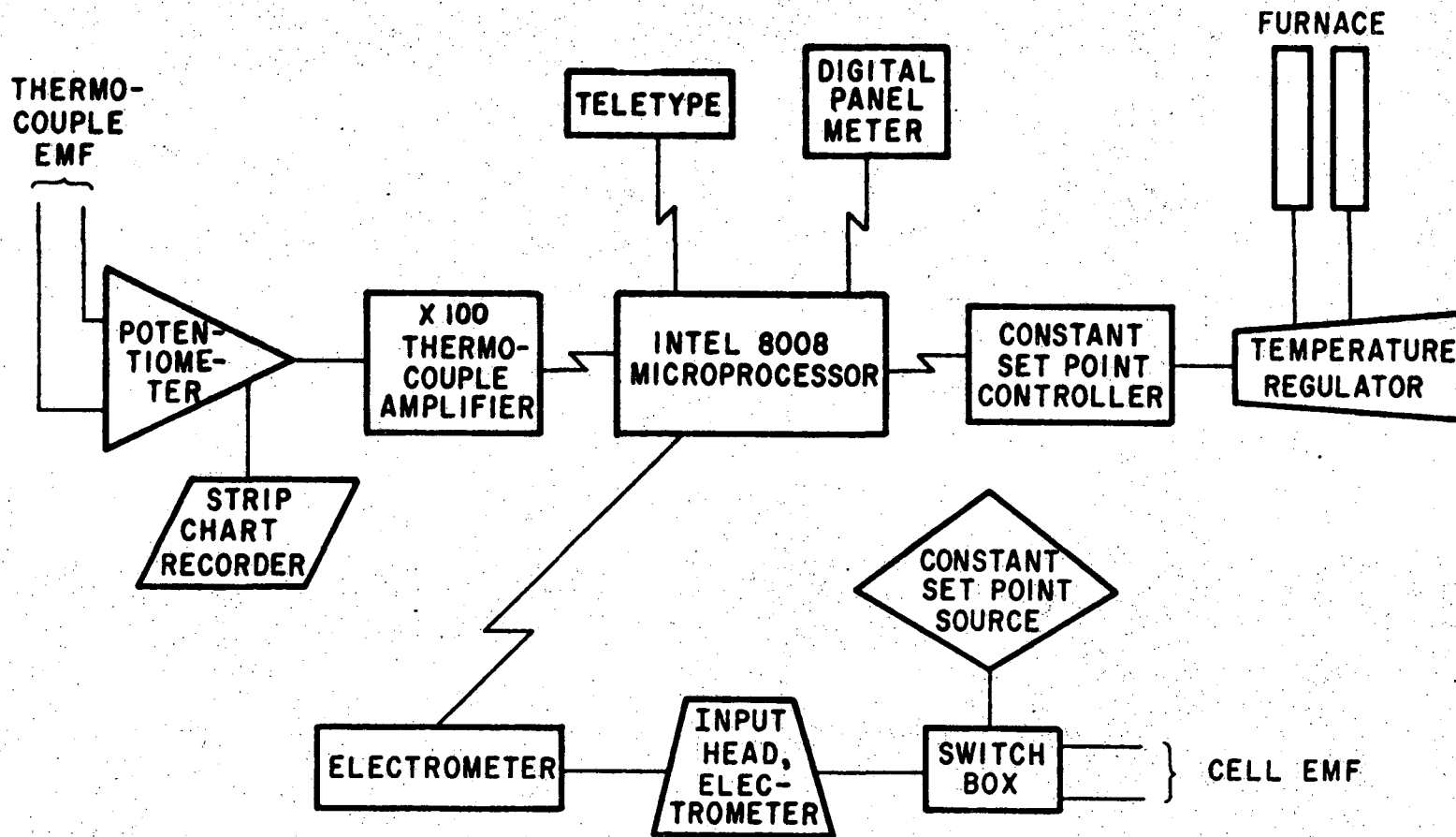


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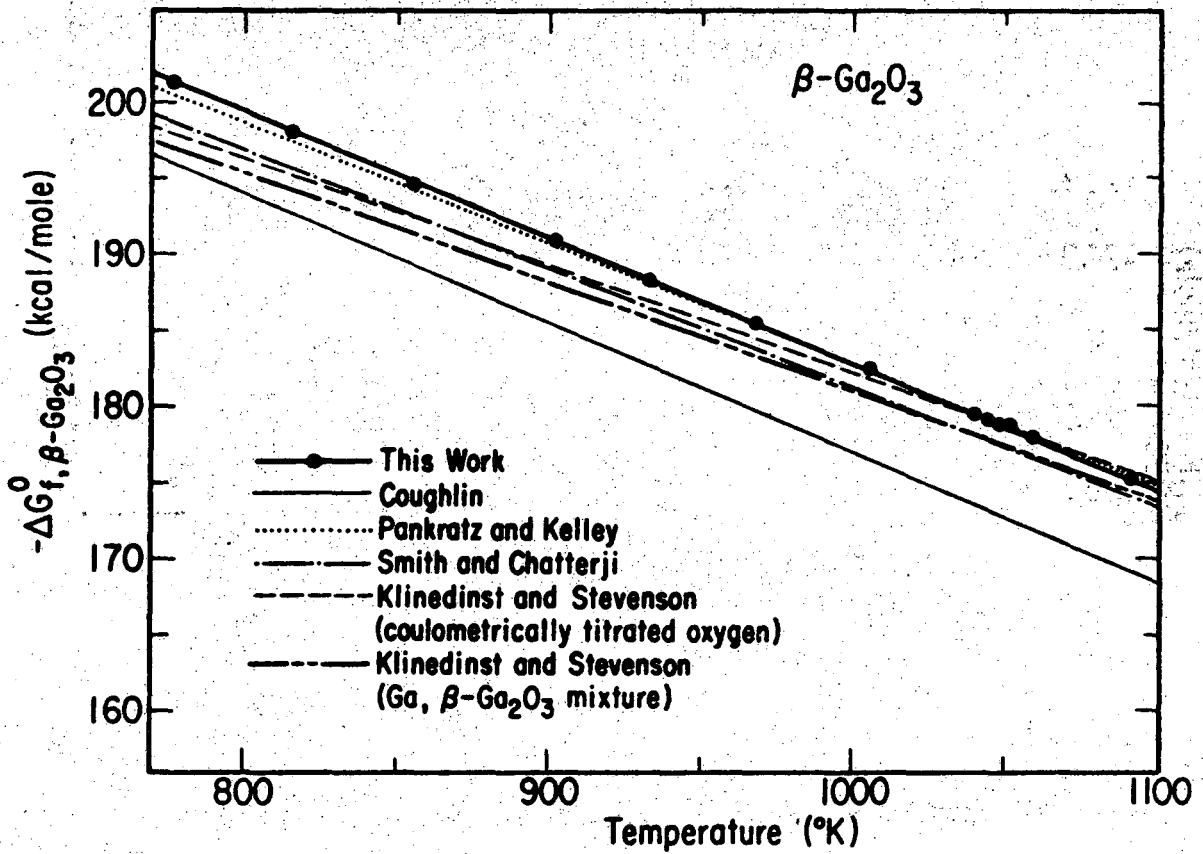
Fig. 1



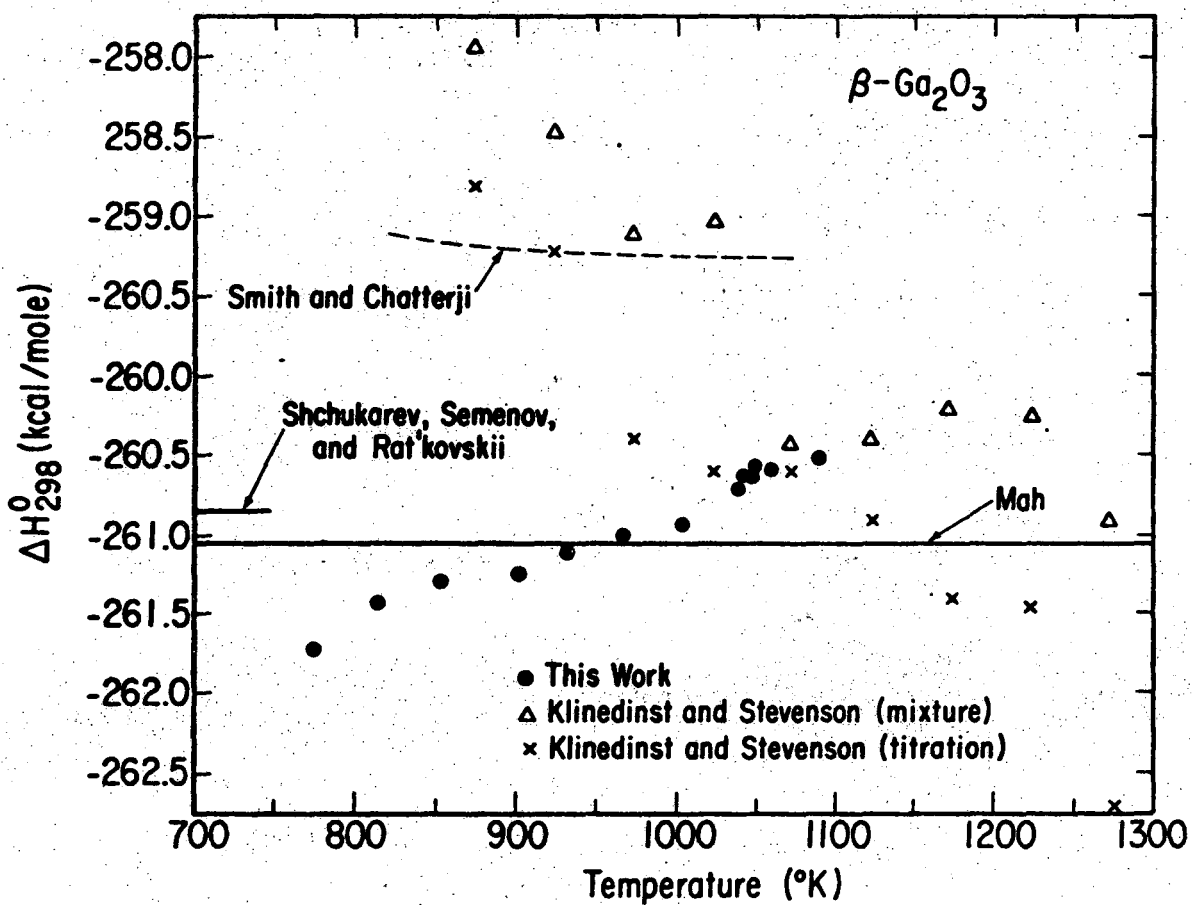
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Fig. 2



XBL 757-6781
Fig. 3

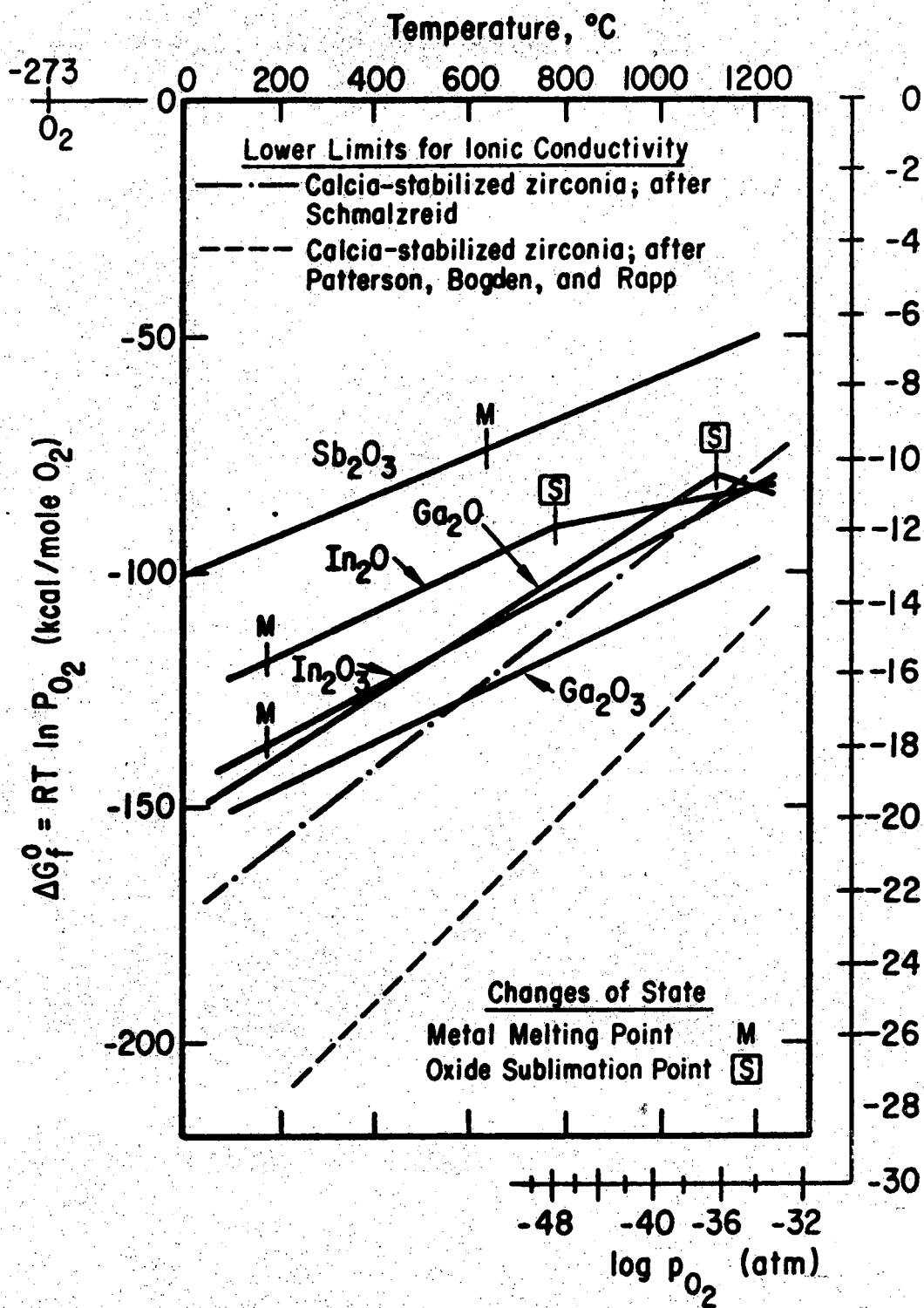


XBL 757-6775
Fig. 4



XBL 757-6774

Fig. 5



XBL 757-6780

Fig. 6

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