large volume of n-butyl alcohol, and its accuracy has been shown by its application to known mixtures of potassium and sodium chlorides.

2. The solubilities of potassium and sodium perchlorates in n-butyl alcohol have been determined.

3. The method was successfully applied in the separation and determination of known mixtures of potassium and sodium chloride including 20 determinations duplicating practical working conditions.

4. The method is particularly well adapted to the determination of potassium in the combined chlorides, the sodium being obtained by difference.

5. The use of n-butyl alcohol in proposed work on the separation and determination of potassium, sodium and lithium is discussed.

URBANA, ILLINOIS

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF ILLINOIS]

THE HEATS OF VAPORIZATION OF MERCURY AND CADMIUM

BY MAYOR F. FOGLER[1] WITH WORTH H. RODEBUSH

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Probably the application of the quantum theory that is of most importance to physical chemists is the calculation of the entropies of the monatomic gases.[2,3] The expression[4,5] relating the vapor pressure of a monatomic liquid to its heat of vaporization also promises to be an application of considerable significance. While it is probable that the numerical results furnished by the quantum theory in these cases will be more exactly confirmed by more accurate data as in the case of other triumphs of the quantum theory, nevertheless there is always a danger in generalizing from meager or uncertain data.

The two elements that offer the best opportunity for a check upon the equation for the entropy of a monatomic gas are mercury and cadmium. The specific heats of these metals have been determined with considerable accuracy both for the solid and liquid, and the heats of fusion are known. There is, however, no direct determination of heat of vaporization of any metal described in the literature, that appears to be at all reliable. It is possible to calculate heat of vaporization from vapor-pressure data by means of the thermodynamic relation,

$$\frac{d\, ln\, p}{dT} = \frac{\Delta H}{RT^2} \qquad (1)$$

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[1] This communication is an abstract of a thesis submitted by M. F. Fogler in partial fulfilment of the requirements for the degree of Doctor of Philosophy in Chemistry at the University of Illinois.

[2] Tolman, THIS JOURNAL, **42**, 1185 (1920).

[3] Lewis, Gibson and Latimer, *ibid.*, **44**, 1008 (1922).

[4] Dushman, *ibid.*, **43**, 397 (1921).

[5] Rodebush, *ibid.*, **45**, 606 (1923).

However, the above equation can be used with exactness only at low pressures, which is precisely where the data are subject to the greatest percentage errors. Since it involves the slope of a curve it requires, for an accurate determination, an empirical equation representing the data to be set up and differentiated. In the case of mercury, Smith and Menzies[6] and Menzies[7] have set up an empirical equation for which they claim an accuracy of 1% at temperatures above 120°. In their equation, there is no term to take care of the variation of the specific heat of liquid mercury with the temperature and, assuming that the accuracy is no greater than they claim, the heat of vaporization calculated at 298° K., from their equation might easily be in error by some hundreds of calories. 300 calories is one entropy unit at 25° C. and is a great enough amount to invalidate any generalization in regard to the entropy of monatomic gases. Since the heat of vaporization is not only the most uncertain factor in the calculation of the entropy of monatomic gases from the experimental data, but is also the fundamental quantity in the equation of Rodebush[5] for the vapor pressure of monatomic liquids, it seemed desirable to make as accurate a determination of it as possible for mercury and cadmium.

Experimental Part

Some time was spent upon what promised to be a simple and rapid method of determining heats of vaporization at higher temperatures. This method consisted in dropping an iron plummet of known specific heat and known initial temperature into the vapor of the boiling metal and determining the weight of metal which condensed upon the plummet. It did not appear feasible to weigh the plummet while suspended in the vapor, although this could probably have been done, so that, after sufficient time had elapsed for the plummet to come to the temperature of the vapor, the vapor was swept away by a current of gas and the plummet was withdrawn and weighed. This operation was not difficult to carry out but the amount of metal collected depended on the time that the plummet was left in the vapor, indicating that the plummet was losing heat by radiation. Radiation shields were introduced in an attempt to remedy this difficulty, but the results were still very uncertain and variable. Apparently, at higher temperatures the transfer of heat by radiation is so rapid that the unavoidable slight difference in temperature between plummet and environment caused serious heat losses.

It was then decided to attempt to use for mercury a modification of the method employed so successfully by A. W. Smith[8] to determine the heat of vaporization of water. In principle, this method consists in determining the difference in the rate of input of electrical energy required

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[6] Smith and Menzies, This Journal, **32**, 1434 (1910).

[7] Menzies, *ibid.*, **41**, 1783 (1919).

[8] Smith, *Phys. Rev.*, **34**, 173 (1911).

to maintain a quantity of liquid in an insulated vessel at a constant temperature when vaporizing at a measured rate and when no distillation occurs. Smith maintained the water at a temperature just below its boiling point and controlled the distillation by bubbling a current of air through the liquid. It seemed undesirable to work with mercury near its boiling point because of the difficulty of preventing excessive heat losses due especially to radiation at high temperatures. It seemed better to work at the lowest temperature at which the mercury could be readily vaporized and this temperature was found to be about 140° where the vapor pressure of mercury is slightly greater than 1 mm. At this low pressure, the distillation could not be controlled by bubbling a current of inert gas through the mercury because it would be necessary to pass through so many moles of gas to each mole of mercury vaporized that serious errors would be introduced because of the heat capacity of the gas itself. Accordingly, the vaporization was controlled by distilling the mercury in a closed system maintained at constant pressure. When it was desired to commence the distillation the pressure was lowered until the mercury just began to vaporize; distillation was stopped by admitting gas and raising the pressure. The operation then consisted in bringing the vessel containing the mercury to a constant temperature, measuring the rate of input of electrical energy necessary to maintain this temperature for an indefinite time, then commencing the distillation and again measuring the rate of input of electrical energy necessary to maintain the same temperature as before, and the amount of mercury distilled in a given time.

The vessel containing the mercury to be distilled was placed in a Dewar tube, but in order to make the heat losses as small as possible, it was found desirable to place this part of the apparatus in some sort of a thermostat. An air thermostat was tried using a heating coil, fan stirrer, and an air thermometer regulator. It was not found possible to maintain uniform temperatures throughout this thermostat. Presumably an air thermostat can be kept at a uniform temperature provided the insulation of the walls is sufficiently good, but with our apparatus it was necessary to have so many openings through the walls that the insulation was poor and the regulation of the thermostat very unsatisfactory. A very satisfactory substitute was found in a vapor bath of dibutyl ether, which boils at 142°.

In Fig. 1 the general set-up of the apparatus is shown.

The vapor bath was made of copper with insulation (A) on the outside. H is the tube containing the mercury to be vaporized. The heating coil was wound around this tube, sheet asbestos was placed over the heating coil as insulation, and a sheath of heavy sheet copper was placed over the asbestos in order to equalize the temperature in different parts of the tube. A thermocouple was attached to the middle of this sheath. The tube H was about 10 cm. long by 2.5 cm. inside diameter; the vapor outlet was a tube of about 7 mm. diameter which was sealed through the bottom of the tube containing the mercury with a ring seal. The level of the mercury in the bulb was kept about 1 cm. below the opening of the vapor tube, and experiments made with the apparatus exposed to view showed that the boiling was not violent enough to throw any drops of liquid mercury into the vapor outlet. The tube H was placed in the Dewar tube which was inverted because it was necessary to maintain the temperature inside slightly higher than the temperature of the vapor bath in order that no vapor might condense inside it. The mouth of the Dewar tube was closed with a loosely fitting cork. In order to make sure that no dibutyl ether vapor might enter the Dewar tube and condense, a very slow current of air was kept passing in through the tube F. To maintain the very highest possible vacuum at all times in the Dewar tube it was connected through a liquid-air trap to a mercury-vapor pump, which in turn was connected to a large evacuated flask, thus eliminating the supporting pump. T_1 and T_2 are thermocouples. G is a magnetic plunger to dislodge any mercury that might condense on the sides of the tube, and N is a series of bulbs in which the distilled mercury was collected, sealed, and weighed. The connections to the heating coil were brought in at J. The source of the heating current was a lead storage battery. The energy input was measured by an ammeter and voltmeter calibrated to 0.1%. Pyrex glass was used exclusively in the apparatus. The mercury was carefully purified.

The arrangement of a Dewar tube inside of a vapor bath as described above gives a maximum of insulation with a minimum heat capacity. This is a very important consideration in an apparatus where it is necessary to maintain steady thermal conditions. It would be possible to decrease the heat losses by the use of insulating material, but such an apparatus would be unwieldy because of the length of time necessary to establish a steady state, and it is more important that the heat loss be constant than that it be small.

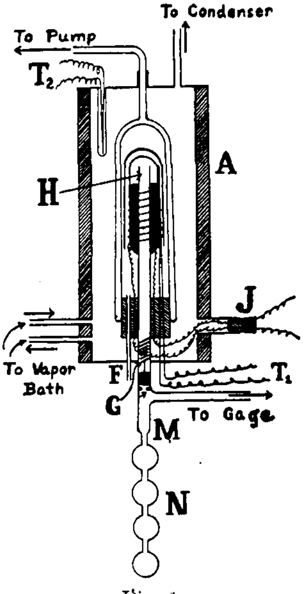

Fig. 1

The mode of operation was as follows. The apparatus was brought to the temperature of the vapor bath, and the pressure in the tube containing the mercury was lowered until the mercury just began to distil. The heating current was adjusted to maintain a temperature inside the Dewar tube slightly above the temperature on the outside. When steady conditions had been established, any mercury adhering to the sides of the vapor outlet tube was dislodged by the magnetic plunger, collected in the bottom bulb, sealed off, and discarded. At the same time a stop watch was started, and at intervals of 15 minutes the mercury was collected in successive bulbs, sealed off and weighed. In the meantime, the current and voltage were maintained constant. No error was involved in the measurement of the time at which the bulb was sealed off, because after the mercury had been shaken down into the bottom bulb with the magnetic plunger, the next portions of mercury which came over would condense in the upper part of the tube. As many as 5 bulbs were filled in succession. At the close of a run the pressure was raised to stop the distillation of the mercury and the current was readjusted to maintain the same temperature inside the Dewar tube as before, in order to correct for heat losses. Runs were made with temperatures inside the Dewar tube ranging all the way from 0° to 7° above the outside temperature. The correction for heat loss appeared to vary consistently with the temperature difference, being zero when the temperature difference was zero, and small in all cases. The value obtained for heat of vaporization appears to be quite independent of the correction made for heat loss. The results of 25 consecutive determinations range from 14,140 to 14,840 calories. These variations are believed to be due almost entirely to accidental errors, such as variation in the heat loss and the amount of mercury that adhered to the vapor outlet tube. The latter error would tend to be eliminated by our method of sealing off successive bulbs one after the other, since if one contained less mercury, the next would probably contain an excess. No results were omitted in this series of determinations, and it can be

TABLE I

HEAT OF VAPORIZATION OF MERCURY

Temp. of vapor bath ° C.	Temp. inside of Dewar app. ° C.	Heat input Cal.	Heat loss Cal.	Wt. of Hg G.	ΔH for Hg Cal.
140.2	142.4	398.48	41.25	5.0670	14,140
140.2	142.4	398.48	41.25	4.9105	14,590
140.2	143.4	398.48	41.25	4.9100	14,595
142.0	145.0	342.3	54.1	3.9690	14,565
142.0	145.0	362.2	54.1	4.2185	14,650
142.0	144.0	363.9	39.0	4.4545	14,610
142.0	144.0	363.9	39.0	4.4930	14,505
142.0	144.0	363.9	39.0	4.5060	14,465
140.1	146.2	404.3	101.0	4.2703	14,250
140.0	147.0	538.0	135.0	5.5990	14,440
140.0	147.0	538.0	135.0	5.5670	14,590
142.3	144.0	385.4	34.0	4.9780	14,555
142.3	144.0	385.4	24.0	4.9040	14,780
142.3	144.0	385.4	24.0	5.0515	14,350
142.4	142.4	370.6	..	5.0100	14,840
142.4	142.4	376.0	..	5.1325	14,695
142.4	142.4	398.1	..	5.5560	14,370
142.4	142.4	377.3	..	5.1200	14,525
141.5	143.2	385.4	27.5	4.9309	14,555
141.3	143.3	395.4	32.6	4.8000	14,745
140.9	143.0	385.4	44.0	4.8080	14,240
140.5	143.1	365.6	48.0	4.3975	14,490
140.5	143.1	372.0	48.0	4.4960	14,455
139.5	143.0	381.1	40.2	4.8150	14,200
139.5	143.0	381.1	40.2	4.8460	14,120

stated with some confidence that the arithmetical mean of the results obtained would not be changed appreciably by any number of additional determinations. The probable error calculated by the method of least squares is about 25 calories. Allowing a possible constant error of 25 calories in the calibration of the instruments, etc., the heat of vaporization of mercury is found to be 14,490 ± 50 calories at 142° C. If this value be corrected to 298° K. we obtain 14,670. The value obtained by differentiating the empirical equation of Smith and Menzies at 298° K. is 14,615 cal. While this close agreement cannot be assumed to prove the accuracy of our results it does, on the other hand, indicate a very high order of accuracy for the vapor-pressure data of Smith and Menzies, since a very slight error in vapor-pressure measurements would cause a much greater error in the calculation of heat of vaporization.

Heat of Vaporization of Cadmium

It would no doubt be possible to make a direct calorimetric determination of the heat of vaporization of cadmium using the same method that was employed in the case of mercury. It would be necessary, however, to work at a temperature of at least 350° where the effects of radiation would be very great and the experimental difficulties would probably be considerable. On the other hand, the results in the case of mercury indicate that heat of vaporization can be calculated with considerable accuracy from vapor-pressure data, and this is especially true in the case of cadmium because we have the very elaborate work of Wüst, Meuthen and Durrer[9] on the specific heats of solid and liquid cadmium at high temperatures. By way of existing data on the vapor pressure of cadmium, there are only the measurements of Egerton[10] on solid cadmium and a measurement of the boiling point of liquid cadmium by Heycock and Lamplough.[11]

The simplest and most promising method for the measurement of the vapor pressure of a liquid at high temperatures appeared to us to be the measurement of the boiling point at various reduced pressures. This has been done, working over a range of pressures from about 10 mm. up to about 80 mm. with very satisfactory results. It was not possible to measure the pressures below 10 mm. with sufficient accuracy, and the measurements in the direction of higher pressures were limited by the softening point of Pyrex glass.

The apparatus used was modeled after that used by the Bureau of Standards[12] for determining the boiling point of sulfur. A sketch of the apparatus is shown in Fig. 2. The cadmium was boiled in a test-tube

[9] Wüst, Meuthen and Durrer, "V. D. I. Forschungsarbeiten." **1918**, p. 204.

[10] Egerton, *Phil. Mag.*, **33**, 33 (1917).

[11] Heycock and Lamplough, *Proc. Chem. Soc.*, **28**, 4 (1912).

[12] Bur. Standards *Bull.*, **6**, 184 (1909).

25 cm. in depth by 3 cm. inside diameter, around the bottom of which a heating coil was wound. This tube was mounted inside of a larger tube that formed a closed system connected to a source of supply of nitrogen, and a gage and a pump. The gage was simply a U-tube manometer containing mercury, made from tubing about 2 cm. inside diameter. A vacuum of about 0.002 mm. was maintained in one limb of the manometer by a connection to an oil pump which was kept running while measurements were being made. A leveling bulb was connected to the bottom of the manometer so that the mercury levels could be raised or lowered.

The thermocouple was placed inside a glass tube, the lower end of which was about 3 cm. above the molten cadmium. Around the lower portion of the thermocouple tube was placed an iron radiation shield which was designed to prevent molten cadmium from running down the thermocouple tube. A platinum-platinumrhodium thermocouple made by Heraeus was used, with a calibration chart furnished by the makers. This calibration was confirmed by checking the thermocouple against the boiling point of sulfur. The thermocouple e.m.f. was read upon a White potentiometer. The heights of the mercury in the manometer were read to 0.02 mm. with a cathetometer. Before each reading the mercury was moved up and down the manometer to free the meniscus and readings taken with the mercury at different heights in the manometer showed no variation due to unevenness of bore. The outside tube for about 3 cm. above the heating coil was uninsulated in order to prevent superheating as far as possible. Above this, the tube was covered with asbestos insulation to prevent too rapid cooling and condensation of the vapor around the thermocouple.

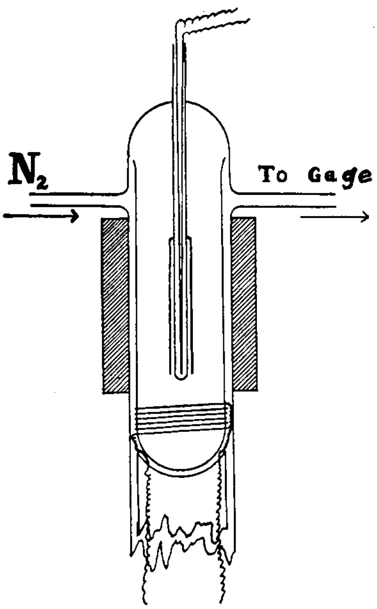

Fig. 2

In the operation the tube was filled with nitrogen at the desired pressure and the cadmium boiled at such a rate that the vapor rose well above the radiation shield, the thermocouple being immersed about 10 cm. in the vapor. Experiment showed that the temperature did not change when the height of the vapor was changed by varying the heating current. When the temperature and pressure had become constant, two sets of readings were made with the mercury at different heights in the manometer. The first question that concerned us was whether the pressure as read by the manometer is the same as that under which the vapor condenses on the thermocouple. This question is not easily answered because a very rapid current of vapor flows past the thermocouple when the cadmium is boiling. It is believed, however, that the average variation in pressure from that read by the manometer is negligible. The question of the accuracy of the temperature measurements is more serious. The thermocouple is not likely to be below the temperature of the vapor, but superheating can very readily take place. The first run was made with the cadmium meniscus about 3 mm. above the top of the heating coil. Results were obtained which when plotted with the logarithm of the pressure as ordinate and the reciprocal of the absolute temperature as abscissa gave a remarkably smooth curve that deviated but slightly from a straight line, as the theory would predict. It seems unlikely that any serious error in the temperature measurements could have occurred in this run, because a variation of even 1° in the temperature of an observation would cause noticeable deviation in the plot and if any considerable superheating were taking place it would have been very remarkable if noticeable irregularities in the data had not appeared. A second run was made with the cadmium surface about 3 cm. above the heating coil. No satisfactory observations were obtained in this run because of the violent bumping and unsteady boiling. A third run was made with the cadmium slightly below the top of the heating coil; in this case, superheating evidently took place and the results when plotted gave a very irregular curve. A fourth run was made with the cadmium surface about 1 cm. above the top of the heating coil, and the results obtained at lower pressures checked very closely the results of the first run. At higher pressures the cadmium began to bump

TABLE II

VAPOR PRESSURES OF CADMIUM

Temperature °K.	Pressure observed Mm.	Pressure calc. Mm.
594.1[a]	0.10	0.10
754.0	9.06	9.14
769.4	12.54	12.70
791.8	20.27	20.10
810.6	28.70	28.84
810.6	29.08	28.84
824.9	37.54	37.58
827.5	39.23	39.45
837.5	47.29	46.99
846.3	54.60	54.95
853.2	61.24	61.66
867.6	78.04	78.34
1039.0[b]	760.00	785.2

[a] Egerton.

[b] Heycock and Lamplough.

and the observations again became uncertain. When the cadmium surface was slightly above the heating coil the boiling was very smooth and the temperature as read by the thermocouple was very constant and responded at once to the slightest change in pressure. It is believed that the temperatures observed under these conditions are not in error by more than 0.5°.

Eleven observations are given in Table II, each observation being the mean of two separate readings taken at the same pressure. The cadmium used was of very high purity.

We should be unable to do better than give a rough approximation of the heat of vaporization of cadmium were it not for the previously mentioned work of Wüst, Meuthen and Durrer[9] on the specific heat of liquid cadmium. If we use their equation for the specific heat of the liquid and assume $C_p = 4.97$ for the vapor, we then have for the heat of vaporization of liquid cadmium

$$\Delta H = \Delta H_0 - 2.453\ T - 0.000716\ T^2 \qquad (2)$$

Substituting this in the Clapeyron equation (1) and integrating, we have

$$ln\ p = -\frac{\Delta H_0}{RT} - \frac{2.453\ ln\ T}{R} - \frac{0.000716}{R}\ T + C \qquad (3)$$

We thus have only ΔH_0 and C to determine.

The value for the vapor pressure at the melting point determined by Egerton[10] is presumably not of high accuracy. Nevertheless, we shall find that it agrees very well with our determinations. The value for the boiling point obtained by Heycock and Lamplough[11] may likewise be in error by 2° or 3°. Furthermore, we can scarcely expect our equation to be strictly applicable at pressures of 1 atmosphere unless cadmium vapor is very nearly an ideal gas in its behavior. Hence, we shall be satisfied if our vapor-pressure equation approximates the value of Heycock and Lamplough. Accordingly, we find that if we assume $\Delta H_0 = 27{,}060$ cal. and take Egerton's value at the melting point as valid we obtain the final equation for vapor pressure in this form,

$$\log p_{(\text{mm.})} = -\frac{5910}{T} - 1.234 \log T - 0.000156\ T + 12.467 \qquad (4)$$

This equation gives excellent agreement with the data and leads to a value for ΔH at 594.1° K. of 25,350 cal. It is believed that the data are not likely to be in error by enough to change the value for ΔH by more than 100 cal.

The Entropy of Monatomic Vapors

We are now in a position to calculate with considerable accuracy the entropy of mercury and cadmium vapors at 298° K. and 1 atmosphere. In the case of mercury the only change that we shall make from the calculation of Lewis, Gibson and Latimer[3] is in the slightly greater heat of vaporization. In the case of cadmium, the specific-heat data of Rodebush[13] indicate an entropy of 11.97 for solid cadmium at 298.1° K. The data of Wüst, Meuthen and Durrer[9] on specific heats and heat of fusion and our determinations of vapor pressure enable us to calculate the entropy of the vapor. Sackur, Tetrode, and others[2] have predicted that the entropies of the different monatomic gases at 298.1° K. and 1 atmosphere would be represented by the equation, $S = 3/2\, R\, ln\, M + C$, where M is the atomic weight and C a constant. Lewis has predicted that the constant would have a value of 25.70. The values we obtained are tabulated together with Lewis's theoretical calculations.

TABLE IV

ENTROPIES OF MONATOMIC VAPORS AT 298.1° K. AND 1 ATMOSPHERE

	Experimental	Predicted
Hg	41.41	41.50
Cd	39.90	39.80

The equation of Rodebush[14] appears to fit the data for mercury and

$$p = \frac{\mathbf{N}RT}{N^2H}\sqrt{2\,M\Delta H_0}\; e^{-\frac{\Delta H_0}{RT}}$$

cadmium remarkably well. It is apparent that we can calculate a very good value of heat of vaporization provided we have only a very rough value for the vapor pressure at one temperature. Thus, for mercury at 298.1° K., we calculate $\Delta H_0 = 14{,}800$ cal. as against 14,670 found experimentally. For cadmium at its melting point we find $\Delta H_0 = 25{,}750$ cal. as compared with the experimental value, 25,350. The extraordinarily high specific heat of molten cadmium indicates that a greater deviation between ΔH_0 and ΔH may be expected than in the case of mercury.

From Egerton's value of 1.13 mm. for the vapor pressure of zinc at the melting point we may calculate the heat of vaporization of molten zinc. The value we obtain, 26,800 cal., we may expect to be somewhat high, as in the case of cadmium. We can check this by calculating the entropy of zinc vapor at 298.1° K. and 1 atmosphere, since we have all the necessary data. The result is somewhat high as may be expected, 39.1 entropy units, as compared with a predicted value of 38.2. This is a further confirmation, if any be needed, of the experimental generalizations that suggested the work of this paper.

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[13] Rodebush, THIS JOURNAL, **45**, 1413 (1923).

[14] Here **N** is the number of molecules per sq. cm. of liquid surface. N is Avogadro's number. h is Planck's constant. M is the molecular weight, and $\Delta H_0 = \Delta E_0 + RT$ where ΔE_0 is the work necessary to remove a molecule from the surface of the liquid which is not in general the same as ΔE, the internal energy of vaporization. In the derivation of this equation (Reference 5) ΔE_0 was assumed to be identical with ΔE. ΔH_0 will usually be larger than ΔH.

Summary

The value of directly measured heat data as a supplement to vapor-pressure data is pointed out.

The heat of vaporization of mercury is determined calorimetrically to be 14,670 ± 50 cal. at 298.1° K.

The heat of vaporization of cadmium is found to be 25,350 ± 100 cal. at 594.1° K.

The generalizations regarding the entropies of monatomic gases and relating vapor pressures to heats of vaporization are confirmed for zinc, cadmium and mercury.

URBANA, ILLINOIS

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF WISCONSIN]

THE ACTION OF SELENIUM OXYCHLORIDE ON VARIOUS METALS AND METALLIC OXIDES[1]

BY WARD L. RAY

Received June 18, 1923

In his studies of selenium oxychloride, Lenher[2] has shown that this inorganic solvent reacts with most of the metals to give the chloride of the metal and selenium monochloride; also that many of the metallic oxides are dissolved by selenium oxychloride.

The object of this research was to study these actions in greater detail and to extend the study, in certain cases, to the action of selenium oxychloride on some closely related substances such as the selenides and the selenites.

The temperatures at which the reactions take place have been varied between room temperature, that of a steam-bath (about 90°), and in some cases the boiling point of selenium oxychoride (175°). A few reactions were studied at even higher temperatures.

Manipulation

About 10 cc. of selenium oxychloride, prepared by the interaction of selenium dioxide and selenium tetrachloride and purified by vacuum distillation,[3] and from 0.2 to 0.5 g. of the metal or oxide were placed in tubes and sealed, to avoid the access of moisture and the consequent hydrolysis. These sealed tubes were allowed to stand at room temperature or were heated on the steam-bath until reaction was complete, or had progressed sufficiently far for our purposes. In some cases a day was required, in others a year.

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[1] Abstract of a part of the thesis submitted to the Graduate School of the University of Wisconsin in partial fulfilment of the requirements for the Degree of Doctor of Philosophy.

[2] Lenher, THIS JOURNAL, **43**, 29 (1921).

[3] Lenher, *ibid.*, **42**, 2498 (1920).