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The Vapor Pressure of Plutonium

Robert N. R. Mulford

University of California

Los Alamos Scientific Laboratory

Los Alamos, New Mexico

Abstract

The vapor pressure of liquid plutonium has been determined over the temperature range 1100° to 1800°K by the Knudsen effusion method. The least squares equation which fits the data is $\log_{10} p(\text{atm}) = -17420/T(^{\circ}\text{K}) + 4.913$. The standard deviation corresponds to about $\pm 10\%$ in the pressures calculated from this equation. The heat of vaporization computed from the temperature dependence of the experimental data is $\Delta H_{298}^{\circ} = 82.3 \text{ kcal/g-atom}$. The heat computed by combining independent entropy and heat capacity data with the present measurements is $\Delta H_{298}^{\circ} = 82.1 \text{ kcal/g-atom}$. Effects of oxygen upon the volatility of liquid plutonium were sought by comparing the vapor pressures observed with the liquid in contact with tantalum, tantalum carbide, magnesia, and plutonium sesquioxide. No differences were found. In addition, the vapor pressure was measured with different degrees of vacuum in the system. No effect was found here either, except that in very poor vacuums a surface film of oxide apparently formed and reduced the volatility by about a factor of 2.

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1. Introduction

The vapor pressure of elemental plutonium has been determined only once previously, by Phipps et al [1] in 1944. Phipps' data appeared to be of good quality and gave reasonable values but it was felt desirable to repeat the measurements with purer metal and with attention paid to the possible influences of oxygen on the observed vapor pressure. Rauh and Thorn [2] have found a lowering of the vapor pressure of metallic uranium, apparently caused by oxygen picked up from the vacuum system. Darnell and McCollum [3] have shown the vapor pressure of thorium to be enhanced by the presence of ThO_2 . The exact explanation of the effect with uranium is currently a somewhat controversial matter but, whatever the explanation, it is clearly desirable to examine insofar as possible any effects of oxygen on the vapor pressure of plutonium. In addition, as knowledge of the vapor pressure of an element is primary to all of the high temperature chemistry of that element, at least two independent measurements of this important property should exist.

With these considerations in mind, a new determination of the vapor pressure of plutonium was made. It turned out that no effect attributable to oxygen other than the formation of an oxide skin at very poor vacuums could be found, and that the vapor pressure observed agreed with Phipps' within 2 per cent, more closely than could be reasonably expected from the accuracy of Knudsen vapor pressure measurements in general. The two independent measurements thus provide vapor pressure data in which we believe some confidence can be placed, particularly as there is excellent agreement with independent thermal and spectroscopic data for the condensed and gaseous phases involved.

2. Experimental

2.1 Apparatus

The apparatus used has been described previously [4]. Briefly, the general arrangement was that the Knudsen cell, heated by a close fitting tantalum strip heater through which electric current passed, was supported by a tungsten stem at the center of a water-cooled shield which surrounded the cell. A collimator hole in the top of the shield was concentric with the cell orifice. The copper target which collected the part of the vapor beam coming out of the collimator was in the form of an annular ring with cooling water on the back side. As many as ten exposures could be made, each on a different sector of the ring, by rotating the target assembly through a vacuum seal. A tantalum shutter between the collimator and target allowed precise timing of exposures. The above parts were located inside a brass can which was evacuated by a pumping

train consisting of a liquid nitrogen filled cold trap, a 300 l/s oil diffusion pump using DC-704 silicone oil, and a mechanical pump. A VGLA ionization gage located close to the cell was used to measure background pressure in the system. A variable leak located between the brass can and the cold trap was used to modify the background pressure as desired by leaking dried air into the system. An ultimate vacuum of 3×10^{-7} torr was attainable with the heater and cell at room temperature. Usually, with the cell hot, the vacuum was about 1×10^{-6} torr; however, on some occasions, it was possible to make exposures with a vacuum of 5×10^{-7} torr.

2.2 Effusion Cells and Heater

The heater was made from tantalum sheet as described previously [4]. The top half was double thickness to provide more heat at the top end of the cell. Tests with a dummy cell showed that the lid was about 10° hotter than the cavity. No difference could be detected between the cavity temperature and the temperature read in the sight hole at the bottom of the cell. With this arrangement, no condensation of vapor on the lid or at the orifice was ever observed.

The cylindrical effusion cells were made of tantalum and were 0.375 inch diameter by 0.750 inch long. The orifice was machined to have a sharp edge with a 15° included angle. The lid was pressed onto a knife-edge seat in the cell body so as to deform the knife edge and make a gas-tight seal. The orifice area was determined by photographing the orifice together with a 0.01 mm scale at 100X, then measuring the orifice diameter on the photograph with the enlarged scale. Cells were assembled, degassed in place at about 2000°C , and then the charge, consisting of short pieces of wire, was added through the orifice.

The cell cavity was 0.250 inch in diameter and 0.500 inch deep. In some of the measurements the charge was contained in a TaC, ThO_2 or MgO cup fitting closely into the cell cavity and having an inside diameter of 0.200 inch. A similar cup was formed of plutonium oxide by tamping PuO_2 powder into the cell, then firing the assembly in vacuum. The vacuum firing in the tantalum cell partially reduced the PuO_2 . Heating the cell with the liquid plutonium charge in place completed the reduction of the oxide, which was seen by X-ray to be all hexagonal Pu_2O_3 after the vapor pressure measurements had been completed. In one series of exposures no cup was used; the plutonium was contained directly in the tantalum cell. This cell had only a short useful lifetime as liquid plutonium wetted the tantalum, climbed the cell walls and crept out the orifice. The liquid also penetrated the grain boundaries of the tantalum. The cell with a tantalum carbide cup was also short-lived, as TaC is easily wetted by plutonium.

2.3 Temperature Measurement

Temperature was measured with a calibrated disappearing-filament optical pyrometer sighted into a 0.070 inch diameter threaded hole in the side of the cell. The hole was 0.125 inch below the bottom of the cavity. The observed temperature was corrected for absorption of the fused silica window in the can. It is estimated that the absolute accuracy of temperature determination is $\pm 5^\circ$. Reproducibility, determined largely by ability to read the pyrometer scale, was $\pm 2^\circ$.

Manual temperature control was satisfactory. The power supply, consisting of a step-down transformer fed by a variable autotransformer operating directly off the 440 volt line, maintained the cell temperature constant within 2° even during two-hour exposures. Most of the time, no temperature drift was detectable during an exposure.

2.4 Determination of Target Deposits

The amount of plutonium deposited on each target sector during an exposure was determined by counting the alpha emission from the sector. A count of the total alpha activity was made with a 51% geometry proportional alpha counter calibrated for the copper sectors. It was also necessary to determine the fraction of the alpha count which came from plutonium, as the plutonium used contained a small amount of americium-241. The high volatility and the high specific activity of the ^{241}Am produced alpha count on the targets far out of proportion to the concentration in the charge. However, this difficulty was only encountered when the bare tantalum or tantalum carbide cells were used. The cells in which the charge was contained in an oxide cup gave at most 2 or 3 alpha per cent ^{241}Am on the targets. Evidently the americium in the charge was absorbed by the oxide and did not volatilize. Nevertheless, every target was pulse-analyzed to determine the $^{239}\text{Pu}/^{241}\text{Am}$ ratio in the deposit. The pulse analysis was done with an Ortec gold-barrier, silicon solid-state detector and a 400-channel pulse analyzer made by the Technical Measurement Co.

2.5 Metal Purity

The plutonium used for the measurements was produced by the electrolytic refining process. Spectrographic analysis showed that of 25 metallic elements looked for, all were below the limits of detection, with the exception of 30 ppm by weight of tungsten and 20 ppm of iron. Carbon and oxygen analyses gave 15 and 10 ppm by weight, respectively. Micrographic examination showed clean metal with virtually no impurity inclusions discernable. Radiochemical assay showed 28 ppm of ^{241}Am at the time the metal was received. Purification by evaporation of

about 10 per cent of the total weight reduced the ^{241}Am to 5 ppm. Thus, the impurities analyzed for totaled less than 100 ppm.

2.6 Calculations

The vapor pressure in atmospheres was calculated by the usual formula:

$$P = k \frac{m}{ta} \sqrt{\frac{T}{M}} \cdot \frac{l^2 + r^2}{r^2}$$

where k is a units constant, 3.760×10^{-8} , m is the micrograms of plutonium on a target, t is exposure time in minutes, a is orifice area in mm^2 , T is the absolute temperature, M is the atomic weight of the effusing species (239.1 in this case), r is the collimator radius, and l is the orifice-to-collimator distance. No correction to the pressures was made for either internal cell geometry or the transmission of the conical orifice, as those corrections do not apply to a collimated beam determination such as the present one.

3. Results

3.1 Primary Data

Table I summarizes the data. The exposures are numbered as A1, A2, etc. The letter refers to a series on a target during which the vacuum was not broken. The number refers to the chronological order in a series. Cup and cell dimensions are given in section 2.2. Figure 1 shows a plot of the data.

Cells 1 and 2, with no cup and a tantalum carbide cup, failed because of wetting after one heating, as described in section 2.2. Cell 3, with a magnesium oxide cup had a moderately long lifetime. However, after series M, the observed pressure began to decrease with time. The cell was opened and it was seen that all that remained of the plutonium charge was a small droplet in the center of the oxide cup. The inner surface of the cup had been converted to hexagonal Pu_2O_3 . The targets exposed with cell 3 had visible deposits of metallic magnesium in contrast to the usually invisible plutonium deposits obtained with the other cells. Although the magnesium deposits were thick enough to have a silvery color, they did not interfere with the plutonium determination by alpha counting, i.e. no degradation of the alpha energies by absorption was seen in the pulse analyses.

3.2 Effect of Background Pressure

In order to test the effect of the quality of the vacuum on the measured vapor pressure, three series of experiments were done. In the first, the sample was contained in a ThO_2 cup within the Ta cell. A series of three exposures was

TABLE I
Plutonium Vapor Pressure Data

Exposure No.	Temperature (°K)	Pu collected (μg)	Time (min)	P _{Pu} (atm.)
Cell 1 - Bare Ta, Charge 0.20 g., $a = 0.526 \text{ mm}^2$, $l = 3.185 \text{ cm}$, $r = 0.676 \text{ cm}$				
A1	1383	0.076	15.10	2.01×10^{-8}
A2	1428	0.047	4.36	4.37 "
A3	1352	0.078	30.01	1.02 "
A4	1377	0.073	16.02	1.81 "
A5	1455	0.274	12.03	9.31 "
A6	1487	0.372	9.33	16.5 "
A7	1527	0.416	5.02	34.7 "
A9	1578	0.592	3.02	83.4 "
Cell 2 - TaC cup, Charge 0.52 g., $a = 0.527 \text{ mm}^2$, $l = 3.208 \text{ cm}$, $r = 0.689 \text{ cm}$				
H1	1488	1.48	30.00	1.99×10^{-7}
H2	1532	1.00	10.00	4.09 "
H3	1588	0.69	3.00	9.59 "
H4	1630	0.68	1.50	19.1 "
H5	1482	1.22	30.00	1.64 "
H6	1531	0.90	10.00	3.68 "
H7	1531	0.98	10.00	4.01 "
H8	1533	0.98	10.00	4.01 "
H9	1533	0.92	10.15	3.71 "
H10	1534	1.00	10.00	4.10 "
Cell 3 - MgO cup, charge 0.45 g., $a = 0.503 \text{ mm}^2$, $l = 3.119 \text{ cm}$, $r = 0.690 \text{ cm}$				
L1	1428	0.37	30.00	4.84×10^{-8}
L2	1465	0.47	20.23	9.23 "
L3	1481	0.45	15.00	12.0 "
L4	1509	0.49	10.23	19.3 "
N1	1526	0.059	4.00	28.4 "
M1	1133	0.00072	61.25	4.11×10^{-11}
M2	1206	0.0052	62.70	2.99×10^{-10}
M3	1250	0.0086	33.03	9.57×10^{-10}
M4	1282	0.0088	15.00	2.18×10^{-9}
M5	1347	0.019	8.00	9.05 "
M6	1348	0.019	8.02	9.03 "
M7	1398	0.032	5.01	24.8 "

TABLE I, continued

Exposure No.	Temperature (°K)	Pu collected (μg)	Time (min)	P _{Pu} (atm.)
Cell 3 - MgO cup, charge 0.45 g., a = 0.503 mm ² , l = 3.119 cm, r = 0.690 cm				
M8	1438	0.040	3.01	52.3 x 10 ⁻⁹
M9	1188	0.0032	60.00	1.91 x 10 ⁻¹⁰
Cell 4 - Pu ₂ O ₃ cup, charge 0.42 g., a = 0.515 mm ² , l = 3.345 cm, r = 0.323 cm				
P1	1477	0.192	30.00	1.26 x 10 ⁻⁷
P2	1530	0.161	10.00	3.25 "
P3	1577	0.107	3.01	7.25 "
P4	1625	0.113	1.51	15.5 "
P5	1478	0.190	30.00	1.25 "
P6	1423	0.070	29.99	0.453 "
P7	1479	0.189	30.01	1.25 "
Q1	1533	0.160	10.01	3.19 "
Q2	1585	0.155	4.00	7.86 "
Q3	1647	0.401	4.00	20.7 "
Q4	1688	0.371	2.01	38.6 "
Q5	1742	0.834	2.01	88.2 "
Q6	1792	1.32	2.00	1.42 x 10 ⁻⁵

Figure 1. Observed Plutonium Vapor Pressure Data

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made with increasing temperature, next the background pressure was increased by leaking dried air into the system and the three exposures repeated. Then the leak was closed, good vacuum restored, and the three exposures repeated. Table II gives the data. Although there is some scatter, no effect of background pressure can be seen.

In the second series, the sample was in a TaC cup. After some exposures at various temperatures, the temperature was held constant, a series of 6 exposures was made at low background pressure giving a constant vapor pressure, next the leak was opened and an exposure was made at high background pressure, then the leak was closed and three more exposures made at low background pressures. The pertinent data are given in the second part of Table II. Again, no effect of background pressure was seen. Any variation is within a random scatter of $\pm 10\%$ which is the standard deviation of all the data.

The third series, F, was run in the same cell with the same charge and ThO_2 cup as was used in series E. Here the temperature was held as constant as possible near 1530°K and the background pressure was varied by a factor of 500. The results are in the third part of Table II. The observed vapor pressure decreased when the background pressure was increased and did not completely recover when good vacuum was restored. Increasing the background pressure (exposure F7) a second time did not appear to affect the vapor pressure. In the poor vacuum of 5×10^{-4} , the tantalum heater oxidized somewhat and changed its resistance, making it difficult to hold a constant temperature throughout the series. After this series, the cell was opened and the charge was seen to be covered with a dark oxide film. Normally the charges had bright surfaces after use.

4. Interpretation and Discussion of Results

4.1 Vapor Pressure

Forty-five usable measurements listed in Table I were obtained over a temperature range from 1133 to 1792°K . The least squares fit to these points gave $\log p(\text{atm.}) = -17420/T + 4.913$ with a standard deviation of ± 0.045 in $\log p$. The maximum deviation of any point was 0.058. This standard deviation is equivalent to ± 10 per cent in the absolute vapor pressure. The corresponding standard deviations of slope and intercept are ± 100 and $\pm .070$, respectively.

4.2 Thermodynamics

The enthalpy and entropy of vaporization may be obtained directly from the above equation as $\Delta H_v = 79.70 \text{ kcal/g-atom}$ and $\Delta S_v = 22.48 \text{ cal/deg/g-atom}$. These values apply over the temperature range of the measurements.

TABLE II
Effects of Background Pressure

Exposure No.	T(°K)	Pu pressure (atm.)	Background pressure(torr)
E1	1451	6.71×10^{-8}	1.1×10^{-6}
E2	1528	2.72×10^{-7}	1.3×10^{-6}
E3	1673	2.72×10^{-6}	2.9×10^{-6}
E4	1446	7.11×10^{-8}	2.3×10^{-5}
E5	1528	2.82×10^{-7}	2.5×10^{-5}
E6	1679	3.12×10^{-6}	2.6×10^{-5}
E7	1448	8.14×10^{-8}	9.9×10^{-7}
E8	1524	2.73×10^{-7}	9.5×10^{-7}
E9	1683	3.12×10^{-6}	2.5×10^{-6}

Above with ThO₂ cup in Ta cell

H6	1531	3.68×10^{-7}	7×10^{-7}
H7	1531	4.01×10^{-7}	3×10^{-5}
H8	1533	4.01×10^{-7}	9×10^{-7}

Above with TaC cup in Ta cell

F1	1532	4.06×10^{-7}	2×10^{-6}
F2	1532	3.94 "	1×10^{-6}
F3	1524	2.38 "	5×10^{-4}
F4	1536	2.43 "	5×10^{-4}
F5	1531	2.55 "	4×10^{-6}
F6	1529	2.76 "	2×10^{-6}
F7	1528	2.68 "	9×10^{-5}
F8	1527	2.72 "	3×10^{-6}
F9	1526	2.80 "	2×10^{-6}

Above with ThO₂ cup in Ta cell. Same charge as run E.

In order to extend the range of usefulness and also to provide a check of the reasonableness of the data, free energy functions are required for the condensed and gaseous phases involved. As these are not available per se in the literature, a discussion of their computation is included here.

Enthalpy increments and free energy functions for monatomic plutonium gas have been calculated by Feber and Herrick [5] from spectroscopic data in the literature. Values appropriate for the present calculations are listed in Table III.

Measurements of the thermal properties of solid plutonium have been made above 298°K by Kay and Loasby [6] as well as by others. Loasby [7] has measured the heat capacity between 17 and 298°. Loasby's is thus the most complete set of data available, all from the same laboratory. Other measurements above 298° are in substantial agreement with Loasby's data. However, Loasby's high and low temperature heat capacity curves fail to match by about 8% at 298°K. An arbitrary smooth join was therefore made between the two heat capacity curves over the range 180 to 320°K. The extrapolation between 17 and 0°K was of the form $C_p = AT^3 + \gamma T$. The enthalpy increments and free energy functions listed in Table III for solid and liquid plutonium were calculated from the smoothed curve up to 320°, Loasby's data was used directly for temperatures between 320 and 913°, and an estimated constant heat capacity of 8.5 cal/deg/g-atom for the liquid was used for all temperatures above 913°.

With the enthalpy increments in Table III, the Second Law heat of vaporization at 298° may be calculated from the enthalpy of vaporization given above. The result is $\Delta H_{298}^\circ = 82.14$ kcal/g-atom. The Third Law heat of vaporization at 298° may be calculated by combining the free energy functions with the experimental pressures. The result is $\Delta H_{298}^\circ = 82.28$ kcal/g-atom. The agreement between Second and Third Law heats is excellent.

We estimate the average heat capacity change for the transition from liquid to gas to be $\Delta C_p = +2$ cal/deg/g-atom over a large temperature range. (The heat capacity of the gaseous plutonium atom appears to have an extraordinarily high electronic contribution as calculated from the energy level data.) Extrapolation of our data yields a normal boiling point of 3600°K and an entropy of vaporization at the boiling point of 23.0 cal/deg/g-atom which agrees well with the Pictet-Trouton rule.

4.3 Effect of Oxygen

The vapor pressure determinations listed in Table I were obtained with the plutonium charge in contact with tantalum, tantalum carbide, magnesia, or plutonium sesquioxide. All these points fit well on the single vapor pressure line and no possible correlation of deviations from the least square line with cup material is evident. We believe that any effect of more than a few per cent in the vapor

TABLE III
Thermodynamic Functions

T °K	$(H_T^\circ - H_{298}^\circ)(s, l)$	$(H_T^\circ - H_{298}^\circ)(g)$	$-(F_T^\circ - H_{298}^\circ)/T(s, l)$	$-(F_T^\circ - H_{298}^\circ)/T(g)$
	cal/g-atom	cal/g-atom	cal/deg/g-atom	cal/deg/g-atom
298	0 (s, α)	0	13.45 (s, α)	42.32
913	7569 (l)	3667	18.87 (l)	44.72
1100	7728	5139	20.42	45.53
1200	8578	5994	21.15	45.95
1400	10278	7842	22.44	46.76
1600	11978	9860	23.55	47.55
1800	13678	12020	24.55	48.31
for Pu(s, α)	$H_{298}^\circ - H_0^\circ =$	1743 cal/g-atom		
for Pu(g)	$H_{298}^\circ - H_0^\circ =$	1482 cal/g-atom		

pressure, caused by oxygen in the charges, would have been detected. It is conceivable that the plutonium charge was saturated with oxygen in all the cells. However, the solubility of oxygen in liquid plutonium is only 20 to 30 ppm by weight at 1500°K [8]. This is an atom fraction of 0.0005, hardly sufficient to cause appreciable lowering of vapor pressure without an incredible deviation from the usually accepted solution laws. In addition, data were obtained over a large range of temperature with no deviation from a straight line plot of $\log p$ vs $1/T$. This is the behavior expected for a simple vaporization process. If there were two vaporizing species, for example $\text{Pu}(g)$ and $\text{PuO}(g)$, or if the oxygen solubility were strongly temperature dependent, deviation from a straight line would be expected because it is unlikely that the various equilibrium constants involved would have the same temperature dependence.

The results of the experiments on the effect of background pressure are interpreted as showing that the observed vapor pressure of plutonium is independent of the partial pressure of oxygen in the system at reasonably low partial pressures. In the one case where the vacuum was poor enough to modify the observed vapor pressure, the effect was physical in that a continuous oxide barrier formed over the vaporizing surface and impeded vaporization.

We thus conclude that there is no significant chemical effect of oxygen on the volatility of plutonium up to about 1800°K. A physical effect, in the form of an oxide skin on the liquid metal surface, can be observed under the right conditions.

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