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THE VAPOR PRESSURE OF STANDARD SAMPLES OF GOLD AND SILVER

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FOREWORD

This report was prepared by the Thermo and Chemical Physics Branch, Materials Physics Division, Air Force Materials Laboratory. The work was initiated under Project No. 7360, "Chemical, Thermal, and Dynamic Properties of Materials," Task No. 736001, "Thermo and Chemical Physics," and was administered under the direction of the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio, with Mr. Gerard L. Haury (MAYT), as Project Engineer.

This report covers work accomplished during the period November 1967 to September 1968. It was submitted by the author in November 1968.

The author wishes to express his thanks to Mr. Bruce Schreiber for aid with the experimental work, to Dr. Joseph Davison for use of a computer least squares program, and to Dr. Emile Rutner for discussions and suggestions on all phases of this effort.

This report has been reviewed and is approved.

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ABSTRACT

In conjunction with a National Bureau of Standards (NBS) program to obtain standard vapor pressures of metals at high temperatures, the vapor pressures of gold and silver were measured by the Knudsen effusion technique. The sample of gold furnished by NBS was 99.999% pure. Vapor pressures were determined for liquid gold in the temperature range 1700° to 2000°K. A value of $(\Delta H^\circ_{298})_v$ = 87.58 ±2.12 kcal/mole (±2.42%) was obtained by combining the results of three separate determinations of the slope of ln (pressure) vs 1/temperature (second law plot). Using the third law of thermodynamics, the above vapor pressure data was combined with available thermodynamic data to yield an average $(\Delta H^\circ_{298})_v$ = 88.01 ±0.30 kcal (±0.34%) for the three separate sets of determinations.

The sample of silver furnished by NBS was 99.9999% pure. Vapor pressures were determined for liquid silver in the temperature range 1370° to 1525°K. A value of $(\Delta H^\circ_{298})_v$ = 72.67 ±1.88 kcal/mole (±2.59%) was obtained by combining the results of three separate determinations of the slope of ln (pressure) vs 1/temperature (second law plot). The average third law value of $(\Delta H^\circ_{298})_v$ for the same three sets of determinations was 67.86 ±0.21 kcal/mole (±0.31%).

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SECTION I

INTRODUCTION

The use of gold and silver as thin film conductors, coatings on satellites, mirrors, and optical filters has increased the importance of an accurate vapor pressure of these substances since they are deposited by effusion onto the desired surface. Furthermore, the vapor pressures of gold and silver can be determined to a high degree of accuracy and therefore these substances can be used as vapor pressure standards. To this end, the National Bureau of Standards (NBS) has initiated a program to determine the vapor pressure of these and other metals at high temperatures. The Bureau's effort is also concerned with refining the various techniques used for measuring low vapor pressures at high temperatures. The work reported herein which covers the vapor pressures of liquid gold and silver is aimed at improving the Knudsen effusion technique for the determination of vapor pressures.

SECTION II

EXPERIMENTAL

1. DESCRIPTION OF APPARATUS

A conventional vacuum evaporator (Figure 1, Reference 1) consisting of a bell jar (a), housed the effusion cell and associated equipment, such as the electron beam furnace. The bell jar was pumped down to pressures of 10^{-5} torr by the diffusion pump (b) and fore pump (c).

A cross section of the type of crucibles used is shown in Figure 2. They were made of graphite and the orifices were machined to the size and contour as shown in Table I.

TABLE I

CRUCIBLE DIMENSIONS

Curves	d_h (in.)	l_h (in.)	t (in.)	I_d (in.)	I_b (in.)	I_a (in.)	I_c (in.)	α (deg)
Curve 1	0.0436	0.0425	0.125	0.28	0.18	0.37	0.13	60
Curve 2	0.0436	0.0425	0.125	0.28	0.18	0.37	0.13	60
Curve 3	0.0308	0.0595	0.125	0.28	0.18	0.37	0.13	30
Curve 4	0.0438	0.0553	0.125	0.38	--	0.37	0.13	30
Curve 5	0.033	0.0505	0.125	0.38	--	0.37	0.13	30
Curve 6	0.042	0.0565	0.125	0.38	--	0.37	0.13	30

NOTE: The symbols correspond to the dimensions of the crucible so designated in Figure 2. The curves may be found in Figures 3, 4, 5, and 6.

Table II gives the areas of the holes A_h, area of the sample A_s, and the overall clausing factor for the cell body.

The crucibles were heated by electron bombardment between 1700° and 2000°K. Filaments (f) and (g) were the sources of electrons which were accelerated through a potential drop of 1000 V by the use of the high voltage power supply (h). The furnace is similar to one utilized by Panish (Reference 2).

TABLE II

SAMPLE AND ORIFICE AREAS AND ORIFICE AND CELL CLAUSING FACTORS

Curves	A_h* (cm^2)	A_s* (cm^2)	W_h*	W_c*
Curve 1	0.00962	0.70	0.5120	0.55
Curve 2	0.00962	0.70	0.5120	0.55
Curve 3	0.00962	0.70	0.3344	0.55
Curve 4	0.00973	0.70	0.4286	0.46
Curve 5	0.0055	0.70	0.3843	0.46
Curve 6	0.00893	0.70	0.4163	0.46

* A_h is the area of orifice cm^2; A_s is the exposed surface area of sample cm^2; W_h is the clausing factor for the orifice; W_c is the clausing factor for the inside of the cell.

The lower tantalum shields (s) were dropped below the ends of side shields (r) about one inch so that the crucible chamber could be adequately pumped out during effusion. Arcing occurred due to high pressures before this was done.

The temperature was controlled by measuring the high voltage current between the crucible and the filaments, and controlling the filament heating current to maintain the high voltage power constant. This was essentially done by a servomotor (n) which moved a resistor (o) to adjust the current to a preset value.

The temperature was read with a Leeds and Northrup (model 8622) pyrometer sighted on blackbody holes in the bottom and top of the crucible. The pyrometer was calibrated against a standard NBS lamp. The temperatures were determined to ±10°K at 2200°K. Temperature readings were taken periodically during the course of an experiment and the sight glass at (p) was cleaned between runs to assure accurate temperature readings.

2. EXPERIMENTAL PROCEDURES

A sample to be evaporated was placed inside a weighed crucible and heated to the desired temperature for a given time. The weight loss was found by weighing the residue and the empty crucible. Mass losses were determined to ±0.1 mg. Corrections were made for the vaporization and oxidation of the crucible when required. The empty crucible was heated at the same temperatures and for the same lengths of time as the metal samples. Weight loss corrections were made for the carbon loss of the crucible for each evaporation experiment (Reference 1).

In order to minimize the temperature gradient between the top and bottom of the crucible, the crucible was either raised or lowered in relation to the filaments until a minimum temperature difference between the blackbody cavities was observed. The crucible was supported by a 1/16 inch tungsten rod inserted into a cavity in the bottom center of the crucible. About 1/4 inch below the crucible a notch was ground into the rod so as to minimize the conductive heat leak. This rod could be raised or lowered as required to position the crucible. The temperature gradient never exceeded 10° and the average temperature between the top and bottom was reported as the temperature of the crucible. To minimize their weight loss, the carbon crucibles were coated after each determination with pyrolytic graphite. The coating was applied by heating the crucible in methane at 1400°C for five minutes.

3. TREATMENT OF DATA

If the system is an evaporating system at equilibrium, the change of the mass rate of flow from the Knudsen cell with temperature is related to the heat of vaporization, and is given by

$$\frac{d(\ln \dot{m}_i)}{d(1/T)} = \frac{d(\ln P_i)}{d(1/T)} + \frac{T}{2} = \frac{\Delta H^{0}_{vi}}{R} + \frac{T}{2} \qquad (1)$$

where ΔH°_{v} is the standard heat of vaporization. The slope of a plot of $\ln \dot{m}$ vs 1/T should give the heat of vaporization, if we assume the difference in the heat capacity of the gas and liquid is zero. The error due to heat capacity differences is minimized if we assume that ΔH_v° is taken at the mean temperature of the experiment.

During the vaporization of gold, only Au is considered to be the effusing species (References 3 and 4); therefore, the vapor pressure was calculated using the following relation (Reference 5).

$$\dot{m} = \frac{W_h A_h}{17.14} P_c M^{1/2} T^{-1/2} \tag{2}$$

where

$\dot{m}$ = mass loss in gm/sec (corrected for crucible loss)

W_h = clausing factor for the orifice geometry employed (Reference 6). In this work, the values are listed in Table II.

A_h = orifice area cm^2 (Table II)

P_c = calculated pressure mm Hg (Table III)

M = molecular wt of gold, 196.97

T = observed temperature °K. °C 'as read' + correction for sight glass (2.5% x °C) + 273.16°.

The pressure, P_c, obtained by the use of Equation 2, was corrected for the actual sample area used (Table II) and the flow through the channel of the body of the Knudsen cell (Reference 7). The relation between the uncorrected equilibrium pressure, P_e', and the observed pressure, P_c, is given by Equation 3 (Reference 7 and 8).

$$P_e' = (1 + \frac{f_c}{W_c} - 2f_c + f_1) P_c \tag{3}$$

where

$$f_1 = \frac{W_h \cdot A_h}{\alpha A_s} \qquad f_c = \frac{W_h \cdot A_h}{A_c}$$

and α = condensation coefficient and is taken as 1 for most metals. The symbols are defined in Table II.

The conditions of the experiment are such that $P_e' = 1.005\ P_c$ for curves 1 and 2, and $1.0017\ P_c$ for curve 3 of Figure 3. Equation 3 gives a positive correction, i.e., $P_e' > P_c$. (See curves 1 and 2, Table III.) Further corrections which were applied were due to the increase in the cavity area, A_h, caused by the thermal expansion of the graphite. The calculated change in A_h varied from 1.36% at 2050°K to 0.90% at 1700°K when the data for the coefficient of expansion of graphite given in Reference 9 was used. This correction reduced the observed value of P_e'. A third correction factor which was applied was due to the effect of pressure on the conductance of the gas through the orifice. This correction is obtained from Figure 7 (Reference 5, p. 108). The ratio of the conductance F/F_t given in this figure is obtained by using the value of the conductance F, under the conditions of the experiment, and the conductance F_t which is calculated by considering the flow to be purely molecular. The correction F/F_t is listed in Table III and is negative when greater than 1, thus decreasing the value of P_e'.

The observed pressures P_c were thus corrected by the above three factors as shown in Tables III and IV. The overall correction varied from 0.5 to 6.0% depending upon the temperature and orifice size. Tables III and IV show the final vapor pressure, P_e, as determined by the above procedures for gold and silver, respectively.

a. Sample calculation:

Gold, Run 6

time-averaged temperature 'as read' = 1704°C
+ 2.5% for sight port loss + 273 = 2020°K

The average temperature = $\frac{\Sigma T \Delta t_i}{\Sigma \Delta t_i}$, where Δt_i is the time interval at the temperature T_i. This average temperature can be used as the temperature for the determination. This approximation is valid because the deviations from the mean were less than 10°C (Reference 10).

TABLE III

COMPLETE DATA, INCLUDING MASS LOSSES, DERIVED PRESSURES, CORRECTION FACTORS, AND EQUILIBRIUM VAPOR PRESSURES FOR THE KNUDSEN EFFUSION DETERMINATION OF THE VAPOR PRESSURE OF GOLD

Raw Data							Correction Factors					
Run No.	Temp (°K)	Time (sec)	Total Mass Loss (g)	Crucible Loss (g)	Net Mass Loss (g)	$\dot{m}$ (g/sec ×10^{4})	(a) P_{mm} Calculated P_c	(b) Correct P_c to P_e'	(c) Orifice Thermal Expansion $1-(\Delta A_h/A_h)$	(d) Flow Factor $1 \div F/F_t$	(e) Total Correction Factor	(f) P_e
							Curve 1, Figure 3					
6	2020	4140	0.2298	0.003	0.2268	0.5478	0.6106	1.005	0.987	1.03	1.022	0.6239
7	1696	25740	0.0309	0.0005	0.0304	0.01181	0.0121	1.005	0.991	1.00	0.996	0.0121
11	1756	12060	0.0288	0.0005	0.0283	0.02347	0.0244	1.005	0.991	1.01	1.006	0.0246
16	1855	3900	0.0361	0.0003	0.0358	0.9179	0.9811	1.005	0.990	1.03	1.030	0.1010
20	1949	1800	0.0584	0.001	0.0574	0.3189	0.3489	1.005	0.989	1.05	1.044	0.3643
21	1877	2640	0.0299	0.0002	0.0297	0.1125	0.1208	1.005	0.990	1.03	1.030	0.1244
22	1783	11100	0.0489	0.001	0.0479	0.04315	0.0452	1.005	0.991	1.02	1.016	0.0459
23	1772	10320	0.0353	0.001	0.0343	0.03324	0.0347	1.005	0.991	1.02	1.016	0.0353
24	2008	1800	0.0784	0.001	0.0774	0.4300	0.4776	1.005	0.987	1.04	1.032	0.4929
25	1902	3600	0.0507	0.0004	0.0503	0.1397	0.1511	1.005	0.990	1.04	1.035	0.1564
							Curve 2, Figure 3					
27	1717	18240	0.0280	0.0003	0.0277	0.01519	0.0156	1.005	0.991	1.00	0.996	0.01557
28	1881	2760	0.0300	0.0003	0.0297	0.1076	0.1157	1.005	0.990	1.03	1.030	0.1192
30	1822	7080	0.0464	0.0005	0.0459	0.06483	0.0686	1.005	0.990	1.03	1.030	0.0702
31	1910	2640	0.0387	0.0003	0.0384	0.1455	0.1575	1.005	0.990	1.04	1.035	0.1633
32	1776	5760	0.0173	0.0005	0.0168	0.02916	0.0305	1.005	0.991	1.02	1.016	0.03096
34	1955	5940	0.1464	0.0034	0.1430	0.01892	0.2639	1.005	0.989	1.05	1.044	0.2755
35	2023	4500	0.2070	0.0030	0.2040	0.4533	0.5058	1.005	0.987	1.03	1.022	0.5169
							Curve 3, Figure 3					
40	1717	10020	0.0054	0.0002	0.0052	0.005190	0.0164	1.0017	0.991	1.08	1.072	0.0176
41	1720	17340	0.0116	0.0003	0.0113	0.006516	0.0206	1.0017	0.991	1.08	1.072	0.0220
42	1864	7380	0.0268	0.0005	0.0263	0.03563	0.1169	1.0017	0.990	1.03	1.021	0.119
43	1924	4620	0.0395	0.0005	0.0390	0.08441	0.2813	1.0017	0.990	1.04	1.031	0.299
44	1818	6960	0.0162	0.0006	0.0156	0.02241	0.0726	1.0017	0.990	1.02	1.012	0.0736
45	1766	11880	0.0133	0.0005	0.0128	0.001007	0.0344	1.0017	0.991	1.07	1.062	0.0366
46	1913	3600	0.0277	0.0005	0.0272	0.07555	0.2513	1.0017	0.990	1.04	1.031	0.259
47	2007	1620	0.0279	0.0008	0.0271	0.1673	0.5535	1.0017	0.987	1.05	1.038	0.594
48	1971	3060	0.0407	0.0010	0.0397	0.1297	0.4330	1.0017	0.989	1.05	1.040	0.451

(a) Equation 2

(b) Equation 3

(c) Linear coefficient of expansion is 0.65% between 298° and 2000° K (Reference 9)

(d) See Section II e.

(e) Product of (b)(c)(d) corrections (See Section II e)

(f) See Section II e.

TABLE IX

COMPLETE DATA, INCLUDING MASS LOSSES, DERIVED PRESSURES, CORRECTION FACTORS, AND EQUILIBRIUM VAPOR PRESSURES FOR THE KNUDSEN EFFUSION DETERMINATION OF THE VAPOR PRESSURE OF SILVER

Raw Data							Correction Factors					
Run No.	Temp (°K)	Time (sec)	Total Mass Loss (g)	Crucible Loss (g)	Net Mass Loss (g)	$\dot{m}$ (g/sec x 10⁴)	(a) P_{mm} Calculated P_c	(b) Correct P_c to P'_e	(c) Orifice Thermal Expansion $1-(\Delta A_h/A_h)$	(d) Flow Factor $1 \div F/F_t$	(e) Total Correction Factor	(f) P_e
							Curve 4, Figure 4					
2	1466	12000	0.1400	0.0045	0.1355	0.1137	0.171	1.007	0.990	1.04	1.038	0.177
3	1430	13800	0.1050	0.0048	0.1002	0.0726	0.109	1.007	0.991	1.03	1.028	0.112
5	1502	3000	0.0599	0.0013	0.0586	0.1953	0.299	1.007	0.990	1.05	1.047	0.314
6	1462	9060	0.1087	0.0033	0.1054	0.1175	0.176	1.007	0.990	1.04	1.038	0.183
8	1483	7200	0.1128	0.0030	0.1098	0.1525	0.232	1.007	0.990	1.04	1.038	0.241
9	1432	14400	0.0992	0.0044	0.0948	0.0658	0.098	1.007	0.991	1.03	1.028	0.101
10	1524	5520	0.1584	0.0026	0.1558	0.2822	0.436	1.007	0.989	1.05	1.046	0.456
11	1399	12720	0.0598	0.0031	0.0567	0.0446	0.066	1.007	0.991	1.02	1.016	0.067
12	1451	10800	0.1160	0.0038	0.1122	0.1038	0.157	1.007	0.991	1.04	1.036	0.162
13	1372	18360	0.0468	0.0046	0.0422	0.0229	0.034	1.007	0.991	1.01	1.006	0.034
							Curve 5, Figure 5					
14	1514	4140	0.0494	0.0017	0.0477	0.1152	0.350	1.0035	0.989	1.05	1.043	0.366
15	1478	10320	0.0744	0.0042	0.0702	0.0680	0.240	1.0035	0.990	1.04	1.034	0.211
16	1450	11280	0.0572	0.0039	0.0533	0.0472	0.141	1.0035	0.991	1.03	1.025	0.144
17	1427	14220	0.0486	0.0043	0.0443	0.0311	0.092	1.0035	0.991	1.02	1.015	0.093
18	1399	15600	0.0314	0.0043	0.0271	0.0173	0.051	1.0035	0.991	1.02	1.015	0.052
19	1370	23340	0.0317	0.0052	0.0265	0.0113	0.033	1.0035	0.991	1.01	1.005	0.033
20	1529	3300	0.0475	0.0017	0.0458	0.1387	0.424	1.0035	0.989	1.05	1.043	0.442
21	1495	5400	0.0470	0.0024	0.0446	0.0825	0.249	1.0035	0.990	1.04	1.034	0.256
22	1471	6720	0.0419	0.0020	0.0399	0.0593	0.178	1.0035	0.990	1.04	1.034	0.184
23	1452	10920	0.0586	0.0038	0.0548	0.0501	0.139	1.0035	0.991	1.04	1.035	0.143
24	1410	15120	0.0409	0.0044	0.0365	0.0241	0.071	1.0035	0.991	1.02	1.015	0.072
25	1384	22200	0.0434	0.0054	0.0380	0.0171	0.050	1.0035	0.991	1.01	1.005	0.050
							Curve 6, Figure 6					
26	1515	3780	0.0627	0.0015	0.0612	0.1619	0.280	1.007	0.989	1.05	1.046	0.293
28	1461	8040	0.0669	0.0026	0.0643	0.0799	0.136	1.007	0.990	1.04	1.038	0.141
29	1478	4680	0.0549	0.0019	0.0530	0.1132	0.193	1.007	0.990	1.04	1.038	0.200
30	1429	10560	0.0556	0.0036	0.0520	0.0492	0.083	1.007	0.990	1.03	1.028	0.085
31	1385	15300	0.0377	0.0041	0.0336	0.0219	0.036	1.007	0.991	1.01	1.006	0.037
32	1398	14040	0.0441	0.0040	0.0401	0.0285	0.047	1.007	0.991	1.02	1.016	0.048
33	1439	11100	0.0745	0.0036	0.0708	0.0638	0.107	1.007	0.991	1.03	1.031	0.111
34	1494	7560	0.1042	0.0032	0.1010	0.1335	0.229	1.007	0.990	1.04	1.038	0.238
35	1418	11520	0.0496	0.0033	0.0463	0.0401	0.067	1.007	0.991	1.03	1.028	0.069

(a) Equation 2

(b) Equation 2

(c) Linear coefficient of expansion is 0.65 % between 298° and 2000 °K (Reference 9)

(d) See Section II a.

(e) Product of (b)(c)(d) corrections (See Section IIa)

(f) See Section II a.

Time = 4140 seconds

Net mass loss = total-crucible loss $= \begin{array}{r} 0.2298 \\ \underline{0.003} \\ = 0.2268 \end{array}$

Using Equation 2

$$\frac{0.2268\text{g}}{4140\,\text{sec}} = \frac{(0.5120)(0.00962)}{17.14} \times P_c \times \frac{196.97}{2020}$$

$$P_c = 0.6106 \text{ mm}$$

Correction Factors:

P_c to P_e', Equation 3

$$f_l = \frac{W_h \times A_h}{A_s} = \frac{0.5120 \times 0.0096}{0.70} = 0.00683$$

$$f_c = \frac{W_h \times A_h}{A_c} = \frac{0.5120 \times 0.0096}{0.50} = 0.00984$$

$$W_c = 0.55 \text{ (Reference 6)}$$

$$P_e' = (1 + \frac{0.00984}{0.55} - 0.01968 + 0.00683)\, P_c$$

$$P_e' = 1.005\, P_c$$

Change in A_h due to thermal expansion from 298° to 2020°K; area increases 1.34% and the correction factor was, therefore, = 1.0000 -0.0134 = 0.987.

Conductance factor (Figure 7):

F/F_t vs aP_a/L_1

$$L_1 = \frac{2.331 \times 10^{-20} T}{P_{mm} \delta^2} \quad \text{(mean free path in cm)}$$

$$L_1 = \frac{2.331 \times 10^{-20} \times 2020}{1 \times 10^{-3} \times (2.88)^2 \times 10^{-16}}$$

a = orifice radius = 0.056 cm

P_a = average pressure in the orifice in microns and

$= \frac{P_c}{2}$ = 309 microns

δ = atomic diameter for Au = 2.88×10^{-8} cm

$aP_a/L_1 = 0.618$

$F/F_t = 0.97$

$1 \div F/F_t = 1.03$

The overall correction factor = 1.005 x 0.987 x 1.03 = 1.022

$$P_e \text{ corrected} = 1.022 \times 0.6106 = 0.6240 \text{ mm}$$

The third law values $(\Delta H^\circ_{298})_v$ of all the curves as listed in Tables V and VI were calculated in accordance with the sample calculation of Table VII. The data used for this calculation is given in Table V, curve 2.

The value of P_e as given in Table III was converted to P atm and then to ln P atm (see Table VII), and -Rln P atm was calculated. From tables of the thermodynamic properties of the elements (Reference 11), values of $\frac{-(F^\circ - H^\circ_{298})\text{ gas}}{T}$ and $\frac{-(F^\circ - H^\circ_{298})\text{ standard state}}{T}$ were obtained, and $\frac{-\Delta(F^\circ - H^\circ_{298})}{T}$ was obtained by the difference. The third law value of $(\Delta H^\circ_{298.13})_v$, expressed as kcal/mole, was then calculated using the relation $\left(-R\ln P + \left[\frac{-\Delta(F^\circ - H^\circ_{298})}{T}\right]\right) \times T$ for each temperature. The root-mean-square deviation for curve 2 was calculated as shown in Table VII.

TABLE V

REDUCED DATA – GOLD

Heats of Vaporization and Vapor Pressures of NBS Standard Reference Material No. 685-60 Mil Au Wire from Spool No. 13-1-60 Mil.

Heat of Vaporization for the reaction: $Au_{(l)} \longrightarrow Au_{(g)}$

Curves	ΔH^{o}_{298} kcal/mole Second Law (± % Error)	ΔH^{o}_{298} kcal/mole Third Law (± % Error)
1	88.52 ± 2.89	88.07 ± 0.32
2	85.29 ± 2.79	88.50 ± 0.36
3	89.30 ± 2.71	87.45 ± 0.34
Composite	87.58 ± 2.42	

Curve 1		Curve 2		Curve 3	
T (°K)	P (mmHg)	T (°K)	P (mmHg)	T (°K)	P (mmHg)
1696	0.0121	1717	0.0156	1717	0.0176
1756	0.0246	1776	0.0310	1720	0.0220
1772	0.0353	1822	0.0702	1766	0.0366
1783	0.0459	1881	0.119	1818	0.0736
1855	0.101	1910	0.163	1864	0.119
1877	0.124	1955	0.276	1913	0.259
1902	0.156	2023	0.517	1971	0.451
1949	0.364			2007	0.594
2008	0.493				
2020	0.624				

Composite: $\ln P_{mmHg} = (-41.22 \pm 1.00)\, 10^3/T\,°K + 19.94 \pm 0.54$

Curve 1: $\ln P_{mmHg} = (-41.69 \pm 1.21)\, 10^3/T\,°K + 20.7 \pm 0.65$

Curve 2: $\ln P_{mmHg} = (-40.06 \pm 1.12)\, 10^3/T\,°K + 19.18 \pm 0.60$

Curve 3: $\ln P_{mmHg} = (-42.07 \pm 1.05)\, 10^3/T\,°K + 20.54 \pm 0.56$

TABLE VI

REDUCED DATA—SILVER

Heats of Vaporization and Vapor Pressures of NBS Silver Standard Reference Material SRM 678 55E-5

Heat of Vaporization for the Reaction: $Ag_{(l)} \longrightarrow Ag_{(g)}$ assuming predominant species monomeric Ag

Curves	ΔH^{o}_{298} kcal/mole Second Law (± % Error)	ΔH^{o}_{298} kcal/mole Third Law (± % Error)
4	72.91 ± 3.10	67.55 ± 0.37
5	71.69 ± 2.29	67.82 ± 0.24
6	72.94 ± 3.56	68.21 ± 0.32
Composite	72.67 ± 2.59	

Curve 4		Curve 5		Curve 6	
T (°K)	P (mm)	T (°K)	P (mm)	T (°K)	P (mm)
1466	0.179	1514	0.365	1515	0.293
1430	0.112	1478	0.211	1461	0.141
1502	0.314	1450	0.144	1478	0.200
1462	0.183	1427	0.0933	1429	0.0848
1483	0.241	1399	0.0515	1385	0.0365
1428	0.101	1370	0.0330	1398	0.0481
1524	0.456	1529	0.442	1439	0.111
1399	0.067	1495	0.258	1494	0.238
1451	0.162	1471	0.184	1418	0.0691
1372	0.0339	1452	0.143		
		1410	0.0717		
		1384	0.0494		

Composite: $\ln P\,\text{mm} = (-34.10 \pm 0.88)\,10^{3} / T^{o}K + 21.50 \pm 0.61$

Curve 4: $\ln P\,\text{mm} = (-34.22 \pm 1.06)\,10^{3} / T^{o}K + 21.67 \pm 0.73$

Curve 5: $\ln P\,\text{mm} = (-33.61 \pm 0.77)\,10^{3} / T^{o}K + 21.17 \pm 0.53$

Curve 6: $\ln P\,\text{mm} = (-34.24 \pm 1.22)\,10^{3} / T^{o}K + 21.47 \pm 0.84$

TABLE VII

SAMPLE CALCULATION FOR THIRD LAW HEATS
(Curve 2, Figure 3, NBS GOLD)

T (°K)	P (mmHg)	p (atm)	ln p (atm)
1717	0.0156	0.205×10^{-4}	−10.795
1776	0.0310	0.408×10^{-4}	−10.108
1822	0.0702	0.924×10^{-4}	− 9.290
1881	0.1192	0.1563×10^{-3}	− 8.761
1910	0.1633	0.2149×10^{-3}	− 8.446
1955	0.2755	0.3625×10^{-3}	− 7.923
2023	0.5169	0.6801×10^{-3}	− 7.294

T (°K)	$-\frac{(F^\circ - H^\circ_{298})\text{gas}}{T}$	$-\frac{(F^\circ - H^\circ_{298})\text{Standard State}}{T}$	$-\frac{\Delta(F^\circ - H^\circ_{298})}{T}$
		(From Reference 12)	
1717	47.72	17.75	−29.97
1776	47.86	18.00	−29.86
1822	47.96	18.19	−29.77
1881	48.10	18.42	−29.68
1910	48.16	18.54	−29.62
1955	48.26	18.70	−29.56
2023	48.40	18.95	−29.45

−R ln p (atm)	T (°K)	$\left[-R\ln p + \left[\frac{-\Delta(F^\circ - H^\circ_{298})}{T}\right]\right] \times T$		Dev^2
21.45	1717	88.29	−0.21	0.044
20.09	1776	88.71	+0.21	0.044
18.46	1822	87.88	−0.62	0.384
17.41	1881	88.58	+0.08	0.006
16.78	1910	88.62	+0.12	0.014
15.74	1955	88.56	+0.06	0.004
14.49	2023	88.89	+0.39	0.152
		avg 88.50		0.648

$$\sqrt{\frac{0.648}{n-1}} = \sqrt{\frac{0.648}{6}} = \pm 0.32$$

ΔH 298 °K = 88.50 ± 0.32 kcal/mol Au

SECTION III
THEORY AND DISCUSSION

The Knudsen effusion technique for the determination of vapor pressures in the range of about 10^{-10} to 10^{-4} atm is one of the principal methods for observing the vaporization behavior of materials at high temperatures. In general, the technique involves heating a substance (i.e., metal) in a small closed container (Figure 1) having a small orifice, and for which the ratio of the area of the orifice to the inside cross section of the container is less than 0.01. The vapor generated from the liquid or solid phase effuses through the orifice into a high vacuum (10^{-5} to 10^{-6} mm). The system, consisting of the vapor and condensed phase of the sample inside the Knudsen cell is often assumed to be in equilibrium. If this is the case, the time and temperature dependence of the mass loss yields the vapor pressure as a function of temperature and also yields the heats of vaporization. The conditions necessary for equilibrium to exist in the system are given below. In the cases of high purity gold and silver the effusing species are predominently the atomic species Au and Ag, respectively (References 3 and 4). Therefore, the following relation between the vapor pressure of the gold or silver in the cell, the mass loss rate and the sample temperature applies (Reference 5).

$$\dot{m} = \frac{W_h A_h}{17.14} P_c M^{1/2} T^{-1/2} \qquad (4)$$

where

$\dot{m}$ = effusion rate from the cell

W_h = clausing factor

A_h = area of the orifice

M = molecular weight of the effusing species

T = absolute temperature

P_c = observed pressure in mm Hg

Since the system is not a closed one, the observed pressure P_c may not be the equilibrium pressure, P_e; therefore, a correction must be made to P_c to obtain P_e. The cell pressure is a function of the orifice and cell geometries, the ratio of the orifice area to the area of the exposed sample surface, and the ratio of the mean free path in the gas phase to the hole geometry. For example, the ratio of the conductance of the gas through the orifice (Figure 7, Reference 5). The ordinate of Figure 7, F/F_t, is the ratio of the conductance (liters/sec) for an average orifice pressure P_a to the conductance for pure molecular flow. To the right of the minimum in curve A, as P_a increases, the conductance, F, increases due to viscous effects, and the correction $(1 \div F/F_t)P_c$ becomes less up to $P_a/L_1 = 1$ where the correction is negligible.

Another effect which is taken into account when correcting P_c to P_e (observed pressure to equilibrium pressure) is the Motzfeldt relations (Reference 7). These take into account the ratio of the orifice area A_h to the cell cross-sectional area A_c and the exposed sample area A_s as follows:

$$P_e = (1 + \frac{f_c}{W_c} - 2\ f_c + f_l)P_c \tag{5}$$

$$f_l = \frac{W_h\ A_h}{\alpha A_s} \tag{6}$$

$$f_e = \frac{W_h A_h}{A_c} \tag{7}$$

$$\alpha = \frac{\text{rate at which molecules condense}}{\text{rate at which molecules strike the surface of the sample}} \tag{8}$$

The condensation coefficient, α, for most metals is found to be unity and has been assumed to be unity for gold and silver. The geometry of the sample surface has an effect on the distribution of the molecules after they exit the orifice. Graphite is not easily 'wet' by gold, and the sample tends to remain globular at least to 1700°K; however, if a thorium oxide cup is used, the gold wets it more readily and the evaporating surface tends to be planar (Reference 12). In this work, the sample surface could not be observed while at temperature and since the effusant was not collected on a target, the distribution of effusant was not determined. Similarly, in the case of silver, the graphite was not wetted by the silver nor the distribution determined.

The clausing factors (see Table II) were calculated using Freeman's "Transmission Probabilities for Conical Orifices" (Reference 6, p. 26). The transmission probability (or clausing factor) is defined as the probability that a molecule which enters the orifice through one end will escape through the other end. The above tabulated probabilities assume that all surface reflections are diffuse, that is, in accordance with the cosine law, and that there are no gas phase collisions and, therefore, the flow through the orifice is steady-state molecular flow. Surface diffusion or creep on the cell or orifice walls is neglected. It should be emphasized that the above clausing factor calculations are only applicable when the orifice diameter is not greater than one-tenth of the mean free path of the effusing molecules as determined by the pressure in the cell directly under the surface containing the orifice. However, for practical purposes, the calculations may be used for pressures that are 10 times greater than that corresponding to this mean free path. The values are no longer valid if the area of the orifice and/or pressure are sufficient to cause mass motion of the gas (Reference 5) due to viscous effects. In the case of the orifice geometries used for gold in this work (Table I and Figure 2) the orifice entrance pressure must be 0.96 mm or less for the mean free path of the effusant to be less than the orifice diameter. It is of importance to consider the l_h to d_h ratio (Figure 2) because the expressions for the mass rate of flow differ with a variation of the ratio (Reference 13). The highest ratio used in this work was 1.93 (curve 3, Table I). Freeman (Reference 6) observed root-mean-square deviations of up to 3.4% when comparing experimental and theoretical clausing factors for l_h/d_h from 0.195 to 0.800.

The silver vapor pressures and heats of vaporization which were obtained when using the larger orifice were not significantly different from those obtained with the smaller orifice. The smaller orifice area was approximately one-half the larger orifice area (curves 4, 5, and 6 of Figures 4, 5, and 6). This would indicate that the cell pressure was near equilibrium. The crucible orifice areas for the three gold runs were the same (curves 1, 2, and 3 of Figure 3). The second law heat of vaporization of the gold for curve 2, Figure 3, was somewhat lower (3 to 4 kcal) than the other two curves (1 and 3) for no apparent reason. However, the second law heats for the other five curves (1, 2, 4, 5, and 6) were all within the statistical error. Upon examination of Tables V and VI, it is seen that the measured pressures agree within 20% for a given temperature, thus lending confidence to the technique. The composite curves of Figures 8 and 9 represent a plot of all the data points taken for each sample. In the case of the gold, the composite statistical error was smaller than that of any of the three individual curves. For silver, however, the composite error was larger than the error for curve 5 (smaller orifice), 2.59% vs 2.29%. The pressures in curve 5 may have been nearer equilibrium.

It should be noted that the second law heats of vaporization were all higher (within the error) when the fully corrected pressures P_e were used instead of the P_c pressures. The statistical error did not change appreciably, however. It can be said then that the total of all the corrections fell within the statistical error.

Ward's least-squares fit of his entire data (Table I of Reference 12) gave a second law heat of 88.84 ±0.55 kcal/mole at 298°K. A least squares calculation utilizing the computer program for this work and Ward's data for the Stackpole graphite cell yielded a second law heat of 88.75 ±0.40 kcal/mole at 298°K. Ward's third law calculation for his samples which were globular in shape was 88.23 ±0.09 kcal/mole at 298°K as listed in Table VIII. There is some evidence that the gold sample surface in this work was globular, even though it could not be observed, since the sample solidified in a globular shape upon cooling. Examination of Hildenbrand and Hall's data (Reference 14) yielded a second law heat of vaporization (88.96 ±0.80 kcal/mole at 298°K) as listed in Table VIII. This value was calculated utilizing the least squares computer program of this work and all their data (cells 3, 4 and 5, Table I of Reference 14) whereas the second law heat of vaporization reported in Reference 14 was 89.3 kcal/mole. Their third law heat of vaporization (88.3 ± 0.9 kcal/mole at 298°K) is listed in Table VIII. Second law calculations which were made in the course of this work, using the data for each individual cell shown in Table I of Reference 14, gave the following results.

Cells	2nd law H°_{298} (kcal/mole)
3	87.48 ±1.66
4	92.28 ±4.46
5	97.32 ±2.72

Again referring to Table VIII, the Panish data for silver (Table I, Reference 2) was treated as noted above giving second law values of the heat of vaporization as shown. All the data listed in Reference 2 was used since the cell and technique used were the same for all points. Panish reported only third law values which are also listed in Table VIII. These were obtained by the same method used in this work. Both the second and third law heats for liquid and solid silver for McCabe and Burchenall (Reference 15) data, as listed in Table VIII, were calculated as noted above. All the data points of Table I, Reference 15, were used since the crucible and technique were the same. The only value reported in Reference 15 is a third law heat of 67.87 ±0.10 kcal/mole at 0°K.

SECTION IV

CONCLUSIONS

This work can be evaluated by making a comparison of the second and third law results of this work with that of other investigators who used different techniques or modifications of the Knudsen technique. The results of this work and other investigators, including the statistical error, are given in Table VIII.

TABLE VIII

ΔH°_{298} DATA COMPARISONS FOR GOLD AND SILVER IN KCAL/MOLE

Investigator	2nd law ΔH°_{298} (kcal/mole)	3rd law ΔH°_{298} (kcal/mole)	Method
GOLD			
J.W. Ward (Ref. 12, Fig. 5)	$88.84 \pm 0.55^{*}$	$88.23 \pm 0.09^{*}$	effusion - tracer target
D. L. Hildenbrand W. F. Hall (Ref. 14)	$88.96 \pm 0.80^{*}$	$88.3 \pm 0.9^{*}$	effusion - torsion
This work	$87.58 \pm 2.12^{*}$	$88.01 \pm 0.3^{*}$	effusion - wt loss from cell
SILVER			
M. Panish (Ref. 2)	$67.63 \pm 2.98^{*}$ $69.60 \pm 0.86^{**}$	$67.70 \pm 0.50^{*}$ $68.14 \pm 0.2^{**}$	tracer technique
McCabe & Burchenall (Ref. 15)	$72.76 \pm 1.24^{*}$ $67.63 \pm 0.43^{**}$	$68.04 \pm 0.46^{*}$ $68.09 \pm 0.4^{**}$	effusion - wt loss from cell
This work	$72.67 \pm 1.9^{*}$	$67.86 \pm 0.21^{*}$	effusion - wt loss from cell
* liquid phase, ** solid phase			

The data referred to in Table V are recent and the techniques used by some of the investigators afforded some advantages over that used in this work. For example, the torsion effusion method (Reference 14) measures the actual pressure of the effusant at the orifice exit, and the cell leakage or surface diffusion do not cause serious errors as they may in the direct cell weight loss technique. The tracer-target method (Reference 12) might be considered to be more advantageous because smaller amounts of effusant can be measured (by radio active tracer) and thus lower vapor pressures can be more accurately measured. The distribution of the gold on the target surface can be mapped and related to the evaporation mechanisms inside the cell. These differences may account for the smaller experimental errors seen in References 12 and 14. Heats of vaporizations were calculated utilizing the second law relation for both liquid and solid silver, using Panish's data (Reference 2) and the statistical methods of this work so that a valid comparison could be made. The data of McCabe and Birchenall (Reference 15) was likewise treated to obtain both second and third law heats of vaporization of silver (Table V).

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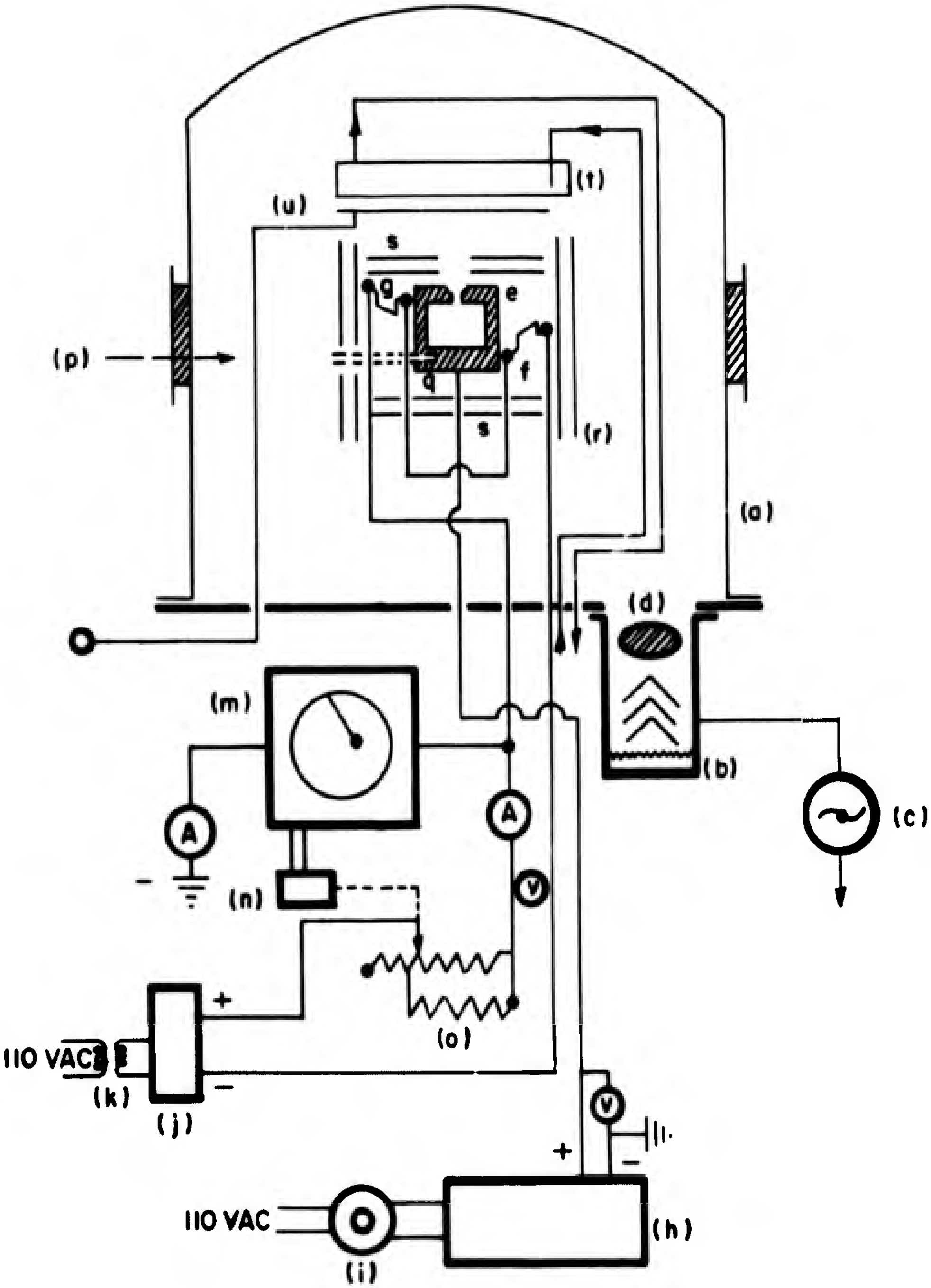

Figure 1. Schematic of Apparatus

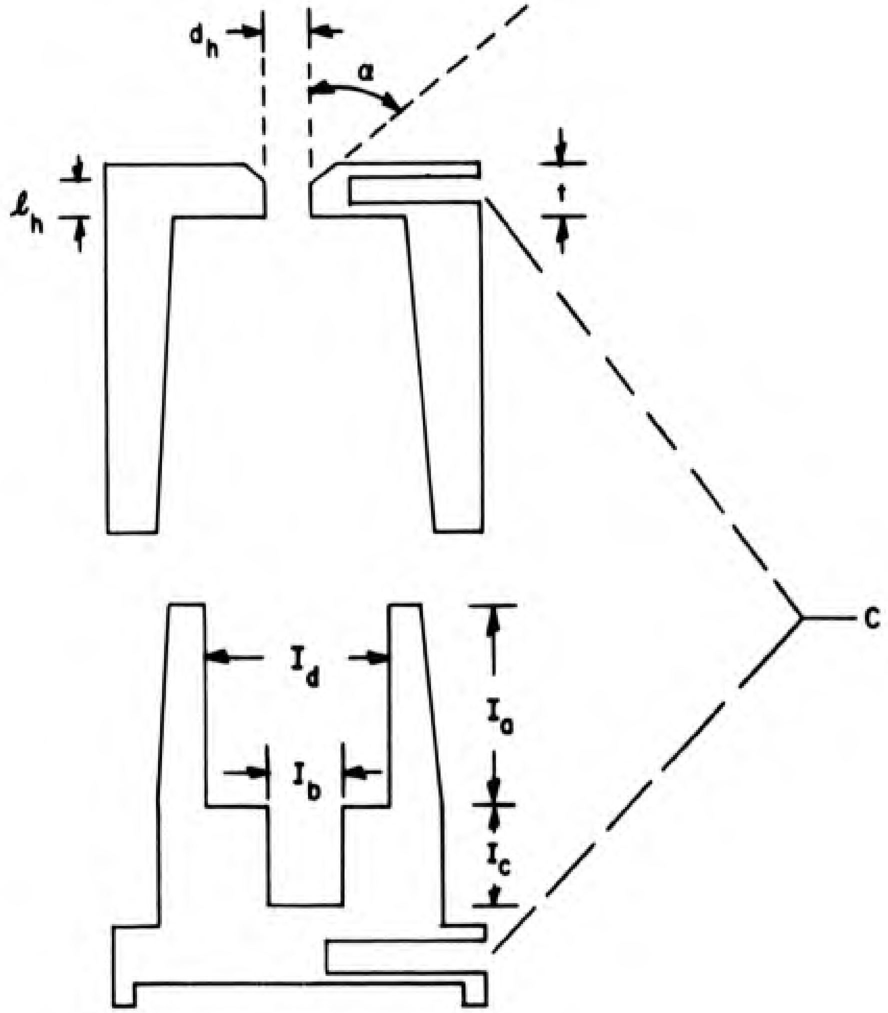

Figure 2. Cross-Section View of Graphite Crucible

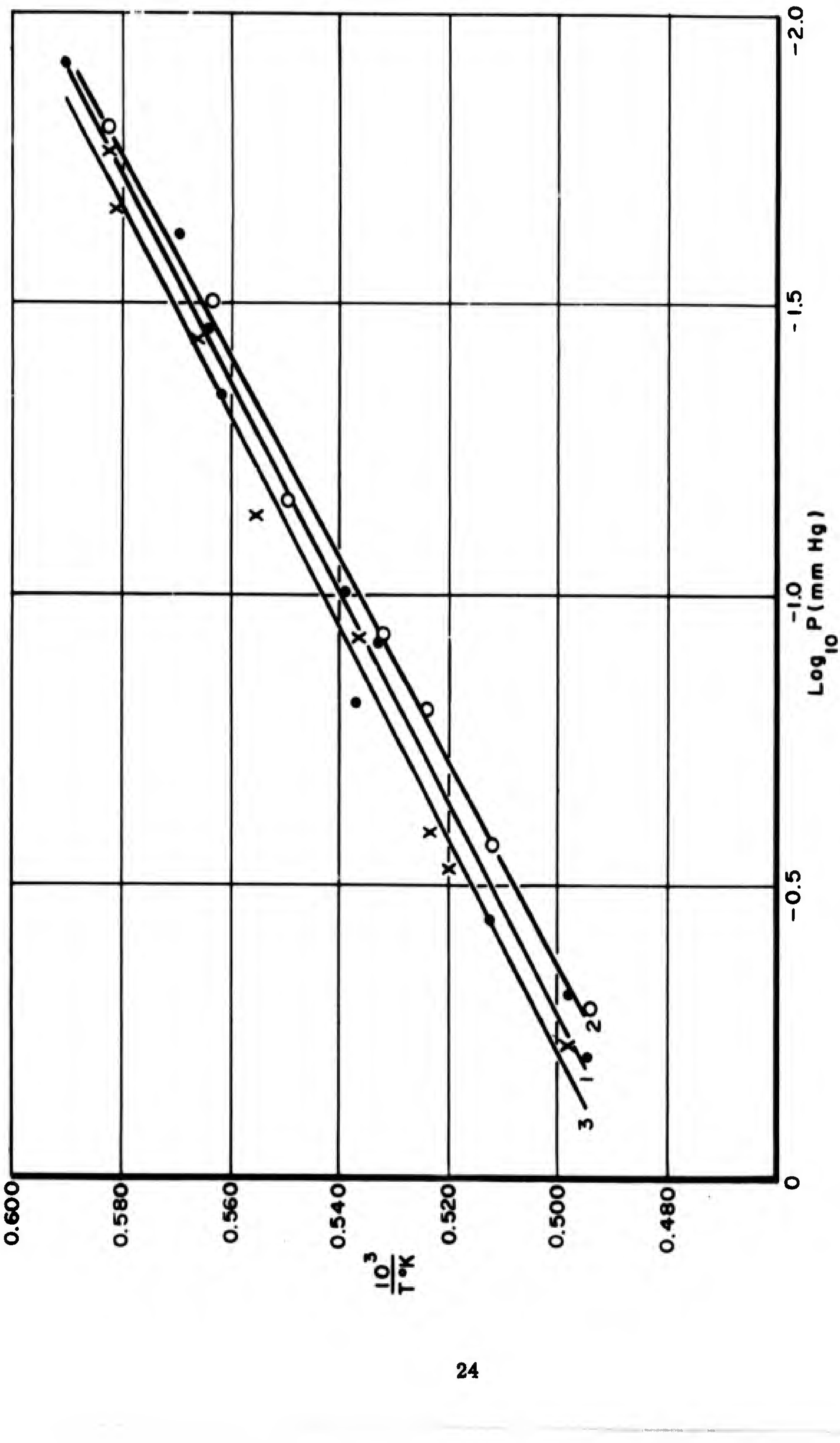

Figure 3. Curves 1, 2, and 3 of NBS Gold

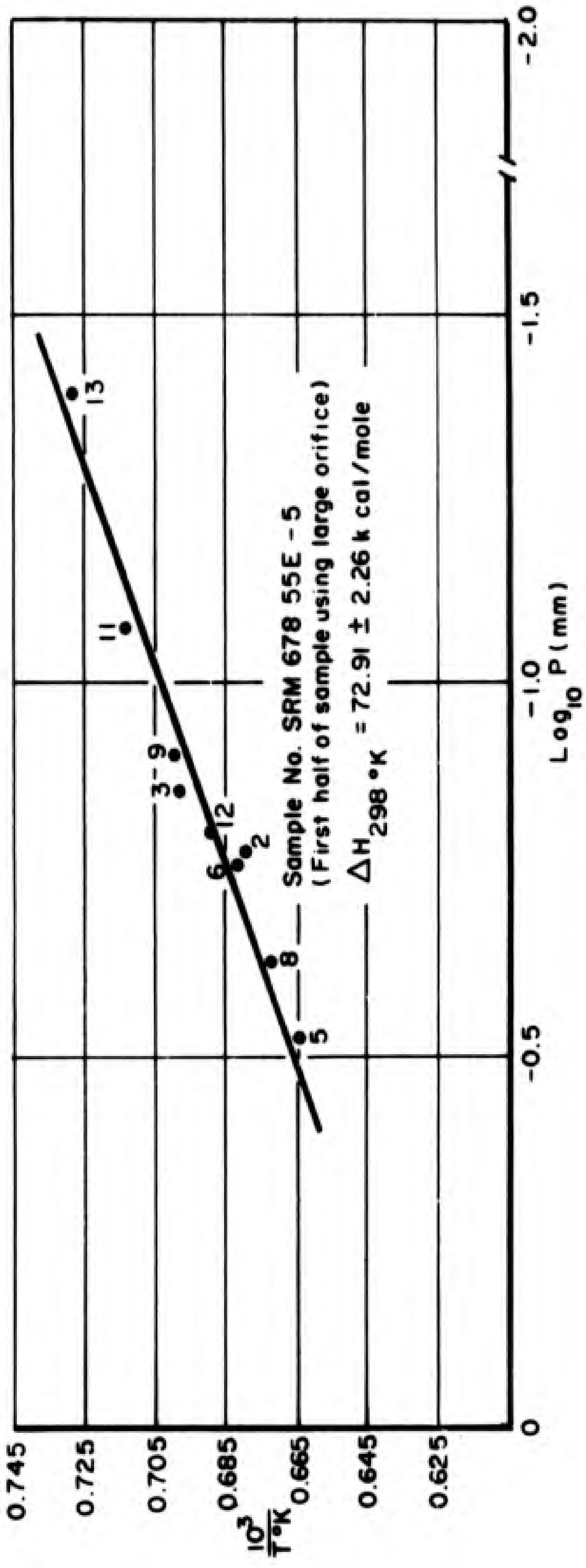

Figure 4. Curve 4 of NBS Silver

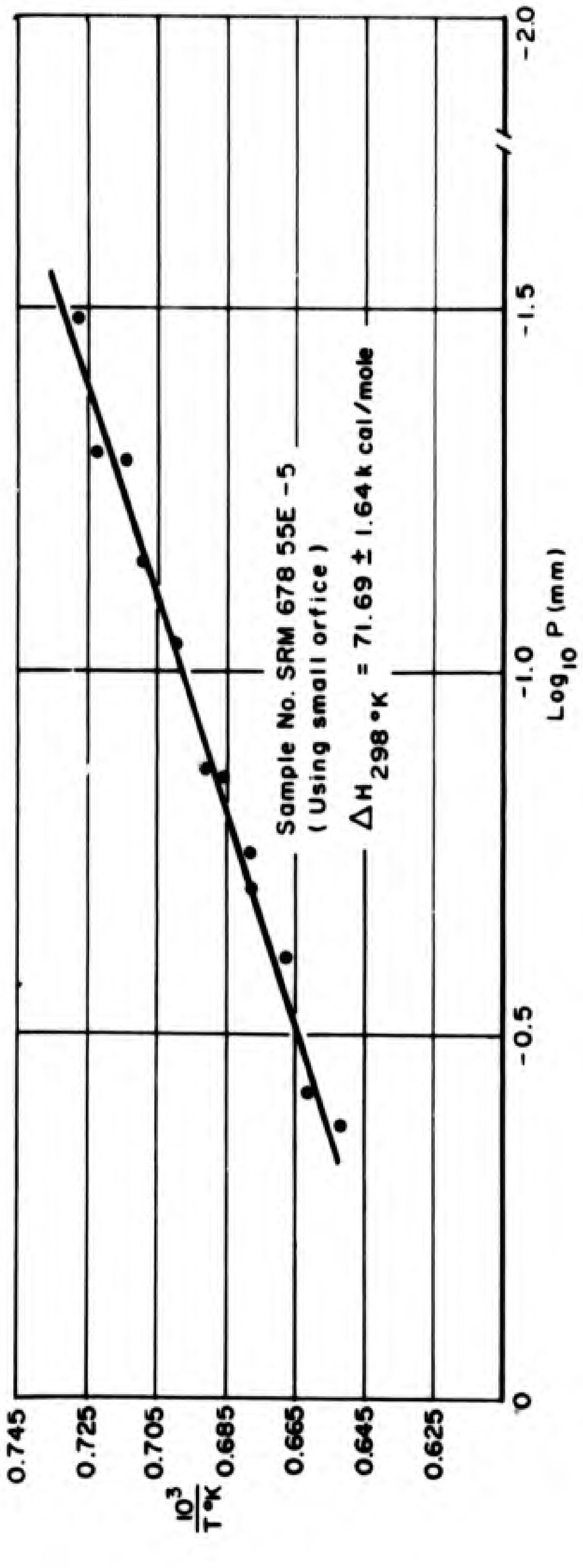

Figure 5. Curve 5 of NBS Silver

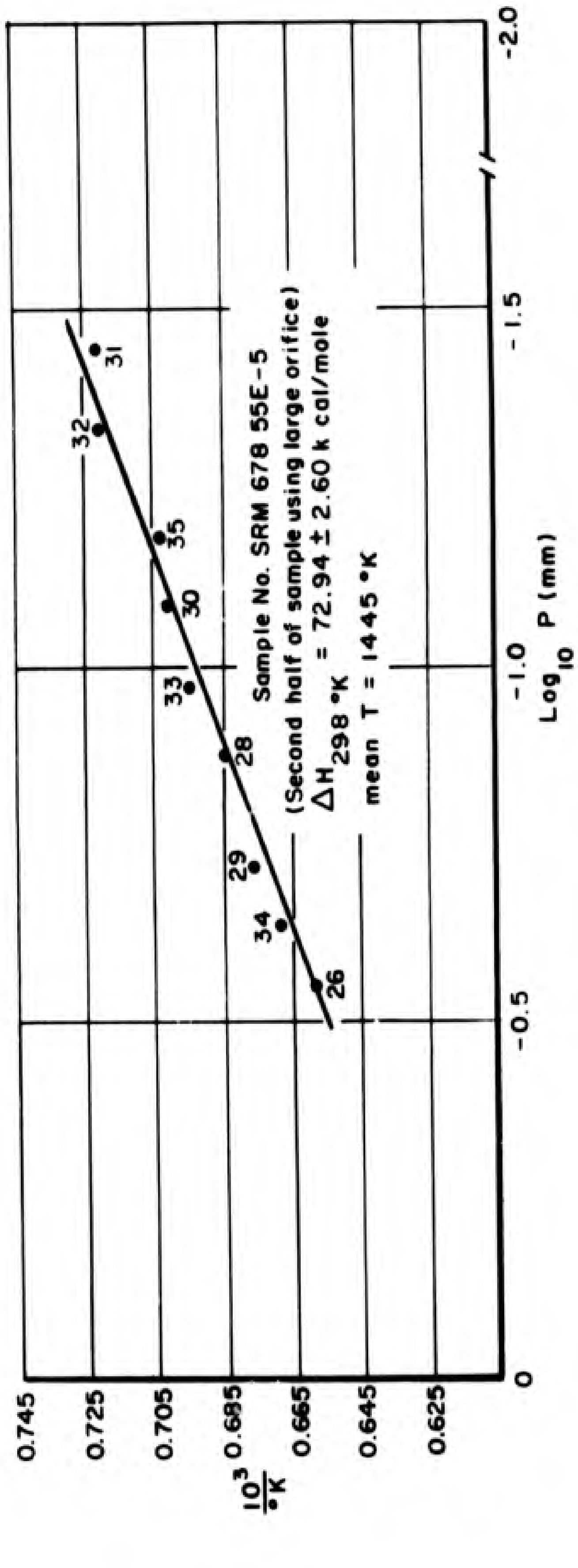

Figure 6. Curve 6 of NBS Silver

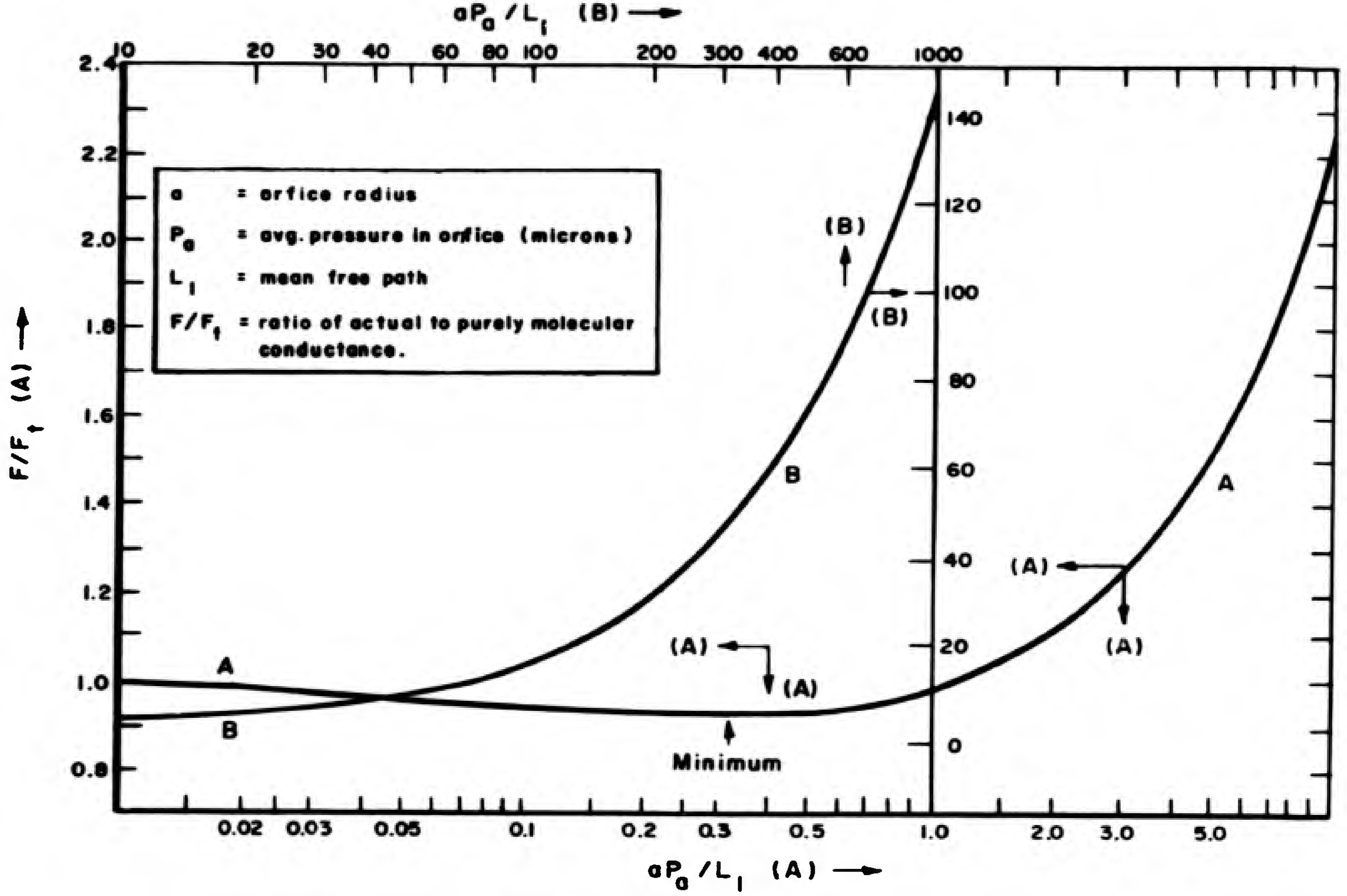

Figure 7. Relative Conductance of Cell. (See Reference 16.)

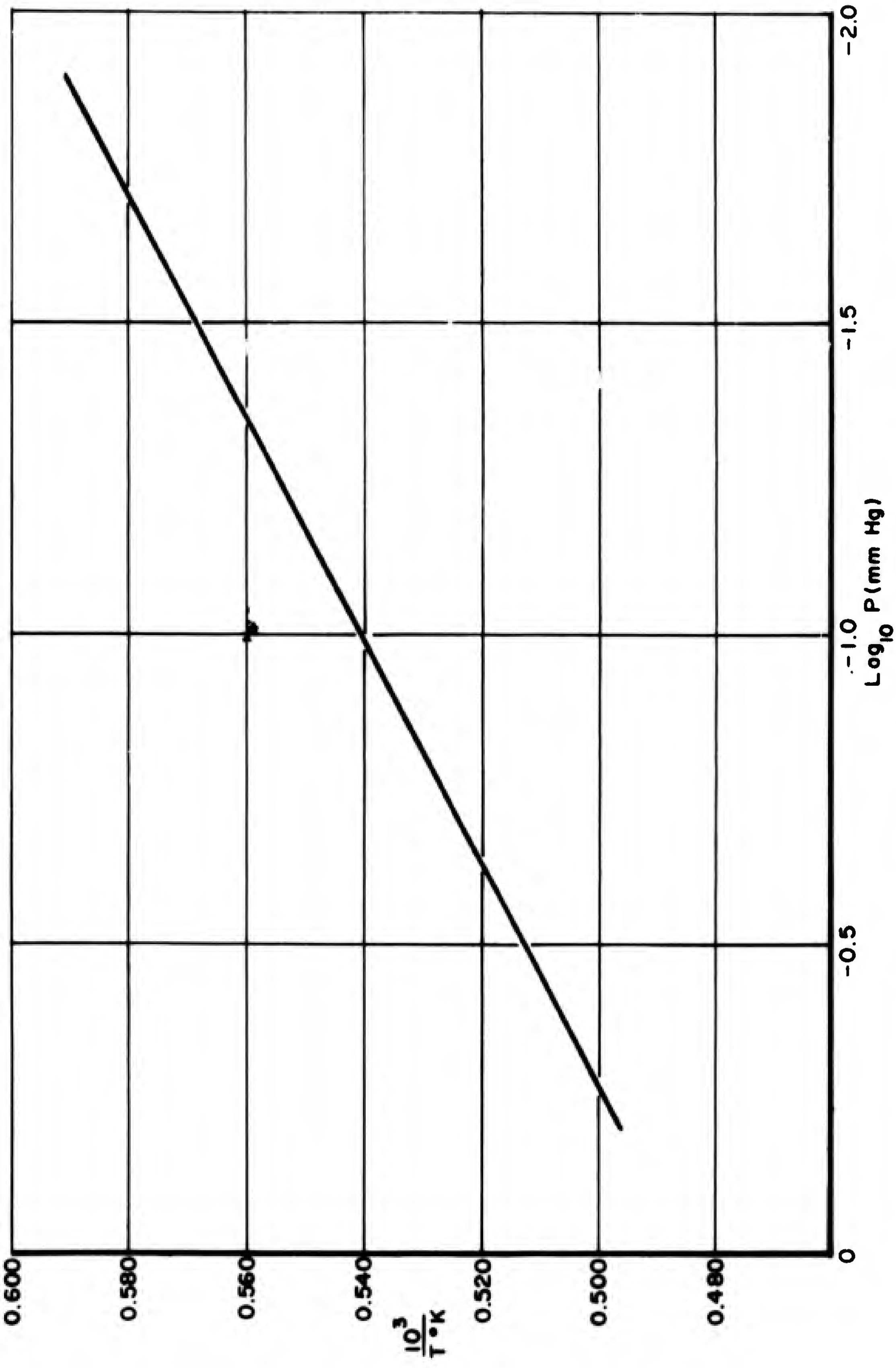

Figure 8. Composite of Curves 1, 2, and 3 of NBS Gold

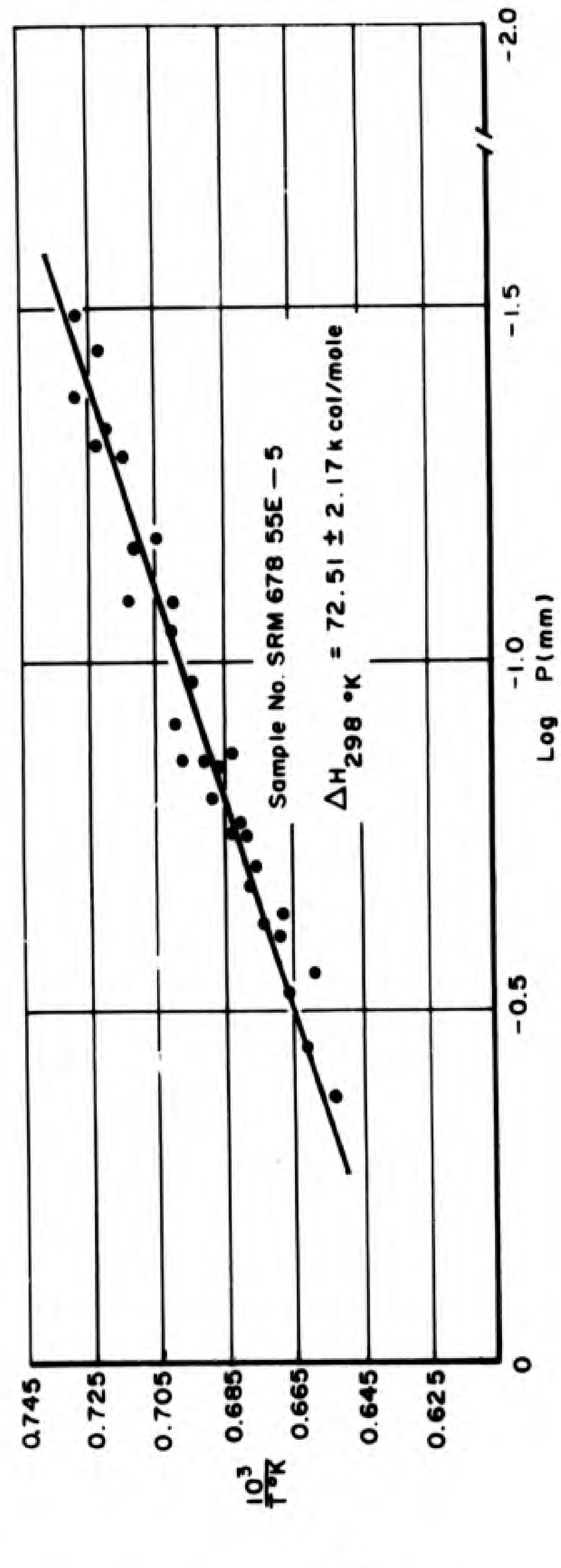

Figure 9. Composite of Curves 4, 5, and 6 of NBS Silver

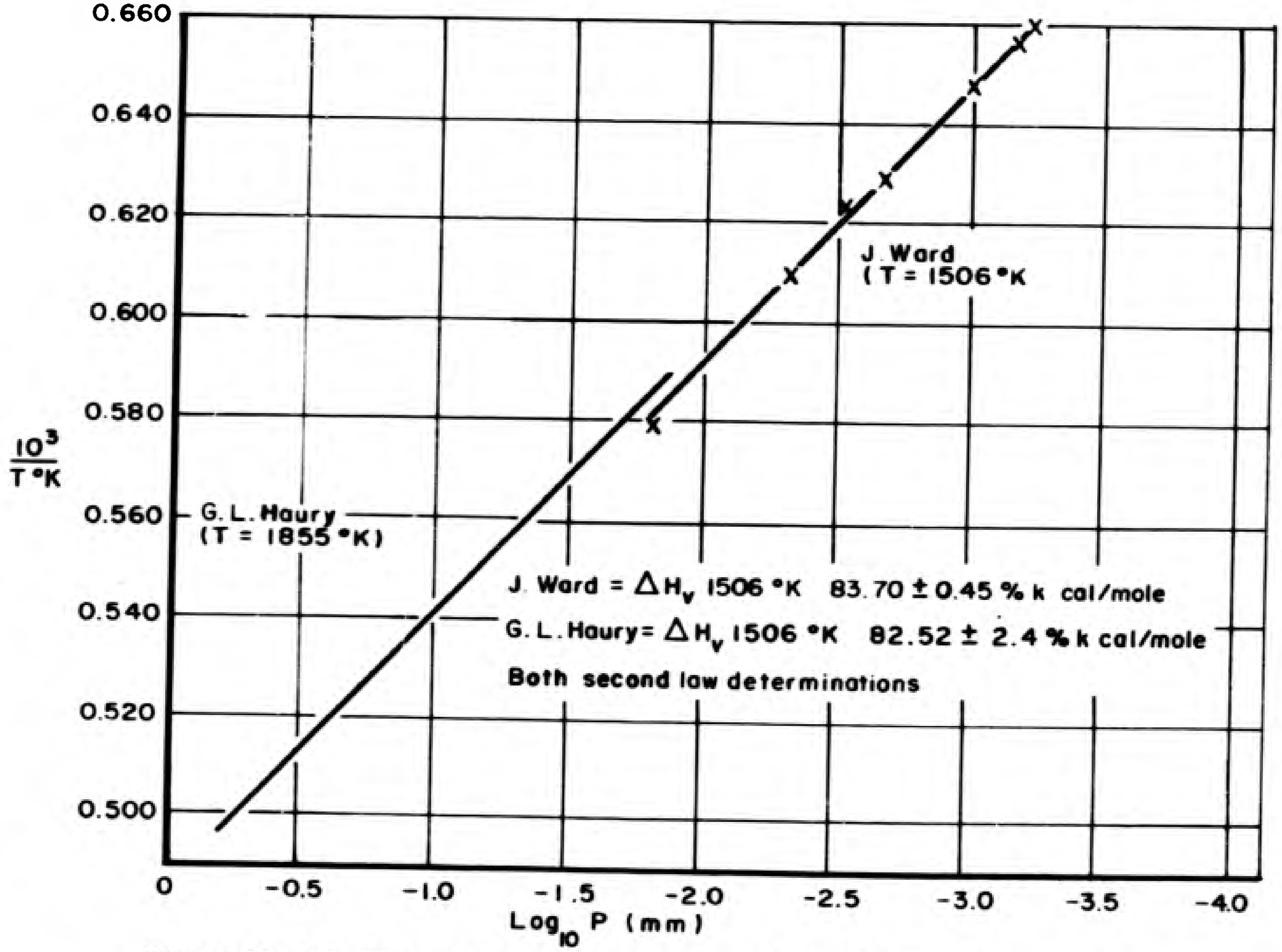

Figure 10. Vapor Pressure of Gold - Comparison of G. L. Haury and J. Ward (Ref. 5) Data

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13. ABSTRACT

In conjunction with a National Bureau of Standards (NBS) program to obtain standard vapor pressures of metals at high temperatures, the vapor pressures of gold and silver were measured by the Knudsen effusion technique. The sample of gold furnished by NBS was 99.999% pure. Vapor pressures were determined for liquid gold in the temperature range 1700° to 2000°K. A value of $(\Delta H^\circ_{298})_v = 87.58 \pm 2.12$ kcal/mole (±2.42%) was obtained by combining the results of three separate determinations of the slope of ln (pressure) vs 1/temperature (second law plot). Using the third law of thermodynamics, the above vapor pressure data was combined with available thermodynamic data to yield an average $(\Delta H^\circ_{298})_v = 88.01 \pm 0.30$ kcal (±0.34%) for the three separate sets of determinations.

The sample of silver furnished by NBS was 99.9999% pure. Vapor pressures were determined for liquid silver in the temperature range 1370° to 1525°K. A value of $(\Delta H^\circ_{298})_v = 72.67 \pm 1.88$ kcal/mole (±2.59%) was obtained by combining the results of three separate determinations of the slope of ln (pressure) vs 1/temperature (second law plot). The average third law value of $(\Delta H^\circ_{298})_v$ for the same three sets of determinations was 67.86 ±0.21 kcal/mole (±0.31%).

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Thermodynamics						
Silver Vapor Pressure						
Gold Vapor Pressure						
Vapor Pressure of Solids						

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