High Temperature Vapor Pressures of Sodium, Potassium, and Cesium

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Saturation pressures were measured for sodium, potassium, and cesium to temperatures of 2540°, 2400°, and 2350° F., respectively, using a static method and a refractory metal apparatus with a diaphragm as a null-point detector. Vapor-pressure equations of the Kirchhoff type accurately represent the observed data for extended temperature ranges with standard per cent deviations of ±0.52 for sodium, ±0.43 for potassium, and ±0.53 for cesium.

FOR extended space flight, turbine power converters are being developed to supply power for auxiliary equipment. The turbines will require high-temperature working fluids, and the alkali metals with high heat-transfer coefficients and heats of vaporization are outstanding candidates. For the effective design of turbine systems, reliable values for the properties of the candidate metals are required. Several years ago some low-temperature properties of sodium, potassium, and sodium-potassium alloys were evaluated at this laboratory. This work was resumed in 1960 when high-temperature measurements of several thermophysical and thermodynamic properties of sodium, potassium, and cesium were undertaken. This article on vapor pressure is the second publication derived from this current series of measurements, and follows the reporting of superheat (*PVT*) results (*10*).

EXPERIMENTAL

Apparatus and Method. The saturation results reported were made in conjunction with the superheat studies. Measurements in both the saturation and superheat regions were made with small, closed chambers using flexible diaphragms as null-point detectors. The columbium-1% zirconium apparatus, the related furnace equipment, and the methods employed have been described (*10*), and only a brief summary is included here.

The measurements were made in a pressurized system with the columbium-1% zirconium apparatus supported at the center of a long longitudinal furnace (*10*). The procedure for a typical experiment was as follows. Equilibrium measurements of pressure and temperature were made for at least one complete cycle from the normal boiling point to the maximum temperature. At each equilibrium temperature, the furnace pressure was adjusted so that the diaphragm was just making or breaking electrical contact with an insulated probe. Under these conditions, the furnace pressure (except for a small deflection correction which was usually below 0.1 p.s.i.) was equal to the pressure of the metal vapor, and was observed externally at room temperature with a sensitive Bourdon-tube gage (*10*). An equilibrium condition for each determination was obtained by taking multiple readings of temperature and pressure at 5- to 10-minute intervals until successive readings showed a temperature drift of 0.07° F. per minute or less.

Heat loss from the apparatus by radiation and convection was reduced by shrouding it with thin sheets of columbium-1% zirconium and by installing shields spaced 1 inch apart to the top of the furnace. The isothermal condition of the apparatus was further assured by the relatively short length (3 inches) and large bulk (¼-inch wall) of the chamber, and by the adjustment of furnace heaters until observed temperatures at the top and bottom of the chamber were matched within 2° F. (generally within 1° F.).

Temperatures were measured with Pt 6% Rh-Pt 30% Rh thermocouples calibrated at this laboratory by direct comparison with two types of NBS standardized couples—Pt 6% Rh-Pt 30% Rh calibrated against primary melting points to the gold point and against optical pyrometers from 1800° to 3000° F.; and Pt-Pt 10% Rh likewise calibrated against primary melting points to 1920° F. with an extension to 2600° F. based on the standard quadratic relationship between e.m.f. and temperature. The use of two types of reference thermocouples for calibration, together with observations of instability (*10*), increased the reliability of the temperature measurements. Errors in observed temperatures (relative to the thermodynamic scale) to the gold point, 1920° F., should not exceed 0.8° F. Above the gold point, the error would be expected to increase with temperature, but should not exceed 3.5° F. at 2550° F.

Purity of Alkali Metals. Since the observed vapor pressure of an alkali metal is sensitive to small amounts of impurities, special techniques were required to purify and transfer the reactive metals. Each metal was distilled under high vacuum from either a nickel (Na and K) or glass (Cs) retort into small, test-tube-like ampoules made of the columbium alloy for subsequent transfer into the measurement apparatus. All transfer and welding operations were performed in a vacuum-inert gas box.

For sodium and potassium, the metals introduced to the still pot were high-purity grades from E. I. du Pont de Nemours & Co. and MSA Research Corp., respectively. Typical spectrographic analyses of these metals after one distillation at this laboratory are presented in Table I. The cesium introduced to the borosilicate glass distillation retort for all the superheat tests and the majority of the vapor-pressure tests was a high-purity grade from the MSA Research Corp. A typical spectrographic analysis of this cesium after one distillation at this laboratory is presented in Table I. Although the metal was distilled at low temperature under high vacuum, the still may have introduced some of the metal impurities, particularly silicon and sodium. A high-purity grade of cesium from Dow Chemical Co. was used for one vapor-pressure experiment. The distilled sample of this cesium for analysis was lost, and the data reported in Table I are for an "as received" sample oxidized on borosilicate glass. It is very probable that silicon, aluminum, and sodium were introduced by reaction with glass under these conditions, and it is recognized that the analysis is unsatisfactory. However, since the volatile

Table I. Spectrographic Analyses of the Alkali Metals

Metal Impurity	Distilled Na (Du Pont), P.P.M.	Distilled K (MSAR), P.P.M.	Distilled Cs (MSAR), P.P.M.	As Received Cs (Dow Chemical), P.P.M.
Na		10–100	100^a	1000^a
K	10–100		< 10^a	1^a
Rb	ND^b	100–1000	500^a	10^a
Cs	ND	ND		
Li	ND	ND	ND	ND
Ca			1–10	10–100
Al			ND	100–1000
B			< 1	
Si		< 1	10–100	100–1000
Mn			ND	< 1
Fe			ND	< 1
Mg		< 1	< 1	< 1
Cu		1–10	ND	10–100
Ba, Sr			ND	ND

^{a} Used standard samples for comparison; figures should be quantitative. ^{b} Not detected.

and nonvolatile inpurities in the MSAR metal (and probably the Dow sample too) are present in concentrations too low to produce a measurable vapor-pressure change, no additional analytical work was performed.

Table II. Saturated Vapor Pressures of Sodium

Temp., ° F.	Pressure, Abs. Atm.	Temp., ° F.	Pressure, Abs. Atm.
VAPOR-PRESSURE EXPERIMENT			
1693.3	1.4283	2511.6	22.297
1800.7	2.3197	2443.4	18.851,
1910.1	3.587	2381.1	16.065
2009.2	5.172	2304.8	13.077
2116.7	7.442	2223.9	10.363
2183.3	9.155	2152.5	8.329
2262.4	11.582	2053.0	6.023
2331.4	14.063	1947.0	4.131
2413.1	17.466	1837.3	2.7035
2470.1	20.164	1722.1	1.6399
2539.2	23.821	1628.1	1.0472
		1593.6	0.8737
VAPOR PRESSURE FROM *PVT* EXPERIMENTS			
1690.0	1.4242		
1769.2	2.0325	1640.5	1.1187
1851.4	2.8729	1772.4	2.0645
1947.4	4.113	1872.0	3.1138
1810.4	2.4081	1976.7	4.607
1661.5	1.2391	2075.5	6.505
1692.9	1.4249	2189.7	9.347
1635.8	1.0867	2299.8	12.916
1709.2	1.5365	2149.2	8.237
1651.5	1.1697	2017.0	5.318
1634.4	1.0744	1906.7	3.562
1645.5	1.1500	1738.4	1.7835
1758.2	1.9291	1636.9	1.1085
1854.1	2.8933	1688.4	1.4017
1958.9	4.301	1633.4	1.0772
2062.0	6.212	1695.8	1.4630
2168.6	8.764	1825.3	2.5681
2135.3	7.831	1760.5	1.9522
2020.6	5.348	1696.9	1.4671
1905.0	3.506	1629.1	1.0629
1716.4	1.601	1636.9	1.0860
1765.5	1.9700	1863.9	2.9831
1873.6	3.1056	1810.4	2.4102
1988.7	4.799	1678.0	1.3371
1937.6	3.994	1648.4	1.1622
1813.7	2.4442	1779.6	2.1366
1700.7	1.4820	1880.6	3.2288
1623.7	1.0221	1998.7	4.986
1639.2	1.0853	1937.0	3.996
1760.3	1.9223	1728.4	1.7052

RESULTS

Eight to 12 *PVT* experiments (*10*) were performed for each alkali metal. For each of these, equilibrium measurements of pressure and temperature were obtained along the saturation curve. The available saturation range was limited for each experiment, since a predetermined weight of the alkali metal was added in order to obtain superheat data for a specific pressure range. In addition to the saturation results obtained from the regular *PVT* experiments, at least one special saturation experiment was performed for each metal. In each of these, a large excess of the alkali metal (over that required for saturation at the maximum temperature) was added to the chamber, and saturation results were obtained for the full temperature range.

The observed vapor pressure for sodium, potassium, and cesium are recorded in Tables II, III, and IV, respectively. Each table is divided into two sections; the results in the top section are those from the one or more special saturation experiments and those in the lower section are from the *PVT* experiments. One feature of the saturation results from the *PVT* study should be discussed. As pointed out previously (*10*), the saturation pressures observed for each experiment near intersection of the saturation and superheat curves were always below corresponding results on the reported saturation curve. It is believed that the pressures are lowered by some physical phenomenon in combination with the diminishing liquid phase and that they do not represent true saturation values. Accordingly, the observed pressures in these regions are not included in the tables.

Calculations indicated that the volume of metal remaining in the liquid phase at the point of deviation from the observed saturation curve was generally around 0.15 cc. For several experiments, steady-state temperature differences up to 10° F. were induced in both directions between the two ends of an apparatus, and pressures measured under these conditions were compared to corresponding values under isothermal conditions. This comparison was improved by controlling the low temperature, whether at the top or bottom, at the temperature of the equilibrium run. For experiments of this type where the remaining liquid volume was small, but greater than 0.15 cc., the effective temperature was invariably the condensing or low value. Observed pressures when based on the condensing temperature were within ±0.2% of the equilibrium value, and even these small deviations were of magnitude and direction to be explained by temperature lowerings of a few tenths of a degree induced at the extremities of the chamber. For similar experiments with larger volumes of the liquid phase, the effective temperature was the condensing value if the bottom of the apparatus was cooler than the top but was the average if the reverse was true. Even though the temperature differences existing between the ends of an apparatus during the saturation

Table III. Saturated Vapor Pressures of Potassium

VAPOR-PRESSURE EXPERIMENTS

Temp., ° F.	Pressure, Abs. Atm.	Temp., ° F.	Pressure, Abs. Atm.
1433.1	1.205	2186.6	17.062
1584.7	2.420	2152.2	15.648
1691.2	3.713	2115.1	14.229
1793.7	5.384	2078.8	12.921
1901.5	7.687	2046.0	11.796
2009.8	10.648	1971.7	9.546
2111.2	14.083	1869.6	6.958
2215.6	18.323	1765.2	4.903
2334.0	24.062	1647.9	3.153
2376.3	26.332	1507.3	1.7379
2393.1	27.309	1421.6	1.1568
2362.3	25.597	1392.8	0.9982
2293.4	21.992		
2292.3	21.943		
2253.0	20.042		

VAPOR PRESSURE FROM *PVT* EXPERIMENTS

Temp., ° F.	Pressure, Abs. Atm.	Temp., ° F.	Pressure, Abs. Atm.
1417.6	1.134	1441.0	1.282
1532.4	1.934	1557.9	2.188
1635.1	2.987	1675.6	3.532
1736.2	4.403	1788.4	5.320
1836.9	6.276	1900.8	7.714
1954.3	9.049	2004.1	10.523
2076.3	12.766	2108.3	13.981
2149.4	15.474	2187.1	17.062
2005.3	10.536	2233.2	18.973
1888.3	7.409	2151.3	15.615
1460.8	1.384	2151.3	15.625
1583.8	2.405	2061.7	12.348
1698.8	3.823	1974.4	9.651
1813.6	5.775	1938.4	8.652
1781.4	5.178	1871.8	7.032
1652.4	3.225	1813.5	5.812
1499.5	1.696	1755.3	4.748
1595.9	2.562	1474.8	1.502
1430.1	1.199	1529.2	1.907
1539.1	2.002	1644.4	3.097
1638.5	3.040	1755.1	4.726
1736.2	4.413	1859.4	6.746
1812.2	5.776	1962.1	9.289
1925.1	8.274	2067.1	12.518
2009.3	10.603	2173.5	16.527
1975.1	9.591	2228.9	18.832
1864.2	6.823	2125.6	14.603
1856.8	6.683	2006.4	10.589
1774.0	5.054	1919.8	8.164
1675.0	3.511	1821.2	5.949
1573.0	2.323	1707.3	3.979
1479.0	1.526	1420.4	1.142
1420.1	1.131	1552.9	2.110
1535.4	1.948	1656.9	3.261
1596.5	2.550	1756.3	4.732
1716.7	4.101	1485.8	1.580
1607.0	2.672	1398.6	1.035

Table IV. Saturated Vapor Pressures of Cesium

VAPOR-PRESSURE EXPERIMENTS

MSA Research Corp. Sample

Temp., ° F.	Pressure, Abs. Atm.	Temp., ° F.	Pressure, Abs. Atm.
1238.0	1.0169	2276.1	29.384
1346.6	1.7426	2218.7	26.195
1428.4	2.5054	2169.5	23.632
1535.0	3.829	2140.1	22.178
1618.3	5.179	2067.7	18.833
1699.8	6.825	2027.3	17.108
1785.9	8.918	1943.3	13.885
1885.2	11.857	1857.1	11.013
1977.8	15.108	1759.2	8.263
2100.8	20.264	1677.6	6.354
2183.5	24.276	1588.6	4.672
2243.1	27.471	1491.5	3.2325
2291.0	30.241	1440.0	2.6191
2345.5	33.530	1318.6	1.5122
2316.6	31.738	1214.5	0.8849

Dow Chemical Co. Sample

Temp., ° F.	Pressure, Abs. Atm.	Temp., ° F.	Pressure, Abs. Atm.
1353.4	1.7991	2247.2	27.730
1505.6	3.438	2177.2	24.015
1654.4	5.905	2130.5	21.720
1806.6	9.543	2034.2	17.392
1948.3	14.068	1888.4	12.039
2091.8	19.909	1761.6	8.396
2201.0	25.215	1601.8	4.925
2322.0	32.067	1431.5	2.5592
2287.9	30.071	1245.5	1.0629

VAPOR PRESSURE FROM *PVT* EXPERIMENTS

Temp., ° F.	Pressure, Abs. Atm.	Temp., ° F.	Pressure, Abs. Atm.
1277.0	1.2421	1284.0	1.2672
1440.6	2.6328	1449.7	2.7180
		1332.1	1.6123
1509.5	3.465	1197.4	0.8110
1377.5	1.9968	1342.0	1.7030
1322.8	1.5498	1480.4	3.1044
1229.6	0.9662	1619.1	5.234
1214.5	0.8794	1546.9	4.026
1361.1	1.8492	1420.4	2.4223
1495.8	3.2740	1312.5	1.4970
1391.8	2.1516	1264.0	1.1811
1510.7	3.503	1404.9	2.2671
1606.9	5.000	1547.7	4.045
1447.6	2.7089	1689.7	6.636
1340.5	1.6879	1262.4	1.1450
1251.9	1.0936	1387.9	2.0987
1403.3	2.2469	1560.5	4.226
1553.9	4.135	1685.6	6.532
1688.5	6.607	1827.6	10.072
1822.1	9.998	1747.5	7.947
1974.5	15.034	1642.6	5.654
1285.4	1.2836	1489.5	3.1996
1436.1	2.5700	1363.5	1.8702
1573.6	4.415	1532.3	3.786
1708.0	7.006	1930.4	13.395
1852.8	10.844	1755.8	8.158
1985.7	15.495	1474.3	3.0178
2131.9	21.796	1281.9	1.2674
2194.4	24.725	1333.3	1.6746
2080.8	19.346	1465.2	2.9205
1923.7	13.164	1480.8	3.1151
1776.1	8.683	1373.5	1.9549
1651.3	5.821	1252.8	1.0887
1514.5	3.533		

experiments were generally less than 1° F., the temperatures recorded in the data tables represent the effective values as defined by the experiments just described.

The vapor-pressure data for all three metals are presented graphically in Figure 1. Grey (5) has shown that a graph of log p *vs.* $1/T$ can be linear over rather wide temperature ranges for an associating gas, if the apparent heat of vaporization (5) is practically independent of temperature. However, since this term is usually dependent upon temperature, log p *vs.* $1/T$ should exhibit curvature. While it is not evident in Figure 1, a larger scale plot reveals that log p *vs.* $1/T$ is not linear for these metals. However, the results for each can be effectively fitted for the full temperature range with one three-term equation of the Kirchhoff type. When the data were precisely fitted with an equation of this type, deviations of observed and calculated pressures were completely random with no temperature-dependent distribution.

The least-squares principle was used in programming a computer to determine the best equation to represent the observed data. In at least one published treatment of vapor pressure (*12*), the values of log p are weighted by p^2. It is true that a change in the dependent variable from p to log p does imply a weighting factor of $(p/E_p)^2$ where E_p is the probable error of p. Accordingly, if the probable error of pressure over its entire range is constant, the correct weighting factor in the least-squares treatment is p^2. However, in the present determinations of vapor pressure with the three Bourdon-tube gages, E_p was roughly proportional to p over the entire pressure range, and no weighting of log p was employed in the least-squares treatments.

In order to evaluate the precision of the vapor-pressure measurements and to establish statistically the requirement for the log T term, several equations were obtained for each metal by various treatments of the observed data.

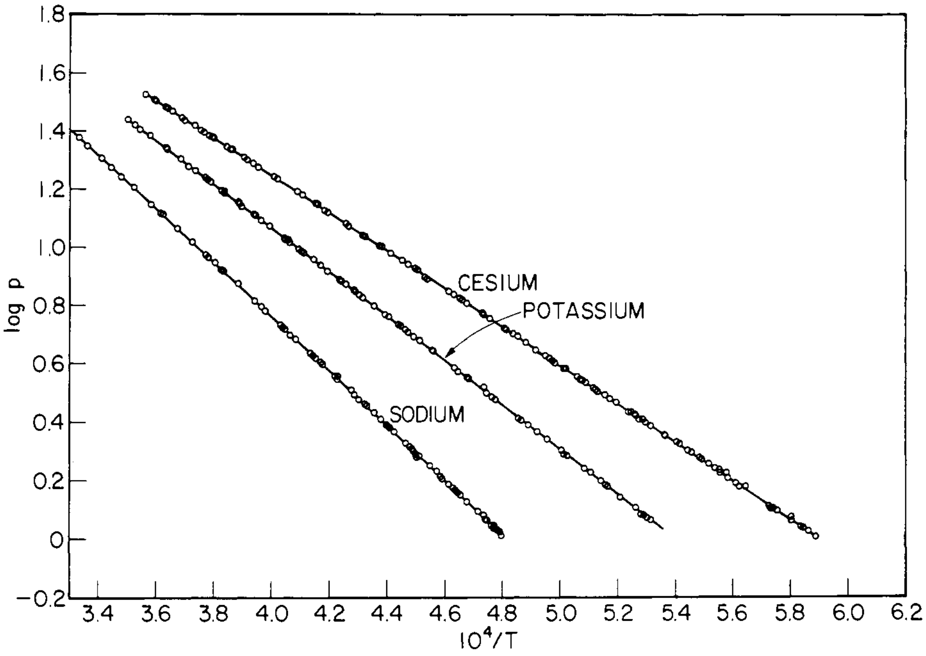

Figure 1. Vapor pressures of sodium, potassium, and cesium

In one case, the coefficients for the Kirchhoff equation were derived using all observed results. In a second case, coefficients for the same equation were derived solely from the data of the special vapor-pressure experiments (first section of each data table). In a third case, coefficients were derived from all of the observed results setting $C = 0$. The derived coefficients for each least-squares equation are presented in Table V along with the corresponding normal boiling point, the standard deviation (of log p), and the standard per cent deviation (of p). The deviation figures reveal that the log T term for each metal has statistical significance and that the Kirchhoff form should be used. The two equations of this type for each metal are effectively equivalent, but those given below, which are based on all data, are preferred.

$$\log p_{\mathrm{Na}} = 6.83770 - 9980.9/T - 0.61344 \log T \quad (1)$$

$$\log p_{\mathrm{K}} = 6.13905 - 8135.3/T - 0.53560 \log T \quad (2)$$

$$\log p_{\mathrm{Cs}} = 5.87303 - 7040.7/T - 0.53290 \log T \quad (3)$$

Throughout the text of this article, p is in absolute atmospheres and T is in ° R., but in the tabulated data, pressure is in pounds per square inch absolute and temperature is in degrees Fahrenheit.

DISCUSSION

The current saturation results are compared to those of previous investigators in Figure 2. For each metal the authors' results are arbitrarily taken as standard, and the per cent deviation of the vapor pressure of each investigator from that of this laboratory is plotted as a function of temperature. It is evident that the authors' results for each metal show good agreement with those of at least two other investigators, and that the deviation between any two sets of results can generally be accounted for by combined experimental error. Kirilov and Grachev have reported results for both sodium (*6*) and potassium (*4*), but the data deviate widely from all published work and have not been included in Figure 2.

Several comments regarding the purities of the alkali metals are pertinent to this discussion. Vapor pressures were observed for cesium metal from two independent sources: the MSA Research Corp. and the Dow Chemical Co. The concentrations of impurities in the MSAR metal by spectrographic analysis (Table I) are too low to affect vapor pressure, but no satisfactory analytical information was obtained for the other metal. However, vapor-pressure results in Table IV were obtained for both metals and are in agreement within experimental accuracy. The stand-

Table V. Derived Coefficients for the Vapor-Pressure Equations of Sodium, Potassium, and Cesium

$$\log p = A + B/T + C \log T$$

Metal	Data Used in Deriving Coefficients[a]	Coefficients A	B	C	Normal B.P., ° F.	Std. Dev. Using All Data	Std. % Dev. Using All Data
Sodium	X	6.83770	−9980.9	−0.61344	1618.6	0.0023	0.52
	Y	7.00980	−10035.2	−0.65769	1619.0	0.0023	0.53
	X	4.49344	−9334.7		(1617.7)	0.0027	0.61
Potassium	X	6.13905	−8135.3	−0.53560	1394.0	0.0019	0.43
	Y	5.91305	−8081.5	−0.47549	1394.2	0.0020	0.45
	X	4.09303	−7572.9		(1390.5)	0.0031	0.70
Cesium	X	5.87303	−7040.7	−0.53290	1236.0	0.0023	0.53
	Y	5.87272	−7039.4	−0.53290	1235.8	0.0023	0.53
	X	3.86469	−6546.4		(1234.2)	0.0031	0.71

[a] X All data, Y Vapor-pressure experiments only.

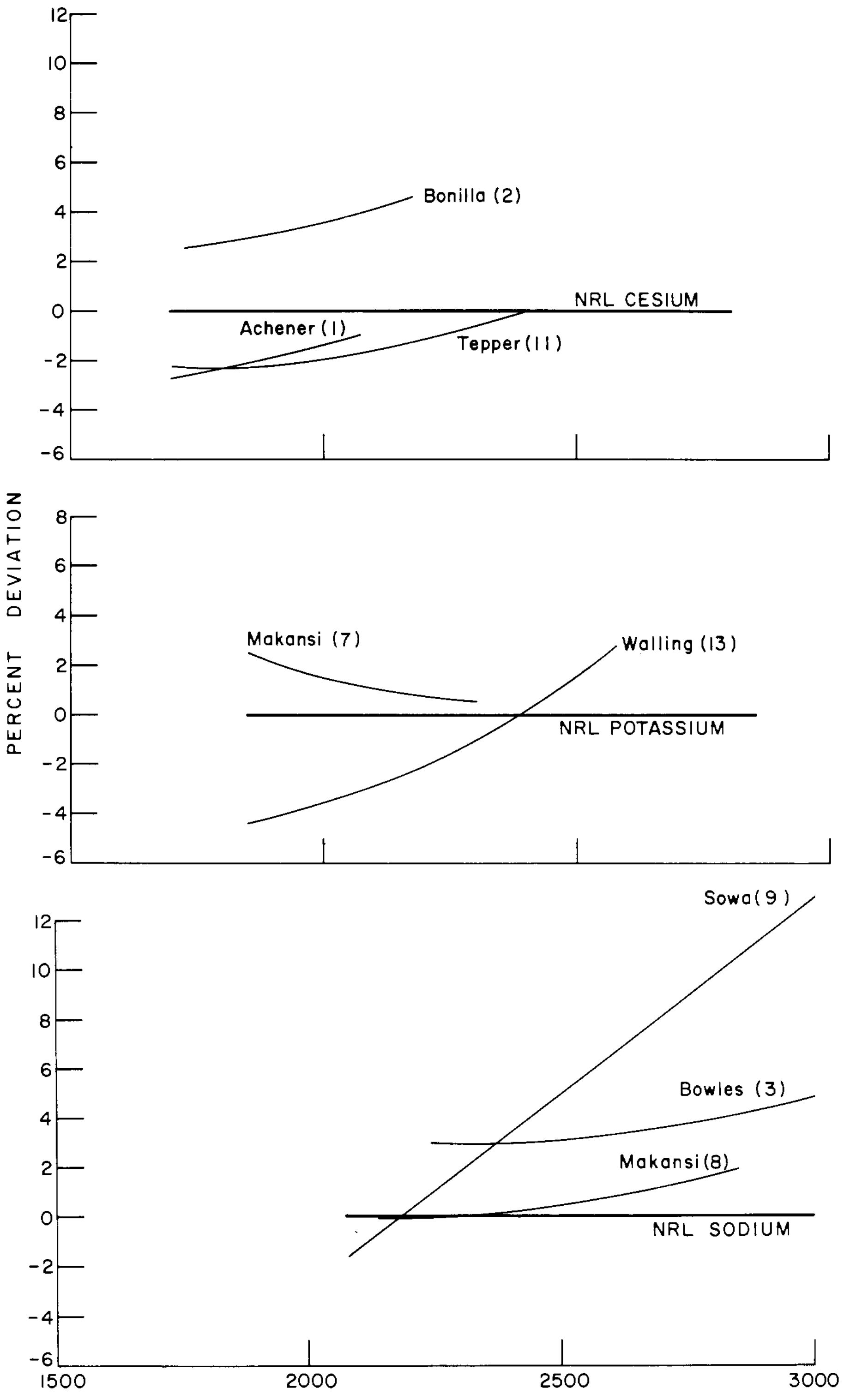

Figure 2. Comparison of vapor-pressure data of alkali metals

ard per cent deviations of the data for the MSAR metal and for the Dow metal (from Equation 3) are ±0.31 and ±0.44, respectively. This agreement implies that the impurities in the Dow metal are also present in low concentrations. Reliable spectrographic analyses were made for typical samples of sodium and potassium (Table I). Metal impurities in these materials are present in low concentrations, and calculations using Raoult's law show that they would not have affected the observed vapor pressures. Alkali oxides are probably present in low concentrations in the original charge of each metal, but according to existing free-energy data, they should be "gettered" at operating temperatures by the columbium and zirconium of the container alloy.

The design of the diaphragm apparatus and all related equipment was approached from the standpoint of obtaining maximum accuracy. The precautions taken to ensure high accuracy in the various measurement parameters were discussed previously (*10*) and are reflected in the precision of the saturation measurements as demonstrated by the low standard deviations.

The measurement of saturation pressure directly with a null-point apparatus of the diaphragm type is new to the high-temperature field, and some discussion of the relative merits of this method is of interest. Since the boiling point apparatus, in various modifications, is so often used with liquids for the measurement of saturation pressures, it is appropriate to compare it with the null-point technique.

The several sources which generally contribute to error in measuring vapor pressures are: temperature measurement, pressure measurement, volatile and nonvolatile impurities, method of selection of the equilibrium temperature for the observed pressure, superheating of the liquid, and Poynting effect. The first three sources are common to all vapor-pressure methods and errors may be controlled by the choice and calibration of measuring devices and by control of the purity of the material being measured. In many measuring devices, particularly of the boiling point type, a temperature difference exists along the length of the apparatus, and errors can be generated by superheating of the liquid, by undetected cold spots, or by a subjective selection of the equilibrium temperature. If the null-point apparatus is designed of short length and large bulk, the complete apparatus may be maintained at the same temperature during a pressure measurement, and no choice as to equilibrium temperature is required. In most apparatus of the boiling point type, an inert gas is used in contact with the vapor and unless proper precautions are taken, error can be generated by diffusion of the inert gas to the surface of the liquid. Since no filling gas is in contact with the contact with the vapor in the null-point apparatus, there can be no Poynting effect.

There are, as one might expect, sources of error which are peculiar to the null-point apparatus. These include: the diffusion of gases through the diaphragm at higher temperatures, the inclusion of inert gas in the chamber during the welding off operation, and sensitivity corrections to the diaphragms. These have been rather fully discussed (*10*), and it has been demonstrated that with the Cb-1%Zr apparatus, these sources of error can be controlled at very low levels.

ACKNOWLEDGMENT

The authors are grateful to the National Aeronautics and Space Administration for support of this work. They also thank Janet P. Mason or the NRL Applied Mathematics Staff for computer programming and Samuel H. Cress of the NRL Metallurgy Division for spectrochemical analyses of metal samples.

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RECEIVED for review August 20, 1965. Accepted March 17, 1966.

High Temperature Specific Volumes of Liquid Sodium, Potassium, and Cesium

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The specific volumes of liquid sodium, potassium, and cesium (at saturation) were measured with pycnometers over the temperature ranges from 1577° to 2491°, 1099° to 2287°, and 1577° to 2304° F., respectively. The results are correlated with those published for lower temperatures, and the specific volume of each metal is well defined by a single equation from its melting point to the maximum temperature of the current measurements.

THE alkali metals are attractive as thermodynamic working fluids in advanced power converters. In order to evaluate the performance of the metals in turbines, boilers, and condensers, an increased knowledge of their liquid and vapor properties at higher temperatures is required. One objective of the program at this laboratory was to derive from *PVT* observations various thermodynamic properties (including Mollier diagrams) for sodium, potassium, and cesium vapors. In the reduction of the *PVT* data, the specific volume of each liquid metal from its normal boiling point to 2500° F. was required. Since there were no published values in the upper part of this range, measurements were undertaken and the results are reported here. This article is the third of a current series on the alkali metals and follows previous ones on *PVT* properties (*9*) and vapor pressures (*10*).