Date 3/22/51 Initials R.G.S.

MLM-529
(Deleted)

Contract Number AT-33-1-GEN-53

MLM-529 (DEL.)

MOUND LABORATORY
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REPORT
FOR

GENERAL RESEARCH

OCTOBER 2 TO DECEMBER 26, 1950

(Supporting Research Volume)

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Classification changed to U
by authority of W.D. Reagan date 1/11/56
by Hugh Kinn date 9/20/09
and W.D. Reagan date 5/28/09

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Date: January 29, 1951

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SUMMARY

Two of the main requirements of a laboratory devoted to process development are the possession of facilities to establish identification and maintain control. The identification and control apply to quality, quantity, purity, and stability of gases, liquids, solids, alloys, and compounds. The establishment, operation, and modification of such facilities, and some of the research with the facilities, are locally termed "Supporting Research" because of the basic support that is supplied for the main projects. The requirements of dealing with small amounts of radioactive materials often requires research directed towards properly modifying standard or developing new techniques and instruments to fit out special needs.

The recently developed amplifier of high gain and wide dynamic range has been used with a boron-wall tube to count neutrons. Plateaus and efficiency of the boron-wall tube are significantly improved by the amplifier when counting neutrons, if there are no strong gamma rays from the neutron source (p. 5). The efficiency of the fast-neutron survey meter, which has been undergoing development, has been improved by a factor of five. This improvement has been achieved primarily by replacing the single-chamber, methane-filled, proportional counting tube by a chamber of double the volume divided into halves (p. 12). The Bradley absorption chamber is windowless and completely surrounds the sample to be counted. This makes it much more useful than end-window counters in measuring weak or soft radiations. The accuracy of absorption measurements made with the Bradley counter has been established by studies on known beta emitters. The instrument is particularly useful in waste disposal research (p. 16).

Sticking, drifting, and fogging of the mirror of the quartz-fiber microbalance, which is used in purity assay, have been significantly reduced by the reduction of moisture levels through the addition of a desiccant. Precalibration of quartz-fiber beams before their installation has been shown to be practicable. Since precalibration permits installation of a beam with the correct characteristics, the average repair time has been reduced from about a week to about a day (p. 23).

The seismic detector, which has been constructed by the Instrument Design Group, consists essentially of a tiny mirror suspended by a five-micron quartz fiber. The detector has been used to show that the local instrument piers are sufficiently stable to warrant the purchase of a very sensitive Coblentz galvanometer (p. 27).

The Mattauch-type mass spectrograph has been focused over the low mass range; and work is now in progress on focusing over the high mass range (p. 32).

An investigation has been made of the possibility of using the calorimeters, [REDACTED] to measure specific heats. The precision and accuracy of the instruments for specific heat measurements has been investigated. Further, the results were evaluated to indicate which calorimeter is most suitable and why. An accuracy of better than one per cent was obtained. The measurements were notable for the rather small temperature interval that was involved (p. 36).

Problem Title - Counting Systems for Pulses of Wide Dynamic Range

Report By - W. Baker

Work Done By - W. Baker

INTRODUCTION

The waste-disposal sample counting and neutron counting with proportional ionization chambers has been shown to be done best with a wide-dynamic-range amplifier having a high-gain characteristic. Improved counting precision results because the high-gain feature allows detection of the very weak pulses which are present, and because the wide-dynamic-range feature prevents development of spurious pulses when the very strong pulses, also present, are detected. Two pre-prototype amplifiers, used for several months as components of standard counting systems, have shown the greater value of the high-gain amplifier as compared to the medium-gain amplifier. It has been found that a high-gain amplifier ($G \approx 20,000$) designed to prevent overloading serves as a universal, proportional-chamber amplifier wherever linear amplification is not required.

With routine absorption methods, weaker beta energies have been detected than ever before within a proportional ionization chamber, when the high-gain, wide-dynamic-range amplifier was used.

This report describes a simple method of obtaining further improvement of the dynamic range of the above high-gain amplifier and shows the results obtained when this amplifier was used with a neutron detector (boron-wall, proportional, ionization chamber and paraffin moderator).

The results which should be realized when this amplifier is used with a B-wall chamber are as follows: (1) the elimination of an expensive laboratory-type voltmeter for adjustment of the chamber voltage; (2) the resurrection of chambers formerly considered useless; (3) the considerable improvement in the stability of the counting system because of relatively long plateaus of lesser slope; and (4) an increase of 20 to 100 per cent in the counting efficiency of the chamber at the gamma threshold.

It has been found that a cathode-follower placed between the first and second inverse-feedback amplifier pairs does not appreciably increase the amplifier noise level. This will allow the design of a small head-amplifier.

The design and construction of a prototype, high-gain, wide-dynamic-range amplifier is now under way.

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DETAILED REPORT

The extended dynamic range of the experimental amplifier was achieved by grounding the cathode of the second triode of the dual triode pair, and by changing from direct coupling to condenser coupling within the pair. For our purposes, it has been found that sufficient dynamic range is obtained with condenser coupling. The grid resistor of the second stage of each amplifier pair was changed from one to ten megohms to obtain a negative grid bias. Grid bias in the new prototype amplifier will probably be obtained from a simple bias supply. Dual triodes have been used instead of pentodes in this amplifier because their circuit lends itself to greater simplicity and economy without sacrificing performance. The circuit diagram of the experimental amplifier is shown in Figure 1.

A CK-5703 subminiature cathode-follower triode was connected between the first and second dual triode pair to check its effect on amplifier sensitivity. With a gain of 0.95 it did not cause any noticeable change in sensitivity. This result should enable the design of a compact head-amplifier.

The characteristics of the amplifier are as follows:

(1) Amplifier gain at midband	21,600
(2) Amplifier gain at 720 kilocycles	3 decibels down
(3) Amplifier gain at 1,100 kilocycles	6 decibels down
(4) Amplifier sensitivity	15 microvolts
(5) Pulse duration	0.3 to 1.0 microsecond
(6) Limited pulse output	9 volts
(7) Amplifier stability	1 per cent change in gain/1 per cent change in line voltage

When these data are compared with the tentative specifications listed in report MLM-502, page 3, it will be noted that the above stability figure is somewhat poorer than the figure listed in the specifications. This does not appear to be a serious limitation as the pre-prototype high-gain amplifier has been in continuous use with an unregulated power supply for several months without requiring undue maintenance. The low current drain of the amplifier should allow connection of its plate supply lead to a VR-105 or VR-150 regulator tube with a resultant improvement in stability.

The results obtained with the high-gain experimental amplifier are best shown with graphs. A direct comparison of amplifier effectiveness when counting neutrons generated in a polonium-beryllium source is given in Curves A and B of Figure 2. The source distance was 12 inches from the chamber as shown in the small figure on the graph. Replacement of the commercial amplifier with the experimental amplifier decreased the slope from 0.11 per cent per volt to 0.057 per cent per volt while increasing the plateau length from 30 to 150 volts. At the same time the counting efficiency at the

gamma threshold increased 20 per cent. The above figures were obtained with one of our best boron-wall chambers. Comparable figures for one of our worst boron-wall chambers are given below (Figure 3). The slope was decreased from 1.0 per cent per volt to 0.16 per cent per volt while the plateau length was increased from 30 to 150 volts. At the same time the counting efficiency was increased 92 per cent. It should be noted that the above results were obtained with a polonium-beryllium neutron source. In contrast, no appreciable advantage is obtained with the experimental amplifier when a radium-beryllium neutron source is used because of the strong pulses developed by the radium-gamma radiation. The effect of this radiation is shown in Figure 4.

fig 2

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FIGURE 2

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COMPARISON OF PLATEAUS OBTAINED WITH STANDARD AND EXPERIMENTAL AMPLIFIER WHEN USING CHAMBER WITH THICK BORON LINING.

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FIGURE 4

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COMPARISON OF NEUTRON-GAMMA PLATEAUS WITH EXPERIMENTAL AND STANDARD AMPLIFIERS.

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Problem Title - Fast Neutron Survey Meter

Report By - J. F. Combs

Work Done By - J. F. Combs

INTRODUCTION

The development of a fast-neutron survey meter was undertaken to provide a suitable instrument for the detection of fast neutrons. At the present time there is no instrument available which is simple in operation, lightweight, and portable.

The first consideration in the design of this survey meter was the detecting device. This detector consists of a cylindrical chamber filled with methane gas. The chamber will operate as a recoil proportional counter. Gamma discrimination is accomplished by limiting the physical size of the chamber. Thus the electron path and the resulting pulse amplitude can be limited to any desired value. The counter is fully described in previous reports.^{1,2}

Recently a proportional counter was built containing twice the counting volume of any counters tested previously.³ This counter is cylindrical in shape. It is two inches in diameter and three inches long. The cylindrical volume is divided into two equal compartments by means of a metal baffle placed normal to the cylinder axis. Each compartment is identical in size and shape to the previously tested counters. The center electrode consists of a 1/2-inch diameter loop of 1/2-mil stainless-steel wire in each compartment. This counter was sealed by means of a threaded cap and Neoprene gaskets.

In previous tests the highest efficiency obtained with a recoil proportional counter was 0.05 per cent. The dual-compartment proportional counter described below was found to have a counting efficiency five times as great as this.

DETAILED REPORT

The dual-compartment, fast-neutron, proportional counter was evacuated and outgassed for 40 hours. The final vacuum reached was 10^{-6} millimeters of mercury.

The counter was then filled with purified methane. The methane supply consisted of a lecture bottle of 99 per cent methane gas, which was further purified in the following manner: The lecture bottle was immersed in liquid nitrogen until the methane had solidified; the oxygen and nitrogen impurities were then removed by pumping off the vapor for two minutes. The needle valve was then closed, and the liquid nitrogen bath was immediately replaced with a dry ice - acetone solution. At the temperature of this solution, any carbon dioxide impurities remain in the solid state as the methane again becomes a gas. At this point the counter was filled with the purified methane from the lecture bottle to a pressure of two atmospheres. The counter was then sealed and removed from the vacuum system.

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The test results of this first dual-compartment counter were unsatisfactory. The Neoprene gaskets could not be satisfactorily outgassed. Therefore, the effects of purifying the methane gas were somewhat nullified. An efficiency of approximately 0.07 per cent was obtained with this counter

A second dual-compartment counter was then built. This counter, Figure 1, has the same counting volume as the previous counter. Its overall design, however, is somewhat different. It is sealed with solder joints at both ends. Both silver solder and soft solder were used to facilitate assembly. The center electrode consists of 1/2-mil stainless-steel wire mounted coaxially in the cylindrical counter. The lower ends of the cylindrical counting volumes were coated with paraffin, 0.030 inch thick, to give the counter a directional characteristic.

After this counter was determined to be leakproof, it was evacuated for 48 hours until a vacuum of 10^{-5} millimeters of mercury was attained. It was immediately filled to a pressure of two atmospheres with purified methane gas.

When tested, the counter was found to detect neutrons at a sensitivity of five millivolts with 2,000 volts applied to the counter. Gamma radiation was not detected at the same sensitivity until the voltage was raised to 2,400 volts. When 2,350 volts were applied to the counter, a neutron counting efficiency of 0.25 per cent was attained at five millivolts sensitivity. This efficiency is approximately five times greater than that obtained with previous counters.

The counter had the desired directional characteristic in that, when the neutron source was placed radially six inches from the side of the cylindrical counter, the counting rate was approximately one half that which was recorded when the source was placed on the axis of and six inches below the counter.

At this point in the development of the fast neutron survey meter, the emphasis was shifted from the detecting device to the necessary associated electronic circuits.

The high voltage power supply is now under development. A regulated supply variable from 2,000 volts to 2,500 volts is desired. It must be battery operated. It must be capable of a current drain of a few microamperes. Its size and weight must be kept at a minimum.

There are several ways of fulfilling the above requirements. The following are under consideration.

- 1 A battery power supply consisting of nine 300 volt dry batteries
- 2 A vibrator type power supply with a voltage doubler
- 3 A multivibrator type, pulsed power supply with a voltage doubler.

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4. A neon-bulb-controlled, oscillator-type, pulsed power supply with a voltage doubler.

5. A blocking oscillator-type, pulsed power supply with a voltage doubler.

More information on the components of the above circuits is required before any choice of power supply can be made.

A compact rate-meter circuit is also necessary for this instrument. At the present time an investigation is also being made into the design and operation of rate meters.

REFERENCES

1. Combs, J. F., Quart. Rpt. Gen. Res., MLM-443-4, p. 77, April 1, 1950.
2. Combs, J. F., Quart. Rpt. Gen. Res., MLM-478, p. 53, August 14, 1950.
3. Combs, J. F., Rpt. Gen. Res., MLM-502, p. 29, November 6, 1950.

DELETED VERSION **FAST NEUTRON PROPORTIONAL COUNTER 2 A**

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Problem Title - Absorption Studies on a Bradley Counter

Report By - M. L. Curtis

Work Done By - M. L. Curtis, M. B. Meyer, and G. D. Stewart

INTRODUCTION

An important contribution can be made to the identification of radioactive materials if the energy of the emitters can be determined. Absorption studies can be made on end-window Geiger counters if the emitter is of high specific activity and if the maximum energy exceeds 0.1 million electron volt. The Bradley absorption chamber has no counter window for the radiation to penetrate, and has essentially a 2π geometry, so that absorption studies can be made on samples of low specific activity and low energy.

DETAILED REPORT

Since for very low energy radiation, the gas absorption between the samples and the absorber could be a significant fraction of the range of the particles, it was found necessary to modify the brass rings supporting the absorbers and to make spacers to use with each absorber. These changes made it possible to keep the distance from the sample to the bottom of the absorber small and constant and at the same time to keep the distance from the top of the absorber to the center wire constant.

Since the Bradley counter counts alpha particles at one voltage, and alpha plus beta plus gamma radiation at a higher voltage, it is possible in the absorption measurements to distinguish between alphas and betas or gammas. Figure 1 shows an absorption study of $\text{RaD} \longrightarrow \text{E} \longrightarrow \text{F}$ in which the alpha count has been subtracted. The maximum energies determined from this curve of 0.03 million electron volt for the RaD and 1.20 million electron volts for the RaE correspond to the values given in the literature.

Figure 2 shows the absorption of phosphorous-32, and the maximum energy of 1.72 million electron volts also compares favorably with values given in the literature. A Feather analyzer¹ has been made from this curve and will be used in interpreting other curves where the maximum energy approaches 1.72 million electron volts.

In Figures 3 and 4, absorption studies of cesium-134 and ruthenium-106 are graphed. Since thick samples were used for these studies, the low energy components of the curves are distorted by self-absorption.

The chamber is being used extensively for the chemists, particularly the Waste Disposal section, for absorption studies which cannot be made with an end-window counter.

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A further application of the Bradley absorption chamber is its use in alpha-ray aluminum absorption studies. Air-absorption studies of alpha emitters can be made in the Logac-S, but since this is a low-geometry, window counter, it has the same disadvantages for alpha-absorption studies as an end-window counter has for betas. Figure 5 shows aluminum absorption of polonium alphas, and illustrates the use of the instrument for this purpose.

REFERENCE

1. Glendenum, L. E., Nucleonics, p. 19, January, 1948.

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ABSORPTION STUDY OF p32

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FIGURE 5

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Problem Title - The Standard Torsion Equal-Arm Balance

Report By - R. G. Olt

Work Done By - M. I. Gray, L. A. Jewett, R. Morgan, R. G. Olt, and E. W. Stautzenbach

INTRODUCTION

The quartz-fiber microbalance in the form used in microassay consists of a completely fused, quartz-fiber beam system of a torsion-fiber, equal-arm, load-balancing type in a case containing the directly associated mechanical and optical systems.

This unit is further enclosed in a vacuum-tight metal housing fitted with the mechanical and optical controls and accessories essential for placement, weighing, and removal of sample-bearing foils while maintaining atmospheric, thermal, and radiation shielding. Since the carriers for the foils also provide atmospheric and radiation shielding, it is possible to operate the balance in low risk areas despite the high level of activity being handled.

Because of the high level of internal contamination, repairs to any portion of the quartz-fiber microbalance or housing requiring the opening or exposure of the housing interior must be performed in a high-risk area, with all health precautions suggested for high-risk work. Since the balance is used in a low-risk area, it is also necessary to follow all interarea contamination-control procedures. As a result, internal balance service is a lengthy procedure which often involves delicate work under difficult conditions.

Incorporation of the mechanical, electrical, and optical refinements described in previous reports^{1,2} improved the operating characteristics and extended the periods of balance availability to about a month.

Need for the cleaning of the area around the load-side pan well became the limiting factor of the operation. Analysis of the observations made in the course of six servicings of pan-well areas indicate that moisture was present and could have been a major factor in all difficulties encountered. Since July, when the use of a desiccant within the balance housing was started, previous recurring difficulties have not been encountered, and balance availability seems to have been extended to at least six months without need for internal service.

Availability of balance MLB-6F for cold work has made practical the establishment of a procedure for calibration of replacement balance beams prior to storage and the calibration of three sets of secondary-standard mass foils for beam-torsion-constant determinations. Calibration of replacement beams prior to storage will reduce out-of-service time for beam replacement in a contaminated balance from at least a week to a matter of about a day. The secondary-standard mass foils are to be used as working standards in the "T" and "R" areas and at Scioto Laboratory.

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DETAILED REPORT**Microbalance Operation Problems**

With the incorporation of a number of improvements to the optical, mechanical, and electrical components of the quartz-fiber microbalance, difficulties interfering with balance operation were reduced to sticking of hangdown and panholders, corrosion of arrest and pan-well surfaces, fogging of the pan-well mirrors, and occasional, erratic drifting of the balance point.

Cleaning of the pan-well areas has been requested only when sticking, drifting, or mirror fogging rendered further use of a balance impractical. The period of time for this to occur has varied from a week to two months, averaging about a month of use per cleaning.

Each of the pan-well-area cleanups has involved removal of the balance from the housing; cleaning of the hangdowns with chromic acid, distilled water, and acetone; polishing of the arrest tops, well openings, and catch pans with metal polish; and cleaning or replacement of the first-surface mirror over the pan well. During these cleanups, moisture has been detected on the corrosion-product films and on the quartz-fiber hangdown and panholder, in one case where drift had been excessive.

Since moisture could accentuate the difficulties encountered, balance desiccation was started in July, 1950, with a cloth sack containing approximately 200 grams of eight-mesh Drierite placed on the operating base under the balance. The six-months operating experience gained since the addition of a desiccant has shown that the previous corrosion, sticking, and fogging difficulties have been eliminated and that other balance deterioration has been arrested.

Beam Calibration

Since the mechanical and electrical torsion-fiber drive now limits twist of the torsion fiber to plus or minus three full turns, it is desirable to have the load-side torsion constant fall between 0.20 and 0.25 micrograms per dial division in order to have a torsion, load-balancing capacity sufficient to permit use of a reasonable number of foils without change of tare and still retain an adequate sensitivity.

All new-beam-calibration experience to date has shown that torsion constants may vary widely beyond the desired limits. This has necessitated replacement and trial of torsion fibers until the desired torsion constant has been obtained. Such installation, replacement, and trial of torsion fibers on a contaminated balance would add from three to five days to the time the balance would be unavailable for use when a beam replacement was required.

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Availability of balance MLB-6F for cold work has made practical the calibration of replacement balance beams prior to storage. Such beams will require only a check of calibration to determine possible minor changes resulting from deterioration or repairs.

Torsion-constant change resulting from beam removal and replacement was checked with Beam No. 11 in balance MLB-6F. During the beam transfer cycle the cemented bushings were removed and rotated randomly, duplicating the normal replacement conditions. Recalibration of the beam after the transfer yielded a torsion constant of 0.23265 micrograms per dial division in comparison with the 0.23266 micrograms per dial division obtained initially. This indicates that precalibration of beams is of adequate precision for normal balance requirements.

Some breakage of fibers may occur during handling or operation of a beam. Only repairs affecting the length or diameter of the torsion fiber or the hinge point of a hangdown are likely to cause changes in the torsion constant. These changes would be in the order of one per cent of the original value. Change of torsion constants with time or with exposure to deteriorating atmospheres has not as yet been investigated. Corrections for the above variations may be determined readily at the time of the return of a balance to service.

Secondary-Standard Mass Foils

To provide working sets of standard mass foils for beam calibration and other experimental work in the "R" and "T" areas and at Scioto Laboratory, three sets of secondary-standard mass foils have been prepared by comparison with our Bureau of Standards calibrated foils. The calibrations given in Table I are subject to correction since the values used for the primary standards have not been rechecked since 1948.

TABLE I
MASSES OF FOILS FOR BEAM CALIBRATION

FOIL NUMBER	BUREAU OF STANDARDS CALIBRATED FOIL MASS	SECONDARY-STANDARD FOILS		
		SET 1	SET 2	SET 3
1	7.4331	7 3738	7.2965	7.0915
2	7.7353	7.6236	7.5562	7.3396
3	7.9940	7.8474	7.8101	7.5940
4	8.1738	8.1411	8.1058	7.8120
5	8 3767	8.3791	8.2849	8.0311
6	8.6152	8 6029	8.5035	8.2898
7	8.7921			

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1. Olt, R. G., Quart. Rpt. Gen. Res., MLM-405-5, pp. 9-21, January 1, 1950.
2. Olt, R. G., Quart. Rpt. Gen. Res., MLM-443-5, pp. 170-177, April 1, 1950.

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Problem Title - **Use of a Quartz-Fiber Seismic Detector in Studying Instrument Pier Stability**

Report By - **J. R. Parks**

Work Done By - **J. R. Parks and T. J. Beasley**

INTRODUCTION

The seismic detector has been designed and built by R. G. Olt and his group. In essence, the detector consists of a small mirror suspended freely on a quartz fiber. It is useful in determining whether a given pier is sufficiently stable for apparatus of a given sensitivity. Stability determines whether the ultimate sensitivity of an apparatus can be used. The seismic detector should prove very useful to the Physics Research Groups. It can be used also for the study of the dynamic properties of quartz fibers.

The dynamic properties of the seismic detector duplicate those of the Coblentz moving-magnet type of galvanometer built by the Leeds and Northrup Company. This galvanometer has been considered for use in the microcalorimeter program because of its extreme sensitivity. There has been some question concerning sufficient stability of the piers in the "R" Building to utilize the Coblentz galvanometer. The seismic detector was built in an effort to determine the stability of the pier in Room 112 in the "R" Building, where it is proposed to use the Coblentz galvanometer in conjunction with a microcalorimeter.

DETAILED REPORT

The seismic detector consists essentially of a 100-micron-diameter quartz staff with a small mirror mounted on it. The staff is suspended from a movable brass plug by means of a five-micron-diameter quartz fiber, as shown in Figure 1. The brass plug rests in the cross member of a brass frame, two inches wide by six inches high by one inch deep. The bars of the frame are one half-inch thick. The frame is attached to a six-inch-diameter brass disk supplied with leveling screws and a level. This assembly was treated with hot oil and cold temperature (refrigerator) through four cycles to improve its stability. The front and back plates are separated from the frame by felt washers. The slot in the front plate for viewing the mirror is covered by a microscope slide. The back plate has a shallow groove down the center in which a small amount of radioactive material is deposited to prevent static charge from accumulating on the staff and suspension fiber. The fiber and staff are each five centimeters long.

The detector and an illuminated scale were mounted on the pier with a distance of 130 centimeters between the mirror and the scale. The deflections of the mirror

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were read with a telescope also mounted on the pier. The detector was protected from air currents by means of a fiber-board box with a viewing slot in the side and a detachable top.

The deflections of the mirror were checked from time to time for a period of two weeks. If the pier were randomly disturbed, the deflections of the galvanometer would also be random. The mean square of the deflections would be a measure of the energy imported to the seismic detector by the pier. Hence the root-mean-square deflection of the detector mirror is a measure of the pier disturbance. The observed total deflection of the galvanometer is 0.2 millimeter. This is the minimum observable deflection since 0.2 millimeter is the step of this setup.

If there were no mechanical disturbance of the pier, the mirror would still show a disturbance (if it were sensitive enough) because the suspension fiber is in thermodynamic equilibrium with its surroundings. According to statistical mechanics, the root-mean-square deflection of the mirror is given by:

$$(\theta)^{1/2} = \left(\frac{kT}{\gamma} \right)^{1/2} \quad (1)$$

where:

θ = Deflection of the mirror in radians.

k = Boltzmann constant = 1.3×10^{-16} erg-degree-Kelvin⁻¹.

T = Room temperature = 298 degree Kelvin.

γ = Torsion constant of the suspension fiber in dyne-centimeter-radian⁻¹

or ergs-radian⁻².

The measure of disturbance given by Equation 1 can be selected as the standard to which other disturbances can be compared.

The torsion constant of the fiber was calculated from the following equation:

$$\gamma = I \left(\frac{4\pi^2}{\tau^2} + K^2 \right) \quad (2)$$

where:

I = Moment of inertia of the suspension system in gram centimeter² or erg-seconds²-radians⁻².

τ = Period of the fiber oscillation in seconds.

K = Damping constant of the oscillation in seconds⁻¹.

The period was found from ten runs by counting oscillations during a measured time interval and averaging the results. The damping constant was calculated from 21 runs by timing the decay of the amplitude of the oscillation from one value to another. They are: τ = 0.97 seconds and K = 0.25 seconds⁻¹.

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The moment of inertia, I , was calculated from the dimensions and mass of the staff and mirror assembly. It was found that the moment was due almost entirely to the mirror. It was found to be: $I = 12.08 \times 10^{-6}$ gram-centimeter² or erg-seconds²-radians⁻².

Dimensions of the mirror are:

2.9 by 2.5 millimeters x 140 microns thick

Mass = 2.3 milligrams

Axis of rotation is parallel to the 2.9 millimeter dimension.

These data allow a calculation from Equation 2 of the torsion constant, γ , as:

$$\gamma = 50.7 \times 10^{-6} \text{ ergs-radians}^{-2}$$

From Equation 1 the root-mean-square deflection of the mirror from thermodynamic disturbance is given by:

$$\left(\overline{\theta^2}\right)^{1/2} = 9 \times 10^{-6} \text{ radians}$$

With an optical lever of 1,300 millimeters and a magnification factor from the mirror of two, the root-mean-square deflection at the scale is given by:

$$\begin{aligned} \left(\Delta S^2\right)^{1/2} &= 2 \times 1,300 \times 9 \times 10^{-6} \\ &= 0.02 \text{ millimeters} \end{aligned}$$

Since this is a root mean-square value peak-to-peak disturbance will be seven times this value or:

$$\Delta S = 0.14 \text{ millimeters}$$

Since the observed and calculated deflections are of the same order of magnitude, it is concluded that the pier in Room 112 in the "R" Building is not significantly disturbed by extraneous forces. This pier should be sufficiently stable for the Coblentz galvanometer.

At this time it is proposed to replace the five-micron fiber with as fine a fiber as can be drawn and worked by the Laboratory Instrument Design Group, with the obvious restriction that it be able to support about four milligrams. Such a fiber, if successful, should allow a more thorough study of pier stability because of its higher magnification of thermal and mechanical disturbances.

A consultation with J. R. Wiesler, Director of Engineering, indicated that the geologic structure of the Mound Laboratory site is very favorable for the construction of stable piers. The structure for several hundred feet deep appears to be a stratification of limestone and shale. The shale is of a type that turns to a clay-like mud when in

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contact with moisture and air. It is highly probable that the disturbances from rail and truck traffic around the base of the site are rapidly damped and scattered before they reach the Research Building.

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Problem Title - Mass Spectrograph

Report By - D. L. Haas

Work Done By - D. L. Haas

INTRODUCTION

A mass spectrograph for chemical analysis of solid samples has been developed. The Mattauch¹ arrangement of a $31^\circ 50'$ electric deflection followed by a 90° deflection in a magnetic field is used in order to obtain a large mass range in focus on one photographic plate.² The electromagnet with 100 milliamperes gave a field of approximately 20,000 gauss across a 1/8-inch gap. The ion source is a Shaw hot-anode type. The preliminary results obtained with this instrument reveal its suitability for experimental and routine qualitative analysis and radioactive and non-radioactive isotopes investigation.

DETAILED REPORT

The schematic arrangement of the two fields and the slit system is shown in Figure 1.

Mattauch¹ has shown that double-focusing will occur over the entire range of the photographic plate when ϕ equals 31° and $50'$.

The voltage required across the condenser plates is dependent upon the accelerating voltage.

It will be noted that ρ_n increases as the square root of the mass, whereas it decreases with the square root of the number of charges. This fact is helpful in the identification of particular lines caused by multiple charges.

Experiments are now being conducted with 11,000-volt ions. The proper ratio between the accelerating potential and the condenser voltage has been experimentally determined. For 11,000-volt ions, a condenser voltage of 820 volts gives optimum focusing of the heterogeneous ion beam as detected by the direct-current amplifier. Focusing and resolution are dependent upon ϕ and the width of the defining slit S_1 . The variable ϕ has been experimentally determined for optimum focusing over the entire mass range. The best focus was obtained with $l = 10.2$ centimeters.

The results of the focus test are shown in Figure 2. Line 1 corresponds to an l of 10 centimeters; line 2 corresponds to an l of 7 centimeters and line 3 corresponds to an l of 12.5 centimeters. The best focus was obtained with an l of 10 centimeters. Results not shown indicated a sharper focus with an l of 10.2 centimeters.

Slit S_2 , which is not required by theory, has recently been added. Focus, without S_2 , was sufficient in the low mass range (1 through 40); however the ions of atomic masses above 100 could not be separated sufficiently for identification. The addition of Slit S_2 has improved the resolution in the higher mass ranges. The following variables have been determined and the proper setting or addition made to the instrument:

1. The correct ratio between the accelerating potential and the condenser potential has now been determined.
2. The optimum distance between the defining slit, S_1 , and the entrance to the electrostatic field has been determined.
3. The addition of S_2 has increased the focusing properties of the instrument in the very high mass range
4. The positioning of the source head is very critical and must be realigned for each sample under investigation.

A positive calibration of the instrument is now in progress. The low mass range has been tentatively calibrated.³ An investigation of the lead isotopes, with finely divided metallic lead in the crucible of the source, is now in progress. Single, double, and triple-charged lead ions have been identified. However, positive identification of the isotopes of lead has not been possible to date.

A preliminary study was also made of the lanthanum prepared by the "Y" Group. Two lines were obtained which indicated masses of 139 (lanthanum) and 155 (lanthanum oxide) This prepared lanthanum will be reinvestigated.

REFERENCES

1. J. Mattauch and R. Hersog, Z. Physik, 89, 786, (1934).
2. A. E. Shaw and Wilfrid Rall, Rev. Sci. Instruments, 18, 278, (1947).
3. A. E. Shaw, *An Intense Positive Ion Source for Solids*, July 8, 1946, (CP-3559).
4. Haas, D. L., Rpt. Gen. Res., MLM-502, p. 85, December 29, 1950.

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a = MEAN RADIUS OF CURVATURE OF IONS PASSING THROUGH CONDENSER.

a_{mn} = MEAN RADIUS OF CURVATURE IN THE MAGNETIC FIELD OF IONS HAVING EFFECTIVE MASS NUMBER n .

l = DISTANCE OF THE DEFINING SLIT S_1 FROM THE ENTRANCE TO THE CONDENSER.

ϕ = ANGLE SUBTENDED BY THE CONDENSER AT THE CENTER OF CURVATURE OF THE MEAN PATH.

ϕ_{mn} = ANGLE SUBTENDED BY ρ_n AT THE CENTER OF CURVATURE OF THE a_{mn} PATH.

ρ_n = DISTANCE FROM THE POINT AT WHICH IONS ENTER THE MAGNETIC FIELD TO THE POINT AT WHICH THEY STRIKE THE PHOTOGRAPHIC PLATE.

D.C. = DIRECT CURRENT AMPLIFIER MONITORING THE HETEROGENEOUS ION BEAM BEFORE THE BEAM ENTERS THE MAGNETIC FIELD FOR ISOTOPIC SEPARATION.

DIAGRAM OF MASS SPECTROGRAPH.

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FIGURE 2

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35 fig 2

Magnification approximately 5x

FOCUS TEST

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Problem Title - Comparison of Resistance-Bridge, Twin-Type Calorimeters for Measurement of Specific Heats of Materials

Report By - K. C. Jordan and J. R. Parks

Work Done By - K. C. Jordan

INTRODUCTION

Calorimeters have proved very useful in determining the thermodynamic properties of materials. These properties have a very direct bearing on research and engineering in chemistry. Therefore the determination of thermodynamic properties of materials precisely and rapidly is an important function of calorimetry

About a year and half ago the idea arose of using the resistance-bridge, twin-type calorimeter, that the Calorimetry Research Group has been investigating, for the determination of specific heats of materials. This problem was not considered as a definite part of the calorimetry research program. It has been carried along as available time permitted.

This paper will deal with certain aspects of the design, construction, and use of the twin-type, resistance-bridge calorimeter for measuring specific heats. Specific heats of various materials have been measured in the laboratory with Calorimeters 46 and 51. Comparison with other types of calorimeters is not attempted here. In Calorimeter 46, experiments were run primarily to determine the inherent accuracy of our method of measuring specific heats.¹ Experiments for determining precision are being made in Calorimeter 51.

It seemed necessary to intercompare resistance-bridge, twin-type calorimeters and to determine how to design such a calorimeter for the measurement of specific heats only. Calorimeters 39, 51, and 53 were compared. The results are presented in this report.

DETAILED REPORT

The accuracy of our method of determining specific heats was investigated by the use of Calorimeter 46. This work showed that the accuracy of the method was better than one per cent. The precision of the method has not been investigated to the point of making a report at present. Some laboratory work is in progress that will lead to a determination of the precision of the method. At this time only the relative precision of different resistance-bridge, twin calorimeters in the determination of thermal capacity has been made

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The heat retained in the sample when the calorimeter is at equilibrium with a given power input is proportional to the difference between the area under the cooling curve (bridge potential versus time) of the empty calorimeter and that under the cooling curve of the calorimeter plus the sample. This relation is given in Equation 1.

$$Q_s = \beta (A_2 - A_1) \quad (1)$$

where:

Q_s = Heat retained by the sample in calories.

A_2 = Area under the cooling curve of the calorimeter plus the sample in microvolt seconds.

A_1 = Area under the cooling curve of the empty calorimeter in microvolt seconds.

β = Constant of proportionality in calories per microvolt seconds.

If the probable error in a single determination of area, A_1 , is p_1 and for area, A_2 , is p_2 , then by statistics the probable error, p_s , in a determination of Q_s is given by:

$$p_s = \sqrt{p_1^2 + p_2^2} \quad (\text{microvolt seconds}) \quad (2)$$

Since the areas considered in this report are of the same order of magnitude, we may write Equation 2:

$$p_s = p_1 \sqrt{2} \quad (\text{microvolt seconds}) \quad (3)$$

The relative error in the determination of Q_s will be:

$$r_s = \frac{\beta p_s}{Q_s} = \frac{p_1 \sqrt{2}}{A_2 - A_1} \quad (4)$$

where:

r_s = Relative error of Q_s .

We may put this into a usable form by dividing the numerator and denominator of the right hand side of Equation 4 by A_1

$$r_s = \frac{\frac{p_1 \sqrt{2}}{A_1}}{\frac{A_2 - A_1}{A_1}} \quad (5)$$

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This equation may be solved for $\frac{A_2 - A_1}{A_1}$ as:

$$\frac{A_2 - A_1}{A_1} = \left(\frac{p_1 \sqrt{2}}{A_1} \right) \times \frac{1}{r_s} \quad (6)$$

Here we see that $\frac{A_2 - A_1}{A_1}$ is proportional to the precision, $\frac{1}{r_s}$, of measure of the thermal capacity of the sample. We shall designate this quantity, $\frac{A_2 - A_1}{A_1}$, by the letter, P, in the discussion of the comparative merits of calorimeters in the determination of specific heats.

$$P = \frac{A_2 - A_1}{A_1} \quad (7)$$

For most calorimeters the precision p_1 of measuring A_1 will be identical if the same technique is used for obtaining the curve and for measuring areas. Also A_1 can be made equal for all calorimeters by choice of heating currents. Thus the r_s in Equations 5 and 6 is dependent upon $\frac{A_2 - A_1}{A_1}$ for variations among calorimeters; here it is seen that the smaller r_s the greater will be the precision, P. Experimental work was carried out to measure this factor of precision for Calorimeters 39, 51, and 52. Also a mathematical treatment was used to derive this precision factor in terms of fundamental parameters of a calorimeter

Experimental cooling curves of Calorimeters 39, 51, and 53 were taken from about 140,000 microvolts maximum bridge potential, first with each sample can empty, and later with each sample can completely filled with brass. These curves are presented in Figures 1, 2, and 3, plotted on a linear scale. The same curves were also plotted on semilog paper in order to measure the time to reach within 0.01 per cent of equilibrium for each curve. Figure 4 gives these curves for Calorimeter 39. Data on these cooling curves and results of calculations are presented in Table 3. The experimental value of $\frac{A_2 - A_1}{A_1}$ is given in Item 17

A mathematical treatment follows by which the value of $\frac{A_2 - A_1}{A_1}$ can be computed with information taken from a single cooling curve of the empty calorimeter. Experimental and calculated values of $\frac{A_2 - A_1}{A_1}$ are presented in Table 2.

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The rate in millicalories per hour of heat flow away from the calorimeter in cooling can be stated as follows

$$\frac{dq}{dt} = - \frac{(3.6 \times 10^6) 2\pi LK (T - T_0)}{\ln \frac{r_2}{r_1}} \quad (8)$$

where

K = Thermal conductivity of gradient medium (air), calories-second⁻¹-centimeter⁻¹-degree⁻¹

r₂ = Radius of inner surface of the jacket centimeters.

r₁ = Outer radius of thermel, centimeters

L = Length of thermel, centimeters

T = Temperature of thermel degrees Centigrade

T₀ = Temperature of jacket degrees Centigrade.

This equation describes the flow of heat across a gradient medium between two, concentric, infinitely-long cylinders with a section L centimeters long being under consideration

Equation 9 relates the average temperature of the calorimeter bridge to the change of resistance of the bridge which changes the potential across the bridge.

$$T - T_0 = \frac{V}{2500 \alpha R} \quad (9)$$

where

V = Bridge potential in microvolts, taking V₀ = 0

α = Coefficient of resistivity of bridge wire, ohms · ohm⁻¹-degree⁻¹.

R = Resistance of bridge at bath temperature, ohms

2500 = One half the bridge current microamperes

Combining Equations 8 and 9 we get

$$\frac{dq}{dt} = \frac{(3.6 \times 10^6) 2\pi LK V}{2500 \alpha R \ln \frac{r_2}{r_1}}$$

which can be reduced to

$$\frac{dq}{dt} = - \frac{9050 KL V}{\alpha R \ln \frac{r_2}{r_1}} \quad (10)$$

At equilibrium the rate of heat leak in millicalories per hour is equal to the heat input, W , where W is the power input to the heater in millicalories per hour, and the bridge potential, V , becomes a maximum at V_m . Thus:

$$W = - \frac{9050 \text{ KL}}{\alpha R \ln \frac{r_2}{r_1}} V_m \quad (11)$$

The sensitivity, S , is equal to the microvolts at equilibrium divided by the heat input in millicalories per hour or:

$$S = \frac{V_m}{W} = \frac{\alpha R \ln \frac{r_2}{r_1}}{9050 \text{ KL}} \quad (12)$$

To show that Equation 12 is approximately correct we can substitute data from one of the calorimeters. Thus for Calorimeter 51:

- $L = 16.25$ centimeters.
- $K = 0.000052$ calorie second⁻¹-centimeter⁻¹-degree⁻¹ (air).
- $\alpha = 0.0054$ ohm ohm⁻¹-degree⁻¹ (bridges are of nickel-99 alloy).
- $R = 1737$ ohms.
- $r_2 = 1.587$ centimeters.
- $r_1 = 1.3716$ centimeters.

and so:

$$S = \frac{0.0054 \times 1737 \ln \frac{1.587}{1.3716}}{9050 \times 16.25 \times 0.000052} = 0.1793 \text{ microvolt-hours-millicalorie}^{-1}$$

The experimental value of S was 0.1328 microvolt-hours-millicalorie⁻¹. Equation 12 was derived, assuming heat transfer by conduction only. In the case of heat transfer by radiation (as occurs in Calorimeter 53), the same equation applies for small temperature differences; but the sensitivity S is equal to:

$$S = \frac{0.0001736 \alpha R}{\epsilon A \sigma T_2^3} \quad (13)$$

where:

- $\epsilon =$ Emissivity, a pure number.
- $A =$ Area of emitting surface, centimeters²
- $\sigma =$ Black body constant, calories-second⁻¹-centimeter⁻²-absolute degree⁻⁴.
- $T_2 =$ Bath temperature, degrees Kelvin.
- $S =$ Sensitivity in microvolt-hours-millicalories⁻¹.

If both radiation and conduction are involved, then the sensitivity can be written as:

$$\frac{1}{S} = \frac{1}{S_c} + \frac{1}{S_r} \quad (14)$$

where:

S_c = The sensitivity as calculated from Equation 12.

S_r = The sensitivity as calculated from Equation 13.

The sensitivity of Calorimeter 51 can now be calculated on the basis of heat transfer by both radiation and conduction. By substituting the following values in Equation 13:

α = 0.0054 ohm-ohm⁻¹-degree⁻¹.

R = 1737 ohms.

ϵ = 0.5 (assumed from best available knowledge).

A = 140 centimeters².

σ = 1.355×10^{-12} calories-second⁻¹-centimeter⁻²-absolute degree⁻⁴.

T_2 = 300 degrees Kelvin.

The sensitivity assuming radiation only is:

$$S_r = \frac{0.0001736 \times 0.0054 \times 1737}{0.5 \times 140 \times 1.355 \times 10^{-12} \times (300)^3} = 0.6360 \text{ microvolt-hours-millic calorie}^{-1}$$

S_c equals 0.1793 from previous calculations. And so the resulting sensitivity:

$$S = \frac{0.1793 \times 0.6360}{0.1793 + 0.6360} = 0.1400 \text{ microvolt-hours-millic calorie}^{-1}$$

This checks much better with the experimental value of 0.1328 given above.

To continue now with our derivations, we can write from fundamental principles of heat, assuming constant pressure:

$$dQ = CdT \quad (15)$$

where:

dQ = An infinitesimal of heat in calories leaving the thermel on cooling.

C = The effective heat capacity of the calorimeter in calories per degree.

dT = An infinitesimal temperature drop of the thermel in degrees Centigrade.

Heat does not flow out over the entire length, L , of the bridge because of the three-section thermel composed of Lucite ends with a central section of metal. The bridge wire is wound over the entire length, and so if the total bridge resistance is used in Equation 9 the average temperature of all three sections of the bridge is obtained. So far as calculated sensitivities are concerned, it is proper to use the total bridge resistance, R , so long as the total length of the thermel is used also. Equation 9 cannot be used in conjunction with Equation 15 where actual temperatures are involved. In view of this, an effective bridge resistance (R_0) is defined which is equal to the total bridge resistance (R) times the ratio of the metal section of the thermel to the total length of the bridge. R_0 is given for the three calorimeters in Item 21 of Table III.

From Equation 9 Equation 16 is obtained in differential form by replacing R with R_0 :

$$dT = \frac{dV}{2500 \alpha R_0} \quad (16)$$

Combining Equations 10, 12, 15, and 16 and remembering that $\frac{dq}{dt}$ in millicalories-hour⁻¹ equals $\frac{dQ}{dt}$ in calories per second $\times 3.6 \times 10^6$ we get:

$$\frac{dV}{V} = \frac{2500 \alpha R_0}{3.6 \times 10^6 \text{ SC}} dt \quad (17)$$

which is the differential equation of cooling in a calorimeter, and by reducing we get:

$$\frac{dV}{V} = \frac{\alpha R_0}{1440 \text{ CS}} dt \quad (18)$$

The solution of (18) with the initial condition that $V = V_m$ at $t = 0$ is given by:

$$V = V_m e^{-\frac{\alpha R_0 t}{1440 \text{ CS}}} \quad (19)$$

From (19) the time, t , for the bridge potential V to fall to 0.01 per cent of V_m may be obtained as shown in Equation 20

$$t = \frac{1.327 \times 10^4 \text{ CS}}{\alpha R_0} \quad \text{seconds} \quad (20)$$

Equation 20 may be rewritten in terms of the heat capacity, C, of the calorimeter giving:

$$C = \frac{\alpha R_0}{1.327 \times 10^4} \left(\frac{t}{s} \right) \text{ calories degree}^{-1} \quad (21)$$

This equation provides an easy method for calculating the effective heat capacity of a resistance-bridge, twin-type calorimeter and also for comparing such calorimeters for heat capacities. Also it provides a fairly good method of measuring heat capacities by comparing the time, t_u , of an unknown material with the time, t_s , of a standard material. To indicate its usefulness, the heat capacity of Calorimeter 53 can be calculated as follows:

$$\alpha = 0.0054 \text{ ohm ohm}^{-1}\text{-degree}^{-1}.$$

$$R_0 = 1150 \text{ ohms.}$$

$$t = 69 \times 60 \text{ seconds.}$$

$S = 1.554 \text{ microvolts-hour-millic calorie}^{-1}$ so that the thermal capacity of the empty calorimeter is:

$$C = 1.25 \text{ calories-degree}^{-1}.$$

When the heat capacity of Calorimeter 53 was calculated from (21) with a brass slug in the sample can (t being 8550 seconds), a value of $2.58 \text{ calories-degree}^{-1}$ was obtained; and since $1.25 \text{ calories-degree}^{-1}$ was due to the calorimeter, it leaves $1.33 \text{ calories-degree}^{-1}$ as the heat capacity of the brass slug. The mass of the slug was 10.8 grams giving a specific heat of brass of $0.123 \text{ calorie-gram}^{-1}\text{-degree}^{-1}$. The handbook value is given as about 0.09.

Repeating the same calculations for Calorimeters 39 and 51, Table I is obtained.

TABLE I

DATA FOR CALCULATING SPECIFIC HEATS FROM EQUATION 21

	CALORIMETER 53	CALORIMETER 51	CALORIMETER 39
α	0.0054	0.0054	0.0054
R_0	1150	868	832
S	1.554	0.1328	0.0616
TIME EMPTY	69 x 60	52.2 x 60	66 x 60
TIME WITH BRASS	142.5 x 60	190 x 60	175.6 x 60
HEAT CAPACITY EMPTY	1.25	8.33	21.80
HEAT CAPACITY WITH BRASS	2.58	30.30	58.10
MASS OF BRASS	10.8	269.7	384.2
SPECIFIC HEAT OF BRASS BY EQUATION 21	0.123	0.0815	0.0945

The heat capacity of a calorimeter and its contents can be related to the area under the cooling curve of the calorimeter. When the power input to a calorimeter that is at equilibrium is shut off, the bridge potential will fall yielding a bridge-potential-time curve as shown in Figure 1

The area under this curve is proportional to the heat capacity of the calorimeter and its contents. This is independent of the form of the bridge-potential-time curve. In general the curve will not be a decreasing exponential function of time; however for practical purposes it can be quite adequately represented by Equation 19. The area under the curve is given in this case by

$$A = V_m \int_0^t \frac{\alpha R_0}{1440 CS} dt \quad (22)$$

where:

A = Area under the cooling curve in microvolts seconds.

Substituting for V_m its value as given by Equation 11 and integrating, we obtain:

$$A = \frac{1440 WCS^2}{\alpha R_0} \quad (23)$$

Here we see that the constant of proportionality is a function of the power input at equilibrium and the constants of the calorimeter. By substituting for S its equivalent as given in Equation 12, Equation 23 may be written as a function of the dimensions of the calorimeter. This is desirable from the standpoint of design, and Equation 24 is obtained.

$$A = \frac{\alpha R_0 CW \left(\ln \frac{r_2}{r_1} \right)^2}{56900 K^2 L^2} \quad (24)$$

Data by which this equation can be checked have been taken on Calorimeter 46. A cooling curve was run on 46 for which the radius of the brass jacket was 1.578 centimeters and the radius of the thermel was 1.372. Also a cooling curve was run for which the jacket radius was increased to 1.896 with the thermel construction remaining as before. The two areas, A_1 and A_2 , should be in ratio as given by Equation 25.

$$\frac{A_1}{A_2} = \frac{\left[\ln \frac{1.578}{1.372} \right]^2}{\left[\ln \frac{1.896}{1.372} \right]^2} = \frac{1}{5} \quad (25)$$

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The experimental ratio was 1:4, indicating fair agreement.

As shown in Equation 7, the precision of a calorimeter for measuring specific heats is proportional to the ratio of the change in area to the area under the cooling curve for the empty calorimeter; we can set up an expression as follows:

$$P = \frac{\Delta A}{A_1} = \frac{A_2 - A_1}{A_1} \quad (26)$$

where:

ΔA = Change in area under the cooling curve for a calorimeter with a sample.

From Equation 23:

$$P = \frac{\Delta A}{A_1} = \frac{C_s}{C_1} \quad (27)$$

where: C_s = Heat capacity of the sample in calories degree⁻¹.

C_1 = Heat capacity of the empty calorimeter in calories degree⁻¹.

Thus the precision index is independent of everything except the ratio of heat capacity of the sample and that of the empty calorimeter.

Also by substituting the value for C in Equation 21 in that of Equation 27 we also have:

$$P = \frac{t_s}{t_1} - 1 \quad (28)$$

where:

t_s = Time in seconds for V to reach 0.01 per cent of V_m for a calorimeter with a sample.

t_1 = Similar time for the empty calorimeter.

Suppose for instance, that one wanted to know as quickly as possible what the precision index of a calorimeter was for measuring the specific heat of some material which will fill the sample can of the calorimeter entirely. Let:

v = Volume in centimeter³ of sample can.

ρ = Density of material in grams-centimeter⁻³.

c = Approximate specific heat in calories-gram⁻¹-degree⁻¹.

Then the heat capacity of the sample is:

$$c\rho v = C_s \quad (29)$$

Now we may substitute in Equation 27 the values for C_s and C_1 , as given in Equations 21 and 29. The result is Equation 30 giving the precision index P as a function of the volume of the sample as well as the unit heat capacity, $c\rho$:

$$P = \frac{13270 \, c\rho \, vS}{\alpha \, R_0 \, t_1} \quad (30)$$

Cooling curves of Calorimeters 39, 51, and 53 were all run for the empty calorimeters and with the sample cans each completely filled with brass. The following table gives the actual precision factor as measured and the calculated one using Equation 30. The density, ρ , and the specific heat, c , for brass was taken at 8.53 and 0.09, respectively, from the handbook.

TABLE II

DATA FOR CALCULATING PRECISION INDICES OF CALORIMETERS

	V	ρ	c	S	α	R_0	$T_{0.01}$	$P \text{ CAL.}$	$P \text{ MEA.}$
CALORIMETER 39	45.10	8.53	0.09	0.0615	0.054	832	66 x 60	1.59	1.59
CALORIMETER 51	27.64	8.53	0.09	0.1328	0.0054	868	52.2 x 60	2.55	2.59
CALORIMETER 53	1.266	8.53	0.09	1.553	0.0054	1150	69 x 60	0.778	1.044

The units of P are dimensionless. The values of P (measured) are taken from Item 17, Table III.

Both the calculated precision factor and the measured one show that Calorimeter 51 is the best calorimeter for measuring specific heats. Calorimeter 53 has the least precision factor.

However, if enough sample material were not available to fill the sample can of Calorimeter 51 completely, then Calorimeter 53 might be better for the specific heat measurement. This situation is analyzed as follows:

Let $P_1 = P_2$ then from Equation 30:

$$\frac{v_1}{v_2} = \frac{R_{01} \, t_{11} \, S_2}{R_{02} \, t_{12} \, S_1} \quad (31)$$

where:

v_1 = Volume of sample in centimeters³ in Calorimeter 51 that will give the same precision as Calorimeter 53.

v_2 = Volume of the sample can in centimeters³ of Calorimeter 53.

P_1 = Precision of the sample volume v_1 in Calorimeter 51.

P_2 = Precision index of Calorimeter 53 for a full sample can. Volume of the sample equal to v_2 centimeters³.

R_{01} = Bridge resistance of Calorimeter 51 in ohms.

R_{02} = Bridge resistance of Calorimeter 53 in ohms.

t_{11} = Time in seconds to reach 0.01 per cent of V_m for Calorimeter 51 empty.

t_{12} = Similar time for Calorimeter 53 empty.

S_1 = Sensitivity of Calorimeter 51 in microvolt-hours-milliccalorie⁻¹.

S_2 = Sensitivity of Calorimeter 53 in same units.

With the following data the volume, v , of a sample in Calorimeter 51 that will give the same precision as Calorimeter 53 with a full sample can may be calculated.

$$R_{01} = 868; \quad R_{02} = 1150; \quad t_{11} = 3132; \quad t_{12} = 4140$$

$$S_1 = 0.1328; \quad S_2 = 1.55; \quad v_2 = 1.266 \text{ centimeters}^3$$

Equation 31 gives for v_1 the following value:

$$v_1 = 6.66; \quad v_2 = 8.44 \text{ centimeters}^3 \quad (32)$$

In other words if the volume of a sample is less than 8.44 centimeters³ then it would be better to run specific heat measurements in Calorimeter 53.

Now, if one had only enough material to fill the sample can of Calorimeter 53, then:

$$\frac{P_2}{P_1} = \frac{S_2 R_{01} t_{11}}{S_1 R_{02} t_{12}} = 6.66 \quad (33)$$

or one would in this case choose Calorimeter 53 in which to run the sample.

This report primarily has been concerned with the precision factor, P , by which calorimeters may be compared for specific heat determinations. However the usefulness does not end there. For instance, indirectly three ways have been presented for determining the heat capacity of a calorimeter. These are:

1. From a single cooling curve by means of Equation 21 as presented in Item 20 of Table III.

2. From a single cooling curve by dividing the area under the cooling curve by the sensitivity multiplied by 3.6×10^6 and then dividing total by the temperature rise as presented in Item 18 of Table III.

3. From two cooling curves, one empty and one with a sample of known heat capacity as presented in Item 19 of Table III.

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TABLE III

INFORMATION OBTAINED FROM THE COOLING CURVES OF
EACH CALORIMETER WITH AND WITHOUT A BRASS SAMPLE

ITEM	CALORIMETER 51	CALORIMETER 53	CALORIMETER 39
1 W IN MILLICALORIES-HOUR ⁻¹	1.090×10^6	9.070×10^4	2.336×10^6
2 V_M IN MICROVOLTS	144,700	140,829	143,622
3 S IN MICROVOLT-HOURS-PER MILLICALORIE	0.1328	1.563	0.0615
4 AVERAGE TEMPERATURE OF BRIDGE IN DEGREES CENTIGRADE CALCULATED FROM EQUATION 9	6.10	9.07	6.65
5 EFFECTIVE LENGTH OF BRIDGE L = TOTAL LENGTH OF BRIDGE	0.5L	1.00 L	0.51 L
6 TEMPERATURE OF THE METAL SECTION OF THE BRIDGE IN DEGREES ITEMS 4/5	12.2	9.07	13.03
7 TIME, T, TO REACH 0.01 PER CENT OF V_M IN MINUTES, EMPTY	52.2	69.0	66.0
8 TIME, T_S , TO REACH 0.01 PER CENT OF V_M IN MINUTES, FILLED WITH BRASS	190.0	142.5	175.6
9 MASS OF BRASS IN GRAMS	234.9	10.8	384.2
10 AREA UNDER EMPTY COOLING CURVE IN MICROVOLT-SECONDS	4.835×10^7	6.23×10^7	6.22×10^7
11 AREA UNDER COOLING CURVE WITH BRASS, IN MICROVOLT-SECONDS	17.36×10^7	12.74×10^7	16.16×10^7
12 AREA RESULTING FROM BRASS ONLY, IN MICROVOLT-SECONDS	12.52×10^7	6.51×10^7	9.94×10^7
13 HEAT IN CALORIES INITIALLY RETAINED IN EMPTY CALORIMETER ITEMS 10 $3 \times 3.6 \times 10^6$	101	11.15	281
14 HEAT IN CALORIES INITIALLY RETAINED BY BRASS ITEM 10/ 3.6×10^6 ITEM 3	261.6	11.64	44.85
15 HEAT CAPACITY OF BRASS 0.09 X ITEM 9 CALORIES PER DEGREE CENTIGRADE	21.13	0.972	34.6
16 TEMPERATURE RISE OF THE BRASS IN DEGREES ITEMS 14/15	12.37	11.98	12.96
17 RATIO OF AREA RESULTING FROM BRASS TO AREA OF EMPTY CALORIMETER ITEMS 12/10 = P	2.592	1.044	1.595
18 HEAT CAPACITY OF CALORIMETER IN CALORIES PER DEGREE ITEMS 13/6	8.28	1.23	21.60
19 HEAT CAPACITY OF CALORIMETER IN CALORIES PER DEGREE ITEMS 14/7	8.15	0.932	21.69
20 HEAT CAPACITY OF THE EMPTY CALORIMETER FROM EQUATION 21	8.33	1.25	21.80

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TABLE III-(CONT'D.)

ITEM	CALORIMETER 51	CALORIMETER 53	CALORIMETER 39
21 EFFECTIVE BRIDGE RESISTANCE R ₀ R X ITEM 5	868	1150	832
22 VOLUME, V, OF SAMPLE CAN IN CENTIMETERS	27.6	1.27	45.1

NOTE: TEMPERATURES IN THIS TABLE ARE IN DEGREES CENTIGRADE.

REFERENCE

1. Jordan, K. C., and Parks, J. R., Quart. Rpt. Gen. Res., MLM-405-5, p. 32, January 1, 1950.

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fig 1

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FIGURE 1

MM-529

DELETED VERSION

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51 fig 2

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FIGURE 2

MLM-529
DELETED VERSION

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52 fig 3

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FIGURE 3

DELETED VERSION
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DELETED VERSION

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$\Delta "A"$, CHANGE IN AREA DUE TO SAMPLE IN THE CALORIMETER.

$"A_1"$, AREA PROPORTIONAL TO HEAT CAPACITY OF EMPTY CALORIMETER.

COOLING CURVES OF EMPTY CALORIMETER
AND WITH A SAMPLE.