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MOUND LABORATORY

Operated By

MONSANTO CHEMICAL COMPANY

MIAMISBURG, OHIO

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REPORT
FOR

GENERAL RESEARCH

DECEMBER 26, 1950 TO APRIL 16, 1951

(Supporting Research Volume)

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Date May 21, 1951

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(Supporting Research Volume)

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SUMMARY

This volume presents the quarterly results of that portion of the research and development which is directed towards establishing new techniques and instruments and towards improving present methods for determining levels of radioactivity accurately and safely

The locally developed amplifier of high gain and of wide dynamic range has been shown to provide plateaus suitable for the simultaneous counting of alpha and beta particles. Commercially available amplifiers have failed to exhibit suitable plateaus when used under the same conditions. This locally developed amplifier in conjunction with the Nuclear Instruments PC 1 chamber has made it possible to estimate beta energies down to 0.02 million electron volt which is to be compared to a former lower limit of 0.1 million electron volt (p. 6)

A fast neutron survey meter must be simple, lightweight, portable, and efficient and must provide discrimination against gamma rays. The first step which is the design of a suitable detecting device has been successfully completed as good efficiency and good discrimination against gamma rays have been achieved. The necessary electronic circuits must now be engineered to provide the requisite simplicity, light weight, and portability (p. 24)

A stable, energy independent gamma monitor has been achieved through the use of a well regulated power supply and a highly degenerative amplifier. The monitor maintains its accuracy within ± 2 per cent when calibrated once per day. The direct coupled amplifier is capable of measuring currents of the order of 1×10^{-13} ampere (p. 28)

A continuously operating air monitor for health protection must be capable of providing rapid response and good counting precision when operating at low-counting rates. These requirements have been met by devising a switching device which permits the accumulation of charge on a sequence of condensers for arbitrary time intervals. Discharge of the sequence of condensers operates an alarm if the rate of accumulation of charge has been excessive (p. 32)

The Nuclear Instruments PC 1 chamber has been under investigation for use in the measurement of the energies of soft beta rays by absorption. RaD and RaE mixtures have been used as "knowns". The results are in substantial agreement with the literature values (p. 34)

Several local problems in the use of the Nuclear Instruments PC 1 chamber are under investigation. The beta rays associated with the polonium alphas are being investigated by studying the behavior of the Nuclear Instrument PC 1 chamber when filled with argon or methane and exposed to natural or artificial polonium. Sulfur 35 or phosphorous 32 Polonium samples are being calorimetered to obtain an alpha plus beta determination and the samples are being counted to get an independent alpha determination so that a comparison can be made to check for the presence of beta activity. The effects of secondary electrons from absorbed radiation is also under investigation (p. 36)

Routine operation of the quartz-fiber microbalance has been improved by the development of interchangeable panholders. The quality of balance parts has been improved by providing better regulation of the pressure in the gas lines for the microtorches (p. 37)

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The mass spectrograph is being modified so that it may operate either as a spectrograph or spectrometer. Operation as a spectrometer is sometimes desirable since isotopic abundances can be determined more precisely with a spectrometer. It will soon be necessary to measure the isotopic purity of polonium 208 samples (p 51)

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Problem Title Counting Systems of Wide Dynamic Range
Report By W H Baker
Work Done By W H Baker

INTRODUCTION

A non-linear high-gain wide-dynamic-range amplifier has been designed and constructed for evaluation as a universal unit for the more complex problems encountered in neutron counting and waste disposal sample counting. It is hoped that this amplifier will extend the scope of counting techniques now limited by the available commercial units. This amplifier greatly improves the operating characteristics of a boron-wall ionization chamber when detecting neutrons. Waste disposal sample counting requires the detection of very weak beta pulses in the presence of strong alpha pulses and the analysis of unknown weak-beta emitters. This can be done with a windowless proportional ionization chamber and this amplifier. With this system it has been possible to make absorption curves of weak-beta emitters that have only one-fourth the energy required for Geiger-Muller tubes with the thinnest available windows.

The entire amplifier consists of a main amplifier mounted on the scaler chassis and a head amplifier mounted on the ionization chamber which may be at a location remote from the operator. This design uses techniques whereby the same subchassis is used as a base for construction of the main amplifier or the head amplifier. Because of the head amplifier's small size it is readily adapted to neutron counting or waste disposal counting. The overall design is such that spare units could be utilized to ease the maintenance problems during heavy work loads, or speed up service operations when breaking in inexperienced personnel in the electronic maintenance group.

In the detailed report the importance of the head amplifier input network design is discussed. Pulse photographs taken at different points in the amplifier are shown, and the amplifier plateaus are compared with those obtained with a commercial amplifier. Absorption measurements of a very weak beta source are also illustrated.

DETAILED REPORT

The amplifier is built around the subchassis (4 in by 2 in by 1/4 in) shown in Figure 1. The completed main amplifier shown in Figure 2 consists of three direct-coupled inverse-feedback pairs in cascade. Direct coupled pairs (Figure 3) were used because of their simplicity and extreme limiting ability and because they do not introduce harmonics into the pulse. Under the conditions of a medium gain of 1,000 in the main amplifier the direct coupled pairs seem to have sufficient pulse-handling capacity to eliminate the development of spurious pulses.

The main amplifier has a banana jack at the input of the amplifier and at the output of each stage so that the gain may be varied in steps of ten by plugging the trigger pair into the proper jack. Vernier gain adjustment is accomplished by control of the overall feedback and control of the trigger-pair sensitivity.

The head amplifier (Figure 4) is condenser coupled as shown in Figure 5. By grounding the cathode of the second stage and increasing the grid bias to prevent over-

loading, it seems possible to obtain the necessary increase in dynamic range required by the increased gain of the head amplifier. Remote mounting of the head amplifier reduces capacitive loading of the ionization chamber and thereby minimizing pulse integration and attenuation but it demands one common ground between the head amplifier and main amplifier. A common ground is accomplished through the use of double shielding, two-way receptacles and common grounds within the amplifiers. The head amplifier case was constructed with copper-plated steel for optimum electrostatic and magnetic shielding, and the outside of the case was chrome plated to minimize corrosion.

It has been found that the configuration of the input network of the head amplifier is very important. The load resistor of the ionization chamber has been set at 1,000 ohms because of the sharp pulse developed across it (0.1-microsecond rise time or better 0.1-microsecond duration at 0.60 maximum amplitude). The effect of the grid resistor and coupling condenser of the input stage should be optimized, so that the pulse at the output of the amplifier has a minimum duration without developing objectionable overshoot. The value of this grid resistor will have to be different with different overall amplifier gains. This is because the normally insignificant trailing decay voltage assumes sizable proportions when amplified 10,000 times in a limited amplifier.

Because of the extremely fast rise time of the pulse, it is possible further to improve the dynamic range and the resolving time of the amplifier by sharply differentiating the pulse before it is applied to the trigger pair. This differentiation and the fast rise time of the pulse increase the sensitivity of the system from 80 microvolts to approximately 50 microvolts. Since spurious counts are not detectable with a maximum usable input pulse of approximately 0.5 volt amplitude it appears that this amplifier has a dynamic range of at least 10,000.

The overall amplifier gain-bandwidth curve is shown in Figure 6. The bandwidth is 3 decibels down at 3 megacycles for an 80-decibel amplifier whose sensitivity approximates 80 microvolts. The main amplifier limits at 3.75 volts, while the maximum usable pulse from the head amplifier approximates 3.5 volts. The amplifier gain changes 2.5 per cent for a 1 per cent change in line voltage.

Figure 7, A, B, and C and Figure 8A show the pulse at the output of the head amplifier, stages 1, 2, and 3, respectively of the main amplifier, and Figure 8B shows the pulse at the output of the differentiating circuit. Figure 9, A and B show the noise output of the amplifier and the noise at the output of the differentiating circuit.

One of the best criterion of a counting system operation is a plateau curve of counts versus voltage. Figure 10 shows the plateau obtained with an alpha sample and a commercial amplifier. It is impossible to obtain an alpha plus beta plateau with this system. Figure 11 shows the alpha and alpha plus beta plateau obtained with the described amplifier and a standard scaler.

Up to the present time, routine beta absorption studies have been limited to energies of 0.1 million electron volt² because of the thickness of mica windows in Geiger-Muller tubes. With a windowless proportional chamber and a high-gain, wide-dynamic-range amplifier it has been possible to measure weak Ra-D beta energies of 0.025 million electron volt. Figures 12 through 16 show the graphs used in determining this energy level with feather analyzers as described by Glendenin.² The measured energy by this method was 0.022 million electron volt. A similar amplifier has been in use for 10 months and has proven very reliable.

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

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REFERENCES

- 1 Elmore and Sands Electronics McGraw Hill, New York, p 159 (1949)
- 2 Glendenin L Nucleonics 2. pp 17-27 (January 1948)

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

FIGURE 1

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SUBCHASSIS

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 2

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A. BOTTOM VIEW

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B. TOP VIEW
MAIN AMPLIFIER

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FIGURE 3

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FIGURE 4

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C. BOTTOM VIEW

A. HEAD AMPLIFIER
MOUNTED ON
WINDOWLESS, GAS-
FLOW, HEMISPHERICAL,
PROPORTIONAL, ION-
IZATION CHAMBER

B. TOP VIEW

HEAD AMPLIFIER

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FIGURE 5

MLM-561

A. OUTPUT OF 1ST STAGE (HEAD AMPLIFIER)
1 MICROSECOND MARKER.

B. OUT OF 2ND STAGE. 1 MICROSECOND MARKER

C. OUTPUT OF
3RD STAGE.
1 MICROSECOND
MARKER

PULSE OUTPUT AT VARIOUS STAGES OF GAIN-OF-10,000. NONLINEAR, WIDE-DYNAMIC RANGE
AMPLIFIER OBTAINED WITH AN ALPHA SAMPLE AND MAXIMUM USABLE GAS AMPLIFICATION.

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A. OUTPUT OF 4TH STAGE, 100 MICROSECOND MARKER

B. OUTPUT OF DIFFERENTIATING NETWORK (INPUT TO TRIGGER PAIR), 1 MICROSECOND MARKER.

PULSE OUTPUT AT OUTPUT STAGE OF GAIN-OF-10,000, NONLINEAR, WIDE-DYNAMIC RANGE AMPLIFIER BEFORE AND AFTER DIFFERENTIATION OBTAINED WITH AN ALPHA SAMPLE AND MAXIMUM USABLE GAS AMPLIFICATION.

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A. NOISE OUTPUT OF AMPLIFIER

B. NOISE OUTPUT OF DIFFERENTIATING NETWORK
(INPUT OF TRIGGER PAIR).

RELATIVE NOISE OUTPUT OF AMPLIFIER BEFORE AND AFTER PASSING THROUGH DIFFERENTIATING NETWORK

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FIGURE 12

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OBSERVED BETA ABSORPTION CURVE RA D→E→F

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FIGURE 15

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DETERMINATION OF BETA RANGE FROM "FEATHER" PLOT (0.155 MEV C-14)

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Project Title: Fast Neutron Survey Meter

Report By: J. F. Combs

Work Order No.: J. F. Combs

INTRODUCTION

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

The design of the detecting device was of the greatest concern initially. This detector 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 any desired value. The counter is fully described in a previous report.¹

The final model of several counters built and tested resulted in a fast neutron counting efficiency of 0.25 per cent at a sensitivity of 5 millivolts when 2,350 volts were applied to its center electrode. Gamma radiation was not detected at the same sensitivity until the counter voltage was raised to 2,400 volts.

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

DETAILED REPORT

Two independent electronic circuits are required to operate the survey meter. Since the instrument is to be portable both circuits must be battery operated and both should be compact and lightweight. To provide a means of indicating the number of fast neutrons detected by the proportional counter, a rate meter circuit is necessary. To provide the 2,400 volts necessary to operate the counter, a high-voltage power supply is required.

A rate-meter circuit (Figure 1) was built which employs a single-stage pulse amplifier with an approximate gain of 10. The output of the amplifier fires a thyratron tube which has a pulse-integrating circuit and a 20-microampere meter in series with its cathode. The variable 500 ohm resistance connected in parallel with the microammeter and 1,000 ohm series resistor allows for calibration of the meter. The circuit provides three ranges of sensitivity for full scale deflection of the meter by varying the time constant in the plate circuit of the type RK-61 thyratron. When switched to its most sensitive range, position C, a fast neutron flux of 100 neutrons per squared centimeter per second produced full scale deflection with 2,500 volts applied to the counter. Gamma radiation caused no deflection under the same conditions. To discriminate against gamma radiation, the gain of the amplifier circuit was made variable. Thus, with a fixed high voltage, this gain is adjusted so that the maximum-amplitude gamma pulses from the counter will cause no deflection of the rate meter. This variation in amplifier gain is accomplished by using a potentiometer as a plate load, connected so that all or part of the voltage developed across it can be coupled to the grid of the thyratron tube. Sub-miniature tubes are used in this circuit, and the number of components have been reduced to a minimum. The circuit is similar to the rate meter circuit reported by Hurst.²

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The high-voltage power supply required to operate the counter must be regulated and must produce a voltage of approximately 2,500 volts. The use of eight or nine 300-volt dry cell batteries to supply this voltage results in a bulky and heavy instrument and the batteries are costly to replace. It was therefore decided to design a miniature pulsed-type, power supply. Of the several types of circuits available the neon bulb-relaxation oscillator type of pulsed power supply was chosen because of its reported high efficiency³ and because it requires the smallest number of components. Several circuits were built and tested utilizing the relaxation oscillator and straight rectifier or voltage doubler circuits. The most satisfactory results were obtained with the circuit shown in Figure 2. The choke used in the plate circuit of the type 5678 tube is a United Transformer type 0-5. It is very small and weighs approximately one ounce. The wave shape of the induced high voltage in the type 0-5 choke was such that the conventional type of voltage-doubling circuit did not operate satisfactorily. The negative portion of the high voltage pulse was much smaller than the positive part. The circuit shown in Figure 2 makes use of only the positive portion of the pulse. While this circuit does not double the voltage, it increases the voltage output by about 33 per cent over that achieved with a straight rectifier circuit. The high-voltage output is regulated by a series of corona regulator tubes. The type 5841 tubes regulate at 900 volts each and the type QF-771 tube regulates at 700 volts giving a total regulated voltage of 2,500 volts. Sub-miniature tubes are used throughout the circuit and all but the type 5678 tube are of the cold cathode type. The power supply operates at an overall efficiency of 10 per cent, a ripple of approximately 10 millivolts appears at its output, and at 5 microamperes 2,500 volts is available from the regulated output.

Knowing the battery current drains required to operate both the rate-meter circuit and the high voltage power supply circuit the number of batteries required for 150 hours of service was determined. The filament voltage for the type 5678 tubes and the type RK-61 tube will be supplied by Mallory type RM-12 mercury cells. The 135-volt plate supply for both the relaxation oscillator tube and the amplifier tube will be supplied by the same batteries. The 67.5-volt plate supply for the thyratron will be supplied by a separate battery. This voltage must remain nearly constant over any one calibration period if the calibration is to remain accurate. The 4.5-volt negative bias for the thyratron tube will be supplied by three Mallory Type 1 RX mercury cells connected in series.

The design of a prototype instrument was initiated. Several parts were under construction when work on this problem was temporarily discontinued.

REFERENCES

- 1 Combs, J. F. Quart. Rpt. Gen. Res. MLM-529, p. 12, Jan. 29, 1951.
- 2 Hurst, G. S. Portable Fast Neutron Survey Meter. ORNL - 485.
- 3 Thomas, A. Electronics, pp. 100-103, Dec. 1948.

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CIRCUIT DIAGRAM—AMPLIFIER AND RATE METER.

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FIGURE 1

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Title Stable, Energy Independent Gamma Monitor
Author A. J. Rogers
Work done by A. J. Rogers

INTRODUCTION

The development of a gamma monitor which is energy independent over wide ranges was required for health protection purposes in areas having a high gamma flux. The monitor was to be operated on alternating current entirely. It was to be capable of quantitatively indicating the gamma-radiation intensity in the ranges of 100 milliroentgens per hour and 500 milliroentgens per hour full scale. Means for the remote operation of the detecting device were to be provided. An instrument incorporating these features was designed and a model is being constructed.

DETAILED REPORT

The completed gamma monitor includes the following major components: a high pressure ionization chamber, a direct-coupled amplifier and associated metering circuit and necessary regulated and stabilized power supplies.

The cylindrical high pressure ionization chamber has a volume of 800 cubic centimeters. It is filled with argon to a gauge pressure of 150 pounds per square inch. Theory indicates that this chamber will reach ionization current saturation when the electrode potential difference is about 200 volts. It is at this potential that electron collection occurs before ion recombination effects can diminish the ionization current. Experimental evidence validates the theory.

With the correct electrode polarity, the flow of ions under action of the electrostatic field existing between electrodes produces an electron current at the center electrode of the ionization chamber. This current flows into an integrating network and is utilized to change the bias of the input stage of the direct coupled amplifier (See Figure 1). This amplifier is essentially a unity voltage gain or current amplifier. The electron current produced in the chamber for a gamma flux of 100 milliroentgens per hour is about 1.5×10^{-9} amperes. With an input resistor of 4×10^9 ohms, this gamma flux results in a potential change of approximately 0.5 volt at the input to the amplifier. Since the amplifier has been designed for unity voltage gain, the equivalent amplifier output is also 0.5 volt. The amplifier has a voltage gain without feedback of 1,200 and a current gain with feedback of about 420,000. The incremental amplifier output-voltage change is applied to a differential cathode follower with an indicating microammeter connected between the cathodes (See Figure 1). Selection of resistors in series with the microammeter permit scaling-factor changes of from 100 milliroentgens per hour to 500 milliroentgens per hour. For a more complete discussion of the metering circuit, see Section II, Chapter 11 of the text "Vacuum Tube Amplifiers" by Valley and Wallman (McGraw-Hill). A portion of the amplifier (V_1 and V_2) is mounted within the base of the ionization chamber. This permits the use of a long connecting cable so that the chamber can be at an appreciable distance from the rest of the equipment. A unique feature of the amplifier is the incorporation of a Victoreen VX-10 vacuum switch for zeroing the instrument when the chamber is in a region of high gamma flux.

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The filament potential for the switch is supplied by a Mallory type RM 4 mercury cell. This potential is applied by closing a switch on the power supply chassis. Calibration of the instrument may then be performed at the power-supply chassis also. The direct coupled amplifier will maintain its calibration to within ± 2 per cent over a 24 hour period.

Two separate power supplies are utilized to provide all necessary electrode potentials. One power supply is cascaded upon the other. The more negative power supply provides a stable ionizing potential for the ionization chamber. The more positive power supply provides all electrode and filament voltages for the amplifier and metering circuit. These power supplies are identical in design (Figure 2) and were suggested by an article by Peter Sulzer appearing in the December 1950 issue of "Electronics," entitled "Stable Electronic Power Supply." The power supplies are conventional vacuum tube direct current amplifier regulated power supplies with increased stability provided for through use of a heater voltage compensating network (V8) and application of a regeneration network (R). Tests have indicated that a ± 10 per cent change in line voltage results in about a ± 0.1 per cent change in output voltage. The drift in output for a 24 hour period is less than 0.1 per cent.

CONCLUSIONS

Extremely stable operation of the gamma monitor is provided for through well regulated power supplies and a highly degenerative direct current amplifier.

The monitor will maintain its accuracy within ± 2 per cent if calibrated once a day.

The direct coupled amplifier and its associated power supply can be useful in many other applications where it is desired to measure currents of the order of 10^{-13} amperes.

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FIGURE 2

MLM-561

Problem Title **Mechanical Integrator for Improving Counting Statistics**
Report By **A. J. Rogers**
Work Done By **A. J. Rogers**

INTRODUCTION

There is a need in health physics applications for a monitoring device that will combine rapid response with good counting reliability at the low counting rates normally found in health physics counting.

A mechanical integrating device which fulfills these requirements was developed for use with a continuous air monitor for health protection where minimum time lag between the occurrence and the detection of changes in the contamination level was required, together with good statistical counting accuracy.

DETAILED REPORT

Since the response of the mechanical integrator is to be dependent on the input pulse rate, it is necessary to convert the input pulses, which are random in time, into voltage pulses that are uniform in shape, amplitude and duration. This is accomplished by a pulse-shaping network which precedes the integrator.

The charging network of the mechanical integrator utilizes these uniform voltage pulses to produce in a capacitor a stored charge that is proportional to the input pulse rate over a definite time interval. This capacitor is then removed from the charging network and a second capacitor inserted. The charge applied to this second capacitor is dependent on the input pulse rate also, and is evaluated over the same definite time interval. This procedure is continued until N capacitors in turn have been charged to energy levels which are in each case proportional to the input pulse rate. Cascading these N capacitors in series provides a total charge that is proportional to the total number of pulses observed by the detecting device over a time interval of NT seconds. T in this case refers to the time interval over which each of the N capacitors has been affiliated with the charging network. It is this total charge that is available for actuation of an alarm circuit. After a short time delay to permit the alarm circuit to evaluate the total stored charge, the first capacitor is discharged and returned to the charging network. In turn the N capacitors are discharged and recharged with the alarm circuit, evaluating the total stored charge at all times. This last feature permits the continuous sampling of a total charge that is equivalent to the preceding $(N-1 + K)$ timing intervals, where $0 \leq K \leq 1$.

A three capacitor mechanical integrator is shown schematically in Figure 1. A series of motor-driven cams mechanically operate switches that provide for the proper sequencing of capacitor charge and discharge cycles. The input to the alarm circuit is permanently connected across the seriesed capacitors to insure continuous sampling of the input pulse rate.

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FIGURE 1

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Problem Title Adsorption Studies on a Nuclear Instruments PC-1 (Bradley) Counter.
Report By M. L. Curtis
Work Done By - M. L. Curtis, and G. D. Stewart

INTRODUCTION

The Nuclear Instruments (Bradley) adsorption chamber has made it possible to contribute to the identification of radioactive materials by determining energies of alpha particles, and of beta particles which are too weak or too low in specific activity to be measured on window type counters. Values which check the literature have been secured on polonium alphas, and on betas which exceed 0.17 million electron volt as reported previously.¹ It remained to test the counter with known beta energies below this figure, and to continue to use it in identification of specific samples presented for measurement by chemists.

DETAILED REPORT

A sample of RaD was prepared for absorption measurements. It contained approximately 16 per cent RaE when the measurements were started, and because of the rapid growth of RaE each measurement had to be individually corrected for growth. Counting rate is plotted against absorber thickness with this sample in Figure 1. Since there was RaE present the observed curve shows two components, and the contribution of the harder component had to be subtracted from the observed curve to determine the range of RaD. A measured energy of 0.04 million electron volt may be compared to a literature value of 0.029 million electron volt. It is believed that back-scattering from the RaE, and secondary electrons from Bremsstrahlung were able to penetrate the very thin absorbers required. This would distort the curve and lead to a high value.

A sample procured from Oak Ridge believed to be nickel-63 (0.05 million electron volt beta no gamma) was found to have a 0.32 million electron volt beta plus gamma. These are properties of cobalt-60.

A sample from the "T" Building, bismuth supernatant solution, was submitted for absorption studies. Maximum energies of 0.14 million electron volt and 0.8 million electron volt plus a stronger beta or gamma were obtained. The material was treated to remove sulfur 35, and a sample of the residue was submitted for absorption studies. A maximum energy of 1.70 million electron volts plus stronger beta or gamma was found. The sample was followed for 40 days for half-life. Two short half-life components were observed (possibly 5 days and 14 days) and a longer half-life component was found which will be followed further.

REFERENCE

- 1 Curtis, M. L., Absorption Studies on a Bradley Counter, Quart. Rpt. Gen. Res MLM-529 p. 16, Jan. 29, 1951.

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Document Title Problems in Beta Counting

Author M. L. Curtis

Revising By M. L. Curtis

INTRODUCTION

Two of the problems involved in counting beta radiation in the type of samples submitted for measurement at Mound are

- 1 To distinguish beta radiation from secondary radiation coming from photons
- 2 To count samples very low in specific activity with a high and variable cosmic-ray background.

Some preliminary work has been done on these problems

DETAILED REPORT

Since methane gas is less efficient for counting gamma or X rays than argon gas measurements have been made in a Nuclear Instrument (Bradley) PC-1 counter with each of these gases. First indications are that there is less "beta" in polonium with methane gas but results are not yet conclusive. Samples of pure polonium natural polonium impure polonium sulfur-35 and phosphorus 32 mounted on both glass and platinum have been prepared and will be measured periodically over a period of time with both gases. This will indicate whether any of the apparent beta is caused by any action of the sample material on the sample mount.

Samples of polonium have been measured on the Logac-S. Five of these samples will be measured simultaneously in the calorimeter. Since the calorimeter will measure beta radiation and the Logac S will not this should give a further indication of any beta impurity in polonium.

The problem of counting low-level samples with a high background requires the means of reducing the background for its solution. A Bradley counter was set up in Room 37 of the "T" Building and it was found to have a beta background of 30 counts per minute compared to an average of approximately 60 counts per minute cosmic-ray background in the E Building.

<i>by the title</i>	The Standard Torsion Equal-Arm Balance
<i>known by</i>	M I Gray R G Olt and J H Wright
<i>known by</i>	M I Gray C L Lesher L A Jewett R G Olt E W Stautzenbach and J H Wright

INTRODUCTION

The quartz-fiber microbalance in the form used in microassays consists of a completely fused quartz-fiber beam system of a torsion-fiber equal-arm load-balancing type in a case containing the directly associated mechanical and optical systems. This unit is further enclosed in a vacuum tight metal housing fitted with the mechanical and optical controls and accessories essential for placement, weighing, and removal of sample bearing foils while maintaining atmospheric thermal and radiation shielding. Since the carriers for the foils also provide atmospheric and radiation shielding, it is possible to operate the balance in low risk areas despite the high level of activity being handled.

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

Incorporation of the mechanical electrical and optical refinements described in previous reports - 2 improved the operating characteristics and extended the periods of balance availability to about a month at which time cleaning of the pan well area would be required. Dessication of the balance interior further reduced the need for cleaning of the pan well areas resulting in further extension of the periods of trouble free balance operation to more than six months.³

A major factor in the increase of balance availability has been the development of interchangeable panholders which eliminate the need for removal of a balance to a high risk area for panholder replacement and subsequent readjustment of pan arrests. The improvements in panholder mass distribution and size control have contributed also to the speed and certainty of the remote loading and weighing of fuels.

Working of quartz fibers has been adversely affected by microtorch flame changes and flashbacks resulting from oxygen and fuel gas supply pressure variation. Measurement of gas line pressures for a 2.4 to 1 range of flow rates showed pressures varying from 11 inches to 9 inches of water in the oxygen line and from 21 inches to 12 inches of water in the propane line when using two stage cylinder gas regulators.

Better control of pressure has been obtained by addition of the third stage component of the Airco Style 1608 three stage nitrogen regulator for oil filled transformers. This component which has not been sold previously as a separate unit shows a pressure regulation within 0.6 inch water at 14 inches over a flow rate range of 0.12 to 1.40 cubic feet per hour.

DETAILED REPORT

Quartz Fiber Panholders

Early attempts to make usable quartz-fiber panholders according to Argonne dimensions and procedures⁴ proved uniformly unsuccessful because of incompatible poise and dimension adjustments. A partial explanation for the lack of success might be that while the Argonne type panholders adequately support spherical pans, the Mound foils which are of a flat flag on staff shape are more difficult to place securely on a ring support particularly with remote loading techniques.

Redesign of the panholder was initially directed towards achieving dimensions of panholders and construction jigs for producing panholders that would release from an arrest without swinging or tilting in either the loaded or unloaded condition. Additional requirements leading towards interchangeability were added as experience was gained in balance operation and maintenance and in quartz fiber working. Added requirements included limitation on overall size so that panholders could be placed on the hangdowns by insertion through the 9 millimeter balance-loading orifice and close tolerances on overall height, ring diameter, ring flatness, and ring parallelism with the feet. The dimensions in Figure 1 are those specified for (A) the Argonne panholder, (B) the 1948 design, and (C) the current design. Figure 2 is a photograph of a current panholder.

The correct location of the microfoil on upper and lower limit panholder rings for minimum tilt on arrest release is shown in Figure 3.

Figure 4 shows panholder jig A which is used for fusing the cross leg to the principal member and for making the two bends that position the ring. The remaining bending operations are performed on panholder jig B. Figure 5.

The current method of constructing and inspecting fused quartz fiber panholders is as follows:

Construction

An 8 centimeter length of 175 micron fiber is cleaned with chromic acid, water, and acetone before application of the working flame. With the fiber held in a near vertical position, a sharp 45° bend is made about 2 millimeters from the free end (Figure 6A) with a soft flame. With the free end of the fiber up and inclined toward the eyes in a vertical plane, the flame is applied to the fiber at the initial bend and formation of the 1 ± 0.2 millimeter circle started (Figure 6B). The flame axis is kept in the plane of the circle in order to avoid side distortion. This bending operation is continued until the initial bend lies over the stem (Figure 6C) and the diameter is within limits specified. With the stem held vertical with the circle down, the junction point is heated so that the circle center falls in line with the stem axis without fusing (Figure 6D).

The stem and circle are placed in panholder jig A for fusion of the circle junction. Both the circle and the stem are held under fibers as indicated in Figure 6E. Use of the minimum heat necessary for the completion of the fusion reduces distortion and necking at the joint. After completion of the joint fusion, the fiber is moved axially to place the circle in the hole in the jig A. In this position (Figure 6F) the circle is inspected for shape and worked with the flame to remove irregularities and misalignment with the stem.

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The inspected circle and stem is repositioned and clamped in jig A so that the circle is centered on the engraved cross lines (Figure 7A). A 6 centimeter length of 175 micron fiber is clamped in the groove which is at 90° to the axis of the stem and 11 millimeters from the center of the circle.

After cleaning the fiber intersection with chromic acid, distilled water, and acetone, the 90° joint is fused with the usual precautions for avoiding distortions and necking in the heated area.

The circle is unclamped, and the jig is rotated so that the circle falls away from the surface of the jig when the stem is heated 1 millimeter from the junction to the cross leg. The bending and jig rotation are continued until the circle lies on the arrest pin (Figure 7B) with the jig horizontal. The jig is rotated 90° and the stem is heated about 1 millimeter from the circle to make the reverse bend, bringing the plane of the circle parallel to and 4 millimeters above the plane of the cross legs and stem (Figure 7C). At this stage, the center of the circle should be directly over the stem and 2.5 millimeters back of the junction of the stem and cross legs.

The bent stem, ring, and cross leg assembly is transferred to jig B (Figure 5), and the cross leg fiber is clamped in the groove next to the cut-out quadrant (Figure 7D) for the forming of the rear foot, column, and hook. The first bend, 90° downward, is made opposite the index groove in the jig (Figure 7E), with the jig in the horizontal position. The fixture is then rotated 180° while forming the U-shaped 1 millimeter by 1 millimeter rear leg (Figure 7F). The column fiber should then be about 1 millimeter from the bend of the circle support fiber. The column is bent toward the circle at the index, 6 millimeters above the fixture surface until it falls in line with the hook index, 10 millimeters above the fixture surface (Figure 8).

With the jig in the horizontal position, the hook is formed by bending the free fiber at a point opposite the index until it reaches the hook angle index (Figure 8B). The bend of the hook should be on the perpendicular line from the plane of the fixture through the center of the circle. The panholder is removed from the jig for adjustment of the hook length. This is done by holding the panholder firmly with one pair of tweezers at a point about 3.5 millimeters from the hook, while another pair of tweezers is used to break off the excess length of fiber (Figure 8C). The hook end is fire polished after the break to length.

The panholder is placed in the leg bending portion of jig B and clamped on either side of the junction (Figure 8D). With the jig level, the leg fibers are bent 90° at each leg bend index (Figure 8E). The panholder is removed from the jig, and the legs are shortened to about 1.5 millimeter length by breaking off the excess fiber with tweezers and fire polishing the ends in the same manner as the hook is adjusted for length. The hook and leg shortening operations are rather critical, since vibration of or through the holding tweezers at this time usually results in loss of the nearly completed panholder by multiple fractures.

Poising and Inspection

The panholder is checked for poise by moving it vertically from and to a clean level surface with a micromanipulator. The three feet should contact and leave the level surface simultaneously, both empty and with a foil placed in the proper position on the circle.

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Slightly unsatisfactory poise and/or shape may be corrected by bending the fibers. Such corrections usually are made by holding the panholder above a level surface with a micromanipulator in such a manner that the panholder will sink to the level surface when heat is applied to the area of the corrective bend.

When the poise and shape are satisfactory, the panholder is rechecked for shape, diameter, parallelness of the circle, overall height, and clearance in a 9-millimeter diameter orifice. Satisfactory panholders are then cleaned with chromic acid, distilled water, and acetone, weighed to the nearest 0.01 milligram, and placed in clean 2 dram screw cap vials for storage until needed for installation.

Microtorch Gas Supply

Construction of quartz fiber instrument elements such as beam systems and panholders involves the making of joints, bends, tapers, shrinks, and stretches of fibers ranging from 4 to 600 microns in diameter. Selection and control of flame sizes, compositions, and velocities for working this range of fiber diameters are rather critical since the masses of fibers being worked to close dimensions at temperatures near 1750°C vary by a factor of over 20,000.

Microtorches used for quartz fiber working at Mound Laboratory have been capillaries drawn from the ends of 10 to 15 centimeter lengths of 5 millimeter outside diameter quartz tubing. Capillary tip orifices ranging from about 0.2 to 0.6 millimeter diameter have been used to obtain flames of 0.2 to 2.0 millimeter diameter according to the composition and pressure of the gas supplied. Torch maneuverability is kept at a maximum by locating the gas control needle valves and mixing chamber on the work bench and by conducting the mixed gas to the torches through small diameter flexible tubing. A pilot torch is attached to each mixing chamber in order to provide a rapid indication of the composition of the mixed gas since a change in composition may take as much as a minute to become stabilized at the tip of the smaller torches.

Variation in pressure of either or both gas supplies influences the flows through the control needle valves and hence the character of the microtorch flame. A change in flame character usually necessitates a readjustment of control valves with an associated delay for torch flame stabilization. If the changes are frequent, the delays increase rapidly along with a greater likelihood of spoiling the work in process.

The most disturbing type of gas-supply pressure fluctuation has been that which is of such speed and magnitude that it causes the flame to flashback into the torches. While each torch is packed with copper turnings to quench a flashback before it causes damage to the tubing and mixing chamber, the capillary orifice usually becomes contaminated with volatilized and condensed copper which spoils the torch for further fiber work. In addition to delays resulting from loss of torches, the possibility of flashbacks induced by sudden pressure changes causes the quartz worker to become unduly tense, a condition which reduces the quality and quantity of fiber work.

A separate oxygen and propane supply system for the fiber drawing machine and the two fiber working benches was installed in late 1949 after measurements of the laboratory propane line pressure showed fluctuations of 3 inches of water in 30 seconds from the nominal of 11 inches. The new system, which was supplied with oxygen and propane from cylinders through Airco Style 8401 oxygen and 8462 propane two-stage regulators, maintained adequately steady line pressures at the torch controls while the regulators were relatively new.

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Gradually increasing difficulty in microtorch control noticed after about nine months local system operation led to a survey of the system gas pressures and flow rates. Manometers were installed in both the oxygen and propane lines at the regulator headers and each of the quartz working benches. Since the bench taps were 22 and 42 feet from the regulators on 1/4-inch copper tube lines, differential pressure manometers were installed at these points to measure pressure drop in the lines from the headers. Manometers indicating pressure in the mixing chambers of the torch controls were used to obtain duplication of flame sizes and gas flows during the survey.

Flow rates of a number of torches and pilots were determined for large and small flame settings by water displacement time measurements. Flow rate values for mixed gas ranged from 0.015 cubic feet per hour for a 0.2 millimeter orifice tip at 0.8 inch mixing chamber pressure to 0.77 cubic feet per hour for a 0.6 millimeter orifice tip at 4.0 inch mixing-chamber pressure. The probable extremes of flow rates likely to be encountered in the system were estimated from the conditions of (A) one bench operating with small torches and flames and (B) two benches operating with large torches and flames. Taking the proportion of one volume of propane to five volumes as the normal ratio of gases consumed, estimated flows range from 0.02 to 0.40 cubic feet per hour for propane and from 0.1 to 2.0 cubic feet per hour for oxygen.

Typical pressures and flows encountered in the headers behind two stage regulators on changing from two bench operation to single bench operation were as follows:

	HEADER PRESSURE in lb. water		HEADER FLOW RATE in ft. ³ /hr.	
	PROPANE	OXYGEN	PROPANE	OXYGEN
TWO BENCHES WORKING	12.3	8.9	0.28	1.40
ONE BENCH WORKING	21.1	31.4	0.12	0.50
HEADER PRESSURE CHANGE	8.8	22.5	0.16	0.80

Inquiry concerning the manufacturer's specifications for the two stage regulators used disclosed that flow rates of the above magnitude were in the class of creepage and that a pressure change of 1 pound per square inch (2.77 inches of water) for shut off is considered satisfactory for new regulators.

A means for obtaining better regulation of pressure at low flow rates was found in the third stage component of the Airco Style 1608 three-stage nitrogen regulator for oil filled transformers. This component, Airco No. 830 1045, which had not been used previously for other gas services, was obtained for trial.

Pressures and flows measured in the headers when using the third stage regulators were as follows:

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	HEADER PRESSURE		HEADER FLOW RATE	
	PROPANE	OXYGEN	PROPANE	OXYGEN
TWO BENCHES WORKING	13.95	13.5	0.28	1.40
ONE BENCH WORKING	14.15	14.1	0.12	0.60
HEADER PRESSURE CHANGE 3 STAGE CONTROL	+0.10	+0.6	0.16	0.80
HEADER PRESSURE CHANGE 2 STAGE CONTROL	+8.8	+22.5	0.16	0.80

Measurement of line losses at the maximum rate of flow showed an oxygen pressure drop between the header and first bench at the 1.4 cubic-foot per hour flow of 0.6 inch of water. Additional drop between the first and second bench at 0.60 cubic foot per hour was 0.35 inch water for a total drop from the header of 0.95 inch of water. The propane line pressure drop at 0.28-cubic-foot per hour flow rate was 0.15 inch water between the header and the second station. Change of the oxygen line from 1/4 inch to 1/2 inch outside diameter copper tube is expected to reduce the line drop to a value comparable to that for the propane line.

The decrease in header pressure change resulting from addition of the third stage of regulation has greatly reduced the difficulties experienced in attempting to control microtorches for quartz fiber work. Installation of adequate flow capacity lines should result in the elimination of detectable interferences with microtorch flame control.

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2. Olt R. G. Quart. Rpt. Gen. Res. MLM 443 5 pp. 170-177 April 1 1950
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MATERIAL: FUSED QUARTZ FIBER 175-MICRON DIA.

SCALE: 1CM. = 5CM.

ARGONNE 1946

FIG. 1 A

MOUND 1948

FIG. 1 B

MOUND 1951

FIG. 1 C

QUARTZ-FIBER MICROBALANCE — PANHOLDER DIMENSIONS.

FIBER DIAMETER, 175 MICRONS

CIRCLE DIAMETER, 4 MILLIMETERS

QUARTZ-FIBER MICROBALANCE PANHOLDER

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FIGURE 3

MLM-561

SCALE: 1CM. = 10CM.

PLACEMENT OF MICROFOIL ON PANCHOLDER RINGS
TO LOCATE MICROFOIL CENTER OF GRAVITY ON
CENTER OF RING.

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PANHOLDER CONSTRUCTION JIG A

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FIGURE 5

MLM-56'

PANHOLDER CONSTRUCTION JIG B

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MLM 561

Problem Title **Mass Spectrograph**
Report by **D Haas**
Work Done By **C Leshner D Haas and K Orr**

INTRODUCTION

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

This aforementioned mass spectrograph is now undergoing a conversion which will result in a mass spectrometer. Retention of the Mattauch arrangement will permit the substitution of an ion collector assembly for the photographic plate. The ion source will be of the surface ionization type. This spectrometer will be used to determine the relative abundance of the artificially produced polonium isotopes (208 209).

DETAILED REPORT

The mass spectrograph has been assigned to the determination of the relative abundance of the 208 and 209 isotopes of polonium produced in the cyclotron at Oak Ridge.

Detection

Because the density of a line produced on a photographic plate by an ion beam is not directly proportional to the magnitude of the ion current producing the line, the photographic method of detection is to be discarded for this experiment. In lieu of the photographic plate, a probe to measure ion currents is to be used in a specially constructed set of pole pieces.

A temporary probe has been constructed and inserted in the present set of pole pieces in an effort to determine the following characteristics:

- 1 The proper design of the shielding necessary to measure the ion current with a minimum amount of background current.
- 2 An exit slit for the current probe.
- 3 The magnitude of the ion current necessary to detect isotopes differing in mass by one mass unit when present in a 1 to 1 000 ratio.
- 4 A calibrated system for varying the magnetic field in terms of the isotopic mass under detection.

A preliminary experiment has been performed with barium oxide as a source material. There are seven isotopes of barium ranging in percentage from 0.1 per cent to 72 per cent.

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NLM 561

in the sample. A total ion current of 5×10^{-22} amperes was measured at the entrance to the magnetic field. This represents total ions from the sample. The least detectable isotopic line barium-134 which is present in 2.4 per cent abundance was detected at the ion collector with an ion current of 1×10^{-24} amperes.

In view of these results the ion collector assembly shown diagrammatically in Figure 1 has been designed. The slit assembly is placed directly on the axis of the former photographic plate. Directly behind the slit a stainless steel Faraday cup has been located. A brass housing is employed here so that the continuity of the air gap may be preserved. Appropriately located apertures permit the entire ion-collector assembly to be operated at analyzer tube pressure. The Faraday cup is connected jointly to the electrometer tube and the grid leak (a resistor of approximately 1×10^{12} ohms). The Faraday cup should suppress secondary electrons quite efficiently. The electrometer tube and grid leak are electrostatically and electromagnetically shielded by the steel housing. The leads to the low potential ends of the electrometer tube and grid leak are brought out of the vacuum chamber through Stupakoff seals. The entire ion collector assembly can be removed from the pole pieces to facilitate the installation of new collector slits.

Source

The Shaw source previously employed is an integral replaceable unit that maintains no direct contact with the slit sections of the analyzing tube. To recharge the source it is necessary to remove the entire mechanism and the following complications result:

- (a) Impossibility of realigning the source without an initial production of ions
- (b) The loss of sample material required for this initial production of ions

Therefore it would appear that a source which could be recharged without disturbing the initial alignment would be conducive to investigations with smaller samples. The ability to use smaller samples becomes a requirement when radioactive contamination is to be minimized.

A source which would fulfill this qualification has been designed and is now in construction. The source employs the techniques of surface ionization for the production of ions. It consists essentially of a filament anode constructed of tungsten ribbon 0.030 inch wide and 0.001 inch thick. This filament has a mechanism which will permit its removal and replacement without disturbing the initial alignment. This filament is also attached to the end of a brass sleeve which slides over the slit system of the spectrometer. Therefore once the source has been aligned with a nonradioactive sample a very small radioactive sample can then be run without the wasteful consumption of sample material.

It is well known that the surface ionization techniques can be applied to the alkali metals, the alkaline earths, and to the rare earths. However the application of surface ionization techniques to the analysis of polonium (208-209) has never been investigated. This investigation has been carried out in the following manner:

A filament constructed of tungsten ribbon 0.030 inch wide and 0.001 inch thick was coated with a 10-lambda sample of polonium (in nitric acid). This filament together

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with two stainless steel foils was then placed in a vacuum envelope. The stainless steel foils have been called foil A and foil B. A potential of 2 000 volts was then applied between the filament and foil A with the filament positive with respect to the foil. The second foil B was operated at the same potential as the filament. The ion current between the filament and foil A was measured and the following results were obtained:

- (a) An ion current of approximately 15 seconds duration was observed for a filament temperature of 2 000°C
- (b) An ion current of approximately 30 seconds duration was observed for a filament temperature of 2 200°C
- (c) A continuous ion current of 11 minutes duration was observed for a filament temperature of 2 350°C
- (d) Above a filament temperature of 2 800 C the ion current could not be observed

Upon the conclusion of this experiment foils A and B were counted with the following results:

1	Foil A	8.53×10^6 counts/minute
2	Foil B	7.04×10^6 counts/minute

Foil A indicates the amount of polonium deposited by the ionized current of material in addition to the amount of material deposited by the process of distillation. Foil B indicates the amount of polonium deposited by distillation. The difference between the counting rates of foils A and B (1.49×10^6 counts/minute) is an indication of the amount of polonium deposited exclusively by the ionization of polonium. Therefore it tentatively appears possible that polonium can be effectively ionized with surface ionization techniques.

The conversion of the spectrograph to a spectrometer will result in an instrument that can measure accurately the isotopic relative abundance of any material that can be ionized with the techniques of surface ionization. By the simple and convenient process of inserting the photographic plate, packing fractions can be determined and qualitative analysis performed on all samples that respond to surface ionization techniques.

REFERENCES

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SURFACE IONIZATION APPARATUS

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END OF DOCUMENT

DATE FILMED: 03-20-80

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