The Specific Heat of Lead in the Temperature Range 1°K to 75°K*

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(Received August 8, 1952)

The specific heat of pure Pb in the temperature range 1°K to 75°K has been measured calorimetrically, both in the normal and superconductive states. The measurements were made using a useful semiautomatic recording system. It was found that at the lowest temperatures (1°K to 4°K) the lattice specific heat of Pb in the normal state was a T^3 function, with a Debye $\theta=96.3$°K. It was found that the variation of θ with temperature over the whole range of temperature employed confirmed the theoretical computations made by Leighton for f.c.c. lattices. The normal electronic specific heat was found to be $7.48\times10^{-4}\ T$ cal/mole-deg, in good agreement with results from magnetic measurements on superconducting Pb. The measured value of the electronic specific heat of the superconducting state was $C_{el,s}=5\times10^{-5}\ T^3$ cal/mole-deg, in the temperature range 1°K to 4°K; this result was also in agreement with the magnetic measurements.

I. INTRODUCTION

NO previous measurements in the liquid helium temperature range have been made on lead in the normal state. Moreover, the only measurements of the specific heat of lead in the superconducting state below 4°K, namely, those of Keesom and van den Ende,[1] indicated the possibility of an anomaly at the lowest temperature. In view of the significance of an accurate determination of the temperature dependence of the specific heat with respect to theories both of lattice vibrations and of superconductivity and in view of the above-mentioned paucity of data for lead, it was considered of interest to carry out the experimental observations that are reported in this paper.

The experimental observations were made using, in the main, well-established techniques of adiabatic calorimetry, as two of us have previously reported.[2] However, by employing a variety of materials for resistance thermometers, by paying special attention to high vacuum isolation, and by using a chart-recording system for semiautomatic temperature observation, we have been able to extend these existing techniques both in range of temperature of observation and in accuracy, and at the same time to increase the speed of experimentation. Some detail is given below of the experimental procedures.

II. EXPERIMENTAL PROCEDURES

Lead of purity 99.99 percent supplied by Mallinckrodt Chemical Works was cast in the form of a cylinder 6.34 cm high and 4.54 cm in diameter, the total content being 5.11 moles. Radial slots parallel to the axis were cut in order to minimize eddy current effects when an axial magnetic field was switched on or off to change the specimen from the superconducting to the normal state, or vice versa. The lead specimen had wound on it a manganin heater wire of mass 0.069 gram and resistance 170 ohms at 0°C. As resistance thermometers we used (a) lead wire of diameter 0.00508 cm and resistance 331 ohms at 0°C, (b) leaded phosphor-bronze wire of diameter 0.00508 cm and resistance 36.2 ohms at 0°C and (c), "1-watt 10-ohm" carbon radio resistors supplied by the Allen-Bradley Company. These thermometric resistors made thermal contact with the specimen through a pure copper mounting. The lead wire was found convenient as a resistance thermometer at temperatures above 10°K, since it had a remarkably high temperature coefficient of resistance.[3] The phosphor-bronze wire, as originally pointed out by Keesom and van den Ende,[4] is satisfactory below about 5°K. The carbon resistor, as shown by Clement and Quinnell[5] and by Brown, Zemansky, and Boorse,[6] was found satisfactory not only at the lowest temperatures but also at temperatures as high as 80°K. In the computations of the data corrections were made, of course, for the heat capacity of the thermometers and their mountings. The specimen was mounted in a vacuum adiabatic calorimeter similar to that described previously by two of us.[2] In the calorimeter used for these measurements, however, a large diameter, high speed, high vacuum pumping line was used, terminating at the top of the cryostat in a DPI, VMF, 20-W, two-stage, fractionating oil diffusion pump. It was found that considerable care had to be taken to maintain vacuum of the order of 10^{-6} mm Hg, owing to the fact that (a) at the higher temperatures, namely, in the liquid nitrogen temperature region, it was found that by the generation of heat in the heater on the specimen gas would be desorbed, presumably from the varnish employed; and (b) at the lowest temperatures, namely, below the lambda-temperature of liquid helium, similar generation of heat would desorb the anomalous helium film. Only by fast and lengthy pumping could the

* Assisted by a contract between the AEC and the Ohio State University Research Foundation.

[1] W. H. Keesom and J. N. van den Ende, Leiden Comm. 203d (1930) and 213c (1931).

[2] A. A. Silvidi and J. G. Daunt, Phys. Rev. **77**, 125 (1950).

[3] de Haas, de Boer, and van den Berg, Physica **1**, 1115 (1934); G. J. van den Berg, Physica **14**, 111 (1948).

[4] W. H. Keesom and J. N. van den Ende, Leiden Comm. 203c (1929); see also W. H. Keesom and P. H. van Laer, Physica **5**, 541 (1938).

[5] J. R. Clement and E. H. Quinnell, Phys. Rev. **85**, 502 (1952); see also Proceedings of the International Conference on Low Temperature Physics, Oxford, p. 51 (1951) (unpublished).

[6] Brown, Zemansky, and Boorse, Phys. Rev. **84**, 1050 (1951).

spurious effects due to such gas desorption be avoided. It is of interest to note that we found it experimentally more difficult to avoid these effects at the higher temperatures. Indeed, it was all too easy at the higher temperatures to observe spuriously high specific heats because of this desorption and to notice apparent hysteresis in the observations.

In measuring temperature with the resistance thermometers, which were calibrated against the vapor pressures of liquid nitrogen, hydrogen, and helium, the potential difference generated across them by a constant current was balanced against a known emf provided by a White single potentiometer (catalog number 7620) before a heating cycle was started. The change in resistance following a heating cycle was observed by measuring the off-balance potential difference with a Liston-Folb dc amplifier (model number 14) and a recording microammeter. In this way temperature changes occurring in the heating cycles were automatically recorded. A typical chart of such an observation is given in Fig. 1, which is a photograph of a record

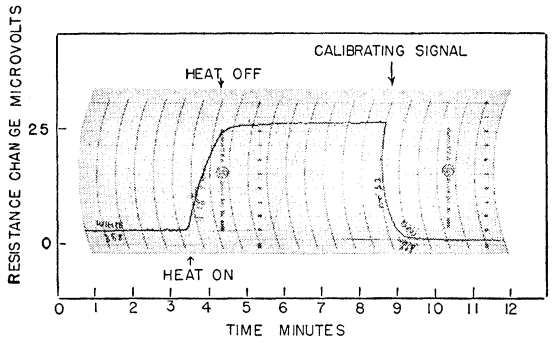

FIG. 1. A semi-automatically recorded heating cycle.

taken at 3.535°K. From the figure it will be seen that at minute 3.7 the heat was switched on and at minute 4.7 the heat was switched off. In this particular cycle the total heat energy provided was 16.56 millicalories. It will be seen further that after the heat was switched off, the temperature of the specimen, as indicated by the potential measurement on the chart, quickly settled down to a constant value. At minute 9, a dc calibrating signal of 25 microvolts was injected in order to correlate the chart difference change with the potential difference change and thus with the change in temperature of the specimen. It is of interest to note that this heating cycle, which is typical of all our observations, shows practically no drift in temperature, either before or after the period of heating. Such lack of drift enables the temperature difference to be estimated with high accuracy, without the need for doubtful extrapolation. This is to be compared favorably with previous techniques, as exemplified, for example, with the heating cycle given by Keesom and Kok.[7]

[7] W. H. Keesom and J. A. Kok, Proc. Amsterdam Acad. Sci. **35**, 294 (1932).

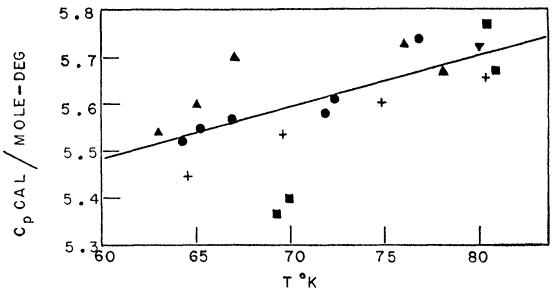

FIG. 2. The specific heat of lead at liquid nitrogen temperatures: ● Our observations; + Meads, Forsythe, and Giauque, J. Am. Chem. Soc. **63**, 1902 (1941); ▼ Griffiths and Griffiths, Proc. Roy. Soc. (London) **A90**, 557 (1914); ▲ W. Nernst, Ann. Physik **36**, 395 (1911); ■ Keesom and Onnes, Leiden Comm. 143.

III. RESULTS

The vapor pressures used in calibrating the thermometers were correlated with temperature using the data provided (a) for liquid helium by van Dijk and Shoenberg[8] [the so-called 1949 scale], (b) for liquid hydrogen by the National Bureau of Standards,[9] and (c) for liquid nitrogen by Henning and Otto.[10]

Our results are collected in Table I which shows all experimental data, such as heat input, temperature differences, etc., as well as the computed specific heats at constant pressure.

It was thought appropriate to make measurements in the liquid hydrogen and liquid nitrogen temperature regions, although many measurements have been made previously at these temperatures, in order to provide a check on the consistency and reliability of our equipment. The results at these temperatures are plotted graphically in Figs. 2 and 3, together with those

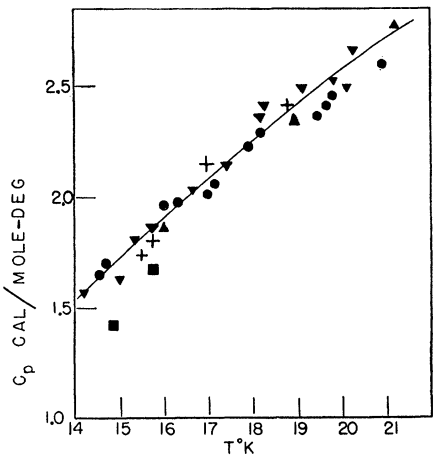

FIG. 3. The specific heat of lead at liquid hydrogen temperatures: ● Our observations; + Meads, Forsythe, and Giauque, J. Am. Chem. Soc. **63**, 1902 (1941); ▲ Eucken and Schwers, Verhandl. Deut. physik Ges. **15**, 578 (1913); ▼ Keesom and Onnes, Leiden Comm. 143; ■ Keesom and van den Ende (reference 1).

[8] H. van Dijk and D. Shoenberg, Nature **164**, 151 (1949).
[9] Wooley, Scott, and Brickwedde, J. Research Natl. Bur. Standards **41**, 379 (1948) RP 1932.
[10] F. Henning and J. Otto, Physik. Z. **37**, 633 (1936).

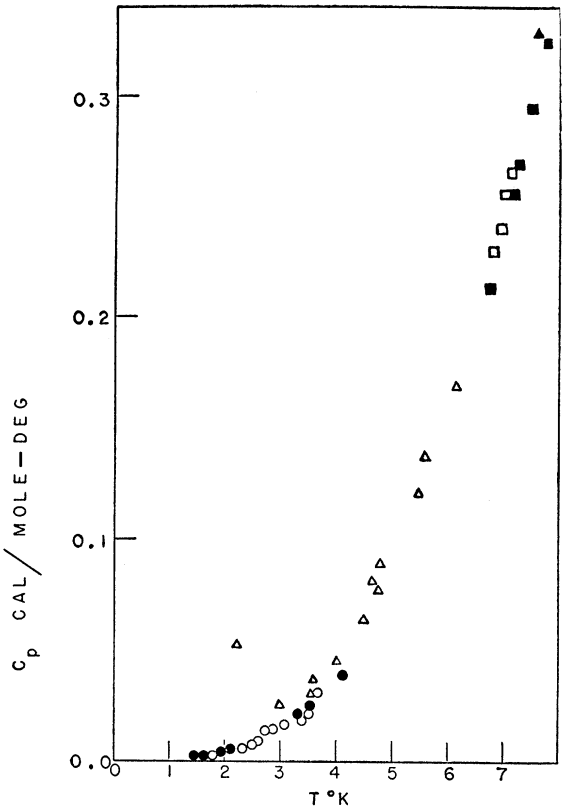

FIG. 4. The specific heat of lead at liquid helium temperatures: ● Our observations in normal state; ○ Our observations in superconducting state; ■ Clement and Quinnell, normal state (reference 5); □ Clement and Quinnell, superconducting state (reference 5); ▲ Keesom and van den Ende, normal state (reference 1); △ Keesom and van den Ende, superconducting state (reference 1).

of previous workers. It will be seen from the figures that the agreement between our results and those previously obtained is satisfactory, and it will be noted that the scatter in previous observations is considerably greater in the liquid nitrogen temperature range than that in the liquid hydrogen temperature range. We feel that this is due to the difficulties enumerated above in Sec. II, associated with gas desorption, which we have noted to be especially troublesome in this temperature region. Our results at liquid helium temperatures for the specific heat of lead both in the superconducting state and in the normal state, which latter was obtained by applying an external axial magnetic field greater than the threshold field, are given in Fig. 4. The figure also includes results obtained previously for lead in the superconducting state by Keesom and van den Ende[1] and the more recent results above 6°K of Clement and Quinnell.[5]

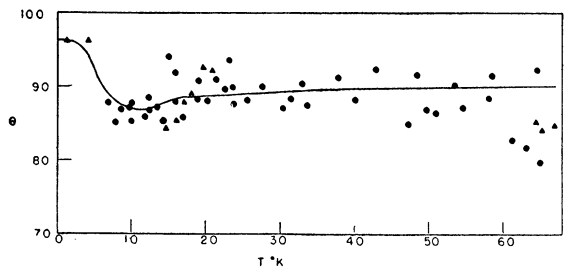

FIG. 5. Debye temperatures for normally conducting lead: ▲ Our observations; ● Previous observations by: Clement and Quinnell (reference 5); Keesom and van den Ende (reference 1); Simon, Z. Phys. Chem. **110**, 572 (1924); Eucken and Schwers, Verhandl. Deut. physik Ges. **15**, 578 (1913); Griffiths and Griffiths, Proc. Roy. Soc. (London) **A90**, 557 (1914); Keesom and Onnes, Leiden Comm. **143**; W. Nernst, Ann. Physik **36**, 395 (1911); Meads, Forsythe, and Giauque, J. Am. Chem. Soc. **63**, 1902 (1941); ——— Theory (Leighton, reference 15).

IV. INTERPRETATION OF RESULTS

Our results (see Fig. 4) for C_p *versus* T for the specific heat of lead in the superconducting state show no sign of any anomaly at the lowest temperatures. It is therefore concluded that the anomalously high value of C_p, in amount 0.0523 cal/mole-deg at 2.20°K reported by Keesom and van den Ende,[1] is probably erroneous.

If it is supposed that at sufficiently low temperatures the specific heat in the normal state can be written as

$$C_p = C_V = 464.4(T/\theta)^3 + \gamma T \text{ cal/mole-deg}, \quad (1)$$

where the first term represents the lattice specific heat and the second term that of the electrons in the normal state, then a plot of C_V/T *versus* T^2 should yield a straight line the intercept of which with the ordinate axis should yield the numerical value of γ. Our results indicate that such a plot made for temperatures below 4.2°K does in fact yield a straight line from which we have deduced that $\theta = 96.3$°K and $\gamma = 7.48 \times 10^{-4}$ cal/mole-deg^2. This value of γ is in excellent agreement with that deduced from magnetic measurements of the threshold curve of superconducting lead, namely, 7.1×10^{-4} cal/mole-deg^2.[11,12] The observed C_V for Pb in the normal state, however, does not follow a Debye function. This can be shown by calculating a Debye θ-value corresponding to each measured value of C_V, The results of this computation[13] are shown in Fig. 5. in which it is seen that the θ-values are not independent of temperature, as they should be if a single Debye function described the whole specific heat curve. It is to be noticed that there is a distinct minimum in the θ *versus* T curve at 12°K and that at the lowest temperature θ flattens out to a value of 96.3°K. This behavior of the specific heat follows the general predictions of Blackman,[14] who showed that at sufficiently low tem-

[11] J. G. Daunt and K. Mendelssohn, Proc. Roy. Soc. (London) **A160**, 127 (1937).

[12] Daunt, Horseman, and Mendelssohn, Phil. Mag. **27**, 754 (1939).

[13] In computing the θ-values for $T > 7$°K the observed specific heat in the normal state (adjusted to constant volume) was equated to $D(T/\theta)$ neglecting any contribution due to the electronic term. Since the γ quoted above is relatively small, this neglect of the electronic term introduces an error which at most [i.e., at 7°K] is 3 percent and which for temperatures above 10°K is considerably less than the experimental error. The θ-value for $T \lesssim 4.2$°K has been found from the C_V/T *versus* T^2 plot mentioned above to be 96.3°K.

[14] M. Blackman, Repts. Prog. Phys. **8**, 11 (1941).

TABLE I. Data on specific heat of lead.

Date	External magnetic field gauss	Type of thermometer	Thermometer dissipation μwatts	Heater current ma	Heater resistance ohms	Heating time sec	Total heat input cal	Heat input to specimen cal	ΔT °K	T °K	C_p cal/mole-deg
At liquid nitrogen temperatures											
8–10–51	0	Pb	0.63	60.93	160	128.3	18.29	15.43	0.539	72.28	5.61
8–10–51	0	Pb	0.63	60.88	160	119.6	17.02	14.38	0.502	71.79	5.58
8–13–51	0	Pb	0.57	60.96	160	119.5	16.91	14.50	0.508	66.82	5.57
8–14–51	0	Pb	0.56	60.86	159	120.0	16.97	14.62	0.516	65.18	5.55
8–14–51	0	Pb	0.55	61.07	159	120.2	17.08	14.76	0.523	64.21	5.52
4–12–52	0	C	5.22	37.32	161	149.1	7.97	7.95	0.273	76.80	5.73
At liquid hydrogen temperatures											
5–9–51	0	Pb	0.96	28.33	154	59.4	1.754	1.722	0.130	20.92	2.59
5–9–51	0	Pb	0.94	28.33	154	59.4	1.754	1.722	0.129	20.79	2.61
5–10–51	0	Pb	0.85	28.33	154	59.8	1.766	1.737	0.138	19.80	2.45
5–10–51	0	Pb	0.19	28.32	154	89.8	2.651	2.607	0.212	19.65	2.41
5–10–51	0	Pb	0.82	28.36	154	60.0	1.776	1.746	0.144	19.45	2.36
5–10–51	0	Pb	0.69	28.29	154	60.1	1.769	1.745	0.149	18.15	2.29
5–10–51	0	Pb	0.16	28.32	154	69.8	2.060	2.033	0.175	18.02	2.26
5–10–51	0	Pb	0.66	28.34	154	45.0	1.330	1.312	0.115	17.86	2.23
5–10–51	0	Pb	0.60	28.32	154	59.8	1.764	1.742	0.166	17.15	2.06
5–10–51	0	Pb	0.58	14.62	154	239.7	1.885	1.861	0.181	16.98	2.01
5–10–51	0	Pb	0.57	28.33	154	45.4	1.341	1.325	0.127	16.83	2.04
5–10–51	0	Pb	0.52	32.28	153	44.8	1.707	1.688	0.166	16.33	1.98
5–10–51	0	Pb	0.51	14.34	153	239.6	1.801	1.781	0.175	16.16	1.99
5–10–51	0	Pb	0.50	28.31	153	60.0	1.758	1.739	0.173	15.99	1.96
8–1–51	0	Pb	0.39	24.22	153	59.8	1.282	1.269	0.146	14.66	1.70
5–10–51	0	Pb	0.37	28.31	153	60.0	1.758	1.741	0.202	14.58	1.69
8–1–51	0	Pb	0.36	24.22	153	60.0	1.287	1.274	0.151	14.51	1.65
8–1–51	0	Pb	0.36	24.22	153	59.6	1.278	1.266	0.149	14.37	1.66
At liquid helium temperatures in superconducting state											
2–16–52	0	Ph–Br	0.19	4.721	152	11.6	0.00942	0.00918	0.171	2.671	0.01046
2–16–52	0	Ph–Br	0.05	2.823	152	43.8	0.01272	0.01246	0.171	2.814	0.01409
2–22–52	0	Ph–Br	0.16	0.901	152	38.1	0.001125	0.001063	0.079	1.781	0.00263
2–22–52	0	Ph–Br	0.16	0.902	152	51.0	0.001506	0.001431	0.093	1.837	0.00301
2–22–52	0	Ph–Br	0.16	0.902	152	52.0	0.001538	0.001464	0.090	1.847	0.00318
2–22–52	0	Ph–Br	0.17	0.902	152	51.6	0.001528	0.001452	0.088	1.903	0.00322
2–22–52	0	Ph–Br	0.17	0.902	152	59.2	0.001750	0.001667	0.096	1.926	0.00339
2–22–52	0	Ph–Br	0.17	0.897	152	102.4	0.003000	0.002883	0.130	1.974	0.00433
2–22–52	0	Ph–Br	0.18	0.902	152	107.8	0.003181	0.003066	0.103	2.312	0.00582
2–22–52	0	Ph–Br	0.18	1.963	152	41.2	0.00578	0.00560	0.147	2.449	0.00743
2–22–52	0	Ph–Br	0.18	1.963	152	47.6	0.00668	0.00650	0.138	2.580	0.00917
2–22–52	0	Ph–Br	0.19	1.963	152	65.7	0.00922	0.00902	0.138	2.704	0.01275
2–22–52	0	Ph–Br	0.19	1.963	152	79.5	0.01115	0.01092	0.150	2.842	0.01423
2–22–52	0	Ph–Br	0.19	1.963	152	73.8	0.01035	0.01013	0.130	2.957	0.01522
2–22–52	0	Ph–Br	0.20	2.231	152	84.5	0.01530	0.01499	0.179	3.078	0.01633
2–22–52	0	Ph–Br	0.20	2.404	152	77.2	0.01632	0.01601	0.163	3.219	0.01917
2–22–52	0	Ph–Br	0.20	2.651	152	90.8	0.02320	0.02270	0.235	3.389	0.01884
2–22–52	0	Ph–Br	0.20	2.652	152	87.2	0.0223	0.02185	0.198	3.509	0.02155
2–22–52	0	Ph–Br	0.20	2.652	152	71.5	0.01830	0.01795	0.148	3.586	0.0237
2–22–52	0	Ph–Br	0.20	2.630	152	67.8	0.01705	0.01679	0.105	3.671	0.0312
At liquid helium temperatures in normal state											
6–6–52	940	C	0.0043	0.787	155	140.1	0.00321	0.00318	0.136	1.892	0.00458
6–6–52	940	C	0.011	1.144	155	23.2	0.001132	0.00112	0.086	1.403	0.00255
6–6–52	940	C	0.0062	1.145	155	36.6	0.001778	0.00175	0.126	1.619	0.00272
6–6–52	940	C	0.0046	1.145	155	63.4	0.00309	0.00306	0.102	1.855	0.00588
6–6–52	940	C	0.0035	1.137	155	107.8	0.00516	0.00512	0.170	2.087	0.00590
6–6–52	940	C	0.00087	4.567	155	30.3	0.0235	0.0234	0.116	4.118	0.0396
3–28–52	830	C	0.0013	1.864	152	64.7	0.00816	0.00813	0.074	3.295	0.0215
3–28–52	830	C	0.0012	2.788	152	49.8	0.01405	0.01400	0.119	3.407	0.0230
3–28–52	830	C	0.0011	2.789	152	58.6	0.01656	0.01650	0.130	3.535	0.0248

peratures a T^3 region would be evident and that as the temperature was raised the θ-value would pass through a minimum. Detailed computations of the specific heat of face-centered cubic lattices have been made by Leighton[15] based on consideration of the normal modes of vibration of the lattice. The results of these computations are shown in Fig. 5 by the full curve for θ *versus* temperature. This curve represents Leighton's computations for an f.c.c. lattice with $\gamma/\alpha=-0.1$, where α and γ are the force constants for central Hooke's law forces which are assumed to act between an atom and its nearest and next nearest neighbors, respectively. Furthermore, in fitting Leighton's com-

[15] R. B. Leighton, Revs. Modern Phys. **20**, 165 (1948).

putations to our results for Pb a θ-value of 96.3°K at 0°K has been taken.[16] It will be seen from the curve that a minimum in the θ-value occurs theoretically at about 10°K, in close agreement with the experimentally observed result and that over a wide range of temperature from liquid helium temperatures up to 70°K there is a close and satisfactory correlation between the theoretical and experimental results for this f.c.c. metal.

Our measurements of C_V in the superconducting state yield approximately a T^3 function. If the lattice specific heat, corresponding to $\theta=96.3$°K, is subtracted from our total observed value, we obtain an electronic specific heat in the superconducting state $C_{el,s}$, given approximately by

$$C_{el,s}=5\times10^{-5}\,T^3 \text{ cal/mole-deg.}$$

It is considered that the temperature range of measurement is too small to allow a conclusive deduction of the full variation of $C_{el,s}$ with temperature. However, in the limited temperature range of $1°K<T<4.2°K$, the computed value of $C_{el,s}=5\times10^{-5}\,T^3$ cal/mole-deg is exactly the same as can be computed from the magnetic measurements on Pb in the superconductive state.[11,12]

This work was supported in part from funds granted to The Ohio State University by the Research Foundation for aid in fundamental research.

[16] *Note added in proof:* The authors' attention has recently been drawn to the elastic data for Pb given by K. Lonsdale [Acta Cryst. **1**, 142 (1948)] from which, using Leighton's formulas, it can be computed that $\gamma/\alpha=-0.12$ and $\theta\approx98$ at 0°K. This further emphasizes the good agreement between Leighton's theory and our results.

PHYSICAL REVIEW VOLUME 88, NUMBER 5 DECEMBER 1, 1952

A Scintillation Spectrometer Study of the Decay of Tl^{204}†

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(Received August 26, 1952)

By means of a special technique of scintillation spectroscopy, whereby radioactivity is introduced into a NaI(Tl) crystal, the β-spectrum of Tl^{204} is investigated and the assignment of the transition as "first forbidden" is confirmed. A reported weak intensity photon radiation of 70-kev energy is uniquely identified as being due to K-electron capture, and the branching ratio of the K capture process to the beta-emission is determined to be ~0.015. Utilizing the formulas for forbidden β-transitions developed by Marshak, the transition energy for the orbital capture is calculated to be ~400 kev. The inner bremsstrahlung associated with the K-capture is detected by coincidence measurements, and its end-point value is found to be in agreement with the transition energy calculated from the branching ratios. An upper limit of 0.2 percent is set for any possible K capture in Au^{198}.

I. INTRODUCTION

THE β-spectrum of Tl^{204} was studied by Saxon and Richards,[1] who obtained a value of 0.783 Mev for its end-point energy and who found that the spectrum shape could best be classified as "first forbidden" ($\Delta I=2$, "yes"). These authors observed that their Kurie plot for the Tl^{204} β-rays, corrected for forbiddeness, diverged from a straight line for energies below ~350 kev to an extent which could not be explained by source thickness. A weak intensity 0.070-Mev photon was observed associated with Tl^{204} by Madansky and Rassetti[2] who suggested the presence of a K capture branch. A recent paper by Lidofsky, Macklin, and Wu[3] confirms the above results and reports a beta end-point value of 0.765 Mev and a Kurie plot which is straight to 150 kev.

A method of studying the decay of radioactive nuclei has been developed at our laboratory which is particularly applicable to the study of beta-spectra and to the detection of weak orbital electron capture branches accompanying β-decay. The technique consists of growing a thallium-activated sodium iodide crystal with a trace of a radioactive element of interest added,[4] so that one obtains a scintillating crystal that has a source of radioactivity distributed evenly throughout it.

A crystal containing a dispersed source of radiations has the following desirable properties: a 4π solid angle geometry; a source thickness which is, for practical purposes, equivalent to zero; the ability to add up simultaneous energy pulses into a single pulse equal in energy to the sum total of the energy of the component pulses. If the activity introduced into a crystal consists of a beta-decay followed by the emission of a prompt gamma-ray, the crystal detects the beta-particles and the conversion electrons arising from the gamma-radiation with ~100 percent efficiency. Each conversion electron is accompanied by a beta-particle which immediately preceded the emission of the gamma-ray;

† Work done under the auspices of the AEC.
[1] D. Saxon and J. Richards, Phys. Rev. **76**, 982 (1949).
[2] L. Madansky and F. Rassetti (private communication).
[3] Lidofsky, Macklin, and Wu, Phys. Rev. **87**, 391 (1952).
[4] Scharff-Goldhaber, der Mateosian, Goldhaber, Johnson, and McKeown, Phys. Rev. **83**, 480 (1951).