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ESTIMATED GAS COMPOSITIONS IN EQUILIBRIUM WITH CONDENSED PHASES OF THE Cs-O SYSTEM

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SUMMARY

Thermionic energy converters with a single additive reservoir furnishing both cesium and oxygen appear promising. In this report, theoretical estimates were made of the relation between the composition of the vapor above the reservoir at various temperatures and the oxygen concentration of the condensate. For example, at 740 K, an oxygen atom fraction of about 0.13 in the condensate corresponded to a cesium/oxygen ratio of about 10^6 in the vapor. The utility of the results is limited by the large uncertainties in the thermal data employed in the analysis.

INTRODUCTION

Compounds and solutions of oxygen with cesium have been proposed as additives for thermionic diodes. The cesium performs its usual role of charge neutralization and lowering of the electrode work functions. The oxygen increases the binding energy of cesium to the refractory metal electrode surface, resulting in lowered interelectrode gas pressures and improved performance.

The binding energy of oxygen to refractory metals is much higher than that of cesium adsorbed on either bare or oxygenated refractory metals. For this reason, the desired ratio of cesium to oxygen in the gas phase has been estimated from observations of the desorption rates of the two species to be about 10^6. The concentration of oxygen in a reservoir containing cesium and oxygen which will provide this ratio is of interest to experimenters. An additional concern is to avoid gaseous oxygen concentrations which will cause corrosion of diode parts.

The preliminary analysis herein is an effort to generate guidelines for the preparation of condensed cesium-oxygen mixtures having desirable cesium/oxygen ratios. The computations, based on the very limited available thermodynamic data, must be considered as tentative.

ANALYSIS AND RESULTS

The Phase Diagram for the Cs-O System

Figure 1 is a phase diagram for the Cs-O system reproduced from reference 1. Some information required for the present study can be deduced from this diagram. Of particular interest is the region marked A. This region consists of a solution containing cesium and oxygen. For the purposes of this analysis the solution is considered to be that of atomic O in Cs liquid. An equally valid assumption would be that Cs_2O is dissolved in Cs liquid. However, difficulties were encountered in constructing an analysis based on the latter view, and this approach has not been pursued. Region A is therefore regarded as a Cs-O solution.

In region B, crystalline cesium is in equilibrium with the Cs-O solution. In region C, crystalline Cs_2O is in equilibrium with Cs-O solution. The melting point of Cs_2O, 490 C or 763.15 K, is at the apex of region C, off-scale on figure 1. Observe that the composition of Cs_2O in figure 1 was incorrectly placed at 32 atomic percent oxygen by the authors of reference 1.

If the solution behavior is ideal, following Raoult's law, the integrated Clausius-Clapeyron equation can be used to determine the heat of fusion ΔH_f of a pure solvent from the suppression of its freezing point by a solute (ref. 2):

$$\Delta H_f = \frac{-RT_0T}{T_0 - T} \ln x_{solv} \tag{1}$$

Here, T_0 is the freezing point of pure solvent in degrees K and R, the universal gas constant, is 1.987 cal/mole K while T is the freezing point after the atom fraction of solvent, x_{solv}, has been reduced from

1.0 by addition of solute. The freezing temperature of cesium is 301.8 K (ref. 3). At $x_{Cs} = 0.908$ or $x_O = 0.092$, on the approximately linear phase boundary between regions A and B, T = 271.15 K. The computed ΔH_f is thus 512 cal/mole Cs, while the measured value is 520 cal/mole (ref. 3). Thus the conclusion is reached that the Cs-O system behaves nearly ideally at low oxygen concentrations in accordance with Raoult's law (ref. 2).

If the constituents of the atmosphere above Cs-O solution behave ideally, Raoult's law for the partial pressure p_{Cs}, of monatomic cesium, Cs_1, takes the form

$$\frac{p_{Cs}}{p_{Cs}(\text{pure})} = x_{Cs} \tag{2}$$

where $p_{Cs}(\text{pure})$ is the partial pressure of Cs_1 for pure cesium. Ideal behavior of the gas phase is assumed herein. However, the wide composition range between pure Cs liquid and Cs_2O, of interest here, makes the applicability of Raoult's law doubtful.

Expressions for the variation of solvent partial pressure with solute concentration more general than Raoult's law (eq. (2)) have been proposed. One of these, due to Margules (refs. 2 and 4), will be used herein,

$$\frac{p_{Cs}}{p_{Cs}(\text{pure})} = x_{Cs} \exp\left(\frac{\beta_1}{2} x_O^2 + \frac{\gamma_1}{3} x_O^3 + \ldots\right) \tag{3}$$

where $\beta_1/2$ and $\gamma_1/3$ are parameters. An expression of similar form applies also to the solute. The determination of parameters $\beta_1/2$ and $\gamma_1/3$ are discussed in a subsequent section. Equation (3) has the feature that when x_O is sufficiently small, the behavior of the equation approaches that of Raoult's law (eq. (2)). This is consistent with the foregoing computation of ΔH_f which depends on an application of Raoult's law.

Composition of the Gas Phase in Equilibrium with Cs_2O

In order to calculate the composition of the gas phase above Cs-O solution, the gas phase composition above condensed Cs_2O was first determined. Thermochemical data for Cs_2O crystal and liquid have been calculated (ref. 5). Also available are thermodynamic tabulations for cesium crystal and liquid, and for gaseous Cs_1 and Cs_2 (ref. 3). Data for O(g) and O_2(g) are contained in reference 6 among other places. Thermodynamic functions for gaseous Cs_2O, Cs_2O_2, and CsO were computed as discussed in the appendix from data in references 7 and 8. Constituents of the gas phase were assumed to be Cs_1, Cs_2, O, O_2, CsO, Cs_2O, and Cs_2O_2. Equilibrium constants for the following reactions were computed and are tabulated in the appendix.

$Cs_2O(c, l) \rightarrow Cs_2O(g)$

$$K_1 = p_{Cs_2O} \tag{4}$$

where c denotes crystal, l denotes liquid, and g denotes gas.

$2Cs(g) + O(g) \rightarrow Cs_2O(g)$

$$K_2 = \frac{p_{Cs_2O}}{p_{Cs}^2 p_O} \tag{5}$$

$2Cs(g) + 2O(g) \rightarrow Cs_2O_2(g)$

$$K_3 = \frac{p_{Cs_2O_2}}{p_{Cs}^2 p_O^2} \tag{6}$$

$Cs(g) + O(g) \rightarrow CsO(g)$

$$K_4 = \frac{p_{CsO}}{p_{Cs} p_O} \tag{7}$$

$2Cs(g) \rightarrow Cs_2(g)$

$$K_5 = \frac{p_{Cs_2}}{p_{Cs}^2} \tag{8}$$

$2O(g) \rightarrow O_2(g)$

$$K_6 = \frac{p_{O_2}}{p_O^2} \qquad (9)$$

The vapor phase above Cs_2O was assumed to contain 2 atoms of cesium for each atom of oxygen. An equation of constraint was thus obtained:

$$p_{Cs} + 2p_{Cs_2O} + 2p_{Cs_2O_2} + p_{CsO} + 2p_{Cs_2} = 2\left(p_{Cs_2O} + 2p_{Cs_2O_2} + p_{CsO} + 2p_{O_2} + p_O\right) \qquad (10)$$

In equation (10), partial pressures replace densities because of the assumption of ideal gases. An implicit assumption herein is that the ratio of condensate to gas volumes at all times is such that the mass of material in the condensed phase far exceeds that in the gaseous phase. Thus, any operation such as temperature change involving a change in composition of the gas phase will not affect the gross composition of the condensate.

Simultaneous solution of the six equilibrium equations and the constraint equation yields the partial pressures of the seven constituents considered. Table I presents results at some temperatures of interest.

Composition of the Gas Phase in Equilibrium with Cs-O Solution

The assumption was made that parameters $\beta_1/2$ and $\gamma_1/3$ of equation (3) were temperature-dependent and could be expressed as

$$\left.\begin{aligned} \frac{\beta_1}{2} &= C + \frac{D}{T} \\ \frac{\gamma_1}{3} &= E + \frac{F}{T} \end{aligned}\right\} \qquad (11)$$

where C, D, E, and F are constants.

At $x_O = 1/3$ for temperatures above the melting point of Cs_2O, 763.15 K, p_{Cs} was required to be that over $Cs_2O(l)$ as given in table I. These data can be represented very well by the form

$$\ln \frac{p_{Cs}}{p_{Cs}(\text{pure})} = A + \frac{B}{T} \tag{12}$$

For $x_O = 1/3$, equations (3), (11), and (12) were combined and differentiated to give a relation between B, D, and F:

$$\left[\frac{\partial \ln \frac{p_{Cs}}{p_{Cs}(\text{pure})}}{\partial \frac{1}{T}}\right]_{x_O=1/3} = B = \frac{D}{9} + \frac{F}{27} \tag{13}$$

where B was found to be -11894 K.

Additional data for evaluating C, D, E, and F were obtained from the x_O, T coordinates of the boundary between regions A and C. The original phase diagram (ref. 9) used to construct figure 1 shows this boundary in its entirety from 443.15 K to the melting point of Cs_2O, 763.15 K. Some approximate coordinates which were deduced from the original diagram are given in table II.

Also required for the evaluation of C, D, E, and F is the pressure of Cs_1 above pure $Cs(l)$. This pressure, from reference 3, is shown in table III for some temperatures in the range of interest.

Using equations (3) and (11), and the data of tables I to III for 443.15, 673.15, and 763.15 K, three additional expressions involving constants C, D, E, and F were obtained. These, together with equation (13) were solved simultaneously to give

$$C = 347.9292$$

$$D = -508083.7$$

$$E = -842.8787$$

$$F = 1203114$$

An expression for p_O above Cs-O solution was derived by combining equation (3) with the Duhem-Margules equation (ref. 2) in the form

$$\left(\frac{\partial \ln p_{Cs}}{\partial \ln x_{Cs}}\right)_T = \left(\frac{\partial \ln p_O}{\partial \ln x_O}\right)_T$$

and integrating. The following expression for the oxygen partial pressure was obtained:

$$\ln \frac{p_O}{p_O(Cs_2O)} = \ln \frac{x_O}{x_O(\mathrm{ref})} - \beta_1 \left[x_O - x_O(\mathrm{ref})\right] + \frac{1}{2}(\beta_1 - \gamma_1)\left[x_O^2 - x_O^2(\mathrm{ref})\right] + \frac{\gamma_1}{3}\left[x_O^3 - x_O^3(\mathrm{ref})\right] \tag{14}$$

where $p_O(Cs_2O)$ is the atomic oxygen pressure above pure Cs_2O. Here $x_O(\mathrm{ref})$ is taken to be 1/3 for $T \geq 763.15$, the melting point of Cs_2O. Between 443.15 and 763.15 K, $x_O(\mathrm{ref})$ is the x_O coordinate of the boundary between regions A and C of figure 1. The data of table II can be used to obtain the following expression between $x_O(\mathrm{ref})$ and T along this line:

$$x_O(\mathrm{ref}) = -0.1067829 + 2.129116\times10^{-3}\ T - 4.308192\times10^{-6}\ T^2 + 2.979734\times10^{-9}\ T^3 \tag{15}$$

Using equations (3), (5), (6), (7), (8), (9), (11), (14), and (15), the data of tables I and II, and the values calculated for C, D, E, and F, the composition of the gas phase above Cs-O solution was calculated at various temperatures and x_O. The results can be expressed in the form of equivalent cesium pressure and equivalent oxygen pressure. The equivalent cesium pressure is

$$P_{Cs} = 2p_{Cs_2O} + 2p_{Cs_2O_2} + p_{CsO} + 2p_{Cs_2} + p_{Cs} \tag{16}$$

and the equivalent atomic oxygen pressure is

$$P_O = p_{Cs_2O} + 2p_{Cs_2O_2} + p_{CsO} + 2p_{O_2} + p_O \tag{17}$$

Table IV contains P_{Cs} and P_O for the gas phase of Cs-O solutions over a range of temperatures and oxygen concentrations.

From the data in table IV, the ratio P_{Cs}/P_O was computed and plotted against x_O for various temperatures in figure 2. Figure 3 shows the equivalent cesium pressure P_O from table IV plotted against x_O for various temperatures.

In figure 3, minima are observed in the curves of P_{Cs} at the higher temperatures. These minima arise as follows: at $x_O = 0$, $P_{Cs} =$ p_{Cs}(pure). With increasing x_O, p_{Cs} declines causing an initial decrease in P_{Cs}. At the same time the contribution of p_{Cs_2O} to P_{Cs} is increasing. At elevated temperatures these counter trends of p_{Cs} and p_{Cs_2O} result in minima for P_{Cs} because of the high vapor pressure for Cs_2O relative to pure Cs, a consequence of thermochemical computation not known to have been observed experimentally.

A simple application of figures 2 and 3 is the following hypothetical example. The equivalent cesium pressure P_{Cs} desired for a certain diode operating condition is 1 torr. A minimum allowable P_{Cs}/P_O is estimated to be about 10^6, dictated by considerations of corrosion and the adsorption properties of the oxygenated electrode surfaces. What Cs-O solution temperature and oxygen concentration are required? Iteration of figures 2 and 3 shows that $T \approx 740$ K and $x_O \approx 0.13$ satisfy the problem.

CONCLUDING REMARKS

The analysis contained herein is based on little factual information, some of it dating back over half a century (ref. 9). Furthermore, the use of the fugacity equations over such wide ranges of temperature and concentration is warranted only by expedience. The information generated therefore can be regarded only as a guide for experimentation.

APPENDIX - THERMOCHEMICAL PROPERTIES OF CONSTITUENTS IN THE Cs-O SYSTEM

Condensed Phases

Reference 3 presents data for the partial pressure of Cs_1 above liquid cesium, a portion of which is contained herein in table III. Equations for free energies of formation ΔF_T^o of Cs_2O from Cs and O_2 are from reference 5. In reference 5 the standard state of O_2 was taken to be $O_2(g)$. The standard state of Cs was taken to be solid below 301.5 K (melting point), liquid from 301.5 to 963 K (boiling point) and gas above 963 K. A more recent computed value of the boiling point of Cs is 937.15 K (ref. 3). The constants of the ΔF_T^o equations in reference 5 pertaining to the region of boiling Cs were adjusted slightly to the 937.15 K boiling point. From these ΔF_T^o equations, F_T^o was computed for condensed Cs_2O using thermal data for Cs from reference 3 and for O_2 from reference 6. This F_T^o was used with data for $Cs_2O(g)$ described below to determine K_1.

Gas Phase

The constant K_5 for the equilibrium between $Cs_1(g)$ and $Cs_2(g)$ is presented in reference 3. Values of K_5 for a range of temperatures appear here in table V.

The constant K_6 for the equilibrium between O and O_2 was taken from reference 6 and also appears in table V herein.

For CsO(g), values of K_4 were computed using data presented in reference 8 together with an estimated atomization energy, D_O, of 60 000 cal/mole given in reference 7 and data for Cs(g) and O(g) from references 3 and 6, respectively. Table V presents K_4.

From estimated vibrational frequencies and interatomic distances in reference 7 $(F_T^o - F_O^o)$ was computed for $Cs_2O(g)$ and $Cs_2O_2(g)$. Reference 7 also contains constant K_p for the following equilibria at 1118 K:

$$Cs_2O(g) \rightleftarrows 2Cs(g) + \frac{1}{2} O_2(g)$$

$$Cs_2O_2(g) \rightleftarrows 2Cs(g) + O_2(g)$$

Using $(F^o_T - F^o_O)$ and K_P, F^o_T for $Cs_2O(g)$ and $Cs_2O_2(g)$ were computed. From these F^o_T, and F^o_T for $Cs_2O(c)$, Cs(g), and O(g), equilibrium constants K_1, K_2, and K_3 were computed. These appear also in table V.

In the case of Cs_2O, the authors of reference 7 assumed that the molecule was straight, while subsequent studies (ref. 10) have indicated that it is bent. However, for the studies herein the linear molecule model was retained.

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TABLE I. - COMPOSITION OF GAS PHASE ABOVE CONDENSED Cs_2O AT SEVERAL TEMPERATURES

Temperature, K	Partial pressure, torr						
	Cs	O	Cs_2	O_2	CsO	Cs_2O	Cs_2O_2
443.15	1.83×10^{-11}	3.80×10^{-35}	2.92×10^{-23}	7.28×10^{-20}	3.28×10^{-18}	2.04×10^{-8}	9.13×10^{-12}
500	4.23×10^{-9}	3.17×10^{-30}	3.81×10^{-19}	9.99×10^{-17}	6.52×10^{-15}	4.59×10^{-6}	2.11×10^{-9}
600	6.86×10^{-6}	3.90×10^{-24}	1.58×10^{-13}	2.81×10^{-13}	8.39×10^{-11}	4.90×10^{-3}	3.43×10^{-6}
700	1.30×10^{-3}	8.91×10^{-20}	1.52×10^{-9}	8.24×10^{-11}	7.02×10^{-8}	6.79×10^{-1}	6.52×10^{-4}
[a]763.15	1.74×10^{-2}	1.30×10^{-17}	1.41×10^{-7}	1.38×10^{-9}	1.96×10^{-6}	7.72	8.71×10^{-3}
800	5.95×10^{-2}	1.75×10^{-16}	1.16×10^{-6}	6.44×10^{-9}	1.02×10^{-5}	2.31×10^{1}	2.97×10^{-2}
900	9.73×10^{-1}	7.00×10^{-14}	1.77×10^{-4}	2.25×10^{-7}	4.46×10^{-4}	2.74×10^{2}	4.86×10^{-1}
1000	9.51	8.25×10^{-12}	7.29×10^{-3}	3.67×10^{-6}	9.30×10^{-3}	2.13×10^{3}	4.76

[a] Melting point of Cs_2O.

TABLE II. - SOME COORDINATES OF THE PHASE BOUNDARY BETWEEN Cs-O SOLUTION AND Cs_2O CRYSTAL

[From ref. 9.]

Temperature, K	x_O
443.15	0.250
573.15	.259
673.15	.283
763.15	.333

TABLE III. - PARTIAL PRESSURE OF Cs_1 ABOVE Cs LIQUID AT VARIOUS TEMPERATURES

[From ref. 3.]

Temperature, K	Partial pressure, atm	Partial pressure, torr
443.15	3.01314×10^{-5}	2.28999×10^{-2}
500	2.94083×10^{-4}	2.23503×10^{-1}
600	5.49759×10^{-3}	4.17817
700	4.32016×10^{-2}	3.28332×10^{1}
763.15	1.18377×10^{-1}	8.99665×10^{1}
800	1.98266×10^{-1}	1.50682×10^{2}
900	6.37354×10^{-1}	4.84389×10^{2}
1000	1.59982	1.21586×10^{3}

TABLE IV. - EQUIVALENT CESIUM PRESSURE P_{Cs} AND EQUIVALENT OXYGEN PRESSURE P_O FOR Cs-O SOLUTIONS

Temperature, K	Oxygen concentration, x_O, torr									
	0.02		0.05		0.10		0.15		0.20	
	P_{Cs}	P_O	P_{Cs}	P_O	P_{Cs}	P_O	P_{Cs}	P_O	P_{Cs}	P_O
443.15	---------	----------	3.74×10^{-3}	7.58×10^{-50}	4.56×10^{-5}	1.00×10^{-29}	1.70×10^{-7}	2.25×10^{-17}	9.21×10^{-10}	6.89×10^{-11}
500	1.71×10^{-1}	5.90×10^{-55}	4.87×10^{-2}	6.69×10^{-41}	1.20×10^{-3}	5.19×10^{-24}	1.10×10^{-5}	1.22×10^{-13}	1.89×10^{-7}	3.49×10^{-8}
600	3.46	2.77×10^{-40}	1.33	1.08×10^{-29}	8.20×10^{-2}	5.33×10^{-17}	2.39×10^{-3}	3.36×10^{-9}	1.67×10^{-4}	4.39×10^{-5}
700	2.92×10^{1}	8.58×10^{-30}	1.39×10^{1}	1.13×10^{-21}	1.63	5.57×10^{-12}	1.09×10^{-1}	5.11×10^{-6}	2.27×10^{-2}	7.40×10^{-3}
763.15	8.38×10^{1}	7.26×10^{-24}	4.40×10^{1}	5.66×10^{-17}	7.07	9.05×10^{-9}	7.15×10^{-1}	1.01×10^{-3}	1.06	4.93×10^{-1}
800	1.44×10^{2}	3.38×10^{-21}	7.96×10^{1}	6.23×10^{-15}	1.50×10^{1}	1.73×10^{-7}	1.88	6.64×10^{-3}	3.93	1.84
900	5.09×10^{2}	4.61×10^{-15}	3.15×10^{2}	3.06×10^{-10}	8.44×10^{1}	1.51×10^{-4}	1.78×10^{1}	4.30×10^{-1}	7.93×10^{1}	3.79×10^{1}
1000	1.32×10^{3}	3.54×10^{-10}	9.15×10^{2}	1.65×10^{-6}	3.32×10^{2}	3.25×10^{-2}	1.26×10^{-2}	1.47×10^{1}	8.39×10^{2}	4.05×10^{2}

TABLE V. - EQUILIBRIUM CONSTANTS FOR THE REACTIONS OF EQUATIONS (4) TO (9)

Temperature, K	K_1	K_2	K_3	K_4	K_5	K_6
443.15	2.683×10^{-11}	9.291×10^{53}	8.322×10^{87}	3.594×10^{30}	6.663×10^{1}	3.830×10^{52}
500	6.040×10^{-9}	4.682×10^{46}	5.167×10^{75}	3.698×10^{26}	1.623×10^{1}	7.554×10^{45}
600	6.441×10^{-6}	1.540×10^{37}	2.099×10^{60}	2.381×10^{21}	2.561	1.403×10^{37}
700	8.936×10^{-4}	2.587×10^{30}	2.121×10^{49}	4.592×10^{17}	6.796×10^{-1}	7.898×10^{30}
763.15	1.016×10^{-2}	1.127×10^{27}	7.407×10^{43}	6.554×10^{15}	3.530×10^{-1}	6.167×10^{27}
800	3.033×10^{-2}	2.145×10^{25}	1.199×10^{41}	7.433×10^{14}	2.501×10^{-1}	1.592×10^{26}
900	3.609×10^{-1}	2.392×10^{21}	4.607×10^{34}	4.977×10^{12}	1.423×10^{-1}	3.498×10^{22}
1000	2.799	1.646×10^{18}	3.394×10^{29}	9.013×10^{10}	6.120×10^{-2}	4.101×10^{19}

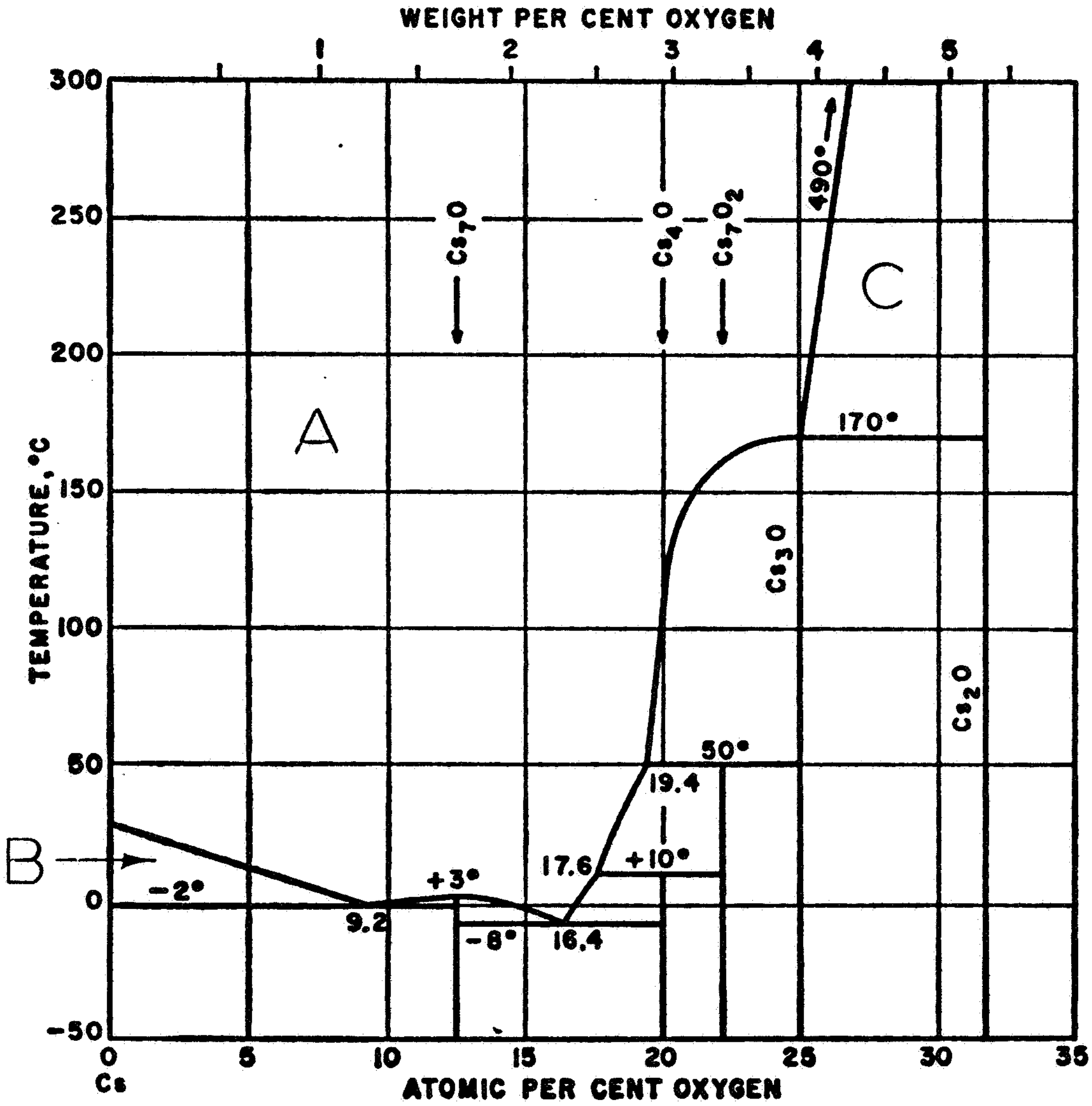

FIGURE 1 - PHASE DIAGRAM FOR Cs-O SYSTEM (ref. 1).

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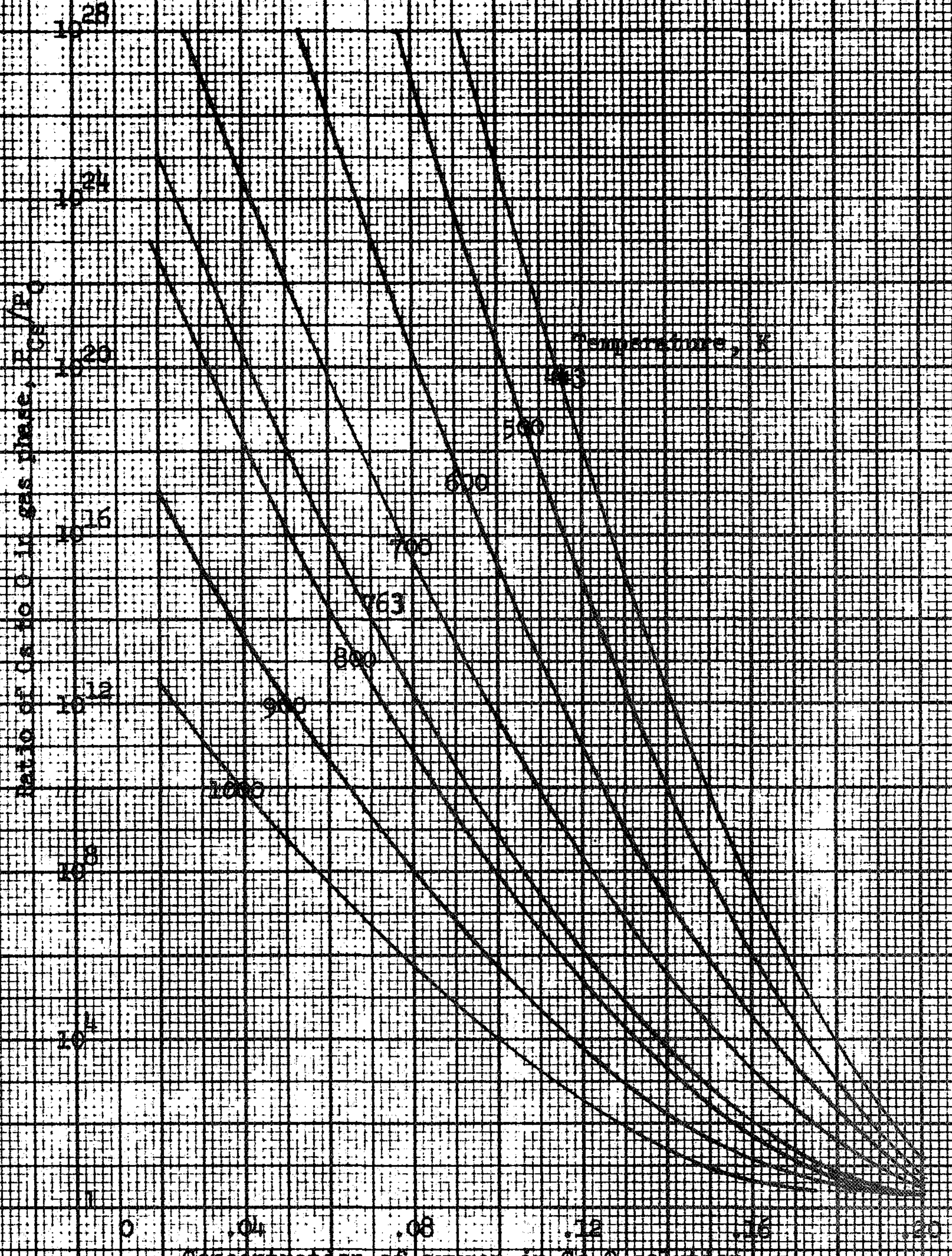

FIGURE 2 - RATIO OF Cs TO O, P_{Cs}/P_O, IN THE GAS PHASE ABOVE Cs-O SOLUTION

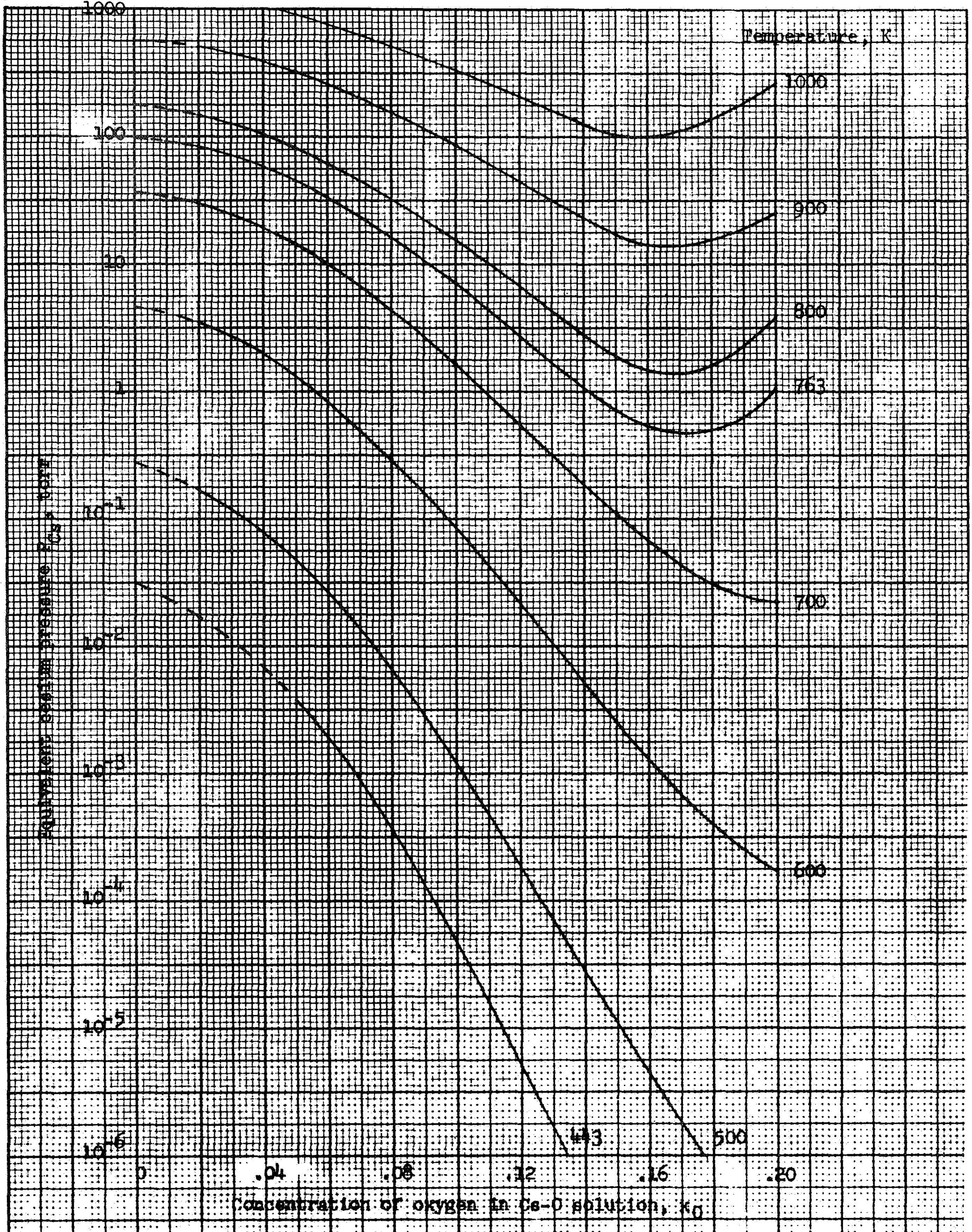

FIGURE 3 - EQUIVALENT CESIUM PRESSURE, P_{Cs}, ABOVE Cs-O SOLUTION