UNCLASSIFIED

MLM-502

DATE 11/10/50 INITIALS R.A.S.

Contract Number AT-33-1-GEN-53

MOUND LABORATORY
Operated By
MONSANTO CHEMICAL COMPANY
MIAMISBURG, OHIO

Classification changed
authority of ASLR
by Paul J. Davis
12/1/72
1/10/73
8/9/79
UNCLASSIFIED

REPORT

FOR

GENERAL RESEARCH

JULY 10 TO OCTOBER 2, 1950

(Supporting Research Volume)

SPECIAL REVIEW
FINAL DETERMINATION

Classification: Unclassified
Category:
Signature: C. W. Huntington
Date: 1/15/80

This document is
PUBLICLY RELEASABLE

Hugh Kinsed
Authorizing Official
Date: 5/26/09

This document contains restricted data
as defined by the Atomic Energy Act of
1954. The release of this information
to an unauthorized person is prohibited.

Date: November 6, 1950

Approved By:

M. M. Haring
M. M. Haring
Laboratory Director

UNCLASSIFIED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

~~SECRET~~

UNCLASSIFIED

MLM-50

DISTRIBUTION

Copy 1. - Dr. Carroll A. Hochwalt
Copy 2. - Dr. M. M. Haring
Copy 3. - Dr. J. J. Burbage
Copy 4. - Dr. R. A. Staniforth
Copy 5. - Mr. E. C. McCarthy
Copy 6. - Dr. D. S. Anthony
Copy 7. - Dr. F. C. Mead, Jr.
Copy 8. - Dr. J. F. Eichelberger
Copy 9. - Dr. J. W. Heyd
Copy 10. - Dr. J. H. Payne, Jr.
Copy 11. - Mr. R. A. Miller
Copy 12. - Mr. D. L. Scott
Copy 13. - Dr. E. A. Rembold
Copy 14. - Mr. R. F. Meehan
Copy 15. - Dr. A. W. Martin
Copy 16. - Dr. W. H. Power
Copy 17. - Dr. R. W. Moshier
Copy 18. - Mr. P. M. Engle
Copy 19. - Dr. D. L. Timma
Copy 20. - Mr. L. G. Fauble
Copy 21. - Mr. J. R. Parks
Copy 22. - Mr. R. G. Olt
Copy 23. - Dr. E. Orban
Copy 24. - Mr. M. McEwen
Copy 25. - Mr. P. E. Ohmart
Copy 26. - Mr. L. B. Gnagey
Copy 27. - Mr. W. L. Hood
Copy 28. - Mr. J. L. Richmond
Copy 29. - Mr. P. J. Lowry
Copy 30. - Mr. W. D. Prather
Copy 31. - Mr. W. H. Waldo
Copy 32. - Area Manager, A.E.C., Dayton, Ohio
Copy 33. - Area Manager
Copy 34. - Area Manager
Copy 35. - Dr. D. T. Vier (Los Alamos)
Copy 36. - Central Files
Copy 37. - Central Files
Copy 38. - Central Files
Copy 39. - Central Files

Issued: DEC 21 1950

~~SECRET~~

-3-

UNCLASSIFIED

UNCLASSIFIED

TABLE OF CONTENTS AND MAN POWER ANALYSIS

(Supporting Research Volume)

	<u>Man Months</u>	<u>Page</u>
SUMMARY		7
ELECTRONIC INSTRUMENTATION		11
Counting Systems for Pulses of Wide Dynamic Range, W. H. Baker	2.5	13
Fast Neutron Survey Meter, J. F. Combs	1.5	29
Scintillation Counters, J. F. Combs	1.0	32
Continuous Alpha Counter for Water Soluble Wastes, J. S. Stanton	2.5	41
ANALYTICAL TECHNIQUES AND INSTRUMENTS		45
Beta and Gamma Absorption Studies on a Bradley Counter, M. L. Curtis	0.5	47
Contribution to Identification of Beta Emitters Found in "WD" Building, M. L. Curtis	2.0	51
Porous-Cup Method of Spectrochemical Analysis of Waste Solutions, D. L. Timma	1.5	67
Spectrochemical Analysis of Aluminum Cans, D. L. Timma	2.0	75
A Mechanism for Drawing Quartz Fibers, R. G. Olt	1.0	79
Mass Spectrograph, D. L. Haas	6.0	85
Development of a Flexible Elevator Coil, R. J. Cote	0.5	91
CALORIMETRY		93
Microcalorimeters 52 and 53, K. C. Jordan	2.5	95

UNCLASSIFIED

~~SECRET~~

MM 502

	<u>Man Months</u>	<u>Page</u>
A Liquid Nitrogen Level Sensing Element, T. J. Beasley	2.0	105
Application of the Laplace Transform to a Calorimeter Bath Control Problem, J. R. Parks	<u>1.0</u>	111
Total	26.5	

~~SECRET~~

SUMMARY

The efficient prosecution of the research and process development on polonium, radium, actinium, waste disposal, and alpha-neutron sources requires the use of a wide variety of instrumental techniques such as: alpha, beta, gamma, and neutron counters and survey meters; emission, absorption, and mass spectroscopy; x-ray and electron diffraction; microbalances; and calorimeters. The complex and kaleidoscopic aspects of our research program require:

1. The application of standard instruments and techniques to a variety of problems.
2. The development of new techniques for the use of standard instruments.
3. The development of new or improved instruments.

The sum of the above three categories constitutes the field of supporting research.

The amplifier for counting pulses of wide dynamic range which was developed for counting beta particles in the presence of alphas has operated so satisfactorily that it has been decided to attempt to adopt it for neutron counting. Substantial improvement at medium counting rates has already been obtained but results at high counting rates are not as satisfactory (p. 13). The development of a survey meter for monitoring fast neutrons has been under way for some time. The attainment of higher efficiency has been attempted by the use of a dual-chambered counter tube. The tube operates with a filling of methane gas at two atmospheres pressure. The achievement of leak-proof seals and highest purity methane has been under investigation as their necessity has been indicated by tests (p. 29). The development of a scintillation counter for surveying and measuring low levels of alpha activity has been continued. Recent efforts have been directed towards

determining the correct voltages to be applied to the photomultiplier tube in order to operate under optimum conditions (p. 32). Recent tests of the coincidence counter to detect scintillations in water containing alpha-active waste material have indicated the necessity of obtaining photomultiplier tubes with low noise level and a better regulated, noise-free power supply (p. 41).

The Bradley counter has been used very successfully in measuring the energies of weak beta particles by determining the absorption in aluminum of known thickness. Recent work with the Bradley counter has been directed towards selecting and obtaining beta active samples of known energy to act as standards. A set of standards covering a wide energy spectra is desirable since unknowns are best evaluated with respect to a standard of about the same energy (p. 47). The Bradley counter has been applied to the determination of the half life and energy of different beta-active samples from various places on the site. Eighty-seven-day sulfur, S^{35} , has been identified tentatively as a constituent of the waste disposal influent (p. 51).

The porous cup method permits the spectrochemical analysis of a solution without requiring the evaporation of the solvent from the sample. This method in conjunction with the semiquantitative method has been applied to the determination of total solids in final effluent solutions from waste disposal. Iron content was determined to be 63.3 ± 2.5 parts per million. However, the absolute error of the semiquantitative method may run as high as 50 per cent even though the probable error may be around two and a half per cent (p. 67). The semiquantitative method has also been applied to the spectrochemical analysis of cold aluminum cans. Fifteen elements have been identified in the can. Iron and silicon were present in the amounts of 0.37 and 0.13 per cent and constituted the major impurities (p. 75).

The mechanism for drawing quartz has been improved with the installation of a cylinder of circular cross-section covered by black felt. The five per cent variation in diameter of the fiber over lengths of one inch has been diminished by the elimination of the twelve-sided reel because of the elimination of variations in velocity and the resultant reduction of impact strains. The new conditions have provided easier starting of the draw, more uniform diameters, less scratching of the fibers, easier visual location of the fibers, and easier storage of the fibers (p. 79).

The mass spectrograph has reached the shake-down stage. The dispersion and resolving power have been measured. The instrument has been brought into good focus over its entire mass range. Experience with the behavior of the machine has been gained by examining the spectra of simple, known substances (p. 85).

A new mounting has been developed for induction heater coils which are used in the "Y" Section process. The new mounting utilizes flexible leads and removable coils and simplifies centering the work coil about the object to be heated (p. 91).

There has long been a need for a microcalorimeter with a sensitive of about a hundred microvolts per millicurie of polonium, since this sensitivity would permit more accurate standardization of the alpha counters over ranges of activity not now accessible to calorimetric standardization. It has also been realized that if actinium is to be assayed calorimetrically, such a sensitivity will be required. Sensitivities in the range of fifty to two hundred microvolts have been achieved by constructing appropriately shaped Dewar vessels to hold the thermels of a resistance-bridge calorimeter. The general dependence of speed and sensitivity upon

dimensions, thermal capacity, and condition of the silvered surfaces of the Dewar have been considered (p. 95).

Highly sensitive calorimeters tend to have high levels of noise, that is random variations of the bridge potential which result from random variations in bath temperature. Thus it is necessary to reduce noise if any benefit is to be obtained from the increased sensitivity now available. Noise levels are usually reduced by operating at low temperatures. Thus some research must be directed towards investigation of the behavior of our calorimeters at low temperatures.

A device for maintaining constant the level of a liquid nitrogen bath has already been constructed (p. 105). A comprehensive mathematical survey has been made on the reduction of bath temperature fluctuations by proper operation of the present control mechanisms (p. 111).

The present study has been directed towards understanding the relationships existing between damping constants, sensitivities, and amplitudes of the various components of the system: room, bath, resistance bridge, galvanometer, photocell, amplifier, heater, and stirrer. The observed results of the experimental study so far appear to be in agreement with the mathematical predictions.

SECRET

U N C L A S S I F I E D

MLM-302

ELECTRONIC INSTRUMENTATION

U N C L A S S I F I E D

Problem Title - Counting Systems for Pulses of Wide Dynamic Range
Report By - W. H. Baker
Work Done By - W. H. Baker

INTRODUCTION

The problem of counting beta particles in the presence of alpha particles, encountered in the assay of waste samples, and the problem of analytically counting neutrons with a boron-walled, proportional, neutron counting tube are related in that both cases involve the counting of small pulses in the presence of large pulses. Thus amplifiers to be used for either one of these applications must be able to amplify weak pulses and at the same time not be overloaded by strong pulses to the point at which spurious pulses are produced and the resolving time of the counter made abnormally long.

The use of a wide-dynamic-range amplifier for counting waste samples has been so successful that attempts are being made to develop such an amplifier specifically for neutron counting. It is hoped that the use of such an amplifier will result in an improved plateau from the tube and thus reduce the required accuracy in setting the high-voltage on the tube when high-precision counting is being done.

To date the experimental neutron counter amplifier has shown a substantial improvement over previous amplifiers at medium counting rates as shown by the greatly improved plateau slope obtained with it (MLM-478, Figure 5). However, at high counting rates the plateau slope again departed from the horizontal at a rate which increased with the

U N C L A S S I F I E D

counting rate. Consequently, it became apparent that suitable test equipment would be required to examine the effect of the pile up of very strong pulses on the amplifier trigger-discriminator circuits. A high-repetition rate pulse generator designed and constructed this quarter is now available for such examination.

Since the principles and advantages of pulse-limited amplifiers are reasonably well understood, work is now being directed toward the design of a usable and stable circuit. Tentative design specifications have been set up and an input circuit has been tested.

DETAILED REPORT

The alpha-plus-beta pulse amplifier now in routine operation is shown in schematic form in Figure 1. This amplifier with a gain of 700 does not cause double pulsing and has a limited output of 13 volts regardless of input voltage. Its gain-versus-frequency curve for a single stage is shown in Figure 2, and the plateau curve for an alpha source is shown in Figure 3. Subsequent routine tests have shown that a good alpha-plus-beta plateau is also obtainable. These results indicate that this amplifier has an excellent dynamic range.

Development of a high-repetition-rate pulse generator led to the circuit of Figure 4. It consists of a 10-megacycle square wave clipper-limiter amplifier, a cathode-follower isolation circuit, a differentiating network, a 0.3-microsecond trigger-discriminator circuit, a phase-splitter and a cathode-follower output stage. The generator operates from 0.01 to 5 megacycles with a sine wave input of 4 volts R.M.S. The 0.3-microsecond output pulse has a variable repetition rate of 0.01 to 5 million, equally

U N C L A S S I F I E D

spaced pulses per second of +15 to +20 volts and -10 to -13 volts. The pulse heights at various repetition rates are shown in Table I. This variation in pulse height is caused by a change in circuit bias with an increase in repetition rate. The output pulse is variable from zero to maximum height without changing the low output impedance of the cathode follower.

The tentative design specifications for the neutron counter amplifier are as follows:

1. Use direct-coupled inverse-feedback pairs for realization of wide dynamic range.
2. Limit the output pulse to 10 volts.
3. If practical, obtain a gain stability of 0.1 per cent per 1 per cent change in line voltage.
4. Use a pentode input circuit for minimum capacitive loading unless its greater complexity outweighs its advantage.
5. Design for high gain (10,000) and low noise background (10 microvolts) for detection of very weak pulses.
6. Design for a gain versus frequency response three decibels down at two megacycles for minimum pulse distortion.
7. Use a cathode-follower output stage of low impedance for connection to a coaxial cable.
8. Design a small unit for easy mounting at the proportional ionization chamber.

Figure 5 is the schematic diagram of an input circuit which has been tested. The output pulses limits at 8.5 volts when a 2-volt input pulse is impressed on the grid circuit. Sine wave limiting of the positive

U N C L A S S I F I E D

MLM-502

portion of the signal occurs at an input of 45 millivolts R.M.S. The gain stability of this circuit is -0.27 per cent per 1 per cent change in line voltage when the filaments are connected to 6 volts D.C. This circuit has a gain of 18.6 which drops to 13.5 at 1.12 megacycles. Its frequency response is shown in Table II.

U N C L A S S I F I E D

Table I

VARIATION OF PULSE HEIGHT WITH REPETITION RATE

Repetition Rate (Megacycles)	Output Pulse	
	-	+
0.01	15	10
0.1	15	10
1.0	20	13
3.0	21	13
4.0	20	11
5.0	13	10

Table II

GAIN VERSUS FREQUENCY OF EXPERIMENTAL INPUT STAGE

Line Voltage	Frequency (Megacycles)	R.M.S. Input (Millivolts)	R.M.S. Output (Millivolts)	Gain	Db. Down
105	0.016	2.05	40	19.5	
105	0.05	2.10	40	19.0	
105	0.160	2.10	40	19.0	
105	0.5	2.15	40	18.5	
105	1.12	2.95	40	13.5	3
105	2.2	4.20	40	9.5	6
105	4.86	8.40	40	4.75	12

-19-

AMPLIFIER DATA

GAIN = 700 @ 1 MILLIVOLT INPUT
OUTPUT = LIMITED TO 13 VOLTS
BANDWIDTH = 3 DB DOWN @ 1.5 MEGACYCLES
FILAMENTS = 6.3 VOLTS A.C.

WIDE DYNAMIC RANGE AMPLIFIER FOR ALPHA-PLUS-BETA COUNTING.

U N C L A S S I F I E D

Figure 1

MLM-502

UNCLASSIFIED

Figure 2

MM-502

UNCLASSIFIED

UNCLASSIFIED

Figure 3

NLM-502

UNCLASSIFIED

-23-

UNCLASSIFIED

-25-

Problem Title - Fast Neutron Survey Meter

Report By - J. F. Combs

Work Done By - J. F. Combs

INTRODUCTION

It is desired by the Health Physics Instrument Section to develop a survey instrument which will detect fast neutrons, but will be insensitive to any other type of radiation. Such an instrument is necessary for fast neutron survey work. At the present time there is no fast neutron survey meter available which is simple, light weight, and portable.

The detecting device, fully described in previous reports,^{1,2} consists of a chamber filled with methane gas which 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 a predetermined value.

Several experimental counters have been designed and constructed. They have differed only in the means of obtaining a gas-tight and vacuum-tight seal. The counters are cylindrical in shape, two inches in diameter, and 1 3/8 inches in length. Originally the inner walls were lined with polyethylene sheathing 1/32-inch thick. The center electrode consists of a 1/2-inch diameter loop of 1/2-mil stainless steel wire.

When the counter was filled with methane to a pressure of two atmospheres, a fast neutron counting efficiency of 0.05 per cent was achieved. Gamma discrimination was obtained, and the necessary applied high voltage was 2,500 volts.

U N C L A S S I F I E D

DETAILED REPORT

In order to attain a higher efficiency a counter was built which utilized two such chambers as previously described placed end to end in one unit. This counter was sealed by means of a threaded cap and Neoprene gaskets.

The counter was checked for leaks by filling it with methane to a pressure of two atmospheres and measuring the pressure with a mercury manometer over a period of four days. During the 96-hour check period the height of the mercury column was observed to remain constant (except for a variation of plus or minus two millimeters caused by atmospheric pressure and temperature changes). It was concluded that there were no leaks in the counter.

It is believed that the low sensitivity of the fast neutron counters tested previously was caused in part by impurities in the methane gas. The methane used to fill the dual compartment counter will be purified in a liquid nitrogen compartment as recommended by Mr. Wm. Hurst of Oak Ridge National Laboratory. The necessary additions to the vacuum system to accomplish this purification were made.

Hurst found that polyethylene cannot be satisfactorily outgassed. Therefore the polyethylene lining of the counter walls was removed, and the present counter will be tested with no such hydrogenous wall lining. Future models may be partly lined with paraffin.

It will be necessary to outgas the counter for five days to a vacuum of 0.01 micron or better. It will then be immediately filled with the purified methane.

U N C L A S S I F I E D

U N C L A S S I F I E D

MLM-502

REFERENCES

1. Quarterly Report for General Research, MLM-443-4, pp. 77-80.
2. Quarterly Report for General Research, MLM-478, pp. 53-57.

U N C L A S S I F I E D

Problem Title - Scintillation Counters

Report By - J. F. Combs

Work Done By - J. F. Combs

INTRODUCTION

The development of a quantitative alpha scintillation counter was continued. Such a counter is desired by the Health Physics Division for low level alpha counting. The scintillation counter offers an instrument with simple construction, high efficiency, and low background.

The counter used for test purposes consisted of a Type 5819 photomultiplier tube with the silver-activated, zinc sulfide phosphor applied directly to its end window, and a Nuclear Instrument and Chemical Corp. Model 162 scaler. Previous results¹ have indicated that a counting geometry of 35 per cent is attainable with this counter. Both voltage and gain plateaus were found to be very satisfactory for samples under 25,000 D/M. These results were recorded with the voltage between the anode and 10th dynode of the photomultiplier tube reduced from the normal value of 50 volts recommended by the manufacturer to 0 volts. There resulted a large improvement in plateau shape with this voltage reduction. It was then desired to determine if there existed an optimum point between 0 volts and 50 volts at which the best results could be obtained.

DETAILED REPORT

In order to vary the voltage between the anode and 10th dynode of the photomultiplier tube, the fixed resistance in the voltage divider across which these electrodes are connected was replaced with a variable resistance.

U N C L A S S I F I E D

Tests were then conducted with the counter to determine the effect on plateau shape of varying the voltage between the anode and 10th dynode from 0 volts to 90 volts. The resulting plateaus are presented in Figures 1, 2, and 3.

The voltage plateaus, Figure 1, were found to decrease in length as the value of V_{AD10} (the voltage between the anode and 10th dynode) was increased from 0 volts to 10 volts. A further increase in the voltage between these electrodes resulted in a slight increase in plateau length. The first effect, the reduction of plateau length, is believed caused by an increase in the amplitude of the noise pulses in the photomultiplier tube as the voltage between the anode and 10th dynode is increased. The latter effect results since V_{AD10} is part of the total voltage applied to the tube and the reduction in the voltage applied across the first nine dynodes becomes noticeable as V_{AD10} increases to voltages of 30 volts and greater. As this reduction occurs the gain of the whole tube decreases slightly and the resultant decrease in noise pulse amplitude causes the plateau length to increase a small amount at the high voltage end.

The full length of the plateau for the $V_{AD10} = 0$ volts setting could not be obtained, since at an input sensitivity of 130 millivolts greater gain than can be obtained with the normal operation of the Type 5819 photomultiplier tube is necessary.

Data were taken at an input sensitivity of 50 millivolts, however, and the resultant plateau with the setting of $V_{AD10} = 0$ volts (not shown in this report) is approximately of the same length and slope as that shown for the setting of $V_{AD10} = 1$ volt in Figure 1.

The gain plateaus, Figures 2 and 3, show that as the value of V_{AD10} is increased from 0 volts to 90 volts, the absolute length of the gain plateaus is increased. This increase in plateau length is caused by an actual increase in the gain of the photomultiplier tube. But since the gain of the noise pulses is also increased, the ratio of the input sensitivity setting at one end of the plateau to the input sensitivity setting at the other end remained essentially the same, 5 to 1, for all values of voltage applied the two electrodes. Therefore, considering only gain plateaus, any of the values of V_{AD10} used will produce satisfactory results. The data for Figures 2 and 3 were taken under the same operating conditions. They were plotted in two figures only for the purpose of clarity.

It was concluded from the above findings that the optimum value of voltage to be applied between the anode and 10th dynode to obtain the most satisfactory voltage and gain plateaus was 1 volt.

A V_{AD10} setting of 1 volt can be considered optimum, however, only in applications where part of the gain of the photomultiplier tube can be sacrificed in order to obtain the improvement in plateau shape.

REFERENCE

1. Quarterly Report for General Research, MLM-478, pp. 59-74.

UNCLASSIFIED

UNCLASSIFIED

Figure 1

MLN-502

UNCLASSIFIED

-35-36

-39-40

UNCLASSIFIED

UNCLASSIFIED

Figure 3

MM-502

Problem Title - Continuous Alpha Counter for Water Soluble Wastes

Report By - J. S. Stanton

Work Done By - J. S. Stanton

INTRODUCTION

At the present time the level of alpha activity at various points in the waste disposal process is measured by making glass slides with 300 lambda of the solution, drying them and sending them to the "I" Building for counting. This system does not fulfill the requirements of a continuous operation in that spot checks only can be made and thus there is no continuous information as to the level of activity. Also there is considerable time lag between when the sample is taken and when the results are received and operating conditions may have become completely altered in that period of time.

To improve this situation it was decided to attempt to develop an alpha detector that could be used in the "WD" Building itself as a continuous monitor of the waste solutions. Originally it was planned to use a large proportional alpha counting tube suspended directly above the solution. However, when it was found that recoil protons caused water to scintillate and when a literature search revealed that scintillations had been observed from alpha particles in water it was decided to attempt to detect these scintillations to see if this effect could be employed in a continuous alpha monitor because the number of disintegrations occurring in several milliliters would be counted in this case thus the overall count would be more accurate and could be made in a shorter length of time.

U N C L A S S I F I E D

In building a scintillation counter to detect a few very weak pulses per minute the big problem encountered is that background pulses resulting from noise in the phototube circuit can easily mask the desired pulses. Therefore to reduce the number of background pulses it was decided to build the counter as a coincidence device with two multipliers scanning the sample. Thus when a scintillation would occur in the water sample as a result of alpha bombardment both tubes would produce simultaneous pulses and the instrument would record the count. However, since most noise pulses would not be emitted from the tubes simultaneously they would not be recorded as counts.

The detection method now being tried consists of two identical multiplier phototubes, and two amplifier and trigger circuits, both coupled into a mixer whose output is connected to a scaler. When supplied with pulses of varying width and amplitude, the trigger circuit performs the function of supplying a very narrow pulse of constant amplitude to the mixer circuit. The mixer combines the pulses from the trigger stage so that discrimination between coincident signal pulses and anti-coincident noise pulses is obtained. The signal pulses are extremely weak, so the problem becomes one of reducing noise to the lowest possible value.

DETAILED REPORT

From the very first test of the first counting equipment to determine if scintillations from alpha particles in water could be detected, it was realized that special techniques, circuits, and equipment were required if these pulses were to be detected in the presence of background noise.

The first step to improve the signal-to-noise ratio was to improve the amplifier and trigger circuits. When the first device was built, it was found that the pulses from the individual amplifier circuits were too broad to give good non-coincidence rejection, and the pulse height from the mixer (when two pulses were received simultaneously) was not sufficiently different to give good coincidence acceptance and anti-coincidence rejection. So a new, fast trigger circuit was inserted between the amplifier and the mixer provided output pulses of 0.3-microsecond in duration. This circuit greatly improved the discrimination between signal and noise.

The mixer was improved by changing the circuit from a cathode follower type to a parallel amplifier type. In this type circuit a discrimination of two to one is obtained between coincident and non-coincident pulses.

During the process of checking the new circuits, a very erratic counting rate was noted when the high voltage to the two multiplier phototubes was turned on. Oscilloscopic examination of the 1,000-volt power supply showed many 0.25-volt (peak to peak in amplitude) pulses on the A.C. line and an occasional transient pulse of 1 volt. This interference was sufficient to cause coincident noise pulses in the output of the photomultiplier tube and to register as a signal pulse. Filters of various types were tried but were not too successful.

A battery source of high voltage power was tried with an Eveready type X-591 (890 volts) for each phototube. These batteries eliminated the noise from the power supply but proved to be disadvantageous since the voltage would drop during the course of a count or series of measurements

U N C L A S S I F I E D

thus the number of counts per unit time would change during the counting period. Also, with the noise level reduced, more gain was needed to bring the signal level up to the value required to trip the trigger circuit.

The gains of the amplifiers were increased to 5,200 and 5,700 by decreasing the feedback of the first stage of both amplifiers. At this gain the dark current would again trigger the scaler but when tested there was no indication that alpha particles were being detected. As a result of this it was decided to have a stronger-than-normal waste-water sample made, so that the increase in "dark noise" due to alpha activity would exceed the statistical variation in count due to dark noise alone. A sample of water having approximately 5,000 counts per minute per milliliter was tried, but there was again no change in the counting rate before and after the sample was introduced.

A measurement of the dark current pulse amplitude was then made and levels as great as 3.5 millivolts were occasionally noted. These levels seemed high; but since no other tubes were available, no other tests were made.

Special low-noise type 5819 multiplier phototubes have been on order, and until they arrive, no further tests will be made. It is not practicable in this case to cool the tubes, since dry-ice temperatures are needed, and the frozen water may cause a severe loss of light.

In the meantime a special, noise free, regulated, high voltage power supply is being constructed by the Electronics Shop for use with the new tubes.

U N C L A S S I F I E D

MLM-502

ANALYTICAL TECHNIQUES AND INSTRUMENTS

U N C L A S S I F I E D

-45-46

Problem Title - Beta and Gamma Absorption Studies on a Bradley Counter
Report By - M. L. Curtis
Work Done By - M. L. Curtis

INTRODUCTION

It is desirable in identifying beta emitters, to be able to make aluminum absorption studies on emitters of lower energies than can be detected on window counters. A Bradley counter has the advantages over window counters of more favorable geometry, and no window for the radiation to penetrate. Work had progressed on the Bradley absorption chamber to the point where good voltage plateaus had been obtained with absorbers in front of the sample. Absorbers available ranged from 0.15 to 1910 milligrams per square centimeter.

DETAILED REPORT

The problems remaining were largely in interpreting the results of the absorption curves obtained with the counter. Feather¹ points out that the visual range of the radiation as read from the absorption curve is often considerably less than the actual range. He suggests that standards be used for comparison which approach the energy of the sample to be analyzed. Accordingly an absorption curve was made on a sample of carbon-14. This curve is given in Figure 1. A maximum energy of 0.16 million electron volts was calculated with the Glendenin range energy relationship.² This value compares favorably with the value of 0.156 million electron volts given in the literature. A "Feather Analyzer" as described by Glendenin² was prepared for use in analyzing radiation approaching this energy.

U N C L A S S I F I E D

A sample of RaE will next be studied for use as a standard for radiation approaching million electron volts, and standards of other energies will be obtained as they are needed.

Glendenin² places a lower limit of 0.1 million electron volts on the energy of radiation that can be determined accurately by absorption measurements with end window counters, assuming the thinnest possible counter window of 1 milligram per square centimeter. Those counters used at Mound have windows approximately 3.5 milligrams per square centimeters. The geometry of our end-window counter is approximately 5 per cent, so that a comparatively high level of activity must be available for measurement.

The Bradley counter geometry approaches 50 per cent, so that measurements can be made on one tenth of the material required by the end-window counter. Energies as low as 0.01 million electron volts are detectable, and it should be possible to determine accurately the energy of radiation as low as 0.05 million electron volts.

REFERENCES

1. Feather, N. Proc. Phil. Soc. (Cambridge) Vol. 34, (1938).
2. Glendenin, L. E., Nucleonics, January, 1948.

Problem Title - Contribution to Identification of Beta Emitters Found
in "WD" Building

Report By - M. L. Curtis

Work Done By - M. L. Curtis and J. F. Hunt

INTRODUCTION

A group of samples were submitted for absorption and half-life studies in an effort to identify the beta emitters found in the "WD" Building.

DETAILED REPORT

Six samples have been studied for half-life and energy determinations. The samples studied were:

1. "WD" influent treated to remove alpha emitters. Sample prepared by R. L. Norton.
2. A supposedly pure polonium standard of 1.2×10^9 alpha disintegrations per minute. The sample was studied with 12 milligrams per square centimeter of absorber to eliminate alpha counts.
3. "HH" filtrate treated to remove alpha emitters. Sample prepared by R. L. Norton.
4. Bismuth scrub, studied with 12 milligrams per square centimeter of absorber to eliminate alphas. Sample prepared by R. L. Norton.
5. Sample of a Hanford aluminum can. Sample prepared by M. T. Ayers.
6. Material separated from "WD" influent solution believed to be radioactive sulfur. Sample prepared by L. L. Bentz.

~~SECRET~~

MLM-502

The half-life studies are shown in Figures 1, 2, and 3. Least squares determinations were made of the half lives of the polonium sample, the "WD" influent, and the "HH" filtrate. The half lives of the other samples were estimated from the graphs. For the sample of the Hanford can, half lives of the sample as a whole, and for the portion able to penetrate 12 milligrams per square centimeter of aluminum were estimated.

Absorption studies were made of each sample as shown in Figures 4, 5, and 6. Maximum energies were determined with Glendenin's¹ range-energy relationship.

A summary of the results are given in Table I.

REFERENCE

1. Glendenin, I. E., Determination of Energy of Beta Particles and Photons by Absorption, Nucleonics, January, 1948.

~~SECRET~~

Table I

ENERGY AND HALF LIFE VALUES OF BETA EMITTERS FOUND IN "WD" BUILDING

<u>Sample</u>	<u>Time Observed</u>	<u>No. of Observations</u>	<u>Half Life</u>	<u>Maximum Energy</u>
1. "WD" Influent	86 Days	58	86.6 $\pm$ 1.2 Days (Least Squares)	0.16 Mev.
2. Po with 12 mg./cm. ² absorber	88	48	169.6 $\pm$ 5.2 Days (Least Squares)	0.19 Mev. Also stronger Beta or Gamma.
3. "HH" Filtrate	62	39	91.0 $\pm$ 6.4 Days (Least Squares)	0.16 Mev.
4. B1 Scrub	51	30	5 Days	1.2 Mev.
5. Hanford Can	65	43	103 Days for total sample. 66 Days for harder component	7.3 Mev. + Strong Beta or Gamma
6. Sulfate	20	11	86 Days	0.16 Mev.

UNCLASSIFIED

Figure 2

MLM-502

HALF-LIFE STUDIES IN BRADLEY COUNTER.

UNCLASSIFIED

-57-58

ABSORPTION STUDIES IN BRADLEY CHAMBER.

UNCLASSIFIED

Figure 6

MLA 502

UNCLASSIFIED

-65-66

~~CONFIDENTIAL~~

MLM-5C2

Problem Title - Porous-Cup Method of Spectrochemical Analysis of Waste Solutions

Report By - D. L. Timma

Work Done By - D. J. Hunt and D. L. Timma

INTRODUCTION

Research on methods of removing beta activity from solution wastes has resulted in the need for analytical control of the various steps in the treatment. Since most of the samples were taken as solutions, the porous cup method of analyzing a solution for its dissolved solids without an isolation step was adapted for the determination of iron. Samples were mixed with an equal volume of a standard cobalt solution to supply an internal standard and then placed in a porous thin-bottomed upper electrode. Analytical working curves have been constructed covering the range from 2.5 to 450 parts per million of iron.

DETAILED REPORT

Original analytical work on the final effluent solutions from the waste disposal process included the determination of total solids. This step was time consuming because of the weighings and evaporation involved. Iron was then determined semiquantitatively in the residue. In later samples it was not necessary to determine the amount of solids dissolved in the effluent solutions, so a more direct method of determining iron was sought.

The porous cup method¹ permits the analysis of a solution without the evaporation of the water from the sample. The method consists

~~CONFIDENTIAL~~

of placing the liquid sample in a porous, thin-bottomed electrode and allowing a spark to strike the solution as it seeps through the bottom of the electrode. By adjusting the parameters in the secondary of the spark source, the discharge may be continued without heating the sample solution in the upper electrode to its boiling point. By placing an internal standard in the sample solution the spectrograms may be treated in the standard manner and working curves constructed.

The exposures were made on a sample solution containing iron, varying the inductance from 25 to 100 microhenries with the capacitance constant at 10 microfarads and the resistance constant at 20 ohms. The intensity of the iron spark lines increased as the inductance was increased. High values of inductance caused the electrodes to be heated until the solution samples boiled out of the electrode. Varying the capacitance from 5 to 25 microfarads with 75 microhenries inductance and 20 ohms resistance again resulted in an increase in iron-line intensity. Again high values of capacitance caused boiling of the sample. Varying the inductance while using a constant value of capacitance of 20 microfarads did not result in a marked variation in iron-line intensity but caused sample boiling. Variation of the secondary resistance did not result in an observable change in iron-line intensity. The conditions selected to give maximum excitation without excessive heating of the electrode were 15 microfarads capacitance, 25 microhenries inductance, and 25 ohms resistance.

Previous qualitative examination of the effluent solutions from the waste disposal plant had not disclosed the presence of cobalt. Because of the similarity of cobalt and iron, the former was selected as an internal standard

~~CONFIDENTIAL~~

MLM-502

for the estimation of iron. A series of cobalt solutions of different concentrations were excited by the porous cup method. A cobalt concentration of 100 parts per million gave spark lines of approximately the correct blackening for densitometric evaluations.

A series of standards were prepared each member of which contained 100 parts per million of cobalt and an amount of iron ranging from 2.5 to 450 parts per million. These samples were examined in duplicate by the porous cup method. The per cent transmission of several spark lines of both cobalt and iron occurring the 2600A region was determined.

The standard method using a stepped sector (2X) was employed for constructing a plate response curve for the S.A. No. 1 emulsion at 2600A. Exposures in the second and third orders were necessary because of the astigmatism of the spectrograph and the inability to place the stepped sector at the stigmatic focus of the grating for the first order of 2600A. All photographic plates were developed in D-19 for three minutes at 68°F.

The per cent transmissions of the iron and cobalt lines from the exposures of the standard solutions were converted to relative intensities, and intensity ratios were calculated for different combinations of iron and cobalt lines. Plotting the ratio of the intensity of an iron spark line to the intensity of a cobalt spark line as a function of iron concentration gave a working curve by which the estimation of the iron content of a sample could be made. By using working curves for four different iron lines, the range of iron concentration from 2.5 to 450 parts per million could be covered (Figure 1).

~~CONFIDENTIAL~~

To analyze a solution, an aliquot was mixed with an equal volume of a standard 200-parts-per-million cobalt solution, and the resulting mixture was placed in the upper porous-cup electrode and sparked for two minutes. The excitation constants were $C = 15$ microfarads, $L = 25$ microhms, and $R = 25$ ohms. The spectra were recorded on a S.A. No. 1 plate with the first order of a three-meter Baird spectrograph. The spectrograms were evaluated by measuring the per cent transmission of the 2598, 2599, 2617, or 2621 iron lines, and of the 2694 cobalt line. The ratio of the intensity of iron to the intensity of the cobalt line was then calculated and used in conjunction with the working curves (Figure 1) to estimate the amount iron present in the sample.

To check the accuracy of this method a synthetic sample was formulated with four milliliters of a sample which had been analyzed previously and found to contain 23 parts per million of iron, four milliliters of a 200-parts-per-million cobalt solution, and two milliliters of one of the standards. The standard used had 250 parts per million of iron and 100 parts per million of cobalt. The resulting synthetic sample contained 59 parts per million of iron and 100 parts per million of cobalt.

By using a synthetic sample containing some of a submitted sample, the impurities which might interfere with the analysis of a submitted sample are introduced into the synthetic sample. Tests made with such a synthetic sample would indicate if extraneous materials in the sample were influencing the accuracy of the analysis.

Ten spectrograms were made with this sample. When iron 2621 and cobalt 2694 lines were used, the average iron content found was 63.3 parts per million with a standard deviation of 2.75 parts per million and a

probable error of 2.5 parts per million. This probable error was 4 per cent of the amount present. Some samples were available which had previously been analyzed for iron by evaporating the solution and then analyzing the residue for iron content by a semiquantitative method. One of these samples in which 94 parts per million of iron had been found by the semiquantitative method gave values of 57, 61, 61, and 64 parts per million of iron by the porous cup method. These results agreed within the limits of error since the semiquantitative method gave answers correct to only ± 50 per cent of the amount present.

REFERENCE

1. Feldman, C., Anal. Chem. 21, 1041-46 (1949).

UNCLASSIFIED

Figure 1

MLA-502

UNCLASSIFIED

-73-74

~~SECRET~~

MLM-502

Problem Title - Spectrochemical Analysis of Aluminum Cans

Report By - D. L. Timma

Work Done By - D. J. Hunt, G. Pish, and D. L. Timma

INTRODUCTION

Investigation of the final effluent from the liquid waste disposal treatment plant showed the presence of beta activity not being entirely removed by the methods employed. In an attempt to account for the amount of activity found, the materials at this site which had been irradiated in the pile were investigated to determine if they contained sufficient quantities of impurities to account for the beta activity detected. The aluminum cans were analyzed spectrographically by a semiquantitative technique. Cans were then dissolved, and the acid-insoluble and base-insoluble fractions investigated separately.

DETAILED REPORT

In an attempt to establish more clearly the source of certain radioactivities which were appearing in the liquid wastes, the composition of the aluminum cans was investigated. Since an approximate analysis was sufficient, a semiquantitative technique¹ was used.

The first analysis was made directly upon the aluminum metal. All of the elements listed in Table I except boron, silver, and nickel were measured by this step. The percentages found are thought to be accurate to ± 50 per cent of the amount present. It was noted that when material from different parts of a can were analyzed separately, such as

~~SECRET~~

the side, top, and weld sections, the results for some elements were different. This would indicate that the top was made from a different batch of raw material than the main body of the can. The weld seemed to have a higher concentration of manganese and vanadium, but was similar to the walls of the can in titanium, nickel, and chromium content. Since the preponderant mass of the can was in its walls, the averaged analysis was taken as that obtained for the sides.

A complete aluminum can was dissolved in hydrochloric acid. The weight of material was 52.5 grams, about 1.5 grams of the original weight having been lost in the process of opening the can to remove the bismuth slug. After the solution was completed, a black, flocculent, insoluble portion remained. This insoluble portion was filtered off, washed, and ignited lightly. The residue weighed 90.1 milligrams. A repeat run gave a residue weighing 94.0 milligrams. These acid insoluble residues were analyzed semiquantitatively, and, in addition to traces of materials found earlier, small amounts of nickel, cobalt, silver, and boron were detected. Because the usable boron lines were too dark for satisfactory densitometric measurement, the amount of boron present in the arc had to be adjusted by diluting the sample one to fifty with pure zinc oxide before the analysis for boron could be made. The results for these elements are also given in Table I.

Sodium hydroxide was added to the filtrate containing the acid soluble portion of the can until the aluminum went back into solution as the complex aluminate ion. The brown precipitate was filtered off, washed, and ignited. It weighed 1.8019 grams. Spectrographic examination of this residue did not disclose the presence of any elements which had not been determined previously.

~~SECRET~~

MLM-502

The results of this investigation are tabulated in Table I. The results are only semiquantitative. However, they do indicate that the aluminum metal was of the 2S type having an aluminum content of at least 99.0 per cent. The presence of impurities, particularly those of high cross section, indicate that the can may be in a factor in absorbing neutrons during irradiation in the pile and in contributing to the presence of spurious types of radiation encountered in the processing of the irradiated bismuth.

REFERENCE

1. Timma, D. L., Quarterly Report for General Research, MLM-478, pp. 111-17.

~~SECRET~~

~~SECRET~~

MLM-502

Table I
SEMIQUANTITATIVE ANALYSIS OF ALUMINUM CAN

<u>Element</u>	<u>Per Cent by Weight of Impurity</u>			<u>Average</u>
	<u>Weld</u>	<u>Side</u>	<u>Top</u>	
Si				0.13
Fe				0.37
Sn				0.0021
Mg	--	0.017	--	--*
Mn	0.043	0.011	0.011	0.011**
V	0.009	0.004	0.004	0.004**
Pb				0.007
Cu				0.04
Bi				0.003
Ga				0.02
Ti	0.030	0.030	0.008	0.030**
Ni	0.0016	0.0016	0.0008	0.0016**
Cr	0.004	0.004	0.0009	0.004**
Co				0.000085***
Ag				0.00009***
B				0.000021***

* Mg contamination probably due to handling.

** Preponderant mass was in the side.

*** Determined in the acid insoluble residue.

~~SECRET~~

Problem Title - A Mechanism for Drawing Quartz Fibers

Report By - R. G. Olt

Work Done By - M. I. Gray and R. G. Olt

INTRODUCTION

Quartz fibers are useful as a material of construction of instruments such as microbalances, electroscopes, electrometers, and galvanometer suspensions because of the chemical inertness and generally excellent physical properties of such fibers.

A variety of methods of producing quartz fibers has been described in the literature, but all of these methods lack means for controlling the size of fiber drawn and hence, result in chance yields of fibers of usable diameters and lengths.

This randomness of resultant fiber size has been a minor problem in the construction of instruments in small quantities or with broad performance tolerances. However, in the construction of instruments in larger quantities and to close performance specifications, the lack of control of fiber size results in the expenditure of large amounts of time in the drawing and selection of usable fibers.

The development of a mechanism for drawing quartz fibers in long lengths to predetermined diameters has been reported as having reached a stage permitting prediction and control of fiber diameter to within limits of plus or minus five per cent of that desired. The residual diameter variations have been found occurring in a random manner within lengths of the order of an inch.¹

In the reduction of known sources of inconsistencies in the fiber drawing process, the provision of thin bands over the reel rungs to form a cylindrical reeling surface has reduced the fiber vibration and velocity variation resulting from the reel rung geometry. In addition to the improved steadiness of the draw, the reel bands facilitate starting of the draw and provide an improved means for handling and storing all sizes of fibers.

DETAILED REPORT

The application of the runged reel to the drawing of quartz fibers made practical the production of long strands of fiber from which a number of shorter lengths could be selected for size, classified, and stored for future use.

More critical study of conditions that influence the drawing of precision fibers has shown that the instantaneous velocity of the drawn fiber for a 12-rung reel varies as much as 3.4 per cent between the extreme reeling conditions of tangent contact with one rung and chordal contact on two rungs. In addition to this variation in velocity which causes up to a 1.8 per cent variation in fiber diameter, the impact of the rungs on the fiber arriving at the reel set up nonaxial vibrations which detract from the desired constancy of drawn conditions.

In choosing a means for reducing reel-induced variations, a number of ways of obtaining a cylindrical drawing surface were considered. The use of paired, removable, flexible bands over the reel rungs provides the desired cylindrical drawing surface and in addition, provides a compact means for transporting and storing fibers with a minimum of handling and attendant fiber deterioration.

Trials of 0.010-inch phosphor bronze; 0.015-inch annealed, Type 303, stainless steel; and 0.005-inch hard-temper, 17-7 PH stainless steel have shown that both a high modulus of elasticity and a high yield strength are desirable in the material used for reel bands. The low modulus of elasticity of phosphor bronze permits sharp bending over the rungs with resultant noncircularity of the band surface. The low yield strength of annealed stainless steel causes the bands to retain the curvature of the reel on removal. The 0.005-inch hard-temper stainless steel permits formation of a true cylindrical surface on the reel with return to the flat storage shape on release from the reel.

As used, the bands have been covered with black felt for prevention of fiber surface scratches and as a background for the location of fine fibers after drawing. Double-faced pressure-sensitive tape is used for attaching the felt to the bands.

The slack formed in the fibers and felt on change from the circular to straight band shape is removed by releasing and refastening one end of the felt to the band. Slack formation may be minimized by the use of thin bands and fabric.

Trial of the 0.015-inch stainless steel bands covered with 1/16-inch black felt for the drawing of five-micron-diameter fibers has shown substantial reduction of vibration at the fusion area. In addition to the improved steadiness of draw, it has been observed that the reduction of reel-induced vibration also makes the starting of the draw easier.

Figure 1 shows a reel of five-micron-diameter quartz fibers drawn on 0.015-inch stainless steel reel bands covered with 1/16-inch

U N C L A S S I F I E D

MLM-502

black felt. One reel band has been removed from the reel and placed on a simple plywood support of a type used in conjunction with fiber storage and shipping containers.

REFERENCE

1. Olt, R. G., MLM-478, pp. 93-109.

U N C L A S S I F I E D

U N C L A S S I F I E D

FIGURE 1

MM 302

FIBER DRAWING REEL AND REMOVABLE BANDS

U N C L A S S I F I E D

Problem Title - Mass Spectrograph
Report By - D. L. Haas
Work Done By - D. L. Haas, C. H. Mellor, and C. L. Leshar

INTRODUCTION

Mass spectrographs have been developed to the point that they can be used as a sensitive detecting and measuring device in many chemical and physical problems. A double-focusing (focusing by both electric and magnetic fields) mass spectrograph has been under construction for some time; because it was believed that the versatility of the instrument would allow it to be used as a tool in the solution of many problems in our development program.

The spectrograph is essentially complete and has reached the shakedown stage of test runs to achieve optimum performance. It is planned at present to use the machine first to investigate yields of ion guns.

DETAILED REPORT

The double-focusing (Mattauch type) mass spectrograph, together with all associated electronic power supplies, regulators and detectors, has been completely assembled and operated. Instabilities in the electronic accessories were remedied and the insulation problems rectified. The magnet has been calibrated as shown in Figure 1 by using standard flip-coil techniques. The dual task of improving the resolving power and calibrating the instrument over the entire mass range is now in progress.

Mittrich¹ has shown that double-focusing over the entire range of the photographic plate occurs in this instrument when

$$\rho = 2(V \cdot m / Ne)^{1/2} / H.$$

Solution of this equation for the mass gives

$$m = \frac{NeH^2\rho^2}{2V}$$

where

ρ = Distance on photographic plate from the entering edge of the pole face to the line in question.

V = Accelerating potential in volts.

m = Mass of ion being analyzed.

N = Number of charges.

H = Field strength in oersteds.

e = Charge of an electron.

Introducing the numerical value of e gives, for $N = 1$,

$$m = 25 \times 10^{-6} \frac{H^2\rho^2}{V}.$$

The dispersion is the separation of the lines corresponding to two effective masses differing by one part in one hundred. Dispersion is inherently dependent upon the radius of curvature of the instrument, and hence cannot be altered. The dispersion coefficient of this instrument, with a maximum ρ of 21.5 centimeters, is 1.07 millimeters. For example, the separation of singly ionized silver-107 from silver-108 will be 107/108 millimeters.

UNCLASSIFIED

Resolution² is the least distinguishable mass difference that can be detected. The theoretical resolving power is dependent upon the width of the defining slit. The present slit width is 0.001 inch which gives a theoretical resolving power of 1 part in 1500 for lines not over exposed. The slit width can be varied and hence the resolution of this machine can be improved.

Spectra have been obtained to develop operational techniques, improve resolution, eliminate electrical breakdowns, and to secure optimum focusing. At the present time, the instrument is being operated with only one slit, the defining slit which theoretically is the only slit required. However, a second slit will be presently installed to increase the sharpness and linearity of the spectral lines. The electrostatic potential on the condenser plates is very critical and the presently employed electronic power supply will be replaced by a battery supply in an effort to improve the sharpness of the line.

Spectra have been obtained of the tungsten crucible and tantalum stem. This characteristic spectrum, once obtained and positively identified can be used as the internal comparator for other analyses in the appropriate mass range. Spectra were then obtained with a platinum stem substituted for the tungsten-tantalum crucible. A gold spectrum was obtained. In each spectrum an effort was made to improve the focusing properties of the instrument by proper alignment of electrostatic field potential values with accelerating potential values. A ratio of 1 to 22 has been experimentally determined as the optimum ratio of electrostatic field potential to accelerating potential.

UNCLASSIFIED

Spectra have also been taken of tin oxide, platinum, and bismuth in tungsten, and identification has been tentatively made. More spectra are necessary before positive identification is possible.

REFERENCES

1. Mattauch, J., Phys. Rev. 50, 617 (1936).
2. Shaw, A. E. and Rall, W., Rev. Sci. Inst. 18, 278 (1947).

UNCLASSIFIED

-89-90

Problem Title - Development of a Flexible Elevator Coil

Report By - R. J. Cote

Work Done By - R. J. Cote and J. F. Haskins

INTRODUCTION

Many times during the "Y" Section development work, progress was delayed while new induction heater coils were formed. It was felt that the installation of flexible leads with a work coil removable from the front of the hood would be advantageous. Therefore, the apparatus described below was fabricated.

DETAILED REPORT

Four three-inch pieces of quarter-inch copper tubing were flared; two were bent at a forty-five degree angle. One inch above the flare nut a woven copper braid was soft soldered to the bent pieces by means of a copper sleeve. About seven inches of white rubber tubing (1 1/4 inch by 1/8 inch) was placed on the four pieces of tubing through the braided tubing. Next the copper braid was pulled snug over the rubber tubing; secured with hose clamps, and finally, soldered to the straight pieces of copper tubing as before. This resulted in two flexible leads which were secured by a plastic clamp. A work coil was made up and screwed to the leads by means of one quarter inch flare to flare unions.

This apparatus was used with fair success, although it became evident that the hose clamps were being heated when current was applied for more than 1 minute. Eventually, the heated clamps melted the rubber tubing causing a leak.

U N C L A S S I F I E D

To remedy this, the hose clamps were discarded and the rubber tubing was bound to the copper tubing with solid copper wire. The wire was insulated from the woven copper braid with Scotch electrical tape. The copper braid was also taped to give a snug contact between the braid and rubber tubing. A metal screw in the plastic clamp which extended between the leads, and was also being heated, was removed. The clamp was secured with plastic dowels.

With these changes the apparatus has proven to be durable. It simplifies the centering of the work coil about the object to be heated and enables one to change the position of the coil readily.

U N C L A S S I F I E D

~~SECRET~~

MM-50

CALORIMETRY

~~SECRET~~ -

-93- 94

~~SECRET~~

MLM-502

Problem Title - Microcalorimeters 52 and 53
Report By - K. C. Jordan
Work Done By - K. C. Jordan

INTRODUCTION

There has long been a need for a microcalorimeter with a sensitivity of about a hundred microvolts per millicurie of polonium, as this sensitivity would permit more accurate standardization of the alpha counters over ranges of activity not now accessible to calorimetric standardization. It has also been realized that if actinium is to be assayed calorimetrically, such a sensitivity will be required. Direct modification of the macrocalorimeters used in Operations assay has so far lead to a sensitivity of only one-tenth the desired value which has indicated the need for more developmental work.

The research on a high sensitivity microcalorimeter (Calorimeter 52), which utilizes Dewar-type flasks as the insulating medium, has continued.

In the Quarterly Report, MLM-478, the effects of various Dewars, as to the degree of silvering and evacuation, the thermel construction, and modes of heat transfer were discussed.

It seemed desirable to know whether the area of the thermel would cause a proportional change in sensitivity as indicated from the fourth power radiation law. A thermel of one-half length was made and tested in the various Dewars. The area of this thermel was half that of the previous one.

~~SECRET~~

~~SECRET~~

MLM-502

The next step was to immerse one of these calorimeters in liquid nitrogen in order to study the change in heat radiation. This step called for a change in Dewar design because of the water condensation inside the calorimeter at low temperatures. Since a half-length thermel had already been made for the preceding experiment, it was easy to make another similar thermel, and design a new Dewar flask around them. This was done, and the result was Calorimeter 53.

The detailed report will deal with the research on Calorimeters 52 and 53 in turn.

DETAILED REPORT

Calorimeter 52

A thermel with a length one half of the original thermel was made by winding four layers of No. 44, 99-alloy enamel-insulated wire instead of two layers of wire as in the previous thermels. Experiments were run with these thermels inserted in both the unsilvered and well-silvered Dewars. The results showed an increase in sensitivity of 1.68 over that of the longer thermel in the case of the unsilvered Dewar. This increase was attributed to the change of the area factor in the fourth-power law of radiation and hereby strengthened the theory that loss of heat was primarily by radiation.

The results on the well-silvered Dewar showed an increase of sensitivity of 1.55 which again indicates loss of heat primarily by

~~SECRET~~

radiation. Other experiments indicate little loss by radiation. Further experimentation will be required to determine this anomaly.

So far all well-silvered Dewars (three in number) have gradually decreased in sensitivity with time in such a way that they all are approaching the same sensitivity. See Table I for these variations in sensitivity.

On the other hand the poorly-silvered Dewar has maintained its original sensitivity. This fact indicates that the emissivity factor of the radiation law changes in the case of the well-silvered Dewars. Air leakage seems improbable.

Also the explanation of the variations in sensitivities of the Dewars as given in Quarterly Report MLM-478, pages 15 and 16, is now invalid. The time of measuring the sensitivities after silvering was not introduced. It is reasonable to believe now that the time element may be a factor instead of the seal-off pressure as presented in that article.

The changing of sensitivity with time of the well-silvered Dewars interrupted the precision runs to be made with our micro-sample. These runs were started again with Dewar No. 1 on the sample side, since it has been in use the longest and is changing sensitivity by less than 0.1 per cent per day, and with Dewar No. 3 on the dummy side. Two runs made to date agreed within 0.23 per cent, the microsample having a curie value of 0.124. These runs will be continued indefinitely in an effort to establish the precision of the calorimeter.

Calorimeter 53

Calorimeter 53, as stated in the introduction, is made to utilize the half-length thermel used in experiments with Calorimeter 52 and also is designed to permit its operation in a liquid nitrogen bath.

The thermels are constructed similarly to those in Calorimeter 52 except for using four layers of No. 44, 99-alloy, enamel-covered wire wound on 1 1/2 inches of Invar thermel form instead of two layers of the same wire wound on the three inches of Invar as in Calorimeter 52. Its heater, sample can, leads, and Lucite sleeve are the same in both cases.

The jackets are made as follows. Two Dewar-type flasks constructed of one-inch outside diameter Pyrex tubing and one-centimeter inside diameter Pyrex tubing seven inches long are connected together at their ends so that both flasks can be evacuated and silvered simultaneously. Ground glass male joints are attached at the openings in each flask and are connected together by a glass tube. This assembly thus permits the leads from the neck of one flask to be brought over to the neck of the other flask without being exposed to the environment. The two or three bridge leads are joined as close to the flasks as possible, allows all lead wires to be brought out through a single ground glass joint, and permit free access for the sample can through the other joint.

It was decided not to silver these flasks because of the long time taken for the sensitivity of silvered Dewars to become stable.

SECRET

Also before evacuating the flasks the sensitivity was measured on both sides and the microsample measured three times. The sensitivities were 25.55 and 24.94 microvolts per millicurie. The curie equivalents of the microsample runs were 0.00, 0.24, and 0.33 per cent higher than the average value obtained with Calorimeter 44. The speed of both sides were 38 and 40 minutes to come within 0.01 per cent of equilibrium.

The calorimeter flasks were then evacuated to seal-off pressure of 2×10^{-5} millimeters. The sensitivities rose to 46.9 and 47.5 microvolts per millicurie with corresponding speeds of 79 and 83 minutes. Two runs on the microsample gave values of 0.40 and 0.53 per cent higher than Calorimeter 44. This indicates that the higher the sensitivity the greater is the deviation with respect to Calorimeter 44. Preliminary runs with Calorimeter 52 have indicated the same results. With a sensitivity of 97, Calorimeter 52 is now giving results 0.85 per cent higher than Calorimeter 44. The reason for this difference has not been determined or even conjectured.

Table I lists the sensitivities of the three best silvered awards with respect to the time, the curie-equivalent of the heater, bath temperature, and the thermal used. All of these are influential in causing sensitivity changes.

Speed Considerations

The equilibrium time (proportional to the inverse of speed) of a highly sensitive calorimeter becomes of major importance. It is

~~SECRET~~

necessary in designing such a calorimeter to insure the greatest speed possible without sacrificing accuracy too severely. These conditions require as low a heat capacity as possible.

In comparing the speeds of calorimeters as to merit, it is not fair to compare the time taken to reach 0.01 per cent of equilibrium as we usually do, because the sensitivities may differ. It seems best therefore to compare speed merits by the ratio of equilibrium time to sensitivity, which to a first order approximation is proportional to the apparent heat capacity of the calorimeter. This proportionality is a constant for any reasonable temperature rise for any one calorimeter and the constants of two or more calorimeters are the same so long as the bridges have the same kind of wire and effective number of ohms.

Table II shows the calorimeters with their ratios of equilibrium time to sensitivity with the units of minutes per microvolt per millicurie. From the table it is interesting to note that the ratio for Calorimeter 46 is 16 in both cases, even though the sensitivity was doubled in changing jackets.

Also it is interesting to know that the ratio of Calorimeters 39 and 37 are not too much different even though from appearances the thermels in Calorimeter 37 had much less heat capacity. The reason probably is that in Calorimeter 37 the heat was allowed to spread out over the entire thermel; whereas in Calorimeter 39 it was allowed to spread only over the central section and thus only the central section was allowed to rise in temperature. Parts of a calorimeter that do not retain heat cannot contribute to reducing the speed. This is the reason for calling the ratio of speed to sensitivity the apparent heat capacity.

~~SECRET~~

~~SECRET~~

MLM-502

Also the reason for the decreasing of the apparent heat capacity with sensitivity in Calorimeters 52 and 53 probably is caused by the fact that the calorimeters are transferring heat by radiation in the cases of the lower sensitivities; and it is conceivable that less of the calorimeter will be heated when heat transfer is taking place by radiation than when heat is forced to be transferred by conduction along the inner tube of the Dewar and the sleeve supporting the thermel.

~~SECRET~~

Table IDEWAR NO. 1(Silvered in April, 1950)

<u>Sensitivity</u>	<u>Date Measured</u>	<u>Curie Equivalent of Heater</u>	<u>Bath Temperature (Degree C.)</u>	<u>Type of Thermal (Inches)</u>
112.0	May 2	0.380	26.5	3
107.46	May 29	3.49	0	3
107.42	May 31	5.45	0	3
107.75	June 1	0.871	0	3
107.80	June 2	0.217	0	3
106.53	June 6	9.49	0	3
98.60	Aug. 29	3.49	26.5	3
97.82	Sept. 8	3.48	26.5	3
97.72	Sept. 22	0.126	26.5	3
97.25	Sept. 26	0.126	26.5	3

DEWAR NO. 2(Silvered June 26, 1950)

170.8	July 5	3.49	0	3
178.3	July 12	0.180	0	3
180.4	July 13	0.180	0	3
180.7	July 18	0.180	0	3
180.6	July 21	0.180	0	3
140.6	Aug. 1	0.165	26.5	3
125.5	Aug. 10	0.154	26.5	3

~~SECRET~~

Table I-(Continued)DEWAR NO. 3(Silvered June 26, 1950)

<u>Sensitivity</u>	<u>Date Measured</u>	<u>Curie Equivalent of Heater</u>	<u>Bath Temperature (Degree C.)</u>	<u>Type of Thermel (Inches)</u>
118.1	Aug. 17	0.149	26.5	3
113.2	Aug. 25	0.872	26.5	3

DEWAR NO. 3(Silvered July 18, 1950)

228.3	July 26	2.00	0	1 1/2
184.9	Aug. 18	0.888	26.5	1 1/2
181.8	Aug. 24	0.888	26.5	1 1/2
176.1	Aug. 31	3.550	26.5	1 1/2
113.9	Sept. 5	3.79	26.5	3
115.8	Sept. 6	0.950	26.5	3
115.2	Sept. 11	0.950	26.5	3

Table II

COMPARISON OF SPEED VERSUS SENSITIVITY OF CALORIMETERS

<u>Calorimeter</u>	<u>Equil. Time Sensitivity</u>	<u>Calorimeter</u>	<u>Sensitivity</u>	<u>Equil. Time Sensitivity</u>
30-I-II-III-IV-V	40-45			
36	63.2	52	178.3	3.82
37	31.2	52	170.8	3.60
38	6.33	52	35.1	3.25
39	34.2	53	228.3	2.37
41	26.4	53	176	2.31
43	17.5	53	47.5	1.56
46 (with 1st jacket)	16.0	53	25.55	1.57
46 (with 2nd jacket)	16.0			
47	37.1			
48	38.8			
51	17.7			

Problem Title - A Liquid Nitrogen Level Sensing Element
Report By - T. J. Beasley
Work Done By - T. J. Beasley

INTRODUCTION

The Calorimetry Research Laboratory has as a goal the measurement of a power of 0.010 calories per hour with a precision of one per cent. This problem is essential to Mound Laboratory since a solution to this problem would aid in purity assay of polonium and in standardizing the alpha counters.

The microcalorimeter research program has been pushed to the point where a calorimeter has been made that has a fair chance of measuring this small power. This calorimeter is No. 53 and is discussed in detail in Paper No. 12 in this volume.¹

So far the limiting factor in the microcalorimetry work has been in the temperature fluctuations of the bath. This part of the problem is also under attack. Research has begun on room temperature and bath temperature fluctuations, their causes and their control. An initial report is also in this volume.²

In addition to the above mentioned study of bath conditions and their effects on the microcalorimeters, it is planned as a part of the research to run Calorimeter 53 in liquid nitrogen. Calorimeter 53 was designed with this in mind. Two problems are involved: first, keeping moisture from freezing inside the calorimeter and second, maintaining the liquid level as constant as possible.

~~SECRET~~

After the servo-mechanism problem associated with the latter requirement was studied, it was decided that an on-off type of level sensing element would be used.

The design of a recently completed liquid nitrogen level sensing element is the subject of this report.

DETAILED REPORT

M. S. Fred and E. G. Rauth reported in the Review of Scientific Instrument³ a liquid nitrogen level controller incorporating a resistor as the level detector.

However, the detector described below depends on dielectric rather than thermal parameters for its operation. Use was made of the fact that the sparking potential of air is about one half that of liquid nitrogen (approximately 30 to 60 kilovolts per centimeter respectively).

Following is a description of the circuit shown in Figure 1. Two tungsten electrodes (0.05-inch diameter) were vertically disposed with the end of the lower electrode fixed at the desired level of the liquid nitrogen so that recession of the liquid would permit the spark to fire while immersion would quench the spark.

Across the electrodes 600 half-wave direct-current volts appear when the spark is quenched, while the current is restricted to about two milliamperes (by two megohms of resistance in the spark circuit) when the impedance of the gap is sharply reduced following the arc-over of the gap. Half-wave direct current is obtained by the use of a 1B3GT tube in the spark circuit.

~~SECRET~~

Since the conditions at the negative lower electrode of a spark gap are more critical than those at the positive electrode (insofar as arcing is concerned) a direct-current source is indicated.

The 1/120-second dead time associated with each cycle of half-wave direct current is long enough to permit the dissolution of any gaseous envelope which might otherwise develop and persist upon slow immersion of the negative electrode. Persistence of such an envelope would permit sub-surface arcing and thus defeat our purpose.

The flow of spark current associated with arc-over generates a voltage across the 0 - 30,000 ohm twin potentiometer which bucks the six-volt negative bias imposed on the grid of an FG-27A thyatron by the "C" battery.

It will be seen from the diagram that the thyatron is across the alternating current line, which means, of course, that it is only capable of passing current every other half cycle (when the cathode is negative and the plate positive). However current cannot pass during the aforementioned half cycles unless the battery bias is bucked sufficiently. In connection with the thyatron operation it should be noted that once it is triggered, changes in the grid potential during the balance of the half wave period are inconsequential.

The biasing signal due to the flow of spark current is shifted in phase by the RC network until the peak occurs at the beginning of the firing half-cycle of the thyatron. Thus we have half-wave direct current passed by the thyatron when an arc exists and no thyatron current when the spark is quenched.

The solenoid has characteristics which cause it to stay in the closed position without chattering when half-wave direct current from the thyatron passes through it.

It will be noted that phase relationships among the various elements of the circuit are important. Therefore connections were made so that 180° shifts cannot be made inadvertently when plugging in the different units.

Special attention was also given to the grounding system so that insulation loads are at a minimum. Positive action of the system has been observed for spark gaps up to about two millimeters.

Quantitative checks for optimal gap separation will be made when the entire servo-system is complete.

REFERENCES

1. Jordan, K. C., Microcalorimeters 52 and 53, Quarterly Report MLM 502, pp. 97-106.
2. Burke, J. R., Application of the Laplace Transform to a Problem of Calorimeter Bath Control, et al. Quarterly Report MLM 502, pp. 111-143.
3. Fred, M. S. and Ruth, E. G., Rev. Sci. Instruments 21, 225, (March, 1950)

~~SECRET~~

Figure 1

UNCLASSIFIED

-109-110

CIRCUIT DIAGRAM FOR LIQUID NITROGEN LEVEL CONTROL.

Problem Title - Application of the Laplace Transform to a Calorimeter Bath Control Problem

Report By - J. R. Parks

Work Done By - J. R. Parks, T. J. Boasley, K. C. Jordan, and F. Shon

INTRODUCTION

Microcalorimetry is a very useful technique in the operation of Mound Laboratory. It is highly useful in combination with the quartz-fiber microbalance in assaying the purity of polonium, since the calorimeters are used in standardizing the alpha counters throughout the project. With the microbalance and a microcalorimeter of sufficient sensitivity and stability, purity may be determined without the use of the counters.

The Logac-L alpha counter has been standardized by calorimetry in the upper part of its range. However, there is a great need on the project for a calorimeter that will standardize the counters in the range of 10^8 to 10^2 counts per minute, and lower if possible.

Group 8, the Calorimetry Research Group, has developed a twin resistance-bridge microcalorimeter, Number 53, that has a sensitivity of 45.9 microvolts per curie of polonium and an equilibrium time of 80 minutes.¹ The step-down Wenner potentiometer used to measure a balance in the bridge is one millivolt. Thus the calorimeter could detect 1/46.9 millicuries of polonium if the background caused by the bath temperature fluctuations were zero, which is equivalent to the detection of a power of 0.00068 calories per hour.

At present, the background caused by the bath is such that the microcalorimeter cannot detect one millicurie of polonium with one per cent precision

To reduce background variations in bath temperature a "proportional" controller was installed on bath number one as shown in Figure IV. The signal to the controller caused by a change in bath temperature is taken from a resistance bridge thermometer and fed to a galvanometer in the controller which reflects a wide beam of light on to a photoelectric cell. The deflecting galvanometer changes the light flux on the cell which changes the current delivered by the thyatron to the bath heater. The heater current is proportional to the galvanometer deflection and therefore proportional to the bath demand. Hence the control has been called a proportional control.

Previous to this time the problem of bath control has been attacked analytically. However, the problem has either been so highly simplified that the value of the results have been nullified for general use, or it has been complicated by the use of the second order differential equation for heat flow applied to models simulating the bath characteristics such as employed by Z. Jelonek in a study of bath control.^{2,3,4,5}

In our attack on the problem we make use of the observed fact that for relatively small temperature differences, Newton's law of heat flow holds very adequately; and our starting point is the first order differential equation for heat flow in a well stirred bath.⁶

The mathematical method known as the Laplace transform has been used to analyze such problems with very good results.⁷ Essentially the Laplace transform is a mathematical tool with which a system of linear

~~SECRET~~

differential equations with constant coefficients may be transformed into a system of simultaneous linear algebraic equations of a complex variable. By a study of the solution of the simultaneous equations in various portions of the complex plane, the properties of the solution of the system of differential equations may be determined without going through the chore of finding the solution. It is also possible more easily to study changes in the control mechanism by working with the Laplace transforms than by working with the differential equations. This type of analysis has been made on bath number one in the Calorimetry Laboratory and is the substance of this report.

DETAILED REPORT

Figure 4 shows a schematic diagram of bath number one and the control. The bath contains approximately 54 gallons of water which has a thermal capacity of 2×10^7 calories per degree. The control heater is placed directly under the stirrer. The efficiency of stirring was checked by introducing small quantities of ink at various points of the bath surface and measuring the time for it to be distributed evenly throughout the bath. The greatest time observed was 15 seconds. This measurement indicates that temperature gradients cannot persist in the bath for times longer than this. The walls of the bath are copper-clad plywood one and one-half inches thick. The top is open.

The bridge thermometer as shown in Figure 1 consists of two thin-walled brass tubes attached to a head where lead connections are made. In one tube are two layers of nickel wire wound on a Lucite rod

which compose two opposite arms of the bridge. The other tube contains two layers of manganin completing the bridge.

The controller as shown in Figures 2 and 3 contains a high sensitivity galvanometer with a period of 16.5 seconds. The output current of the controller thyatron is put through a 300-watt industrial heater.

When the bridge thermometer, the controller, and output heater are considered as a unit, the sensitivity of the controller was measured as 15×10^4 watts per degree temperature change at the thermometer.

The critical factors in the operation of any controller and controlled system are the damping constants of the components of the whole system. These constants were measured experimentally for the bath and controller components or calculated from known data. Table I gives the damping constants.

At this point let us discuss the operation of the bath and controller. We may assume that the ambient conditions of humidity and temperature have remained constant for a long time and that the bath is under control. This means, one, that the galvanometer in the controller is maintaining a steady deflection because of the steady output of voltage by the bridge thermometer for a steady bath temperature, U , which is at the control temperature, U_0 . Steady deflection of the galvanometer reflects a steady portion of light on the photoelectric cell which controls the thyatron at a steady output of power. This power is the input to the bath which, when added to the power of stirring and other constant sources of power, just balances the power leak from the bath to the environment thus maintaining the bath temperature at the control point, U_0 . Bath number one is controlled at approximately 2.5° above room temperature.

~~SECRET~~

Now, let us assume that at some instant the room temperature begins to oscillate with an angular velocity and amplitude to which the bath will respond. At a time later the bath temperature, U , will begin to deviate from the control point by an amount, $\delta(t) = U - U_0$ where $\delta(t)$ is the deviation of the bath temperature as a function of time, t . At a time still later the bridge thermometer will send a signal to the galvanometer which in its response will change the amount of light on the photocell which in turn will change the power output of the heater by an amount, q_c . This power, q_c , is called the corrective power input to the bath and is positive or negative depending on whether the bath temperature deviation, $\delta(t)$, is negative or positive, i.e. the bath temperature, U , falls below or rises above the control point, U_0 .

If the time of input and the value of q_c is right, the deviation $\delta(t)$ may be properly corrected. The deviation, $\delta(t)$, cannot be reduced to zero. However, its amplitude can be made small by control. This is the object of bath control. This also is the object of this study.

In what follows, the terminology and method are that used by Gardner and Barnes in their book "Transients in Linear Systems."⁷

The output of the bath is the temperature deviation, $\delta(t)$. This deviation is the input of the controller. The output of the controller is the corrective power, q_c , which is the input to the bath completing the controller and controlled system loop. This system is unusual in that the corrective power input, q_c , is disturbed by an input disturbance given by

$$d_1(t) = K \gamma(t) \quad (1)$$

~~SECRET~~

where $d_1(t)$ = Input disturbance in calories second⁻¹

K = Lumped thermal conductivity of the entire surface of the bath
in calories degree⁻¹ second⁻¹

$\theta(t)$ = Room temperature deviation in degrees

t = Time in seconds

The reaction of the bath to power input and room disturbance is given by the following equation:

$$C \frac{d\delta(t)}{dt} + K \delta(t) = q_c + d_1(t) \quad (2)$$

where

C = Thermal capacity of the bath = 2×10^5 calories degree⁻¹

q_c = Corrective power input in calories second⁻¹

The controller can be described better as a system of differential equations.

1. The bath output affects the bridge thermometer by the following equation

$$c_1 \frac{du_1}{dt} + k_1 u_1 = k_1 \delta(t) \quad (3)$$

where

c_1 = Thermal capacity of the thermometer in calories degree⁻¹

k_1 = Thermal conductance of the jacket in calories degree⁻¹ second⁻¹

u_1 = Temperature change of the thermometer at time, t , in degree

Since the thermometer is a bridge, its output caused by temperature change, u_1 , is given by

$$V = \sigma_1 u_1 \quad (4)$$

where:

V - Voltage output of the bridge in volts

σ_1 - Sensitivity of the bridge to temperature change in volts degree⁻¹

2. The voltage output, V , of the thermometer is the input of the high sensitivity galvanometer whose equation of motion is:

$$\frac{d^2\theta}{dt^2} + a \frac{d\theta}{dt} + \beta\theta = \gamma V \quad (5)$$

where:

θ = Galvanometer deflection in radians

a = Electrical damping coefficient of the galvanometer in seconds⁻¹. The galvanometer is critically damped.

β = The unit restoring torque of the galvanometer suspension in seconds⁻²

γ = The electromechanical coefficient of the galvanometer coil in radians volt⁻¹ seconds⁻²

The sensitivity of the galvanometer is given by

$$\sigma_2 = \frac{\gamma}{\beta} \text{ radians volts}^{-1} \quad (6)$$

3. The galvanometer deflection, θ , through the photocell changes the grid voltage of the thyatron in the controller, giving a corrective power to the heater as shown by:

$$q_h = \sigma_3 \theta \quad (7)$$

where

q_h Power supplied the heater in calories second⁻¹

~~SECRET~~

σ_3 - Sensitivity of the thyatron amplifier in calories second⁻¹ radian⁻¹

4. The output power, q_h , of the controller acts on the heater by the following equation

$$c_3 \frac{du_3}{dt} + k_3 u_3 = q_h + k_3 \delta(t) \quad (8)$$

where

u_3 - Temperature change of the heater in degrees

c_3 - Thermal capacity of the heater in calories degree⁻¹

k_3 - Thermal conductivity of the heater sheath plus insulation in calories degree⁻¹ second⁻¹

At this point it is seen that the corrective power supplied to the bath at time, t , is given by:

$$q = k_3 [u_3 - \delta(t)] \quad (9)$$

The sensitivity, σ , of the controller is given by the product of the sensitivities of the bridge, galvanometer, and thyatron amplifier.

$$\sigma = \sigma_3 \sigma_2 \sigma_1 \text{ calories degree}^{-1} \text{ second}^{-1} \quad (10)$$

The controller sensitivity has been experimentally determined as

$$\sigma = 3.6 \times 10^{-4} \text{ calories degree}^{-1} \text{ second}^{-1}$$

Now by solving our equations (1) to (10), we may obtain an expression involving the bath temperature variation, $\delta(t)$, the room disturbance, $T(t)$, and the output of the bath and controller. The interpretation of the result can best be done with the Laplace transform.

However there is a simple case we may consider at this point. Let us assume that the thermometer, the galvanometer, and the heater act

~~SECRET~~

instantaneously. This is equivalent to saying that the coefficients of the derivatives in Equations (3), (5), and (8) are zero. In this case we get the following for the corrective power input to the bath:

$$q_c = -\sigma_1\sigma_2\sigma_3 \delta(t) = -\sigma \delta(t) \text{ calories second}^{-1} \quad (11)$$

Equation (11) indicates that under the assumed conditions the controller is a true proportional controller.

Equations (1), (2), and (11) form the basic system of equations of the bath with a proportional control. If we combine them as indicated, Equation (12) is obtained.

$$C \frac{d \delta(t)}{dt} + \sigma \delta(t) = K \tau(t) \quad (12)$$

The term, $K \delta(t)$, in Equation (2) is neglected since it is very small compared to the others.

Now assuming the bath to have been under control, $\delta(t) = 0$, up to time $t = 0$ and assuming the room disturbance to begin at $t = 0$ to oscillate with one degree amplitude and an angular velocity of ω as given by Equation (13), we may apply the Laplace transform to Equation (12).

$$\tau(t) = \sin \omega t \text{ degrees} \quad (13)$$

From the transform we may deduce the following things about a bath that is controlled by a control that has zero lag.

1. The deviation, $\delta(t)$, of the bath from the control temperature will be a sine wave that lags behind the room temperature oscillation and has the same angular velocity, ω .
2. The amplitude, A , of oscillation of the bath deviation is given by

$$A = \frac{K}{\sqrt{C^2 \omega^2 + \sigma^2}} \text{ degrees} \quad (14)$$

~~SECRET~~

This equation indicates that the amplitude will be smaller for

- a. Better insulated baths, where K is small.
- b. Larger apparent thermal capacity baths, where C is large.
- c. A more sensitive controller, where σ is large.

3 The bath is self-regulating in that with no controller the amplitude is, since $\sigma = 0$ in this case,

$$A = \frac{K}{C\omega} \text{ degrees} \quad (15)$$

Here we see that if $\frac{K}{C}$, the damping constant of the bath, is small (a very slow bath), the amplitude of the bath temperature variation will be small excepting for the very slow oscillations of room temperature. If the damping constant of the bath is equal to 2×10^{-5} seconds⁻¹, and if the amplitude of the room temperature sinusoid is one degree with a period of 80 hours the bath will respond with an amplitude of one degree.

A room temperature oscillation of 80-hour period is not of experimental interest since practically all experimental work in the laboratory is of not more than eight hours duration. From Equations (14) and (15) we may compare the reaction of bath number one to a room temperature oscillation of one degree amplitude and 80-hour period with and without control. The angular velocity is given by

$$\omega = \frac{2\pi}{T} = 0.00021 \text{ radians second}^{-1} \quad (16)$$

where

T = Period of oscillation of room and bath = eight hours

Without control using Equation (15) we get the amplitude of the bath temperature variation to be

~~SECRET~~

$$A = 0.1 \text{ degree} \quad (17)$$

With a control that has zero time lag, the amplitude of the bath temperature variation is, using Equation (14)

$$A = 0.0001 \text{ degree} \quad (18)$$

It is evident that control helps considerably and it is also evident that control may be worse with lags in the controller.

It should be noted at this point that the thermal capacity of the bath, C , is an apparent thermal capacity in the case with control. This apparent thermal capacity can be made quite small by putting the heater very near the thermometer, in which case the amplitude of the bath temperature variation will tend to be larger. Also the thermal conductivity, K , of the bath is apparent and will tend to be larger the greater the exposure of water at the top of the bath and the greater the stirring of the bath. A larger K will tend to make the amplitude of response greater. Increasing K and decreasing C makes the bath more sensitive to higher frequency room temperature oscillations. However, the relative positions of the heater, the stirrer and the thermometer are such in bath number one in the Calorimetry Laboratory that the apparent thermal capacity approximates the real value.

The case of a controller with zero lag is approximated by a real bath of low thermal conduction to the room and high thermal capacity (small damping constant) equipped with a fast thermometer, a fast galvanometer, and a fast heater (large damping constants). However in a real case in which the controller has lag, it is known that beyond a certain

~~SECRET~~

controller sensitivity an increase may produce large oscillations in the bath temperature.

This problem is of considerably greater complexity than the previous one. However considerable information can be obtained by a study of it.

Before proceeding we will assume that C, the thermal capacity of the bath, approximates the thermal capacity of bath number one in the Calorimetry Laboratory; that the damping constant of the bridge thermometer is the determining factor in lag of the controller; that the damping constants of the galvanometer and heater are of the same order of magnitude and are very large compared to that of the bath thus making their action practically instantaneous. From Table I which lists these damping constants it can be seen that the last two assumptions are not too far off.

From Equation (2) the damping constant, λ , of the bath is given by:

$$\lambda = \frac{K}{C} \text{ seconds}^{-1} \quad (19)$$

Since the galvanometer is critically damped, the damping constant of the galvanometer is from Equation (5).

$$\lambda_2 = \frac{a}{2} \quad (20)$$

The damping constants of the thermometer and the heater are from Equations (3) and (8).

$$\lambda_1 = \frac{k_1}{c_1} \text{ and } \lambda_3 = \frac{k_3}{c_3} \text{ respectively.} \quad (21)$$

From our assumptions we have:

$$\lambda_3 = \lambda_2 \gg \lambda \text{ and } \lambda_1 > \lambda \quad (22)$$

From these assumptions and from Equations (3) to (9) inclusive for a bath with a lagged controller we get the following equations which lead to the equation for the corrective power, q_c , as a solution of a first order linear differential equation.

$$c_1 \frac{du_1}{dt} + k_1 u_1 = k_1 \delta(t) \quad (23)$$

$$V = \sigma_1 U_1 \quad (24)$$

$$\theta = \sigma_2 V \quad (25)$$

$$q_c = \sigma_3 \theta \quad (26)$$

$$q_c = -\sigma \lambda_1 e^{-\lambda_1 t} \int e^{\lambda_1 t} \delta(t) dt \quad (27)$$

The integral is a function of the upper limit, t , only since the initial condition is zero.

Here we see, in comparing Equations (27) and (11), that the controller is not truly proportional since q_c is not a linear function of $\delta(t)$. However as λ_1 increases without bound (bridge thermometer becoming more instantaneous), it can be shown (see Appendix A) that Equation (27) reduces to Equation (11), since in physical actuality $\delta(t)$ and its derivatives exist, are continuous, and are bounded.

Equations (1), (2), and (27) form the basic equations for a bath with a lagging thermometer. Now assuming as before that the system has been under control to time $t = 0$ and that the room temperature at $t = 0$ begins to oscillate with an amplitude of one degree and angular velocity, ω , and applying the Laplace transform to Equation (28) which is a combination of (1), (2), and (27),

$$C \frac{d\delta(t)}{dt} + \sigma \lambda_1 e^{-\lambda_1 t} \int_0^t e^{\lambda_1 t} \delta(t) dt = K \tau(t) \quad (28)$$

(Here, again, the term $K \delta(t)$ of Equation (2) has been dropped because of its negligible size.)

where $\tau(t) = \sin \omega t$ degrees, we get as the transform:

$$CS \Delta(S) + \frac{\sigma \lambda_1}{S + \lambda_1} \Delta(S) = K \frac{\omega}{S^2 + \omega^2} \quad (29)$$

where,

S = Complex argument of the Laplace transform

$\Delta(S)$ = Laplace transform of the bath deviation, $\delta(t)$

The Laplace transform of the integral in Equation (28) can be constructed by reference to the theorems on the transform of an integral and of complex translations in the complex domain as given in "Transients in Linear Systems" by Gardner and Barnes on Pages 129 and 245.

Solving (29) for $\Delta(S)$ we get

$$\Delta(S) = - \frac{K \omega (S + \lambda_1)}{(S^2 + \omega^2) (CS^2 + C \lambda_1 S + \sigma \lambda_1)} \quad (30)$$

Now the characteristic of $\delta(t)$ will depend on the poles of Equation (26) which are the roots of the denominator. The following may be deduced for a bath with a lagging thermometer:

$\delta(t)$ will be composed of the sum of two components. One is a sinusoid with the same angular velocity as the room deviation but behind it in phase. The other component, which is induced by the controller, will depend on the roots of the following quadratic equation where S is the unknown.

$$CS^2 + C \lambda_1 S + \sigma \lambda_1 = 0 \quad (31)$$

~~SECRET~~

Three cases will arise.

1. If $c^2 \lambda_1^2 - 4c \sigma \lambda_1 > 0$ the roots of (31) are real and unequal and lie in the left-hand half of the complex plane.

Consequently the second component of $\delta(t)$ will be the sum of two damped exponentials. Hence if the room oscillations persist long enough, the first component will remain only, and we should have to determine its amplitude to find the dependence on λ_1 and the sensitivity of the controller.

2. If $c^2 \lambda_1^2 - 4c \sigma \lambda_1 = 0$ the roots of (31) are real and equal and lie in the left half of the complex plane. In this case the second component becomes one damped exponential. This is generally the critical situation.

3. If $c^2 \lambda_1^2 - 4c \sigma \lambda_1 < 0$ the roots of (31) are complex conjugates and lie in the left half of the complex plane. In this case the second component will be a damped sinusoid with a damping constant equal to $\frac{\lambda_1}{2}$ and an angular velocity Ω given by

$$\Omega = \left[\frac{\sigma \lambda_1}{c} - \left(\frac{\lambda_1}{2} \right)^2 \right]^{1/2} \quad (32)$$

In essence Equation (31) says that if the sensitivity, σ , of the controller is large then the damping constant, λ_1 , of the thermometer should be as large as possible to make damping rapid but not so large that Ω is small since if the controller induces a long period temperature variation in the bath, the calorimeters will respond to this extra disturbance.

The general thought is that the sensitivity of the controller and its damping constant should be such that $(C^2 \lambda_1^2 - 4C \sigma \lambda_1)$ should be as nearly zero as possible. This would be good if at the same time the amplitude of the sinusoid induced by the room variation is as small as possible. At this point we should study the amplitude of the room-induced sinusoid when the controller also induces a sinusoid of angular velocity, Ω , as given by Equation (32). In this case Equation (30) becomes:

$$\Delta(s) = - \frac{\lambda \omega (s + \lambda_1)}{(s^2 + \omega^2) \left[\left(s + \frac{\lambda_1}{2} \right)^2 + \Omega^2 \right]} \quad (33)$$

where

$$\lambda = \frac{K}{C} \text{ the time constant of the bath in seconds}^{-1}$$

Now the amplitude of the room-induced sinusoid is obtained from the

$$\text{Real part of } \frac{\lambda \omega (s + \lambda_1)}{\omega \left[\left(s + \frac{\lambda_1}{2} \right)^2 + \Omega^2 \right]} e^{i\omega t} \quad (34)$$

This gives the amplitude as in Equation (35), using Equation (32)

$$A = \lambda \left(\frac{\lambda_1^2 + \omega^2}{\omega^2 \lambda_1^2 + \left[\left(\frac{\lambda_1}{2} \right)^2 + \Omega^2 - \omega^2 \right]^2} \right)^{1/2} \quad (35)$$

At this point we remember the amplitude assumed for the room oscillation was one degree. We see that if the damping constant of the bath, λ , is small, we get a decrease in the undamped sinusoid induced into the bath by the room, free of charge so to speak. Also we get a decrease in amplitude for any room temperature angular velocity ω , for a large

~~SECRET~~

thermometer damping constant, λ_1 , and for a large controller sensitivity, σ : an achievement we pay for in increased cost of control equipment. Also Equations (35) and (32) tell us that we can get better control over the long period changes induced in the bath by making the thermometer as fast as practicable and increasing the sensitivity of the controller to the point of critical damping.

Now let us discuss what has been done experimentally. About three months ago the "proportional" controller was put into operation on bath number one. It was found to improve the operation of the bath. For intervals of a week the bath temperature has been so steady that a change in temperature could not be detected by observing a Beckman thermometer even though the room temperature had undergone considerable variation in this time. During this time the controller showed considerable oscillation from zero to maximum corrective power input with a period approximating two minutes.

From the analysis we see that the lag and sensitivity of the controller must have been such that the roots of Equation (31) were complex conjugates and the controller was inducing a sinusoid into the bath. Also the lag and sensitivity were such that the amplitude of the sinusoid (even of very long period) caused by the room were controlled to the point where they were undetectable by a Beckman thermometer. The oscillation of the control was deemed undesirable. At this point we may check the reasonableness of the analysis by calculating the period of the induced sinusoid from the data on the bath and controller.

~~SECRET~~

The period of the controller-induced oscillation is given by

$$T = \frac{2\pi}{\sqrt{\lambda}} = \frac{2\pi}{\left[\frac{0 \lambda_1}{C} - \frac{\lambda_1^2}{4} \right]^{1/2}} \quad (36)$$

where

$$C = 2 \times 10^5 \text{ calories degree}^{-1}$$

$$\lambda_1 = 0.015 \text{ seconds}^{-1} \text{ (Table I)}$$

$$\sigma = 3.6 \times 10^4 \text{ calories degree}^{-1}$$

When substitutions are made and calculation performed, we get for the period of the induced sinusoid

$$T = 123 \text{ seconds} \quad (37)$$

The close agreement with the observed value of approximately two minutes is probably fortuitous, however, our faith in the analysis is strengthened, and we will proceed to calculate the amplitude of the response of the bath to a room temperature oscillation of one degree amplitude and a period of eight hours and compare it with calculated amplitudes for the bath without control and with a control that has zero lag.

Substituting the following values in Equation (35) and making the calculation,

$$\lambda = 2 \times 10^{-5} \text{ seconds}^{-1} \text{ (Table I)}$$

$$\lambda_1 = 15 \times 10^{-3} \text{ seconds}^{-1} \text{ (Table I)}$$

$$\omega = 21 \times 10^{-5} \text{ radians second}^{-1} \text{ [Equation (16)]} \quad (38)$$

$$\sigma = 3.6 \times 10^4 \text{ calories degree}^{-1} \text{ second}^{-1}$$

$$C = 2 \times 10^5 \text{ calories degree}^{-1}$$

we get

$$A = 0.0001 \text{ degrees} \quad (39)$$

~~SECRET~~

This value agrees with the control that has been observed on bath number one since the "proportional" control was installed. We are planning to build a much faster and more sensitive bridge thermometer to replace the Beckman thermometer, which will not detect a temperature change of 0.001 degree.

Here we see that a great measure of control is achieved even though the controller induces a comparatively high frequency oscillation in the bath. We feel that the calorimeters themselves will damp out and not be affected unduly by the induced oscillation. The induced oscillation is objectionable since it obscures the control point. From the equation for the angular velocity, Ω , Equation (32) it is seen that Ω is zero if the sensitivity, σ , of the controller is reduced to the point where

$$\frac{\sigma}{c} = \frac{\lambda_1}{4} \text{ for } \Omega = 0 \quad (40)$$

In this case Equation (35) gives the amplitude of the sinusoid induced by the room as

$$A = \lambda \left(\frac{\lambda_1^2 + \omega^2}{\left[\frac{\lambda^2}{4} - \omega^2 \right]^2 + \lambda_1^2 \omega^2} \right)^{1/2} \quad (41)$$

Substituting in values of λ , λ_1 and ω and calculating A, we find

$$A = 0.005 \text{ degrees} \quad (42)$$

In other words we would get 50 times worse control in eliminating the controller-induced oscillation by reducing the sensitivity, σ , of the

~~SECRET~~

controller to satisfy Equation (40). This reduction could be achieved by reducing the bridge current in the bridge thermometer thus reducing σ by reducing σ_1 , the sensitivity of the bridge thermometer.

This analysis is correct since Equation (41) has Equation (14) as a limit as λ_1 is increased, thus indicating, as it should, that a controller with lag has as its limit an instantaneous controller when the damping constant, λ_1 , of the controller is increased without limit. This fact is shown in Appendix B.

Comparing the amplitudes calculated from Equations (35) and (14) shows that operating the bath with a controller induced oscillation is as good as instantaneous control. Hence it would be a distinct advantage to devise some method of eliminating the controller induced oscillation other than reducing the sensitivity of the controller.

From the study by our group of the analysis of control by means of the Laplace transform, we considered means of adding an integrating or differentiating control component to the present "proportional" control in order to increase stability of the control without reducing controller sensitivity.

T. J. Beasley and K. C. Jordan suggested the following method of obtaining a measure of differentiating control. In essence, as shown in Figure 5, the method is to feed a portion of the corrective heater current into a milliammeter causing it to deflect at a rate proportional to the rate of change of the heater current. The pointer of this milliammeter is tied mechanically to the pointer of a second milliammeter and thus drives it at a rate proportional to the rate of change of the heater current.

~~SECRET~~

The voltage generated in the second milliammeter is fed into the bridge-thermometer galvanometer circuit of the controller and in opposite phase to the thermometer voltage. This component of the apparatus supplies negative rate feedback to the controller. This component has been installed on the controller, and the objectionable oscillation of the controller has been removed. We have not had sufficient time to observe the effect of this method on the bath temperature. We are also planning to study, by means of this type of analysis, the effect of negative rate feed back on the controller and the bath temperature.

Appendix A

The problem here is to show analytically that as the damping constant, λ_1 , is increased without limit

$$q_c = -\sigma \lambda_1 e^{-\lambda_1 t} \int_0^t e^{\lambda_1 t} \delta(t) dt \quad (23)$$

approaches

$$q_c = -\sigma \delta(t) \quad (11)$$

as a limit.

This can be shown by applying partial integration repeatedly to Equation (23).

The result is

$$q_c = -\sigma \delta(t) + \sigma \left[\frac{\delta'(t)}{\lambda_1} - \frac{\delta''(t)}{\lambda_1^2} + \frac{\delta'''(t)}{\lambda_1^3} - \dots \right]$$

Now if $\delta(t)$ and all of its derivatives exist, are continuous and are bounded, then as λ_1 is increased without limit, we get the required result.

Appendix B

The problem is to show that as λ_1 is increased without limit

$$A = \lambda \left(\frac{\lambda_1^2 + \omega^2}{\left[\frac{\sigma \lambda_1}{c} - \omega^2 \right]^2 + \lambda_1^2 \omega^2} \right)^{1/2} \quad (31)$$

reduces to

$$A = \frac{K}{\sqrt{c^2 \omega^2 + \sigma^2}} \quad (14)$$

This can be accomplished by dividing the numerator and denominator of the fraction in Equation (31) by λ_1^2 . The following is obtained

$$A = \frac{K}{c} \left(\frac{1 + \frac{\omega^2}{\lambda_1^2}}{\left[\frac{\sigma}{c} - \frac{\omega^2}{\lambda_1} \right]^2 + \omega^2} \right)^{1/2}$$

which, as λ_1 increases without limit, reduces to Equation (14).

REFERENCES

1. Jordan, K. C. Miscellaneousimeters 52 and 53, MIM-502.
2. Fairchild, C. Temperature Control, Temperature its Measurement and Control in Science and Industry
3. Sligh, T. S. Jr., Some Characteristics of the Group Thermo-regulator, J. Am. Chem. Soc. 42, 60, (1920).
4. Jelonek, E. Analysis of a Thermostat with Contact Thermo-regulator, Proc. Cambridge Phil. Soc. 42, (I), 62-72, (1946).

~~SECRET~~

~~SECRET~~

MIM-502

5. Carslaw, H. S. and Jaeger, J. C., Conduction of Heat in Solids, pp. 320-325.

6. Carslaw, H. S. and Jaeger, J. C., Conduction of Heat in Solids, p. 16.

7. Gardner, M. F. and Barnes, J. L., Transients in Linear Systems, pp. 191-201.

~~SECRET~~

Table IDAMPING CHARACTERISTICS OF THE BATH AND CONTROLLER COMPONENTS

<u>Component</u>	<u>Damping Constant-λ</u>	<u>Remarks</u>
Bath	2×10^{-5} seconds ⁻¹	Measured
Bridge Thermometer	15×10^{-3} seconds ⁻¹	Measured
Galvanometer	26×10^{-2} seconds ⁻¹	Calculated from the period
Heater	2 seconds	Measured

~~SECRET~~

UNCLASSIFIED

Figure 1

MLM-502

BRIDGE THERMOMETER

500231

UNCLASSIFIED

500229

PROPORTIONAL CONTROLLER - FRONT VIEW

500230

UNCLASSIFIED

Figure 3

MLM-502

PROPORTIONAL CONTROLLER - REAR VIEW

UNCLASSIFIED

-139-

BLOCK DIAGRAM OF CALORIMETER BATH AND
PROPORTIONAL TEMPERATURE CONTROL.

END OF DOCUMENT

DATE FILMED: 03-20-80

FILMED AT 24X