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ASSAYING OF
RADIOACTIVE ROCKS AND ORES

by

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INTRODUCTION.

The information presented in this bulletin is based on experience gained by the Bureau of Mineral Resources, Geology and Geophysics, in certain types of radioactive measurements. From the physicist's point of view, the contents will probably appear to be rather disconnected. However, the topics discussed have been chosen as being of interest to organizations such as mining companies desirous of employing physical methods of measurement in connexion with the search for, and development of, deposits of radioactive minerals, without any particular interest in the purely scientific aspects of the subject.

The following topics are treated:—

1. Naturally occurring radioactive elements, with a brief note on isotopes.
2. Principles of the design of instruments for measuring radioactivity.
3. Radiometric assaying.
4. Fluorimetric assaying.

The discussion is based mainly on the experience of the Geophysical Section of the Bureau, but reference is also made to published material from other sources. A brief bibliography refers to literature which has been found useful. It is stressed, however, that this bibliography is by no means a complete one.

The technique of prospecting for radioactive minerals is not discussed in detail, but is merely mentioned incidentally, where required. This subject is adequately covered in a joint publication by the Australian Atomic Energy Commission and the Department of National Development (1954).

NATURALLY OCCURRING RADIOACTIVE ELEMENTS.

Before discussing the naturally occurring radioactive elements, it is desirable to summarize the findings of modern investigations on isotopes. These are:—

1. Elements occurring in nature are composed, in general, of atoms having identical chemical properties, but having atomic weights which may be one of a limited number of different values. These atomic types are known as isotopes.
2. The isotopic constitution of an element, i.e., the percentages of the isotopes having different atomic weights, is invariable in elements occurring naturally, except where such elements have been subjected to conditions which would allow partial separation of the isotopes by processes depending on differences in density. It appears that such processes only affect elements of atomic weight less than about 40.

The isotopic constitution of certain elements varies with time, due to the processes of radioactive decay. Thus natural uranium consists of two isotopes, which decay radioactively at different rates. Natural rubidium and potassium each consist of several isotopes, only one of which is radioactive. The established fact of the present invariance of the isotopic constitution of such elements all over the earth leads to the conclusion that all these elements at present existing were originally formed simultaneously. This time of formation of the elements must not be confused with the time of their introduction into their present geological environment, which can be anywhere within the whole range of geological time.

Radioactive isotopes of most, if not all, elements can be formed in modern nuclear laboratories by bombardment of the elements with energetic particles. However, as indicated above, a few naturally occurring elements are radioactive. These consist of three series of elements, derived originally from the breakdown of uranium₂₃₈(U₂₃₈), uranium₂₃₅(U₂₃₅) and thorium₂₃₂(Th₂₃₂), respectively, and certain isotopes which break down with emission of single particles, changing to inactive isotopes. Details of these elements are shewn in Tables 1, 2 and 3, the

data for which were obtained from a publication of the U.S.A. National Bureau of Standards (1950). In addition, the following elements are subject to single disintegrations:—

1. Naturally occurring rubidium contains 27.2 per cent. of Rb_{87} , which emits a beta particle and leaves Sr_{87} , which is inactive. The half-life of Rb_{87} is about 6.1×10^{10} years.
2. Naturally occurring potassium contains 0.012 per cent. of K_{40} , which is subject to a double decay process. Eighty-eight per cent. of the K_{40} decays to Ca_{40} with emission of a beta particle, and 12 per cent. decays by K-electron capture with gamma ray emission to A_{40} . The half-life of K_{40} is 1.27×10^9 years.
3. It is known that one isotope of samarium decays with alpha-particle emission. However, the radiation is so weak that no satisfactory determinations of the half-life, the atomic weights of the active isotope, or the decay product have yet been made.

For ordinary prospecting purposes, the uranium and thorium series are of most interest. The other disintegrations mentioned are of interest only in connexion with the measurement of geological age.

PRINCIPLES OF THE DESIGN OF INSTRUMENTS FOR MEASURING RADIOACTIVITY.

INTRODUCTION.

Instruments for the detection of ionizing radiation present a great diversity in appearance, but all are based on certain general principles, which can be described in simple terms. The details of design of many are very complex and require great skill and experience. Discussion of these matters cannot be envisaged here. Several standard works, which may be consulted for more detailed information, are listed in the bibliography.

DETECTION OF RADIATION.

The methods of detection used in radiation-measuring instruments are all fundamentally electrical. However, it is convenient to distinguish between so-called electrical detectors and scintillation detectors. These two types will be considered separately.

Electrical Detectors.—All electrical methods of radiation detection depend on the ionizing effects of the radiation. The radiation enters a vessel containing gas at a suitable pressure, and two electrodes, between which a potential difference is established. The radiation ionizes the gas and the positive and negative ions are attracted to opposite electrodes by electrostatic attraction. Each ionized particle thus causes a voltage pulse between the electrodes, which is registered by the methods described later. When a continuous flux of radiation is entering the vessel, the passage of ions causes a steady electrical current between the electrodes, and this can be measured.

The behaviour of detectors of this type is extremely complex. However, for the present purpose, only the effect of increasing voltage is of interest, and this can be described simply in general terms.

Suppose a single ionizing particle enters the gas. It produces ions, the number of which depends on the charge and velocity of the incident particle. When the voltage between the electrodes is low, some ion pairs are able to recombine so that all the ions are not collected at the electrodes. As the voltage is increased, a value is reached at which no recombination takes place, and all ions are collected. This is called the saturation voltage. This condition persists over a

certain voltage range, which is called the "ionization chamber region." A detector working at a voltage within this region is called an ionization chamber, and produces pulses whose amplitude is dependent only on the energy of the original particles. The amplitude of the pulses is of the order of microvolts.

As the voltage is still further increased, the ions in their passage to the electrodes have sufficient energy to ionize atoms with which they collide. These secondary ions are also attracted to the electrodes, with the net result that the pulse collected is greater than that which would be obtained by collecting only the total ionization due to the original particle. This process is known as "gas amplification." The amount of amplification depends on the voltage applied.

A gas amplification of 10^2 to 10^3 is commonly employed. Higher amplification is possible, but is not generally used, for the following reasons:

1. As the gas amplification increases, the amplitude of the output pulses tends to level out so that proportionality with the energy of the original ionizing radiation is gradually lost.
2. At high gas amplification, the dependence of the amplification on the applied voltage becomes more and more critical. This imposes severe demands on the stability of the applied voltage.

A detector using a gas amplification of up to 10^3 is called a "proportional counter," and provides output pulses whose amplitude may be of the order of millivolts.

At still higher voltages, ionization-by-collision and similar processes becomes the main factor in the functioning of the detector. The original ionizing particle acts merely as a trigger to set the process going, the final result being a gas discharge between the electrodes. Assuming that this discharge is stopped after a very short time by one of the methods mentioned below, it produces a pulse at the electrodes, the amplitude of which may be 1 volt or higher. The amplitude of the pulse is determined by the voltage applied to the tube, and is quite independent of the energy of the ionizing radiation. Proportionality is therefore entirely lost. A detector operating under these conditions is known as a Geiger tube, and the voltage range over which the tube operates is called the "Geiger region."

The behaviour of a Geiger tube, exposed to a steady flux of ionizing radiation, working into a circuit capable of recording pulses of amplitude of the order of a volt, is shown in Fig. 1.

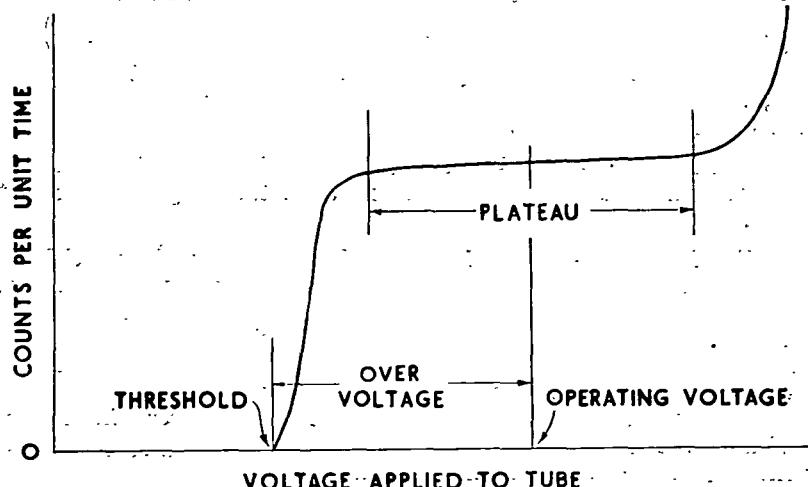


Fig. 1. Operating characteristics of G.M. tube in Geiger region.

Until the voltage reaches a certain minimum value, known as the "threshold," no counts are recorded. As the voltage increases above the threshold value, the count rate increases rapidly to a value which is maintained relatively constant over a range of voltage, known as the "plateau." The length of the plateau is generally 100–200 volts. Further increase of voltage causes a rapid and erratic increase in count rate, and finally the complete breakdown of the tube. Geiger tubes are invariably used on the plateau. The amplitude of the pulse produced depends merely on the "overvoltage," which is the difference between the operating voltage and the threshold, and is quite independent of the energy of the ionizing radiation.

In the Geiger tube as described so far, there is no provision for stopping the discharge once it has been started. In the earlier types of tube, this had to be done externally, by some means which dropped the voltage below the threshold value for a short period. This can be done by connecting the voltage supply to the tube through a very high resistance (of the order of 10^9 ohms or higher). When the tube fires, the passage of the discharge current through the resistor causes a voltage drop sufficient to reduce the applied voltage below the threshold value. This introduces a long time-constant into the circuit, so that the tube cannot be used for rapid counting. Alternatively, an electronic means of dropping the voltage may be used. The one-shot multivibrator is very convenient for this purpose. (For notes on the functioning of the multivibrator see p. 11.) The multivibrator is designed to be triggered by the pulse from the Geiger tube and the large negative pulse developed at the plate of the "off" tube is applied to the Geiger tube, causing the voltage to drop below the threshold value for a time determined by the constants of the multivibrator. The Geiger tube is thus made inoperative for a time which is accurately known and a correction for this "dead time" can be applied to the observed count rate if necessary.

Most Geiger tubes now incorporate a quenching medium in the gas filling, usually consisting of a small amount of a heavy organic gas. This quenches the discharge very rapidly. Such tubes are known as "self-quenching," and may be used with series resistances as low as 100,000 ohms, which makes them capable of rapid counting. They are subject to certain minor disadvantages, which may become apparent in very precise work. In such cases, the use of a multivibrator quenching stage, as described above, may be preferable. The original self-quenching tubes worked generally at voltages ranging from 1,000 to 1,500, and tubes of this type are still widely used. A more recent development has been the halogen-quenched tube, which works generally at about 400 volts. Such tubes are more difficult to manufacture, but have the advantage that their life is theoretically unlimited. Because of their low operating voltage, they are especially suitable for use in portable equipment.

Due to the nature of the discharge, the Geiger tube cannot be used for very high counting rates. The resolving time of a Geiger tube is generally about 200 microseconds.

Scintillation Detectors.—It has long been known that alpha particles cause scintillations when they fall on screens coated with certain crystalline materials, and this property was used as a means of detection in many classical experiments on radioactivity. In recent years, it has been found that radiations of all types can cause scintillations in a wide range of materials, both organic and inorganic. The properties of such materials, which are generally termed phosphors, are, in the main, outside the scope of these notes. The main interest, in the present connexion, lies in the fact that crystals of sodium iodide, activated with thallium, are very efficient detectors of gamma radiation.

This method of detection has become generally useful only since the development of the photo-multiplier tube, the method of functioning of which need not be discussed here. The tube acts as a detector, which converts the scintillations into voltage pulses, combined with an amplifier of very high gain, so that a gamma ray detected by the crystal is registered as a voltage pulse, the amplitude of which may be of the order of millivolts.

For laboratory work in nuclear physics, the combination of phosphor and photo-multiplier tube has great advantages over any other particle detector. The response to beta and gamma rays is proportional, and relatively simple forms of beta and gamma ray spectrometers can be constructed. The response is so fast that ordinary electronic techniques are quite inadequate to take full advantage of the speed available. As a gamma-ray detector, the sodium iodide crystal is much more efficient than a Geiger tube. For gamma radiation of the energies associated with uranium minerals, the detection efficiency of a Geiger tube is much less than 1 per cent., while that of the crystal may be of the order of 30 per cent.

However, there are certain special problems in the use of scintillation detectors. These are not difficult to solve in laboratory work, but seriously affect the construction of portable scintillation equipment. Sodium iodide crystals are hygroscopic and are quickly ruined by exposure to moisture. They are also very expensive. The photo-multiplier tubes are expensive and fragile, and can easily be damaged by exposure to light, or by incorrect operation. A high voltage (usually 1,000 to 1,500 volts) must be applied to the tube, the noise background of which increases rapidly with increasing voltage. It is essential that the voltage supply be very stable, and that the associated electronic equipment also be stable in operation. For these reasons, portable scintillation detectors are much more expensive and more difficult to maintain than portable Geiger counters.

For work of this type, the scintillation detector has two main advantages over the Geiger counter, both being due to its high detection efficiency. It is generally about three times as sensitive, i.e., an outcrop giving a reading of twice background on a Geiger counter would give a reading of about six times background on a scintillation instrument. Also, due to its much higher count rate, its response time can be made much shorter than that of the Geiger counter. Neither of these advantages is sufficient, in ordinary prospecting work, to justify the extra cost. The rapid response is a capital advantage in equipment for airborne surveying, in which scintillation counters are used exclusively.

EQUIPMENT FOR MEASURING IONIZATION CURRENTS.

The oldest and most familiar instrument for the measurement of ionization currents is the electroscope. The equivalent circuit for this instrument is shown in Fig. 2.

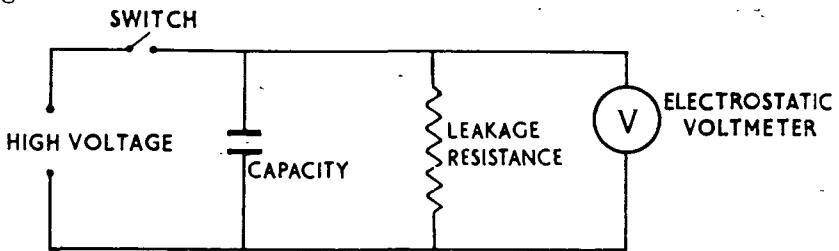


Fig. 2. Equivalent circuit of electroscope.

It consists essentially of an ionization chamber, the centre electrode of which is charged electrically to a high potential. The leaves act as an electrostatic voltmeter, which measures the potential of the electrode. This potential will fall

gradually, due to leakage of the charge from the electrode across the surface of the insulator. However, if the gas in the chamber is ionized, the rate of leakage of charge will be increased, due to the ionization current. Measurement of the rate of fall of the leaves will show the total rate of leakage of charge and, provided the natural leak is small compared with that due to the ionization current, accurate current measurements are possible.

In the hands of a skilled experimenter, the electroscope is capable of results of high accuracy and, in fact, was used in many classical experiments on radioactivity. It is essentially a laboratory instrument, and has largely been superseded by more flexible equipment.

Equipment generally used for measuring ionization currents is shown schematically in Fig. 3. The current through the ionization chamber is passed through a very high resistance of about 10^{11} ohms, and the resulting voltage drop is applied to a D.C. amplifier. The amplified current is measured on an ordinary current meter.

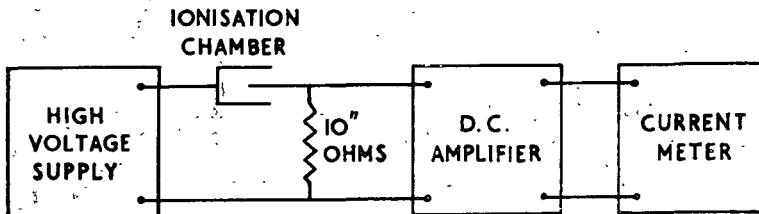


Fig. 3. Equipment used for measuring ionization currents.

It is obvious from the schematic diagram that, in order to obtain accurate current measurements, the input impedance of the D.C. amplifier must be large in comparison with the resistance of 10^{11} ohms. This cannot be obtained by conventional methods because of the grid current that flows in the first stage of the amplifier. This is of the order of 10^{-8} amps in an ordinary tube and gives an input impedance of only 10^8 ohms. Considerable improvement is obtained by using the tube in the manner shown in Fig. 4.

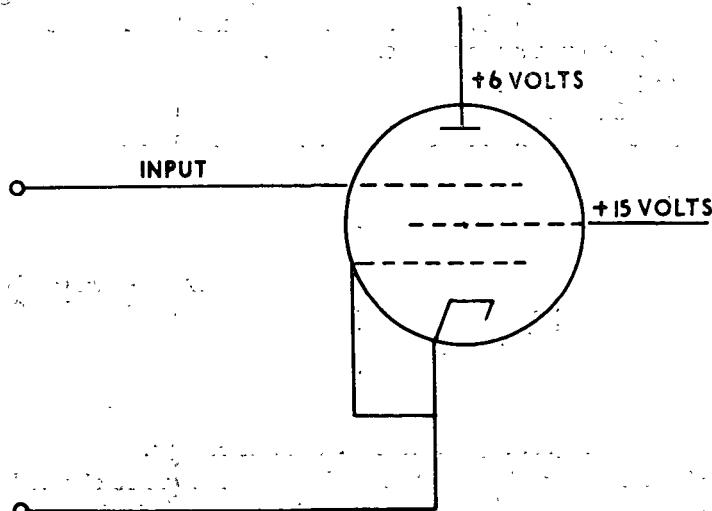


Fig. 4: Typical operating conditions for electrometer tube.

Here, the suppressor is used as the control grid. The low plate voltage of 6 volts is chosen to prevent the formation of positive ions in the gas of the tube. A potential of about 15 volts is applied to the screen to prevent positive ions formed at the cathode from reaching the plate. Further improvement is obtained by operating the heater at reduced current, by mounting the tube in a light-tight box, and by coating the glass of the tube with insulating wax to cut down surface leakage.

A tube used in this way is known as an "electrometer tube." Several types of commercial radio tubes, used in this manner, can have input impedances of the order of 10^{13} ohms. If higher impedances are required, specially constructed electrometer tubes may be used. In such tubes the input impedance may be as high as 10^{17} ohms.

Electrometer tubes are difficult to use because the steady currents show a tendency to drift and this is often very difficult to remedy. Suitable circuits, and discussion of the precautions necessary, may be found in the standard texts.

In recent years, a considerable advance in the measurement of small currents has been made with the development of the vibrating reed electrometer. This consists of a condenser, composed of a fixed plate and a vibrating reed which is driven at its resonant frequency by a suitable oscillator. The D.C. voltage developed across the high resistance is applied to this condenser and the resulting A.C. voltage is suitably amplified. The instrument is much less subject to drifts than the ordinary D.C. amplifier using electrometer tubes. The accuracy of measurement is limited only by the accuracy and stability of the high resistance used.

In all apparatus of this type, the greatest care is necessary with insulation. An insulation resistance of 1,000 megohms would virtually short circuit the whole set-up. In order to obtain reliable measurements, the insulation resistance must be high in comparison with the input impedance of the amplifier. To obtain and maintain such resistance of the order of 10^{15} ohms requires the utmost care in the construction and use of the equipment.

EQUIPMENT FOR REGISTRATION OF PULSES.

Most radioactive measurements are made using equipment which registers voltage pulses caused by individual ionizing particles. Two types of equipment may be used, and these are shown schematically, in Fig. 5.

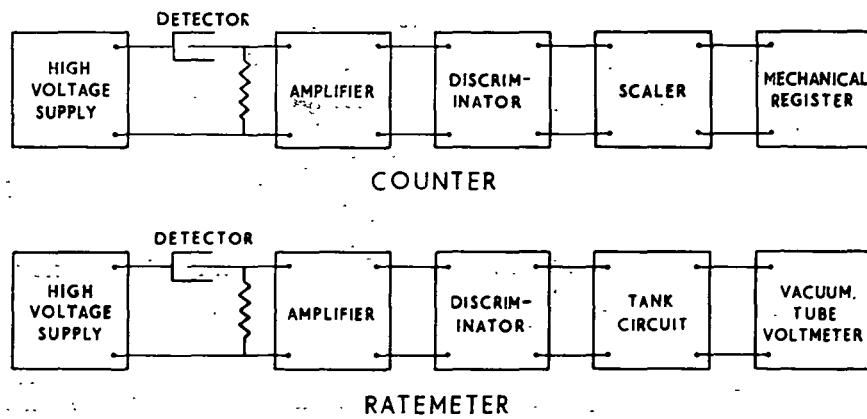


Fig. 5. Equipment for registration of pulses.

The detector may be an ionization chamber, a proportional counter, a scintillation detector or a Geiger tube, depending on the type of information required. The choice of detector is governed by the properties of the various types, which may be summarized as follows:—

—	Voltage Required.	Gain of Amplifier Required.		Proportionality.	Speed.
Ionization Chamber ..	Low	Very high	Good ..	Fast	
Proportional Counter ..	Moderate ; highly stable ..	High	Good ..	Fast	
Scintillation Counter ..	High ; highly stable ..	Moderate : wide band ..	Good ..	Very fast	
Geiger Counter ..	High	Low	Nil ..	Limited	

Where discrimination between ionizing particles of different energies is required, either an ionization chamber, a proportional counter or a scintillation counter must be used. The great disadvantage of these detectors, however, is the nature of the amplifier required. This limits their use outside the laboratory.

The Geiger counter, although at a disadvantage to the other detectors in most respects, has the major advantage that it requires very little amplification. It is generally used in portable equipment, and largely in the laboratory. Briefly, the general practice is to use Geiger tubes whenever possible, and to have recourse to other detectors only when the properties of the Geiger tube make it unsuitable for a particular application.

The various circuit elements shown in Fig. 5 are discussed in more detail below.

High Voltage Supplies.—High voltage supplies ranging from a few hundred to two thousand volts are required for various types of detectors. The current demands of the detectors are very small. However, allowance must be made for the current drawn by any stabilizing circuit which may be required.

The voltage may be supplied from dry batteries, which give a very stable supply, but have several disadvantages. Dry batteries are expensive and bulky. Also, the current drawn is so small that the life of the batteries is limited only by their shelf life, and their actual capacity is not used.

Various electronic devices are successfully used for supplying high voltages. For laboratory use, the most convenient is to generate the voltage by an oscillator at radio frequency and use an R.F. transformer to obtain the necessary voltage level. This R.F. voltage is then rectified and filtered. The voltage can be readily controlled over a wide range by detuning the oscillator, or by using the ordinary voltage divider.

A method which has been used in compact portable equipment makes use of the properties of the free-running multivibrator. It is well known that if a current I be passed through an inductance L , the voltage developed across the inductance is $L \frac{dI}{dt}$ (in consistent units). It is a fundamental property of the multivibrator that the change-over between tubes occurs almost instantaneously, so that dI/dt is very large. It is found that a practicable supply giving a high voltage may be obtained by using a free-running multivibrator, with a small choke as the plate load of one tube. The voltage developed across the choke is rectified and filtered. A disadvantage of this circuit is that the voltage is not easily controlled, and the supply has therefore to be designed to cover a relatively small voltage range.

For methods of stabilizing such voltage supplies reference should be made to the standard texts.

Amplifiers.—The pulse as furnished by the detector must be amplified to a voltage level sufficient to trigger the discriminator. The functioning of the discriminator is described below; at present, it is sufficient to state that it requires a pulse of a few volts to trigger it. For rough comparison purposes, this figure may be taken as 10 volts. In any particular experiment, sufficient amplification must be provided to raise the level of the minimum pulse to be detected to at least this figure.

The limit to the amount of the amplification that can be used is fixed by noise in the tubes of the amplifier, which cannot be completely eliminated. The amount of noise varies from amplifier to amplifier, but for one designed and constructed with maximum precautions, noise equivalent to about 3.5 microvolts R.M.S. at the first grid has been observed. This is equivalent to a peak amplitude of about 10 microvolts. It follows that a pulse of amplitude less than 10 microvolts cannot be detected, no matter how much gain is used, as it cannot be distinguished from the noise. Also, it is undesirable that the noise pulses be amplified to a level sufficient to trigger the discriminator. The maximum useful gain may therefore be taken as the gain which would raise a signal of 10 microvolts to 10 volts, i.e., a voltage gain of 10^6 . Provision is always made for adjusting the gain by means of attenuators.

As well as having sufficient gain, an amplifier for use with an ionization chamber must fulfil several other requirements. It should be capable of delivering output signals of a fairly wide range of amplitude. An upper limit of 50 volts may be taken as a not unreasonable figure. Over this range the amplitude of the output signal must be accurately proportional to that of the input at all settings of the gain control, if full use is to be made of the proportional characteristic of the detector. The gain must be very stable. Furthermore, the amplifier should preserve the sharpness of the detector pulses. In other words, it should be sensitive to frequencies as high as possible. An upper, half-power frequency of 1 megacycle is desirable. The low frequency response is generally limited by the insertion of a coupling with a short time-constant at the input to one stage, to prevent the amplifier being overloaded by relatively slow variations in the detector output voltage.

The design and construction of an amplifier having all the above properties is an extremely difficult matter. When used with a proportional counter, the extreme gain of 10^6 is unnecessary, a gain of 10^4 being usually sufficient. However, an amplifier with a gain of 10^4 , satisfying the other requirements mentioned, is still a high-grade amplifier. This is the main problem in the use of ionization chambers and proportional counters.

The design of amplifiers for use with laboratory scintillation detectors is a separate problem. Such amplifiers need have only moderate gain, but, if it is intended to take anything like full advantage of the speed of the scintillation counter, should have a band-width of the order of 100 megacycles. Only modern tubes of special construction are of any use in such service.

With the Geiger tube, this difficulty does not arise. The output pulse is of the order of one volt, and as there is no proportionality and the tube is not capable of very high counting speeds, no particular care is necessary with regard to gain stability or high frequency response. Some amplification is usually desirable. This can be provided readily by means of a single pentode stage giving a voltage gain of about 50.

Discriminators.—A discriminator is a circuit which, on receipt of an input pulse of suitable amplitude, provides an output pulse of amplitude and wave form depending only on its own circuit constants. Discriminators are invariably based on some form of the "one-shot" multivibrator. When this circuit has adjustable bias, it performs the following two functions:—

1. *Discrimination.*—The circuit can be triggered only by pulses of amplitude greater than a certain voltage, which depends on the applied bias. By varying this bias, a "spectrum" of the amplitudes and relative frequencies of the pulses from the detector may be obtained.
2. *Pulse Shaping.*—Any input pulse which triggers the discriminator appears at the output as a pulse of standard wave form and amplitude, quite independent of the shape of the input pulse.

In different applications, emphasis may be laid on one of these functions to the exclusion of the other. Thus, in the counter set-up shown in Fig. 5, discrimination is the main requirement, since the requirements for triggering the scaler are not critical. In fact, when a Geiger tube is used as a detector in this set-up, the discriminator section is not required. In the ratemeter set-up, both functions are essential, except when a Geiger tube is used. Then, the pulse shaping function is the essential one, as there is no proportionality in a Geiger tube, and therefore no possibility of discrimination. Once again, the design of extremely fast discriminators for laboratory scintillation detectors involves special problems.

Scalers and Mechanical Registers.—The most convenient way of recording pulses is by means of a mechanical register. However, these are limited in the rate at which they can record pulses. Available registers vary widely in resolving time. Great improvements have been made in recent years and several registers now constructed will count 50 random pulses per second, with negligible losses. In fact, some registers are known which will count 100 random pulses per second quite reliably. The mechanical design of such instruments need not be discussed in detail here. Increased resolving power may be obtained in them by making the working parts lighter, so as to reduce inertia, or by using a relatively robust movement and supplying more driving power. Both these methods have limited possibilities. If the working parts are made lighter, the instrument becomes difficult to adjust, and subject to wear. On the other hand, the power demands cannot be made too high, otherwise the design of the driver stage becomes difficult.

A register which counts 100 pulses per second is still quite inadequate, even for a relatively slow detector such as a Geiger tube, which may have a resolving time of some hundreds of microseconds. To meet this situation, a scaling circuit is used between the discriminator and the register. Scaling circuits are based on the "scale of 2" flip-flop multivibrator. The fundamental property of this circuit is that, when pulses are applied to the input, half the number of pulses appear at the output. Thus, if n such stages are used in series, the number of pulses appearing at the output of the last stage is $\frac{1}{2^n}$ of the number at the input. It is therefore a simple matter to calculate the number of scaling circuits required to reduce the maximum rate of input pulses to a figure within the capacity of the register used.

Such scaling circuits, having a resolution time of 5 microseconds, can be readily constructed and are quite reliable in use. It is found, however, that it is difficult to obtain resolution times shorter than this. Scaling circuits with resolution times of less than one microsecond have been constructed, but they must be carefully set up, and operated by a skilled technician.

The fundamental "scale of 2" circuit can be used as the basis of a "scale of 10" counter, with a little extra complication. Details of this circuit are given in the standard works.

Recently, alternative types of scalers have been developed, based on the multi-element gas-filled tubes. These are simple in design and economical in components, but have considerably larger resolving times than hard valve scalers. They are generally adequate to the requirements of assay work, and are discussed in more detail later.

Tank Circuits.—The fundamental tank circuit is shown in Fig. 6. The theory of the circuit is discussed in the standard works.

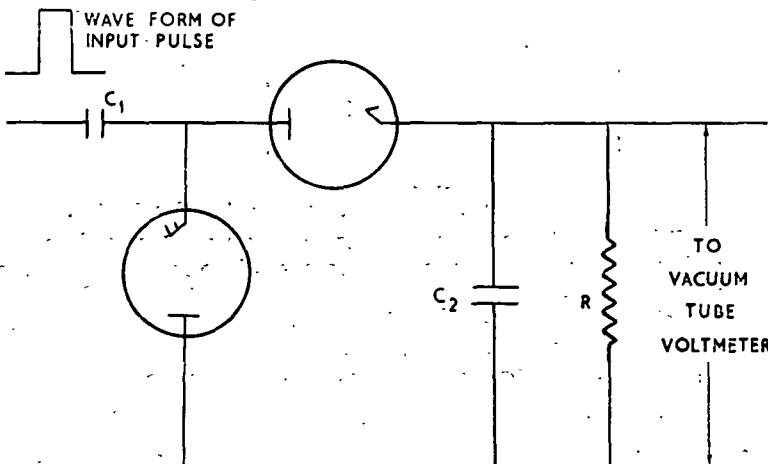


Fig. 6.—Ratemeter tank-circuit.

Provided certain restrictions on the size of the components are satisfied, it may be shown that:—

1. The average voltage appearing across C_2 is proportional to the product $C_2 R$. It is thus convenient to set the sensitivity of the instrument by altering the value of R .
2. The accuracy of the instrument (measured by the steadiness of the reading) is proportional to $\sqrt{C_2}$. It is convenient to adjust the accuracy of the reading by altering the value of C_2 . For rough work, a lower value may be used, and a rough reading obtained rapidly. For accurate work, a large value must be used, with consequent increase in the time required to obtain the reading.
3. The time required for the reading to be obtained is proportional to the time constant RC_2 .

Notes on the Multivibrator.—The following notes are intended to describe briefly the functioning of the multivibrator, which, in one form or another, is an essential part of most instruments used in the detection of ionizing radiation. The detailed design of these circuits requires great skill and experience.

(a) The "free-running" multivibrator is shown in Fig. 7.

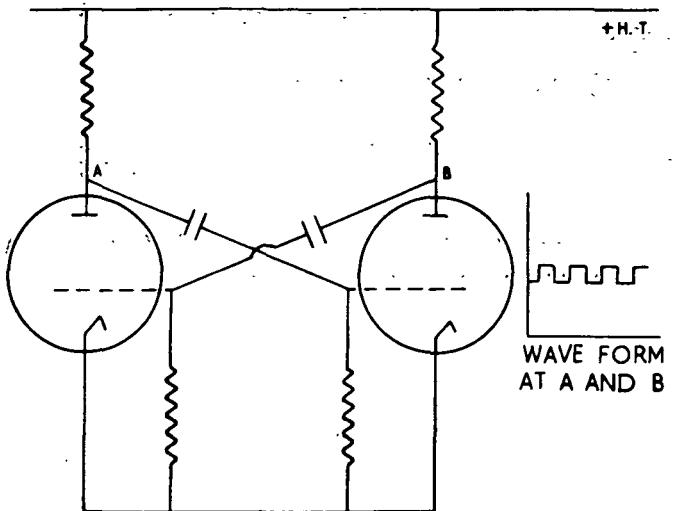


Fig. 7. Circuit diagram of "free-running" multivibrator.

This circuit has no stable state. It has two extreme states, in one of which tube A is conducting and tube B non-conducting, while in the other, the reverse condition holds. The circuit oscillates between these states, at a frequency which depends on the circuit constants. The change-over occurs almost instantaneously, so that the wave form is roughly rectangular.

(b) The "one-shot" multivibrator is shown in Fig. 8.

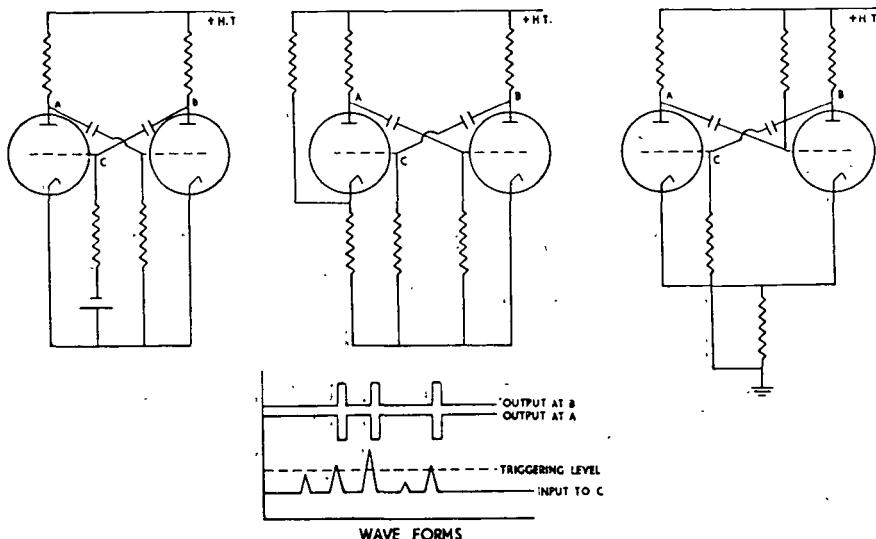


Fig. 8. Circuit diagram of "one-shot" multivibrator.

Three forms of the circuit are shown, which are identical in operation. The circuit consists essentially of a multivibrator in which a bias is applied to one tube. The circuit then has a stable state in which, in the circuits shown, tube A is "off" (non-conducting) and tube B is "on" (conducting). If, however, a

positive pulse of sufficient amplitude is applied to the grid of tube A; the bias is overcome, and the tube "runs free" for one cycle. The output wave form at the plates of tubes A and B is shown in the diagram. By adjustment of the amount of bias, the level of the pulse needed to trigger the circuit may be varied. The output pulse depends only on the circuit constants, and not on the shape of the input pulse (within certain limits). It must be remembered, however, that the triggering mechanism of such a circuit is not as simple as it sounds. Reference should be made to the standard works for details of practical discriminator circuits.

(c) The "flip-flop," which is the basis of most scaling circuits, is shown in Fig. 9.

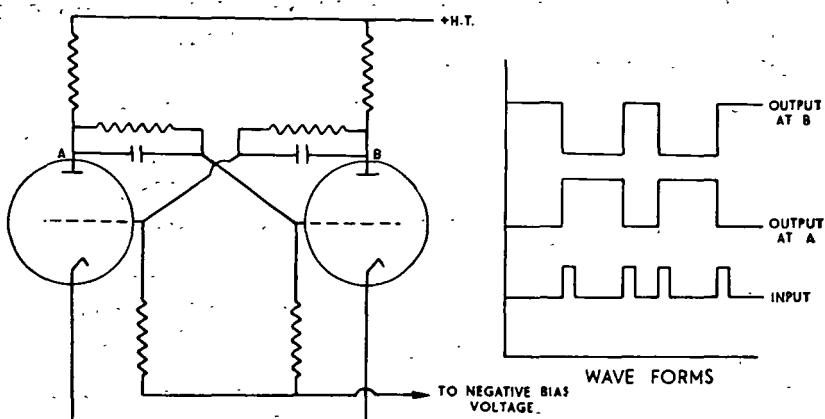


Fig. 9. Circuit diagram of "flip-flop."

The circuit has two stable states, in which one tube is "off" and the other "on." It will rest in either of these states, until the arrival of a suitable triggering pulse. This causes the circuit to change over to the other state, in which it will remain, until the next input pulse arrives. The wave forms are shown on the diagram, from which it is obvious that the number of pulses appearing at A or B is half the number of input pulses received. For details of the functioning of the circuit, and methods of applying input pulses, reference should be made to the standard works.

Illustrative Circuits.—As an illustration of the principles discussed, circuits of simple types of equipment constructed by the staff of the Bureau are shown. Nothing more is claimed for these circuits than that their functioning has fulfilled the design requirements, and that they can be constructed without any special difficulties.

1. Demonstration model G.M. ratemeter.

This is a mains-operated laboratory ratemeter, and includes a small loudspeaker. The circuit diagram is shown on Plate 1. It is designed to operate from halogen-quenched Geiger tubes, the voltage for which is obtained from a voltage doubler of conventional design. Provision is also made for injecting calibration pulses of suitable shape from a pulse generator. The circuit includes two stages of voltage gain, a one-shot multivibrator for pulse shaping, a cathode follower for coupling the loudspeaker, a diode pump, tank circuit and vacuum tube voltmeter. The only portion of which the functioning is not obvious in the light of the foregoing discussions is the 6SH7 valve. This is a feed-back, capacity

multiplier, which effectively multiplies the capacity of the tank circuit condensers by a large factor. By this means, the use of large electrolytic condensers in the tank circuit is avoided. The functioning of this circuit is discussed by Elmore and Sands (1949).

It would be more in line with modern practice to replace the valves shown by their miniature equivalents. This can safely be done for all valves except the 6H6 in the voltage doubler, which is working considerably above its rated heater-to-cathode voltage. Experience shows that this practice is safe for a 6H6 valve, but it would probably be unsafe if a 6AL5 were used.

2. Regulated E.H.T. power supply.

This is a stabilized, high-voltage supply of the multivibrator type, designed for use in vehicle-mounted equipment. The circuit diagram is shown on Plate 2.

3. Four Geiger sets suitable for portable use.

These are simple sets, of a type suited to construction by amateurs. The circuit diagrams are shown on Plates 3, 4, 5 and 6. The circuit on Plate 5 is a ratemeter with a fairly long time-constant, designed for field assay work. Plate 6 shows the same circuit, modified for use as an ordinary portable counter.

Notes on Servicing.—It will be obvious, from the foregoing discussion, that the servicing of equipment of this type presents problems rather different from those encountered in ordinary radio servicing. However, it is felt that, once the functioning of the various portions of the circuits is appreciated, an experienced radio serviceman can readily detect faults in the simple types of equipment described, by measuring voltages at various points on the circuit.

The minimum equipment required for such servicing is an electrostatic voltmeter for measuring the high voltages applied to the detector, and a high-impedance multimeter for testing circuit voltages generally. The electronic type of multimeter is very convenient for this purpose. For calibrating ratemeter circuits, a suitable pulse generator is required. A cathode-ray oscilloscope is very useful for servicing equipment of this type, and is absolutely essential for the more complicated types of laboratory equipment. However, commercially available oscilloscopes do not generally cover a wide enough frequency range for this purpose. Also, the proper use of a cathode-ray oscilloscope in testing this type of equipment requires considerable skill and experience.

RADIOMETRIC ASSAYING.

INTRODUCTION.

From the discussion on naturally occurring radioactive elements, it is obvious that any uranium or thorium mineral emits a complex series of alpha, beta and gamma radiation (see Tables 1, 2 and 3). The possibility immediately suggests itself that the physical methods of radiation detection previously described could be used for assaying ores for uranium or thorium content, thereby avoiding the use of long and difficult chemical procedures.

The mathematics of radioactive decay are treated in most text-books on physics. It can be easily proved that:

1. When the process has been going on for a sufficient time, a stage is reached at which each decay product is produced at the same rate at which it decomposes. The series is then said to be in radioactive equilibrium. The time required for the establishment of radioactive equilibrium depends on the half-life of the longest lived product in the series. In the uranium and thorium series, the elements uranium

II and mesothorium I are the longest-lived products respectively. The time required for establishment of equilibrium in the uranium series is of the order of 10^6 years, and is considerably less in the case of the thorium series. It may be assumed, therefore, that the primary radioactive minerals have in all instances been deposited in their present environment for a sufficiently long period for equilibrium to have been attained.

2. When radioactive equilibrium has been established, the intensity of any particular radiation or group of radiations is directly proportional to the amount of the longest lived element present. In the case of the uranium and thorium series this is the parent element in each instance.

THEORY OF RADIOMETRIC ASSAYING.

The above-mentioned facts constitute a basis for radiometric assaying for uranium and thorium. If a characteristic radiation from one member of the series could be isolated, and its intensity measured, it might appear possible to calculate directly the amount of the parent element present, and identify it without any doubt. However, for practical reasons, this simple method is impossible. Owing to the difficulty in determining the efficiency of the detector and the corrections for geometry, absorption, back-scattering, &c., which would be required, no absolute counting method can be considered. The only practical method of assaying is to compare, under identical geometrical conditions, the radiation from the unknown sample with that from the same amount of a standard sample, whose uranium or thorium content has been checked by other means. This effectively introduces another unknown into the problem, because, before a suitable standard for comparison can be chosen, it is necessary to know whether the sample contains uranium or thorium or both.

The ideal requirements for radiometric assaying are, therefore:—

- (1) Radioactive equilibrium.
- (2) Information whether radioactivity is due to uranium or thorium.

These requirements are not generally satisfied, however. As stated previously, it can be assumed that any primary deposit of radioactive minerals has been in place long enough for the establishment of equilibrium. It cannot be assumed that it has not been subjected to conditions which would tend to disturb equilibrium. Since uranium compounds are more soluble than the compounds of some of the disintegration products, notably radium, it is obvious that the action of ground water could disturb radioactive equilibrium. The effect might be a solution and removal of uranium, leading to a deficiency in uranium as compared with its decay products, or a solution and redeposition of uranium leading to an enrichment of uranium in the oxidized zone. Both of these effects have been observed. As thorium compounds are not generally soluble, similar effects do not occur with thorium minerals.

As regards distinguishing between uranium and thorium, it was thought at one time that this could be done safely on mineralogical grounds, but experience has shown that this is not so. All thorium minerals contain a little uranium. Uranium minerals frequently occur without any detectable amount of thorium, but it has also been found that thorium mineralization can be associated fairly closely with ores rich in uranium.

If a radiometric assay procedure is to be of any value, it must therefore include means of detecting whether the radioactive sample contains uranium or thorium or both, and whether or not there has been any serious disturbance of radioactive equilibrium. Since both uranium and thorium emit alpha particles

only, the most direct method of approach would appear to be to isolate the alpha radiation characteristics of each of these elements, and to use this as a means of comparison with standard materials. For several reasons, however, this is impracticable. The measurement of alpha-ray energies is a complicated operation, which is best performed using an ionization chamber of special design. The auxiliary equipment required is generally beyond the scope of any but a well-equipped physics laboratory. A further difficulty, which is probably more serious, arises from the fact that alpha particles are very quickly absorbed in any fairly dense material. Self-absorption in the grains of the sample itself is considerable, and impossible to estimate. The usual method used in alpha counting is to have the sample in solution and to deposit a very thin uniform layer of the active material by evaporating the solution. To obtain a film of uniform density is a difficult process. In any case, any such operation as this would greatly reduce the speed of the process, which is the main advantage of radiometric assaying.

For the above-mentioned reasons, alpha-counting methods are never used for assaying. All assaying procedures are based on beta and gamma counting; which can be performed on powdered, solid samples. In principle, the procedure adopted is to compare the sample with a standard sample containing a known amount of uranium in equilibrium with its decay products, by beta counting, and to repeat the process using gamma counting. If the sample contains uranium in radioactive equilibrium, the results should be the same. If this is not the case, it may be assumed that either radioactive equilibrium has been disturbed, or the sample contains thorium. Samples may be separated into the following four classes:—

1. Samples in which radioactivity is essentially due to uranium in equilibrium.
2. Samples in which equilibrium has been disturbed, with enrichment in uranium.
3. Samples in which radioactivity is essentially due to thorium. As stated previously, disturbance in equilibrium is not usual in thorium minerals.
4. Samples which may contain a mixture of thorium and uranium, or in which equilibrium has been disturbed, with impoverishment in uranium. The elementary methods to be described do not distinguish between these two cases.

In as much as the types of radiation used in assaying are not emitted by uranium and thorium, but come from the decay products, the results do not offer the same degree of certainty as do the results of accurate chemical assays, although their inherent accuracy may be of the same order. It is possible to envisage a combination of uranium not in equilibrium and thorium, which could lead to a sample being wrongly assigned to the first of the groups listed above. However, no other type of incorrect indication is likely to arise. The main justification of the methods described is that they have been used by the Bureau and many other organizations for several years, without any serious errors being encountered.

The process as described has been carried to a degree which can be achieved without any special precautions, and is sufficient for the requirements of those for whom the present notes are intended. This does not by any means exhaust the possibilities of radiometric methods of assaying, but special care, and in some cases special equipment, are required for any considerable increase in accuracy. One direction in which extra precision may be sought is discussed by Eichholz, Hilborn and McMahon (1953).

LABORATORY ASSAYING.

Introduction.—The practical aspects of radiometric assaying are described in some detail, with the object of providing the information which would be required by an organization, such as a mining company, wishing to establish a radiometric assay laboratory.

The method to be described consists basically of a comparison between the intensity of the beta and gamma radiation of an unknown sample and that of a standard radioactive sample (of known composition) under identical conditions. A measure of the intensity of radiation is obtained by the use of a detector which produces an electric pulse each time a beta particle or gamma photon passes into, and is absorbed within, the sensitive part of the detector. The number of pulses produced by the detector in a given time is counted electronically.

The number of counts per minute obtained from a sample will therefore depend on the efficiency of the detector and the geometrical relationship of the sample to the detector, hence the necessity to determine the number of counts per minute from a standard sample, using the same detector and geometry as for the sample.

The assay procedure is not difficult and requires no special scientific training, although considerable care is necessary to obtain reliable results. However, the maintenance of the electronic counting equipment and high-power voltage supplies requires the services of a skilled radio technician with special experience in pulse circuits.

The following description is considered in three sections, dealing with:—

1. The essential equipment required to set up a radiometric assay laboratory.
2. The assay technique adopted in the radiometric laboratory of the Bureau of Mineral Resources.
3. The interpretation of the assay results, with special reference to the criterion of radioactive equilibrium and the distinction between uranium and thorium.

Assay Equipment.

Introduction.—The complete assay process involves three distinct operations, namely, crushing the rock samples, preparing the pulverized samples for assaying, and assay counting and calculation of results. It is preferable that these three operations be carried out in separate rooms. In particular, it is essential to keep the rock-crushing equipment well separated from the assay laboratory, in order to prevent contamination of the assay equipment by radioactive dust. Contamination would increase the background count, and thus make the assaying of weakly radioactive samples more difficult.

Equipment for Preparation of Samples.—The requirements of the rock-crushing equipment are not peculiar to radiometric assaying, and suitable equipment is in general use in Mines Departments' laboratories and elsewhere. The most important features of a rock-crushing installation are:—

1. If many samples are to be handled, two motor-driven machines are necessary. First, a rock-cracking machine is required to reduce the rock samples to a size (about —8 mesh) which can be handled by the second machine. This is a pulverizing machine, which is required to reduce the cracked rock to a fine powder with a grain size of about —100 mesh.

2. The cracking and pulverizing machines should be designed so that all surfaces where pulverized material may settle can be easily cleaned. A compressed air supply is desirable for blowing all dust out of the machine between each crushing.
3. An ore sampler is required in the crushing room for quartering bulky samples down to a size convenient for storing and assaying. A final crushed sample of the order of 200 gm. is generally suitable.
4. Because of the large silica content of most rocks, the operator must be protected against silicosis of the lungs. If a large number of samples are to be handled, an efficient dust-extracting system is necessary in the rock-crushing room; otherwise, an efficient dust mask may be sufficient. In either case, advice should be obtained from Health Departments of the State or Territory concerned.

The only equipment required for the sample-preparation room is a small, accurate ore sampler to handle up to about 200 gm. of material, a balance or scales capable of weighing masses up to about 100 gm. with an accuracy of about 0.1 gm., and a supply of sample holders.

Two types of sample holder in use by the Bureau are shown on Plates 7 and 8. The annular type of holder shown on Plate 7 is used with the tubular type of thin-walled, Geiger tube. The important requirement in this case is that the thickness of the wall separating the sample from the detector should be as small as possible in order to minimize the absorption of beta radiation from the sample. Wall thicknesses of 0.001 inch of copper or 0.03 inch of a plastic material have been found to be suitable. The other type of sample holder shown on Plate 8 is used with end-window Geiger tubes and scintillation counters. This is a reversible piece of equipment, the shallow holder at one end being used for very small samples and the deep holder at the other end for larger samples.

Equipment for Sample Counting.—The principles of the design of the detectors and counters used in the assay room have already been described. However, some of the particular requirements for assay work are now mentioned briefly.

Detectors.—When a large range of uranium or thorium values is to be assayed, three types of detector are desirable, namely:—

1. A cylindrical beta-sensitive Geiger tube such as the B6 (20th Century Electronics). When used with an annular sample holder, this type of detector provides the most favourable geometry which can be achieved in a simple arrangement, and it is suitable, therefore, for assaying weakly active samples containing less than about 0.5 per cent. equivalent uranium oxide (eU_3O_8).
2. An end-window, beta-sensitive tube such as the GM4 (English General Electric Company) is suitable for assaying more active samples; using an end-window sample holder. This type of holder is more convenient to use, and the less favourable geometry is desirable for reducing the count rate (from very active samples) to a level which can be more readily handled by the Geiger tube and counting equipment.
3. The gamma count rate from samples containing more than 0.05 per cent. eU_3O_8 can be obtained with a Geiger tube by placing a beta absorber (about 1/16 inch of brass) between the sample and the tube. However, due to the low efficiency of the Geiger tube as a gamma ray detector, very long counting times are required to obtain the required statistical accuracy, and the method becomes impracticable for weakly

active samples. For this reason a scintillation detector, consisting of a sodium iodide crystal and photo-multiplier, is desirable for the gamma counting.

In each instance the detector should be mounted inside a lead castle, the wall thickness of which should be sufficient to absorb most of the local background gamma radiation and the soft cosmic radiation. A considerable reduction in background count can be achieved with a wall thickness of 1.5 to 2.0 inches, but because of the intense penetrating power of the hard cosmic gamma radiation, little additional improvement can be gained by increasing the wall thickness much beyond this figure.

The lead castle must be fitted with a rigid support for both the detector and the sample holder, so that consistent geometry between the sample and detector can be maintained.

Pulse Amplifiers.—The amplifier following a Geiger tube need have only a low gain (about 50), but provision is required for quenching the Geiger tube for a short period of about 300 to 500 microseconds immediately after each pulse. This is done by momentarily dropping the voltage across the tube to a value less than the threshold voltage. The reasons for this are:—

1. After a discharge in a Geiger tube, spurious pulses may be produced up to a few hundred microseconds later, by the emission of secondary electrons from the cathode. This is caused by a positive ion cloud drifting back from the centre wire. By momentarily dropping the voltage on the Geiger tube below the threshold, these secondary electrons cannot produce any pulses.
2. It has been stated earlier that Geiger tubes have a "dead time" of about 200 microseconds after each discharge. At high count-rates, the number of counts missed during this dead time is significant; but allowance can be made for the loss if the duration of the dead time is accurately known. The dead time of Geiger tubes is subject to considerable variation, however, and a more accurate correction to the observed count-rate can be made by making the quenching (paralysis) time, imposed by the amplifier circuit, longer than the natural dead time of the Geiger tube. This accurately known quenching time can then be used to make the correction.

More gain is required for the amplifier for a scintillation counter; but the requirements of the amplifier are not severe, as full advantage need not be taken of the speed of response of the scintillation counter. A gain of about 100 and a band-width of 1 to 2 megacycles is satisfactory.

Because the output impedance of scintillation counters and Geiger tubes is very high (usually about 10. megohms), long output leads would cause severe capacitative loading of the detector. It is therefore desirable to place the amplifiers as near as possible to the detectors. By using a cathode follower stage in the output of the amplifiers a large step-down in impedance is achieved, and long leads may be used between the amplifier and the counting circuit in order to obtain the most convenient layout for operating the equipment.

High-voltage Power Supplies.—The voltage required for the operation of Geiger tubes is generally between 1,000 and 1,500 volts. The high-voltage tubes are generally more stable than the halogen-quenched tubes, and are therefore more suitable for accurate assay work. The current drain imposed on the supply is negligible; but regulation of the output voltage to within 2 per cent. is desirable.

Somewhat higher voltages may be required for photo-multiplier tubes (up to 2,000 volts) and the current drawn by the detector may be as much as 0.5 milliamps. Regulation is even more important than for Geiger tubes and should be within 1 per cent.

The power supply should contain a built-in voltmeter across the output, and coarse and fine control of the output is desirable.

Scaling Equipment.—Since the output pulse rate of assay detectors is generally much too high to be handled by a mechanical register, it must be either counted electronically or scaled down electronically to a rate which can be handled by a mechanical device. The choice of such a scaler is determined mainly by the highest count rate to be recorded. In general, the cost and size of scaling equipment becomes progressively greater as the resolving time becomes smaller. For use with Geiger tube detectors, a scaler with a resolving time of the same order as the natural dead time of the tube is adequate, and counters based on cold-cathode, decade scaling tubes such as the Dekatron (resolving time 250 microseconds) and the Nomotron (resolving time less than 100 microseconds) are satisfactory.

In recent years, much progress has been made in the development of several types of vacuum decade counting tubes with resolving times considerably less than those possible in gas-filled tubes. Of these, the only one commercially available at present is the Philips E.I.T., which has a resolving time of about 30 microseconds. A resolving time of one microsecond is claimed for the Trochotron, which is now under development in Sweden.

All the decade counting tubes referred to above provide a visible indication of the number of counts received, and if sufficient stages are used in the scaler, no mechanical register is required.

At present, the only type of scaler which is suitable for handling very high count rates is one based on the scale-of-two, flip-flop, multivibrator circuit. Resolving times of about one microsecond may be obtained, but the circuitry involved makes such counters very bulky and expensive. However, when a scintillation detector is used, the count rates for samples containing more than 0.1 per cent. eU_3O_8 are high enough to be measured with sufficient accuracy by a well designed and accurately calibrated laboratory ratemeter.

A discriminator circuit is usually included in the input stage of the scaling unit or ratemeter, and a control for the bias setting is provided on the front panel of the unit.

Assay Procedure.

Setting up the Equipment.—Before accurate assays can be made, a set of standard samples must be prepared and the equipment adjusted to obtain the most stable operating conditions. When suitable supplies of aged pitchblende are obtained from overseas it is hoped that the Bureau will be able to supply uranium standards to organizations wishing to do their own radiometric assaying. At present, such standards can be obtained only from overseas.

It is desirable to use a set of uranium standards covering the expected range of assay values. For values usually encountered in ore assays, the following range, expressed as percentage eU_3O_8 , should suffice—0.01, 0.1, 0.5, 1.0, and 5.0. Actually, the calibration curve for the detector will be very nearly a straight line, so no great error is likely to be introduced by using only one standard, e.g., 1.0 per cent. However, any calibration errors which do occur can be minimized by always choosing a standard which has about the same value as the sample to be assayed.

The most convenient method of handling the standards is to fill a set of each type of sample holder to be used, with known amounts of the various standards. Provided the holders are reasonably well-sealed, the standards can be permanently kept in this way, without danger of equilibrium being disturbed.

The mass of each standard contained in the holders should be found, and it is also desirable to keep the weights of the standards as uniform as possible. Control over the weight of sample filling the holders is obtained by varying the tightness of packing.

To determine the correct operating voltage and the condition of a Geiger tube, it is desirable to determine the characteristic curve by increasing the anode voltage (from below threshold) in steps of about 20 volts and observing the count rate produced by a fairly active source at each voltage setting. The usual shape of the curve so obtained is shown in Fig. 1. For a tube in good condition, a plateau length of about 200 volts and a plateau slope of about 3 per cent. per 100 volts can be expected. The life expectancy of most high-voltage Geiger tubes is about 10^8 counts. When finding the plateau curve, it is important to avoid raising the anode voltage above the value found at the end of the plateau. Too high a voltage will destroy the tube.

The correct operating voltage for a photomultiplier tube must be found in a similar manner. However, as the scintillation counter is a proportional detector, the bias setting of the discriminator also has a considerable effect on the count rate. A typical set of curves showing count rate versus E.H.T., for different bias values is shown in Fig. 10. The best operating voltage and bias setting must

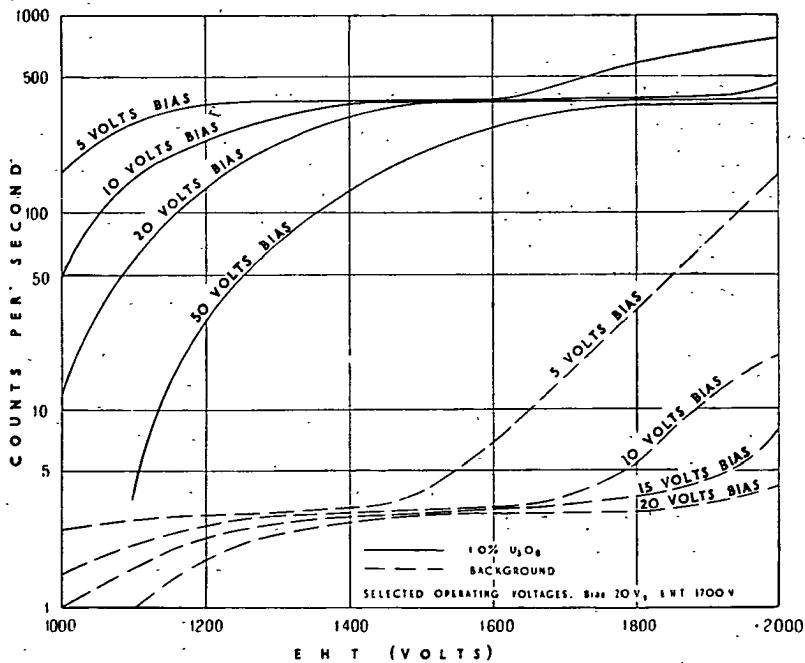


Fig. 10. Voltage characteristics for E.M.I. photomultiplier tube, Type 6260, with sodium iodide crystal.

be a compromise between high counting efficiency on a "flat" part of the curve and low background count. The background count is due to photomultiplier noise (dark current), stray local radiations and cosmic radiation.

Preparation of Samples.—Before pulverizing each rock sample, it is desirable to obtain a rough estimate of the activity. This can be done quickly, using a beta-sensitive Geiger tube and a ratemeter. This preliminary test is a useful guide for selecting the most suitable detector and sample holder. When pulverizing a batch of samples, it is also useful to know which are the more active ones, so that these may be pulverized last, thereby minimizing inter-contamination of the samples. Cleaning of the cracker and pulverizer, after very active samples have been put through, is particularly important. A convenient method of cleaning the plates of the pulverizer is to pass through a quantity of clean, inactive sand.

Careful sampling of the powdered material is very important, in order to obtain a representative sample for assaying. Care is also required in filling the sample holder in order to ensure even packing of the material. The mass of the sample is obtained by weighing the sample holders before and after filling.

The choice of sample holder depends on the activity of the sample and the type of detector to be used. Using a scintillation detector for the gamma count, samples with activity greater than about 0.1 per cent. eU_3O_8 can be assayed conveniently in the deep, end-window holder. For samples with activity less than 0.1 per cent. eU_3O_8 , it is necessary to use the annular holder for the beta count..

Sample Counting.—Assay counting consists essentially of observing the count rate of the background, the sample and the standard; the net count rate for the sample and standard being obtained by subtracting the background count. However, there are several sources of error involved in obtaining the true relative count rates of the sample and standard. Some of these errors can be reduced by taking certain precautions in the counting procedure, and corrections can be applied for others. The five main sources of error are:

1. *Statistical Fluctuations.*—Because of the random nature of the radiation, the count rate observed over a short period may differ greatly from the average count rate. The counting time required for a given statistical accuracy in the net sample count rate depends, therefore, on the count and the excess of the sample count above background. A useful nomogram, giving the counting time required for 5.0 per cent. accuracy, for different samples and background count rates, is shown in Fig. 11 (Davidon, 1953).

The counting time varies inversely as the square of the accuracy required. Thus, to obtain accuracy of 2.5 per cent., the times shown by the nomogram should be multiplied by four. When the background count is small compared with the sample counts, two or three observations of the background per day are sufficient, a counting time of about ten minutes being adequate. For very weak samples, a series of alternate counts of sample, background and standard is desirable.

2. *Variations in Detector Sensitivity.*—Even when the high-tension supply is well regulated, appreciable variation in the output of a Geiger tube, exposed to a constant source, has been observed during the course of a day. It is desirable, therefore, to compare the count rates of sample and standard within as short a period as possible. When long counting times are involved, it is preferable to take short counts of the sample and standard alternately, continuing the sequence long enough to obtain the required aggregate counting time. This method is also a safeguard against drift in photomultiplier outputs.

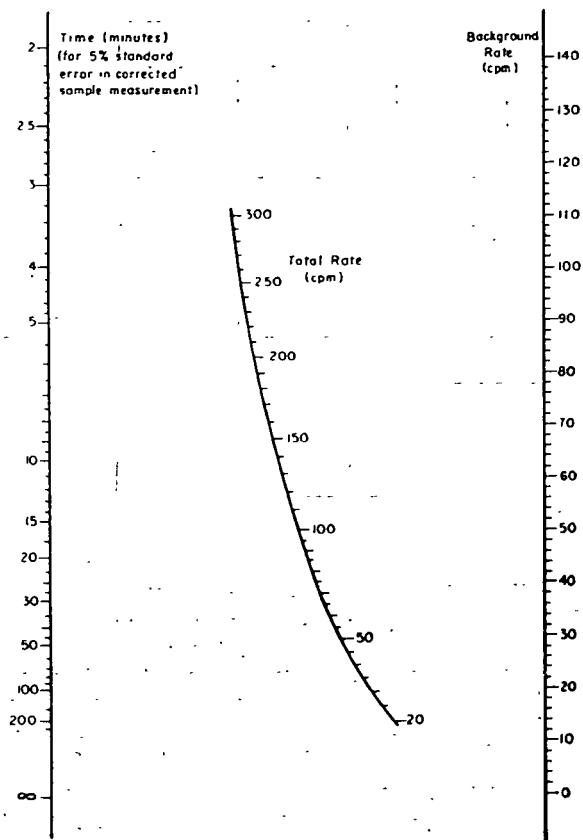


Fig. 11. Nomogram for counting times.

3. *Instrumental Faults.*—The practice of taking several counts on each sample also provides a safeguard against the less obvious instrument faults which may produce a greater variation in the several count rates than would be expected from a purely statistical variation.
4. *Paralysis Losses.*—At high count rates it is necessary to make a correction to the observed count rate for pulses missed during the resolving (paralysis) time of the detector. Provided the average pulse interval is large compared with the paralysis time, the "true" count rate can be found with sufficient accuracy by means of the simple equation:

$$N = \frac{n}{1 - nt},$$

where N = true count rate
 n = observed count rate
 t = paralysis time.

It will be found more convenient, however, to use the paralysis time correction curves given in Fig. 12.

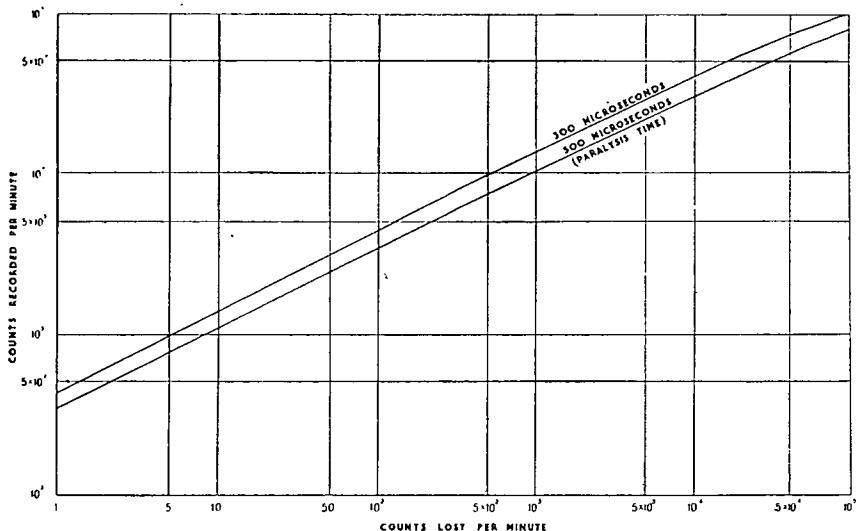


Fig. 12. Correction curve for paralysis time losses.

5. *Self-absorption of the Sample.*—A correction must also be applied to the observed count rate to allow for the effect of any difference in density between the sample and the standard. Much of the beta radiation and some of the gamma radiation is absorbed within the sample itself, the absorption being roughly proportional to the sample density, in the case of gamma absorption. Since sample holders of the one type all have the same volume, it is more convenient to consider this correction in terms of the mass of the sample, which depends not only on the density of the original rock specimen, but also on the tightness of packing of the powdered material in the holder.

Correction curves have been obtained experimentally in the Bureau's laboratory for the annular and deep end-window holders (see Figs. 13 and 14). These curves were obtained by preparing several 1·0 per cent. eU_3O_8 samples, using filling materials of different density. The curves show that the beta count is almost independent of the mass of the sample, the gamma count on the other hand being roughly proportional to the mass. This is due to the range of the beta particles in the sample being short compared with the thickness of the sample, and the range of the gamma particles being much greater than the sample thickness. The corrected count rate, R_c , for a sample of mass M_1 is found by multiplying the observed count rate, R_o , by the factor C_2/C_1 , where:—

C_1 = count rate for a 1 per cent. sample of mass M_1 .

$$C_2 = \dots \dots \dots \dots \dots \dots \dots \dots \dots M_2$$

M_s = mass of standard.

C_1 and C_2 are found from the correction curve.

Gamma Counting with a Geiger Tube.—When a scintillation counter is not available, the gamma counting must be done with a Geiger tube by placing an absorber between tube and sample. If a suitable thickness is chosen for the absorber, all the beta radiation can be absorbed, without causing any appreciable reduction in the gamma radiation. It has been found in the Bureau's laboratory

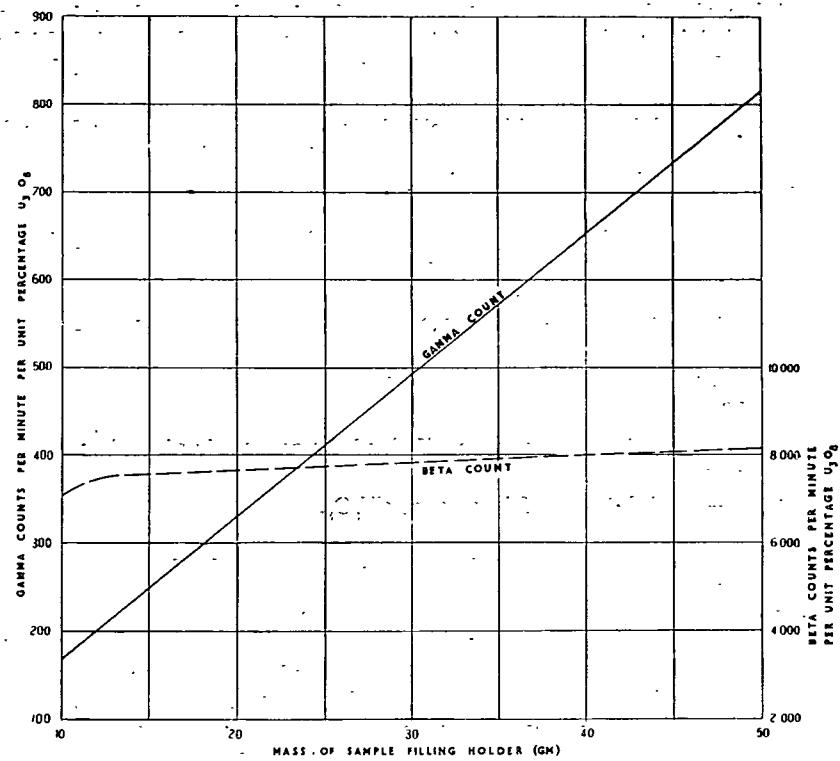


Fig. 13. Count rate correction curves for sample self-absorption, using annular holder (13.0 c.c.).

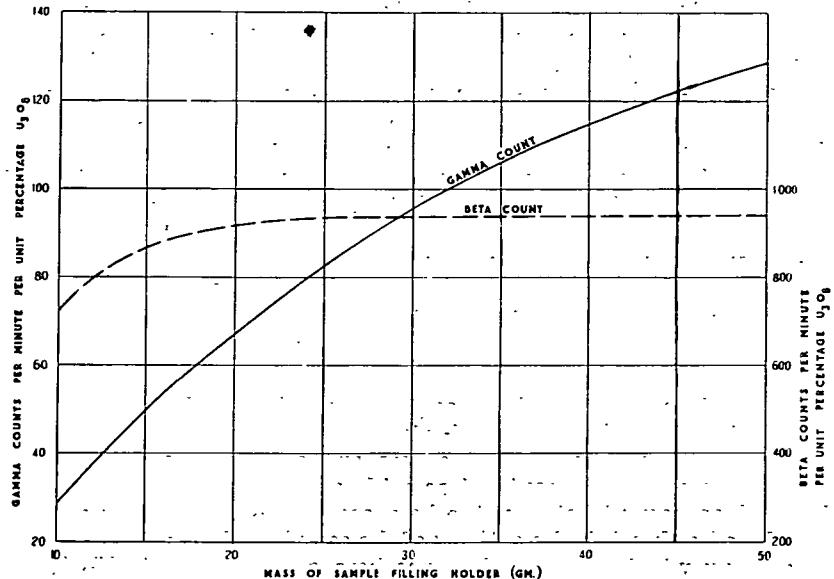


Fig. 14. Count rate correction curves for sample self-absorption, using deep end-window holder (13.0 c.c.).

that a suitable absorber for the B6 Geiger tube is a brass tube with a wall thickness of 1/16 inch. For the GM4 tube, a brass plate of 1/32-in. thickness provides a suitable beta absorber. A drawing of the annular absorber is shown on Plate 7.

Interpretation of Results.—When all the necessary corrections have been applied to the observed count rates, the calculation of the percentage eU_3O_8 content of the sample is a matter of simple proportion, e.g.:—

$$\text{Beta count of sample} = 478 \text{ counts per minute.}$$

$$\text{Beta count of standard (0.1 per cent. } \text{eU}_3\text{O}_8) = 196 \text{ counts per minute.}$$

$$\text{Gamma count of sample} = 4,900 \text{ counts per minute.}$$

$$\text{Gamma count of standard} = 2,080 \text{ counts per minute.}$$

By the beta method, the assay value (U_B) of the sample is given by:—

$$U_B = \frac{478}{196} \times 0.1 = 0.24 \text{ per cent. } \text{eU}_3\text{O}_8.$$

By the gamma method, the assay value (U_G) of the sample is given by:—

$$U_G = \frac{4,900}{2,080} \times 0.1 = 0.24 \text{ per cent } \text{eU}_3\text{O}_8.$$

In practice, the beta count obtained with a Geiger tube is really a beta + gamma count. However, as the tube is much less efficient as a gamma counter than as a beta counter, the gamma contribution is relatively small, and little error is introduced by using the combined count in the calculations. If desired, however, the true beta count can be obtained by subtracting the gamma count found by the use of a beta absorber, as described in the previous section.

The practice of expressing the results in terms of equivalent U_3O_8 is adopted because it may not always be certain that the activity is due to uranium. In fact, all that is claimed for the result is that the observed activity of the sample is equivalent to that which would be produced by a certain percentage (0.24 in the above example) of pitchblende (U_3O_8). It is shown later, however, that in some instances it can be determined with reasonable certainty whether or not the activity is due entirely to uranium.

It has been found in the Bureau's laboratory that, in general, radiometric assays can be repeated to within ± 5 per cent., without taking any extreme precautions. The main source of error appears to be in the sampling of the pulverized material. In a few instances, larger sampling errors have been observed, probably due to uneven distribution of the uranium in the original rock specimen.

Uneven distribution of uranium in the assay sample can also cause discrepancies between beta and gamma assays, as the beta assay, in effect, indicates the percentage of uranium in the top few millimetres of the sample, whereas the gamma assay gives the percentage of uranium in the whole of the sample.

Figures for the percentage eU_3O_8 content are of little value unless there is a means of finding out whether the activity is due to uranium or thorium. For this reason, it is necessary to find both the beta and gamma assays. If the assay sample contains uranium in equilibrium with all its daughter products, there should be good agreement between the beta assay (U_B), and the gamma assay (U_G). On the other hand, if the activity of the sample is due entirely to thorium, the gamma assay will be greater than the beta assay, the ratio U_B/U_G being approximately 0.55, if a scintillation counter is used for the gamma count. If a

Geiger tube is used for both the beta and gamma counts this ratio is reduced to about 0.5. The reason for the different values of the U_B/U_G ratio is probably that some of the softer gamma radiation is not counted by the scintillation counter, which has been used with a discriminator setting of 20 volts bias.

If the presence of thorium is established in this way, then the percentage of thorium expressed as ThO_2 can be found by multiplying the percentage eU_3O_8 (found by the beta count) by the factor 4.0. This will give a reliable figure for the percentage of thorium, but it can always be checked by comparing the sample with an accurate thorium oxide standard sample.

The ratio U_B/U_G is also the criterion used to determine whether or not a uranium sample is in equilibrium. If equilibrium has been disturbed, large errors may occur in the beta and gamma assays. Table 1 shows that the uranium series consists of two main groups which will be referred to as the uranium group (U I to Io) and the radium group (Ra to RaF). The radium group is responsible for nearly all the effective gamma radiation and each group provides approximately equal amounts of the effective beta radiation. Effective radiation means radiation of sufficient energy to be detected by the assay equipment. The radium group is therefore the major source of radiation in the uranium series, and any natural process such as selective leaching of uranium or loss of the gaseous member of the radium group (radon) which can alter the relative amounts of the two groups of elements can cause serious errors in the radiometric assays.

Instances of extreme disequilibrium in natural deposits of uranium are rare, but it is possible for the ratio U_B/U_G to range between about 5.0 and 0.56. Very few samples have been assayed in the Bureau's laboratory with a U_B/U_G ratio greater than 2.0. The effect of disequilibrium on the accuracy of the beta and

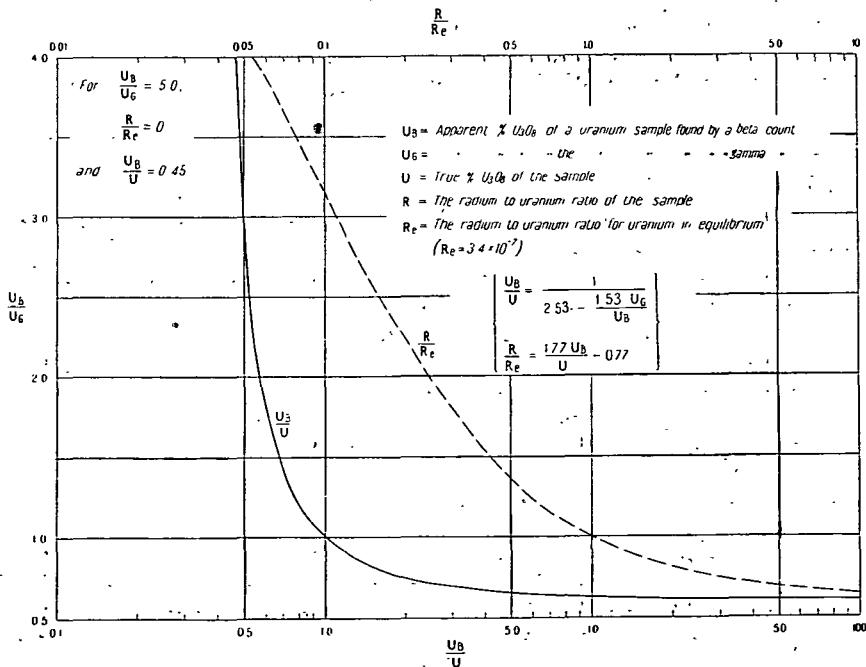


Fig. 15. Curves showing $\frac{U_B}{U}$ and $\frac{R}{R_e}$ as functions of $\frac{U_B}{U_G}$ for uranium samples.

gamma assays has been investigated theoretically and is shown in Figs. 15 and 16. The ratio of the apparent uranium percentages determined by the radiometric assays to the true uranium percentage is shown as a function of U_B/U_G . The radium to uranium ratios corresponding to the various values of U_B/U_G are also shown in Fig. 15.

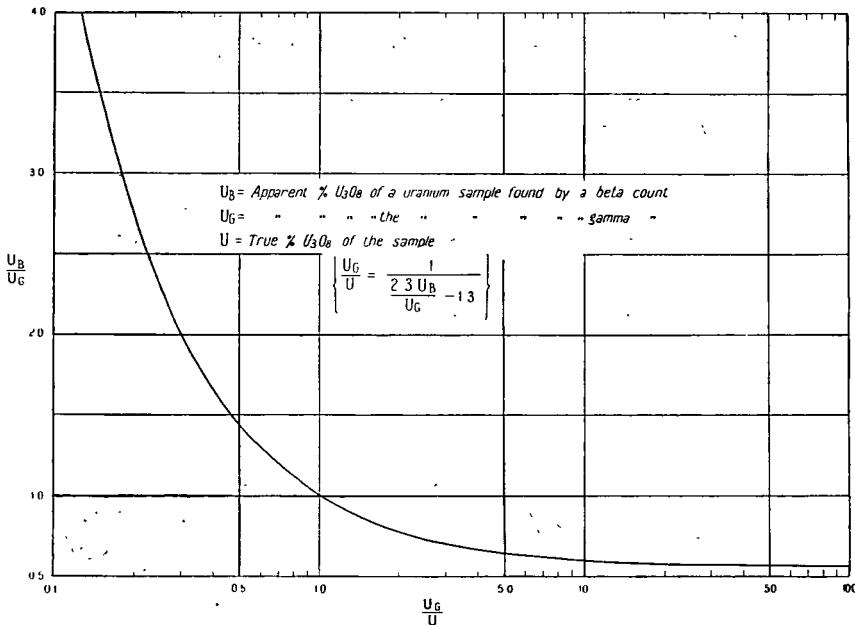


Fig 16. Curve showing $\frac{U_G}{U}$ as a function of $\frac{U_B}{U_G}$ for uranium samples.

The following conclusions can be made from an examination of these curves:

1. For values of U_B/U_G greater than 1.0, no really serious errors are produced in the beta assays, but the gamma assays are greatly affected.
2. For values of U_B/U_G less than 1.0, the assay errors rapidly become very great in both instances, and a more positive test for uranium should be used, e.g., fluorimetric assay.
3. When the equilibrium is disturbed in such a way as to result in a large excess of radium relative to uranium, the value of U_B/U_G approaches that for thorium. In such rare cases, therefore, a uranium sample (essentially radium) may be mistaken for thorium. In either case, however, the assumption that the sample is deficient in uranium is valid.
4. The curves provide a means of correcting the radiometric assays for the errors caused by a disturbance in equilibrium, at least for high values of U_B/U_G . However, where there are errors of the order of ± 5.0 per cent. in the beta and gamma assays, the errors in the observed values for U_B/U_G will be ± 10.0 per cent. No great improvement can therefore be expected from applying this correction, except in instances of extreme disequilibrium. However, if precautions are taken to reduce other errors in the assay procedure this method of correcting for disequilibrium could be valuable, and it has, in fact, been adopted in Canada and elsewhere (Eichholz et al, 1953).

The importance of obtaining extremely accurate ore assays, however, must be considered in relation to the larger errors which may occur in field and mine sampling. Uranium is frequently found in association with thorium, and this further complicates the interpretation of the assay results. The effect of the presence of thorium in a uranium assay sample, on the value of U_B/U_G and the accuracy of the uranium assay, has been calculated, and is shown in Fig. 17. These

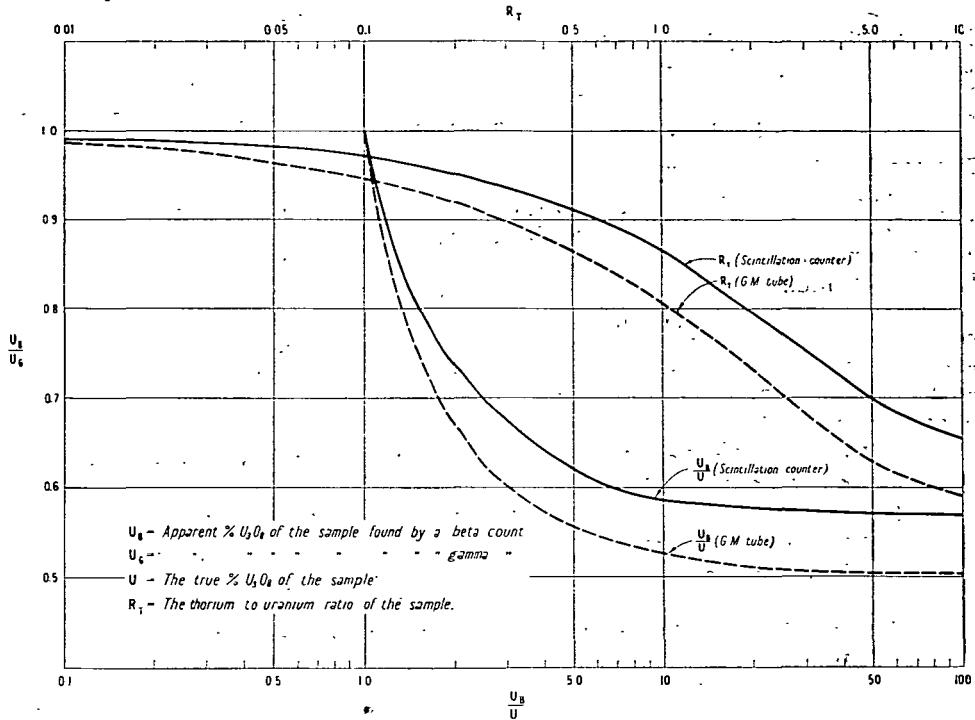


Fig. 17. Curves showing $\frac{U_B}{U}$ and R_T as functions of $\frac{U_B}{U_G}$ for uranium, thorium samples.

curves are based on the assumption that the uranium is in equilibrium and, if the ratio U_B/U_G could be determined with sufficient accuracy, they would enable a rough estimate to be made of the percentage of both uranium and thorium present in the sample. If the uranium is not in equilibrium, there is no unique solution to the problem. However, calculations have shown that, even in the most unfavourable combination of radium:thorium and thorium:uranium ratios, the error due to this cause is not greater than about 20 per cent. and is such as to make the calculated percentage of U_3O_8 less than it should be. The possibility of such an occurrence must, in any case, be remote.

The interpretation of radiometric assays can be summarized as follows:—

1. $U_B/U_G = 1$. If the value of U_B/U_G is between 0.9 and 1.1, it can be assumed that the sample contains uranium, virtually in equilibrium, and the beta assay can be accepted with confidence.
2. U_B/U_G greater than 1.1. In this instance the beta assay is not likely to be greatly in error, but may tend to be low.
3. $U_B/U_G = 0.5$. If U_B/U_G is between 0.5 and 0.6 for a scintillation counter assay, or between 0.45 and 0.55 for a Geiger tube assay, the activity is almost always due to thorium, but in very rare instances may be due to radium. In either case the sample will contain little or no uranium.

4. U_B/U_G between 0.6 and 0.9. In this instance the activity may be due to the uranium family, deficient in uranium, or to a mixture of uranium and thorium. The beta and gamma assays may be completely misleading (tending to be high) and it is essential to use another method for the uranium assay, e.g., a straight chemical analysis, fluorimetric assay or colorimetric analysis.

Three typical assay calculations are shown at the end of this report. Sample No. PR 55/17 contains uranium in equilibrium, PR 55/8 and PR 54/174 appear to contain a mixture of uranium and thorium. A Geiger tube was used for both the beta and gamma assays of PR 54/174, and a scintillation counter was used for the gamma assay of the other two samples.

RADIOMETRIC FIELD ASSAYING.

It is often desirable to obtain a rough "on-the-spot" assay of rock samples, in the field. This calls for assay equipment which is light and simple to operate. A high standard of accuracy is not essential, but a method of distinguishing between uranium and thorium is most desirable.

To meet these requirements, a simple battery-operated Geiger tube ratemeter for field assaying has been designed by the Bureau. The circuit diagram is shown on Plate 5, from which it can be seen that the instrument is basically similar to many survey ratemeters, but has been modified to suit the requirements of assay work in the following ways:—

1. The range has been increased to cope with the higher count rates which can be expected in assay work.
2. Long time-constants are provided in the counting circuit.
3. Metering of the filament, H.T. and E.H.T. voltages is provided. The E.H.T. voltage is continuously monitored, so that any change in the Geiger tube voltage will be quickly observed.
4. Controls for adjusting the H.T. and E.H.T. are provided on the front panel.
5. The Geiger tube is mounted vertically at the front of the unit so that annular type sample holders can be easily placed over the tube.
6. A beta-sensitive, Geiger tube is required. The 20th Century Electronic B6H or B12H low voltage, Geiger tubes are suitable.

A suitable sample holder for use with the B6H tube has been designed and is shown on Plate 7. The length of this holder should be doubled for use with the B12H tube. A brass tube with a wall thickness of 1/16 inch provides a suitable beta-ray absorber. A drawing of an absorber for the B6H tube is also shown on Plate 7.

Before the ratemeter is used it must be carefully calibrated on all ranges. This is most conveniently done by means of a pulse generator with an accurate and variable pulse repetition rate.

Although the samples need not be reduced to the same grain size as for laboratory work, some means of pulverizing to about —25 mesh is required. Sufficient quantity of a standard U_3O_8 sample (e.g., 1.0 per cent.) is required to fill one of the sample holders.

The assays should be made in the same way as described in the laboratory method. That is, count rates are obtained for background, sample and standard, with and without the absorber. The corrections for paralysis may be made on the assumption that the Geiger tube dead time is about 250 microseconds. A density correction for the gamma count can be obtained by assuming the correction to be proportional to the mass of the sample relative to the standard.

Although the equipment has not yet been used very much, tests which have been made indicate that an accuracy of the order of 20 per cent. may be expected; and the discrimination between uranium and thorium, as provided by the absorption test, is satisfactory. The following precautions need to be taken when using this equipment:—

1. A careful check must be kept on the E.H.T. voltage when each count is made. The voltage may drop slightly at high count rates and an adjustment may be necessary to maintain the correct operating voltage.
2. The tube should be shielded from strong light while counts are being taken, as some low-voltage Geiger tubes are photo-sensitive.
3. A reading time of about five minutes is required on the lowest range of the ratemeter, because the time constant of the tank circuit is so long that the instrument takes about five minutes to reach its final reading.

TESTING OF HAND SPECIMENS.

It is common for those possessing portable Geiger counters to endeavour to use them for assay purposes at least to the extent of determining which of two hand samples of rock is more strongly radioactive. Very little experience is necessary to prove that such instruments are quite insensitive when used in this way. However, it is desirable that the limitations of this method of assaying be fully realized, otherwise hopelessly misleading results may be obtained.

Most portable instruments detect gamma rays only. As gamma radiation is relatively penetrating, the reading obtained from a particular sample is greatly influenced by the size of the sample. This is often a source of disappointment to prospectors inexperienced in the use of the Geiger counter, as they find that although an outcrop may give a reading of several times background on a portable counter, a hand specimen of the same rock causes no detectable increase in count rate. Similarly, the indication obtained on a hand specimen from equipment detecting gamma rays only is so much dependent on the size and shape of the sample that it is quite impossible to use such equipment to obtain even a rough estimate of the grade of material.

Beta counting equipment has considerable advantages for this type of work. Due to the higher efficiency of the Geiger tube as a beta counter, the beta counting equipment is considerably more sensitive. Also, as beta radiation is much more easily absorbed than gamma radiation, it is relatively easy to confine the test to a small area of the sample and shield the tube from the radiation emitted by the remainder. However, the fact that beta counting is essentially a surface effect renders such a test unreliable as an estimate of the activity of the bulk of the sample. It is frequently found that secondary radioactive minerals are deposited along cracks in the sample. When fractured, the rock tends to break along such cracks, thus exposing a surface which is considerably more radioactive than the bulk of the sample. For this reason beta counting tests on samples, especially those containing secondary minerals, frequently give much too high a value for the uranium content of the bulk of the rock.

The most convenient type of equipment for testing hand specimens uses a General Electric type GM4 Geiger tube. This is a beta tube of the end-window type, and is mounted in a metal probe, with a grille covering the thin end window of the tube. The hand sample is held against the grille. Provided the sample is rather larger than the area of the grille, and not less than one-quarter of an inch thick, the conditions approach those of a sample of infinite thickness and infinite size, as far as beta radiation is concerned, and all samples fulfilling these

conditions may be considered as being tested with the same geometry. The count rates are read on a mains-operated ratemeter, such as the Austronic Beta Gamma Monitor type BGR/1, or the General Purpose Ratemetér manufactured by E. K. Cole Ltd. These instruments include power supply and ratemeter, and only require the attachment of the Geiger tube.

As an indication of the accuracy obtainable, Fig. 18 shows the results of a number of tests on samples which were later assayed by standard radiometric methods. It will be observed that the scatter of the points is considerable, and that this method of testing can be relied on to give a rough indication only.

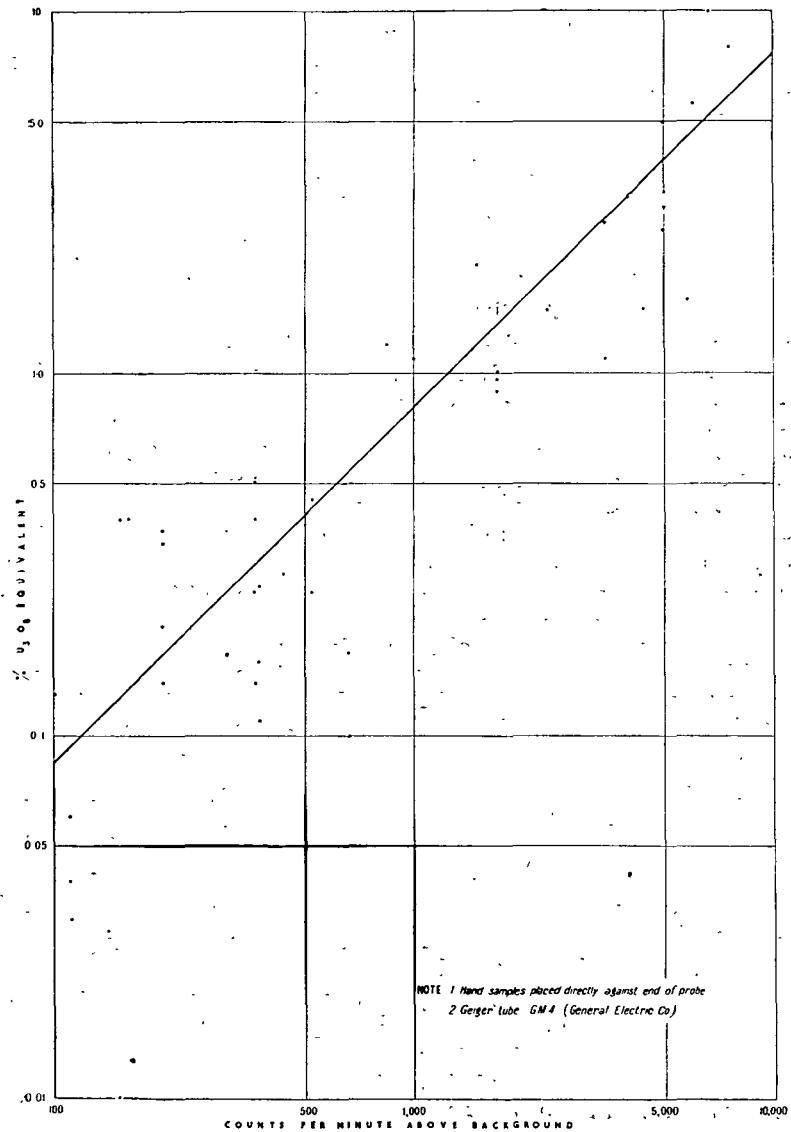


Fig. 18. Approximate calibration of BGR/1 ratemeter for hand samples.

FLUORIMETRIC ASSAYING.

INTRODUCTION.

When a substance is fused with sodium fluoride under suitable conditions, and the fused bead is examined under ultra-violet light, the presence of uranium, even in traces, is revealed by a characteristic green fluorescence. This fact has been utilized for a long time as a quick test for uranium. The capability of the photomultiplier tube to detect light fluxes of very low intensity has made possible the development of a very sensitive and accurate method of assaying for uranium, based on fluorescence. Details of this method are discussed below.

This discussion is based on several overseas reports, which are listed in the bibliography, and on a considerable amount of experience gained by the Bureau. It appears that a considerable personal element is involved in the technique, and some difficulty has been experienced in reconciling some of the statements made by various authorities. The assaying should be in the charge of a competent chemist, who can develop his own technique, based on available information.

The theory of fluorescence is beyond the scope of this report, and is, in fact, still obscure in many respects. It has been established, however, that the intensity of the fluorescent light emitted is directly proportional to the uranium content over quite a wide range, for beads fused under the same conditions. This fact provides a basis for an assaying method.

The process of fluorimetric assaying is to take the sample into solution as a nitrate, fuse a known amount of solution with a suitable flux in a standard dish, irradiate the resulting disc with ultra-violet light, and compare the intensity of the fluorescent light with that emitted under the same conditions by a similar disc containing a known amount of uranium. A difficulty arises, because several other elements quench the uranium fluorescence. The mechanism of quenching is not completely understood, though it appears that some of the effect is due to a shift of the wavelength of the fluorescent light, thus altering the efficiency of the photomultiplier tube. The quenching effect depends on the actual amount of the quenching element present in the final disc, relative to the amount of flux used. For samples rich in uranium, the effect may be overcome by diluting the sample, so that the final concentrations of quenching elements are below the tolerance. For samples very low in uranium this cannot be done. In such instances the uranium is extracted from the solution by a chemical process, leaving behind all the quenching elements. The presence of quenching in a sample may be checked by a process known as "spiking," which involves testing the original sample, then adding a known quantity of uranium and repeating the test. If the difference between the two readings is less than that which should be caused by the amount of uranium added, it can be assumed that the discrepancy is due to quenching, and the effect may be corrected for on this basis.

At present, acid treatment is used in the decomposition of rock samples in the Bureau's laboratory, but fusion methods have been employed elsewhere. It has been the experience here that the acid treatment method is more reliable. However, it is proposed to make further investigations into the fusion technique, and if this can be perfected, a considerable saving in time will be achieved.

The procedure to be described requires the services of a skilled inorganic chemist. If many samples are to be handled, a staff of at least five (e.g., three chemists and two laboratory assistants) is required to make efficient use of one fluorimeter.

ASSAY EQUIPMENT.

There is insufficient space in this report to describe in detail all the equipment which is required for fluorimetric assaying. The major items are an accurate balance, an electric muffle furnace and a fluorimeter. The fluorimeter (Type 1080A) at present in use in the Bureau's laboratory was designed at the Atomic Energy Research Establishment, Harwell, England. It is a reflection type of instrument, the major components of which are:—

1. *Sample Holder.*—The platinum sample dishes are held in six holes in a rectangular slide, which is fitted into the base of the instrument. The slide may be locked in any one of the six reading positions. When the slide is pushed into the instrument, the dish in the selected reading position is located at the junction of the optical axes of the ultra-violet and visible light systems.
2. *The Ultra-violet Light System.*—The source of the ultra-violet light is a quartz tube, mercury vapour, arc lamp. This is followed by a system of quartz lenses and filters which project a uniformly illuminated disc of light of wavelength 3650A onto the sample dish.
3. *The Visible Light System.*—A second system of lenses and filters projects a uniform disc of fluorescent light (in the region 5520A to 5680A) from the sample dish onto the cathode of a photomultiplier tube. The filters are required to remove any reflected ultra-violet light.
4. *Detector.*—The detector consists of a photomultiplier tube followed by an amplifier and vacuum tube voltmeter. As the ultra-violet lamp is excited by 50 cps. A.C. power, the light is modulated at 100 cps. An A.C. amplifier can therefore be used. A filter network is incorporated into the amplifier so that the response is a maximum at 100 cps. and falls off rapidly above and below this frequency. The effect of spurious signals is thereby greatly reduced. The output meter is a moving coil rectifier type with a full-scale deflection for 25 volts R.M.S.

The sensitivity of the detector can be varied by switching the anode load of the photomultiplier tube in steps of powers of ten, between 10 ohms and 10 megohms, the output meter deflections thus being multiplied by factors of 10^5 to 1.

5. *Power Supply.*—Two separate mains-operated power supplies are required for this instrument. One supplies power for the 100 cps. amplifier and the mercury lamp, and the other supplies regulated high voltage power for the photomultiplier tube.

ASSAY PROCEDURE.

Introduction.—The procedure adopted in the Bureau's laboratory will be described in some detail. Twelve separate steps are given, in the order in which they should be carried out. Other subsidiary information, such as the preparation of salting agent and fusion mixture, is given separately, later in the bulletin. The procedure involves the following four main stages:—

- Stage 1.*—Decomposition of the rock samples (steps 1 to 7).
- Stage 2.*—Extraction of the uranium (steps 8 and 9).
- Stage 3.*—Fusion of the uranium with a sodium fluoride flux to form a fluorescent disc (step 10).
- Stage 4.*—Comparison of the fluorescence of the "unknown" disc with that of a standard disc, and calculation of the uranium content of the former (steps 11 and 12).

With the equipment and staff available, it has been found convenient to assay the samples in batches of eight. At present, one analyst takes about four days to assay each batch, most of this time being consumed in decomposition of the sample.

Procedure.

Step 1.—If a radiometric assay has not previously been made, the material to be assayed is reduced to grain size of less than -100 mesh.

Step 2.—The amount of sample to be treated depends on the expected uranium content, as determined previously by radiometric assay. When the ratio U_B/U_G is less than one, the curves shown on Fig. 17 can be used to make a rough estimate of the true uranium content of the sample. The amount to be treated can then be chosen so as to obtain the desired amount of uranium in the final phosphor disc, after allowance has been made for the dilution factor used later in the procedure. It is desirable to use as high a dilution factor as possible (e.g., 100,000:1), but it may have to be reduced for weak samples, as it is also desirable to avoid having to treat a large mass of sample. The weight of the sample should not exceed 5 gm. or be less than 0.5 gm. The choice of sample size and dilution factor is therefore a matter of compromise, e.g.:—

If a sample is expected to contain 1.0 per cent. U_3O_8 , and it is desired to obtain a phosphor disc containing 0.1 micrograms of uranium, a convenient sample size would be 1.0 gm. This would enable a dilution factor of 100,000:1 to be used. The sample to be treated should be weighed to an accuracy of better than ± 1.0 per cent.

Step 3.—After weighing, the sample is transferred from the weighing bottle to a 250-ml. beaker. It is then moistened with about 1.0 ml. of water, and 10 ml. of concentrated HCl are added. The beaker is covered with a watch glass and the sample digested for five minutes on a hot plate. The drops of solution condensed on the walls of the beaker and on the cover glass are washed into the solution with a jet of 1.0 per cent. HNO_3 from a wash bottle, 5 ml. of concentrated HNO_3 and 5 ml. of concentrated (1 + 1) H_2SO_4 added. The uncovered beaker is replaced on the hot plate and heated at 70° C. until the HNO_3 and HCl are fumed off. If, at this stage, the solution is heated to a temperature greater than 70° C. it tends to spit as the last traces of HNO_3 and HCl are driven off. The temperature is then increased to approximately 100° C. and maintained until almost all the SO_3 is fumed off. When the residue is almost dry, frequent stirring is necessary to prevent bumping and the tendency of the residue to form a caked, hard mass. The residue is then strongly heated on the hot plate to drive off the last traces of SO_3 . Droplets of H_2SO_4 adhering to the walls of the beaker are removed by flicking a bunsen flame around the sides of the beaker.

Step 4.—To the dry, powdered, residue 5 ml. of concentrated HNO_3 and 50 ml. of water are added and the mixture boiled on the hot plate for five minutes. Bumping is avoided by placing a small piece of filter paper in the solution under the tip of the stirring rod. The solution is then filtered, while hot, through a buckner funnel with a sintered glass plate, equivalent to a No. 1 Whatman filter paper. The residue is washed five or six times with 1.0 per cent. HNO_3 . If this residue is white it is discarded. Sufficient HNO_3 is added so that when the solution is made up to the mark it is 29.0 per cent. HNO_3 .

Step 5.—If the residue is not white and does not contain any elements or compounds which will react with platinum it is washed into a platinum crucible and evaporated to dryness on the water bath. Two ml. of H_2F_2 and 1-2 drops of concentrated H_2SO_4 are added. This solution is evaporated to dryness on the

water bath. If the residue is not decomposed this treatment is repeated. Any undissolved material is then fused with a small amount of Na_2CO_3 . To this final residue, 1 ml. of concentrated HNO_3 and 5 ml. of water are added and the sample is digested on the water bath for ten minutes. This solution is then added to the bulk solution in the standard flask and the solution made up to the mark as in step 4.

Step 6.—If the residue is *not white* and contains an element or compound, such as an excess of iron, which will react with platinum, the element or compound is removed by an appropriate chemical method and the remaining material is treated as in step 5.

Step 7.—A predetermined aliquot of the solution in the standard flask is pipetted into a 50-ml. standard flask and the acidity is adjusted so as to be 29·0 per cent. HNO_3 when the solution is made up to the mark.

Step 8.—Two ml. of this 29·0 per cent. HNO_3 solution are pipetted into a ground glass, stoppered test tube which contains 9 ml. of $\text{Al}(\text{NO}_3)_3$ salting solution (3 grams $\text{Al}(\text{NO}_3)_3$, 9 $\text{H}_2\text{O}/\text{ml. H}_2\text{O}$). The salted solution is gently agitated until it is homogeneous, 10 ml. of ethyl acetate are added, the mixture shaken vigorously for 30 seconds, the stopper removed and the aqueous and ethyl acetate layers allowed to separate for four minutes. Approximately 7 to 8 ml. of the ethyl acetate layer are pipetted off and filtered through a No. 42 Whatman filter paper into a test tube.

Step 9.—0·5 ml. of the ethyl acetate extract is pipetted into each of four platinum dishes using a micro-pipette. The dishes are then placed under a radiant (infra-red) heater at a distance of 20 cm. and evaporated to dryness.

Step 10.—To each of the dishes $0\cdot5 \pm 0\cdot02$ gm. of fusion mixture is added (45·5% K_2CO_3 ; 45·5% Na_2CO_3 ; 9% NaF). After addition of the fusion mixture the dishes are placed on a stainless steel rack and heated in a muffle furnace at exactly 650° C. for ten minutes in the following manner:—

- (a) Fuse for 3 minutes, then swirl the dishes for 15 seconds.
- (b) Fuse for 3½ minutes and swirl for 15 seconds.
- (c) Fuse for 3 minutes and swirl for 15 seconds.

The swirling is done by gently rotating the rack supporting the dishes so that the molten flux swirls up to the lip of the dish and mixes with the material which has crept up the walls of the dish during the evaporation stage.

Step 11.—After fusion, the dishes are removed from the muffle furnace and allowed to cool in a desiccator for four minutes. The fluorescence of each disc is then determined. Four readings are taken of each disc by rotating the discs through successive angles of 90°.

Step 12.—The percentage U_3O_8 is then calculated by subtracting from the mean of the sixteen readings so obtained, the fluorescence of a blank disc (i.e., $0\cdot5 \pm 0\cdot02$ grams of fusion mixture only) and comparing this result with that obtained from a disc containing a known amount of uranium. This standard disc is made by pipetting 2 ml. of a 29·0 per cent. HNO_3 solution containing 1 microgram/ml. into 9 ml. of salting solution in a ground glass, stoppered test tube and extracting and fusing in the same manner as the unknowns. The standard disc then contains 0·1 microgram of uranium.

Preparation of Standard Solution.—The standard solution is prepared as follows:—2·112 gm. of AR uranyl nitrate is dissolved in 1 litre of distilled water and sufficient HNO_3 to make 29·0 per cent. HNO_3 . This solution contains 1 microgram of uranium per ml.

Preparation of Fusion Mixture.—The purest AR NaF should be used as a constituent of the fusion mixture. Different batches of NaF even from the same manufacturer differ markedly in their "fluorescent" properties and degree of contamination. The analyst should obtain samples of NaF from various suppliers and after testing the purity of each sample, purchase a large stock of the purest.

NaF.	Fluorescence.	
	1 gm fusion mixture, other constituents constant.	1 gm fusion mixture, other constituents constant, ± 0.25 μ grams of uranium.
Sample A	6×10^2	2.5×10^5
Sample B	4.5×10^3	8.0×10^4
Sample C	4.0×10^3	8.0×10^4

It will be noted from the above table, that the difference between the fluorescence of the blank disc, and that containing 0.25μ gm. of uranium is less, using the most contaminated sodium fluoride (sample B), than the difference between the fluorescence of the blank disc and that containing 0.25μ gm. of uranium using the least contaminated sodium fluoride (sample A). This effect is probably due to other contaminants in the less pure sodium fluoride quenching the fluorescence of the uranium. No uranium contamination has been detected in reagent grade Na_2CO_3 or K_2CO_3 used in the Bureau's laboratory. The ingredients of the fusion mixture (Na_2CO_3 , K_2CO_3 , NaF) are ground separately using a pestle and mortar and mixed for approximately two hours in a mixing mill.

Preparation of Salting Agent.—The salting solution is prepared by dissolving $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ in distilled water in the ratio 3 gm. to 1 ml. Gentle heating is necessary to dissolve the aluminium nitrate completely.

General Precautions.—(1) Since the Bureau's laboratory is not air-conditioned, great care is exercised to avoid air-borne contamination of the fused discs. A small piece of air-borne fibre added to a blank disc (i.e., fusion mixture only) increased the fluorescence of the disc from 6×10^2 to 1.1×10^4 . As the amount of uranium measured in the fused disc is very small (10^{-7} grams), glassware, especially glassware used during the ethyl acetate extraction stage, and apparatus used subsequently in the procedure should be cleaned and handled with more than usual care. The fused discs are slightly hygroscopic and as a disc absorbs moisture its fluorescence increases, e.g., breathing on a fused blank disc increased its fluorescence from 6×10^2 to 1.7×10^3 .

(2) Special care must be taken to ensure that all platinum ware is thoroughly cleaned. After use, rinse the platinum dishes in water, boil for fifteen minutes in 6NHCl, rinse, boil in concentrated HNO_3 for twenty minutes, rinse, boil in concentrated HNO_3 , rinse thoroughly with distilled water and store in a desiccator until required. If, after this cleaning, the platinum dishes are still discoloured, fuse with a little Na_2CO_3 , dissolve the residue with water, rinse, and boil for fifteen minutes in 6NHCl. Should the dishes still be discoloured, fuse with KHSO_4 , rinse with water, boil for fifteen minutes in 6NHCl and rinse with distilled water. The dishes must be handled with platinum tipped tongs during the final rinsing with distilled water and must not be touched with the fingers until the discs they contain have been read in the fluorimeter. Dishes which develop an etched, rough surface after repeated fusions should be polished with jeweller's rouge. Platinum crucibles stained with iron are best cleaned by heating a gram or two of AR, NH_4Cl in the covered crucible for two to three minutes.

Suggested Improvements in the Procedure.—As stated previously, the procedure given is that which is in current use in the Bureau's laboratory. Experience gained there and information from overseas reports indicate that improvements in the technique could probably be effected, although additional equipment would be required for this purpose. The following suggestions are made to indicate the lines along which an improved technique could be developed:—

- (1) Observations made show that a decrease in the mass of fusion mixture in the fused disc increases the fluorescence of the disc even though the mass of the uranium in the disc is kept constant, e.g., with a disc of 0.25 micrograms of uranium and 1 gm. of fusion mixture, the fluorescence was 4.6×10^4 , whereas with a disc of 0.25 micrograms of uranium and 0.5 gm. of fusion mixture, the fluorescence was 9.1×10^4 . The smallest mass of fusion mixture tested was 0.5 gm., the comparatively large size of the platinum dishes making it impossible to swirl smaller amounts of fusion mixture around the dish while the flux was molten. Further experiments with dishes of a smaller capacity are necessary to determine the maximum fluorescence for this uranium/fusion mixture ratio.
- (2) Fused discs cannot be read in the fluorimeter if their uranium content is appreciably less than 10^{-8} gm. of uranium per disc. The fluorescence of such a disc is approximately 4×10^3 . The least fluorescent blank disc read in this laboratory gave a reading of 6×10^2 and the usual dark current of the fluorimeter is 5×10^2 ; i.e., the dark current reading and the blank disc fluorescence are approximately 1/6 of the fluorescence obtained with a disc containing 0.01 micrograms of uranium. The dark current can be decreased appreciably by cooling the photo-multiplier tube. A report by Price, Ferretti and Schwartz (1953) suggests cooling the photo-multiplier tube to dry ice temperature. In the opinion of the authors a small refrigerating coil would be more efficient than dry ice in keeping the temperature of the photo-multiplier constant. Enquiries should also be made of suppliers to ascertain if photo-multiplier tubes have been developed which give lower dark current readings than those tubes now in use in the fluorimeter. If the dark current can be decreased by a factor of 10, then discs containing 1×10^{-8} gm. of uranium could be read in the fluorimeter.
- (3) The dilution technique suggested by Price et al (1953), based on the fact that quenching of fluorescence can be avoided by altering the ratio of quencher to fusion mixture, does not entail the salting of the solutions of unknowns or extracting with an organic liquid. From observation, some high-grade samples (i.e., 0.25 per cent. uranium and above) should lend themselves to this technique. However, if the percentage of any of the quenching elements is greater than the percentage of uranium in the sample a salting-extraction procedure will be necessary.

The Quenching Elements.—Some disagreement is to be found in the literature on the subject of quenching and it is suggested that, until empirical results have been obtained, any samples which contain a high proportion of quenching elements should be assayed using the salting-extraction technique. The strongest quenching is produced by the following elements: Cr, Mn, Fe, Co, Ni, Cu, Zn, Ag, Sn, La, Pt, Au, Pb, Th, Ce, Pr, Nd.

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TABLE 1.—URANIUM SERIES (Main Sequence).

Name	Isotope	Half Life	Principal Emission Energies in Mev.		
			Alpha.	Beta.	Gamma
UI*	U 238	$4 \cdot 5 \times 10^9$ y	4.18
UX ₁	Th 234	24.1 d.	..	0.2, 0.1	0.09
UX ₂	Pa 234	1.18 m.	..	2.32, 1.5	2% 0.78, 0.82
UII	U 234	$2 \cdot 6 \times 10^5$ y	4.76
Io	Th 230	$8 \cdot 0 \times 10^4$ y	4.68, 4.61	..	(11%) 0.07
Ra	Ra 226	1600 y.	4.79, 4.61	(0.09)	(0.19)
Rn	Em 222	3.825 d.	5.49
RaA	Po 218	3.05 m.	6.00
RaB	Pb 214	26.8 m.	..	0.7	(0.047, 0.035)
RaC	Bi 214	19.7 m.	..	1.65, 3.17	1.76, 2.19, 1.2, 0.6
RaC'	Po 214	164 μ sec.	7.68
RaD	Pb 210	22y.	..	0.026	10% 0.007
RaE	Bi 210	5.0 d.	..	1.17	..
RaF	Po 210	138.3 d.	5.30
RaG	Pb 206	Stable

NOTE.—Energies in brackets indicate low intensity. Branch products of less than 0.2 per cent. of decays are omitted.

* Relative abundance : 99.3 per cent. in natural uranium.

TABLE 2.—ACTINIUM SERIES (Main sequence).

Name.	Isotope.	Half Life.	Principal Emission Energies in Mev.		
			Alpha.	Beta.	Gamma.
AcU*	U 235	$7 \cdot 07 \times 10^8 y.$	4·6, 4·4	..	0·16
UY ..	Th 231	25 h.	..	0·21	0·03
Pa ..	Pa 231	32,000 y.	5·0	..	0·09, 0·3
Ac ..	Ac 227	13·5 y.	..	0·02	(0·04)
RdAc ..	Th 227	18·9 d.	5·7, 6·0	..	0·03 to 0·5
AcX ..	Ra 223	11·2 d.	5·6, 5·7	..	0·02 to 0·4
An ..	Em 219	3·92 s.	6·5, 6·8	..	0·2
AcA ..	Po 215	0·0018 s.	7·36
AcB ..	Pb 211	36 m.	..	1·4, 0·5	0·4, 0·8
AcC ..	Bi 211	2·16 m.	6·6, 6·2	..	0·35
AcC"	Tl 207	4·76 m.	..	1·45	(0·87)
AcD ..	Pb 207	Stable

NOTE.—Energies in brackets indicate low intensity. Branch products of less than 1·0 per cent. of decays are omitted.

* Relative abundance : 0·71 per cent. in natural uranium.

TABLE 3.—THORIUM SERIES (Main sequence).

Name.	Isotope.	Half Life.	Principal Emission Energies in Mev.		
			Alpha.	Beta.	Gamma.
Th ..	Th 232	$1 \cdot 39 \times 10^{10} y.$	3·98
MsTh 1	Ra 228	6·7 y.	..	0·053	..
MsTh 2	Ac 228	6·13 h.	..	1·55	0·96, 0·91
RdTh ..	Th 228	1·9 y.	5·42, 5·34	..	0·08
ThX ..	Ra 224	3·64 d.	5·68, 5·45	..	(0·25)
Th ..	Em 220	54·5 s.	6·282
ThA ..	Po 216	0·16 s.	6·774
ThB ..	Pb 212	10·6 h.	..	0·33, 0·57	0·24, 0·30
ThC ..	Bi 212	60·5 m.	34 % 6·04, 6·08	66 % 2·25	0·72, 0·8, 1·8
ThC'	Po 212	0·3 μ sec.	66 % 8·78
ThC"	Tl 208	3·1 m.	..	34 % 1·72	2·62, 0·58, 0·51
ThD ..	Pb 208	Stable

NOTE.—Energies in brackets indicate low intensity. Branch products of less than 0·2 per cent. of decays are omitted.

BUREAU OF MINERAL RESOURCES
RADIOMETRIC LABORATORY
Port Hope

SAMPLE NO. *PR.55/17*
DATE. *8/1/1955*

RADIOMETRIC ASSAY.

ASSAY COMMENCED *4/10* CONCLUDED *4/14*
SAMPLE: Description *Received in pulverized form. Known to contain uranium minerals.*
Location *Northern Territory* Grain size -100.

G.M. TUBE SET UP						SCINTILLOMETER SET UP					
HOLDER: Type	<i>Deep end window</i>					HOLDER: Type	<i>Deep end window</i>				
Size	<i>12 c.c.</i>	No.	<i>D5</i>	Pos.	<i>3</i>	Size	<i>12 c.c.</i>	No.	<i>D5</i>	Pos.	<i>2</i>
CASTLE: No.	<i>2</i>					CASTLE: No.	<i>1</i>				
SCALER: Type	<i>1000 A</i>	No.	<i>158</i>			SCALER:					
Bias	<i>.5V</i>		Paralysis			RATEMETER: Type	<i>10,000 A</i>	No.	<i>1065</i>		
G.M. TUBE: Type	<i>G.M.G.</i>	No.	<i>1836</i>	E.H.T.	<i>1940 v</i>	Bias	<i>.20V</i>		Paralysis	<i>.5 spec.</i>	
ABSORBER:			PARALYSIS	<i>.500</i>	<i>usec.</i>	PHOTOMULTIPLIER: Type	<i>6260</i>	No.	<i>5530</i>		
						E.H.T.	<i>1700</i>	Volts			

	B + G		G		B + G		G		B + G		G	
	COUNT	MINS										
	168	16										
Counts/Min	10.5		162									

	SAMPLE NO.				U_3O_8 STANDARD, 1.0 %				ThO_2 STANDARD, ... %				
	Mass.	<i>25.0</i> Grams			<i>24.3</i> Grams								
		B + G		G		B + G		G		B + G		G	
		COUNT	MINS	COUNT	MINS	COUNT	MINS	COUNT	MINS	COUNT	MINS	COUNT	MINS
Count 1		<i>5900</i>	0			<i>3689</i>	2						
Count 2		<i>6099</i>	0			<i>3685</i>	2						
Count 3		<i>6115</i>	2			<i>3680</i>	2						
Count 4		<i>6134</i>	2			<i>3662</i>	2						
Total		<i>24248</i>	8			<i>14716</i>	8						
Counts/Min		<i>3031</i>		<i>31320</i>		<i>1839.5</i>		<i>18600</i>					
Paralysis Loss		<i>80</i>		-		<i>30</i>		-					
Total		<i>3111</i>		<i>31320</i>		<i>1839.5</i>		<i>18600</i>					
Background		<i>11</i>		<i>162</i>		<i>10.5</i>		<i>162</i>					
Difference		<i>3100</i>		<i>31158</i>		<i>1829</i>		<i>18438</i>					
Gamma Count													
Difference													
Corrected for Density													

BETA ASSAY (U_B). 1.7 % U_3O_8 Equivalent or ... % ThO_2 Equivalent

GAMMA ASSAY (U_G) 1.6 % U_3O_8 Equivalent or ... % ThO_2 Equivalent.

$\frac{B/G}{B/G \text{ (Sample)}} = \frac{0.103}{0.101} = 1.0 \dots = \frac{U_B}{U_G}$ (*Sample probably contains uranium in equilibrium*)

Beta, gamma assay for samples { ... % U_3O_8 + ... % ThO_2
not in equilibrium or containing Th }

Assayed by Checked by

BUREAU OF MINERAL RESOURCES

RADIOMETRIC LABORATORY

February

SAMPLE NO. P.R. 55/8

DATE 10.12.1955.

RADIOMETRIC ASSAY

ASSAY COMMENCED 11.30 CONCLUDED 12.30

SAMPLE Description Received in pulverised form known to contain thorium minerals

Location Northern Territory

Grain size -106

G.M. TUBE SET UP				SCINTILLONOMETER SET UP			
HOLDER	Type	Deep end window		HOLDER	Type	Deep end window	
Size	12 c.c.	No. A5	Pos. 3	Size	12 c.c.	No. A5	Pos. 2
CASTLE	No.	2		CASTLE	No.	1	
SCALER	Type	1009A	No. 158	SCALER			
Bias	5V	Paralysis	-	RATEMETER	Type	1037A ₂	No. 1065
G.M. TUBE	Type	GM.4	No. 8214	E.H.T.	1260 v.		
ABSORBER:		PARALYSIS	500 μsec	PHOTOMULTIPLIER	Type	6260	No. 5538
					E.H.T.	1700	Volts

BACKGROUND

	B + G		G		B + G		G		B + G		G	
	COUNT	MINS										
Counts/Min	12.0	10										
	12.0	186										

Mass.	SAMPLE NO. 21.9 Grams				U ₃ O ₈ STANDARD 0.1%				ThO ₂ STANDARD %			
	B + G		G		B + G		G		B + G		G	
	COUNT	MINS	COUNT	MINS	COUNT	MINS	COUNT	MINS	COUNT	MINS	COUNT	MINS
Count 1	1687	2			1322	5						
Count 2	1667	2			1333	5						
Count 3	1715	2			1290	5						
Count 4	1630	2			1260	5						
Total	6699	8			5205	20						
Counts/Min	837.4	9000			260.2	2190						
Paralysis Loss	6	-			-	-						
Total	843.4	9000			260.2	2190						
Background	12.0	186			12.0	186						
Difference	831.4	8814			248.2	2004						
Gamma Count												
Difference												
Corrected for Density	831.4	10,000										

BETA ASSAY (U_3) 0.34 % U₃O₈ Equivalent or 1.1 % ThO₂ EquivalentGAMMA ASSAY (U_C) 0.50 % U₃O₈ Equivalent or 1.0 % ThO₂ EquivalentB/G (Sample) : $\frac{0.0831}{0.124}$ = 0.67 = $\frac{U_B}{U_C}$ (Sample probably consists mainly of thorium)Beta, gamma assay for samples } % U₃O₈ + % ThO₂
not in equilibrium or containing Th }

Assayed by

Checked by

BUREAU OF MINERAL RESOURCES
RADIOMETRIC LABORATORY
Footcrag

SAMPLE NO. P.R. 54/174DATE 9/11/1954RADIOMETRIC ASSAY.ASSAY COMMENCED 9.10 CONCLUDED 11.30SAMPLE: Description MolybdeniteLocation Grain size -100

G.M. TUBE SET UP				SCINTILLOMETER SET UP			
HOLDER	Type	<i>Annular holder</i>		HOLDER	Type		
Size	12.4 c.c.	No. C8	Pos.	Size	c.c.	No.	Pos.
CASTLE	No.	3		CASTLE	No.		
SCALER	Type	1009A	No. 158	SCALER			
Bias	5V	Paralysis		RATEMETER	Type	No.	
G.M. TUBE	Type	B6	No. JA1613 E.H.T. 1110 V.	Bias	Paralysis	
ABSORBER	1/16" Brass	PARALYSIS	.500 plate	PHOTOMULTIPLIER	Type	No.	
				E.H.T.	Volts	

BACKGROUND

	B + G		G		B + G		G		B + G		G	
	COUNT	MINS										
	125	10	89	10	133	10	99	10				
Counts/Min	12.5		8.9		13.3		9.9					

	SAMPLE NO. <u>20.6</u> Grams				U ₃ O ₈ STANDARD, 0.1% %				ThO ₂ STANDARD, ... % Grams			
	B + G		G		B + G		G		B + G		G	
	COUNT	MINS	COUNT	MINS	COUNT	MINS	COUNT	MINS	COUNT	MINS	COUNT	MINS
Count 1	4517	2	1458	10	1902	2	509	10				
Count 2	4509	2	1440	10	1854	2	508	10				
Count 3	4526	2	1431	10	1898	2	526	10				
Count 4	4506	2	1508	10	1854	2	504	10				
Total	18051	8	5897	40	7448	8	2047	40				
Counts/Min	2256.4		145.9		931		51.2					
Paralysis Loss	5.0		-		7		-					
Total	2306.4		145.9		938		51.2					
Background	13.0		9.4		13		9.4					
Difference	2297.4		136.5		922		41.8					
Gamma Count	126.5				41.8		-					
Difference	2157				883.2		-					
Corrected for Density	-		144		-		41.8					

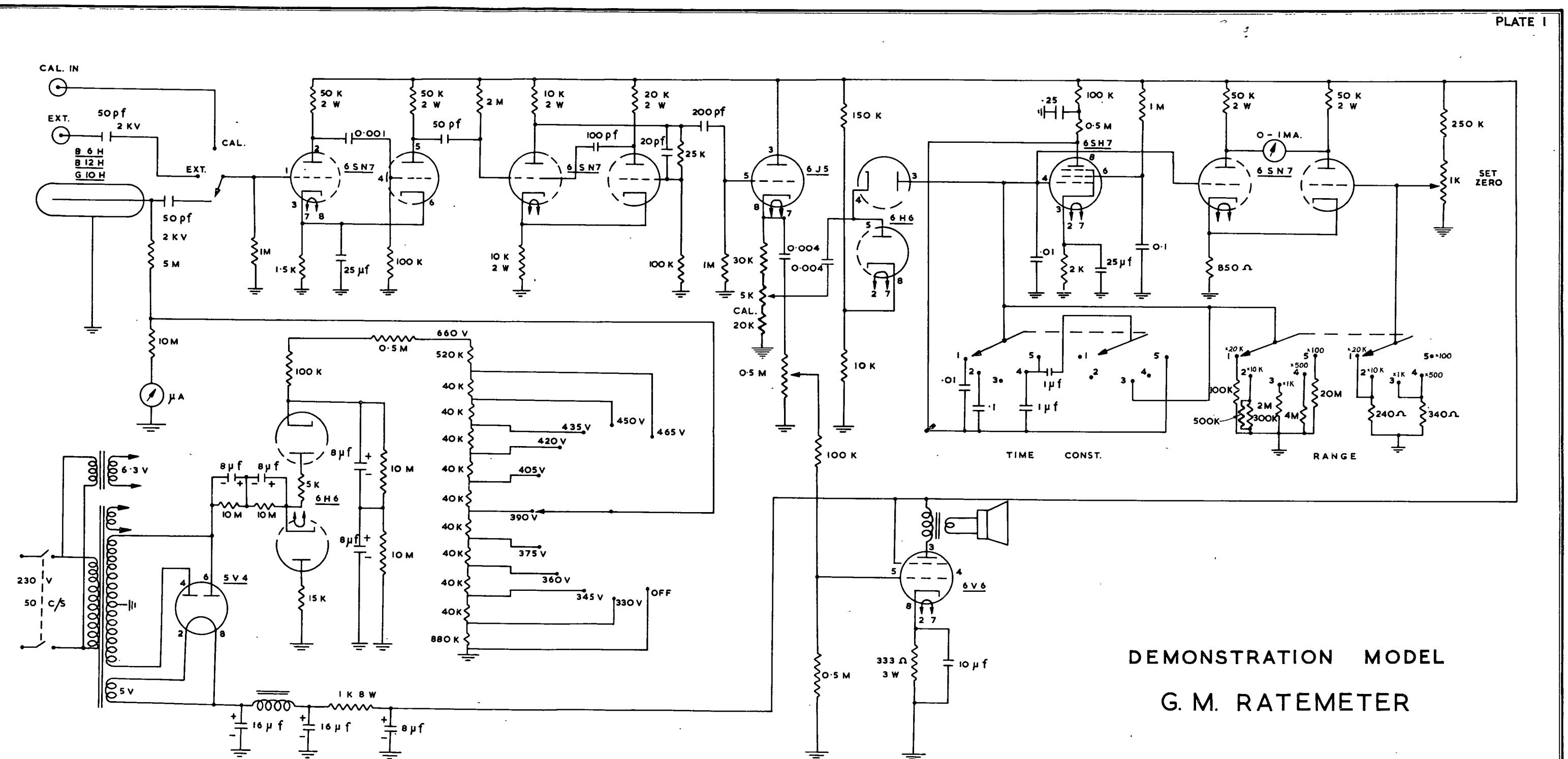
BETA ASSAY (U_3) : 0.24. % U₃O₈ Equivalent or % ThO₂ EquivalentGAMMA ASSAY (U_3) : 0.34. % U₃O₈ Equivalent or % ThO₂ Equivalent

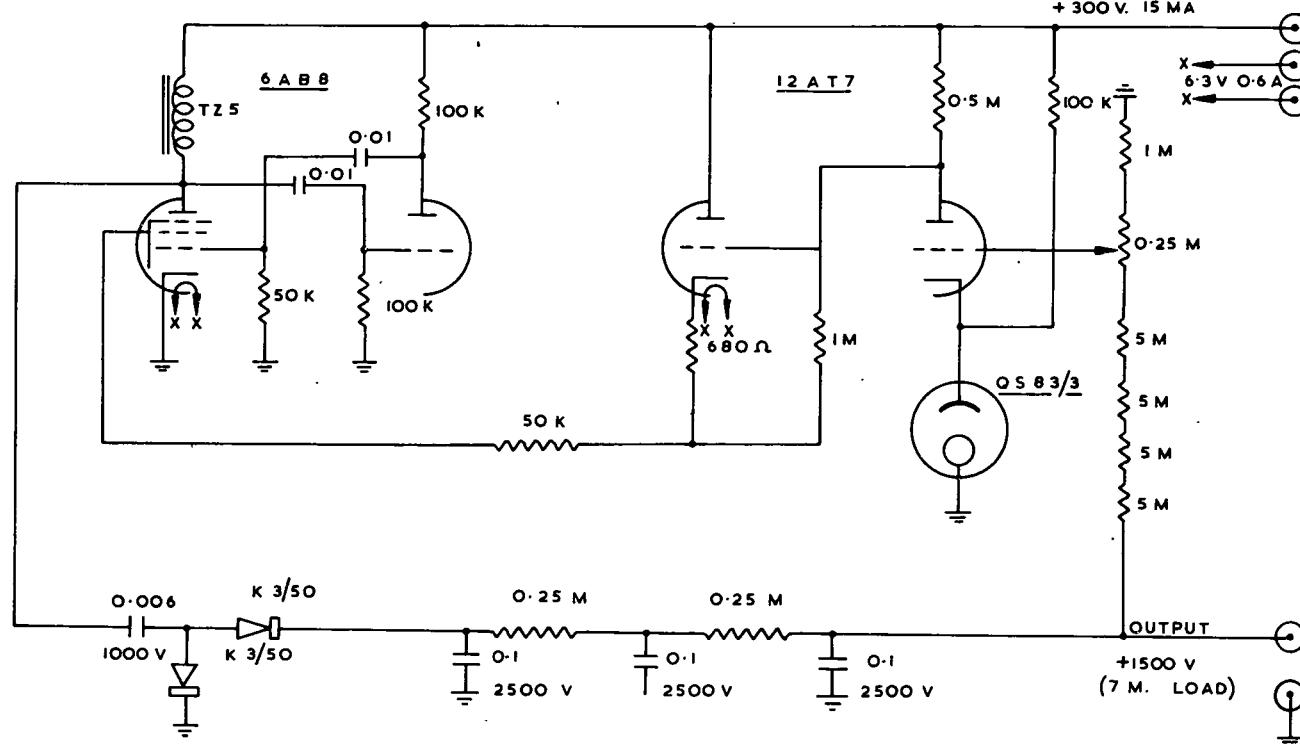
$$\frac{B/G}{B/G \text{ (Sample)}} = \frac{15.0}{21.1} = 0.71 = \frac{U_3}{U_3} \text{ (Sample probably contains uranium and thorium)}$$

Beta, gamma assay for samples } 0.15 % U₃O₈ + 0.31 % ThO₂
 not in equilibrium or containing Th } (Rough estimate only).

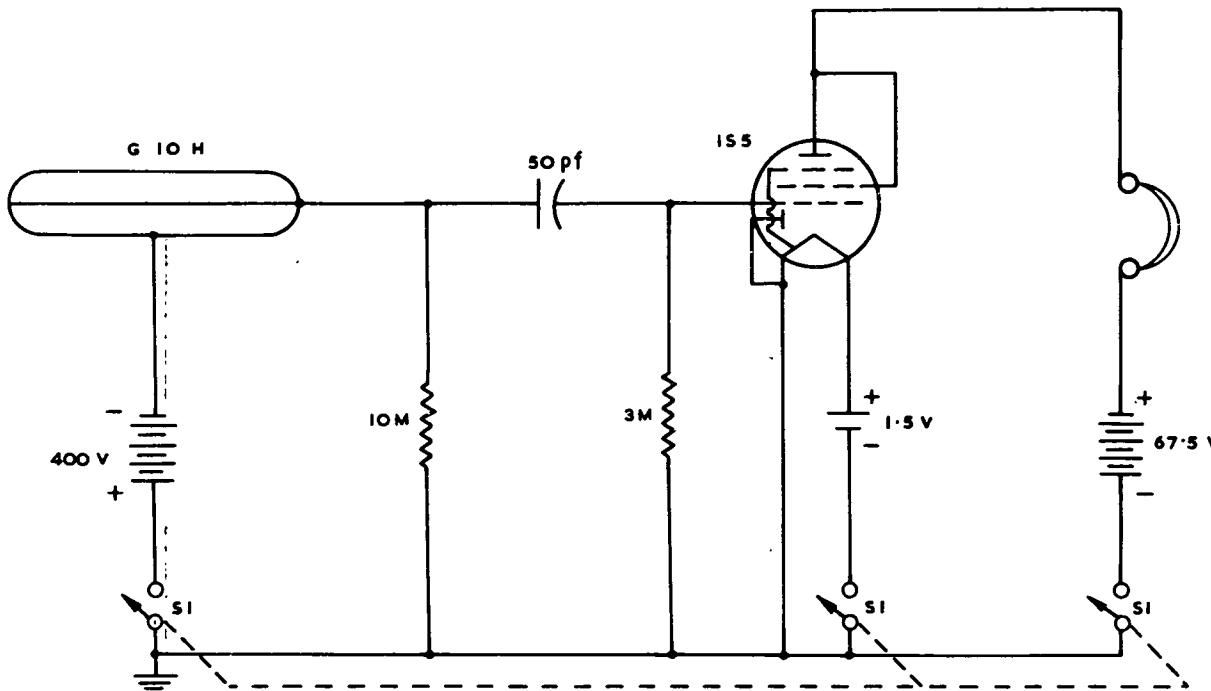
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Government Printer, Melbourne

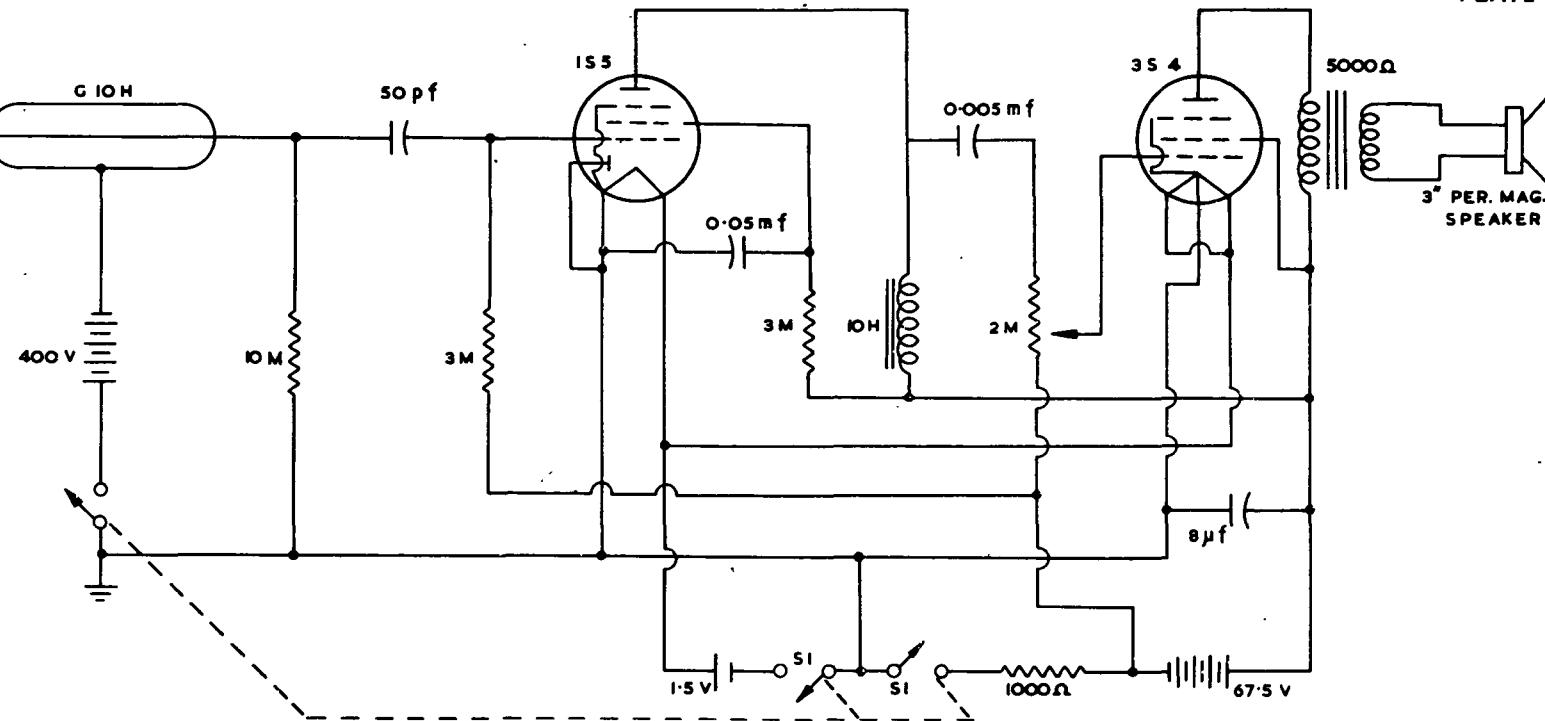




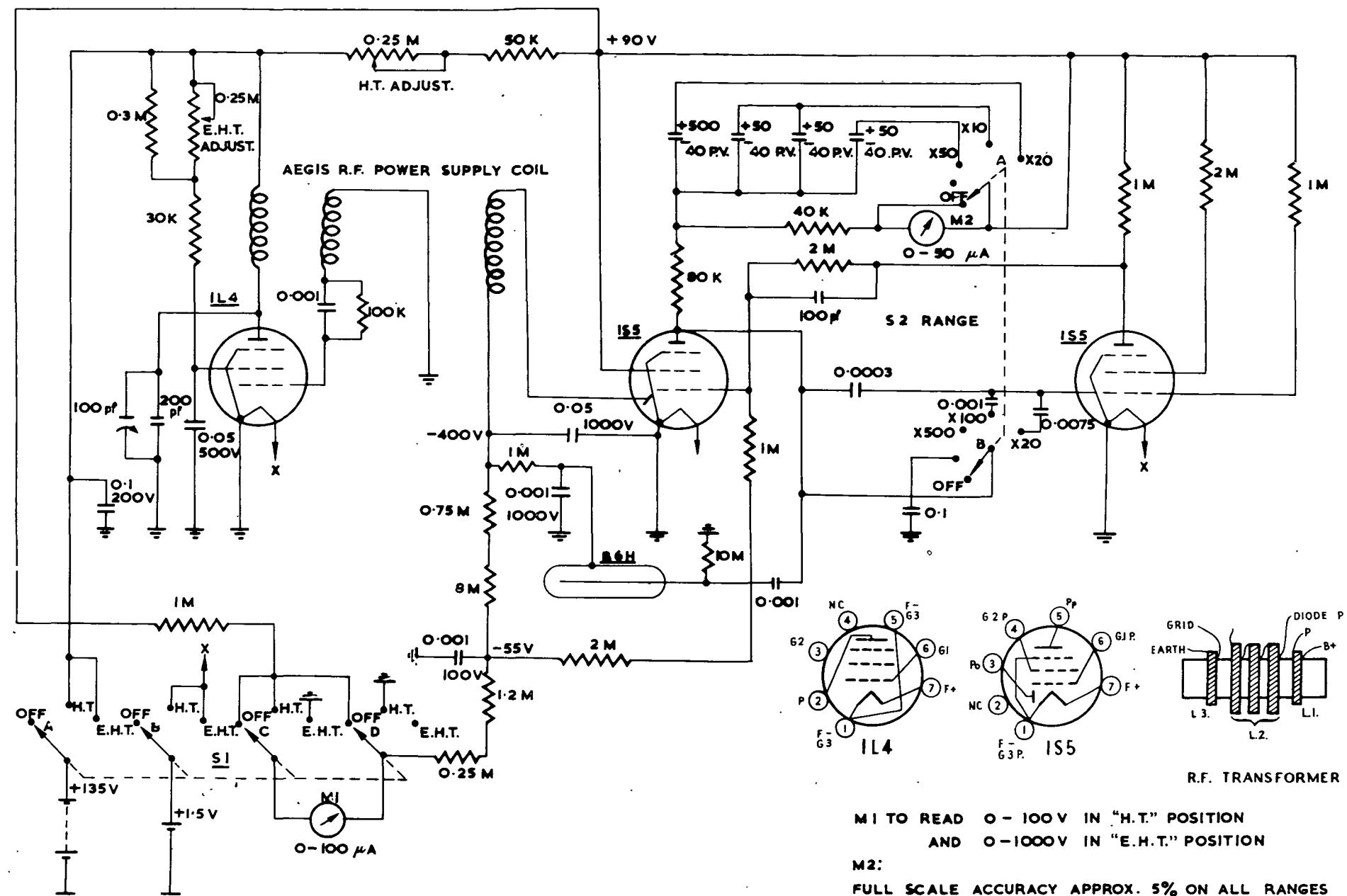
REGULATED E.H.T. POWER SUPPLY



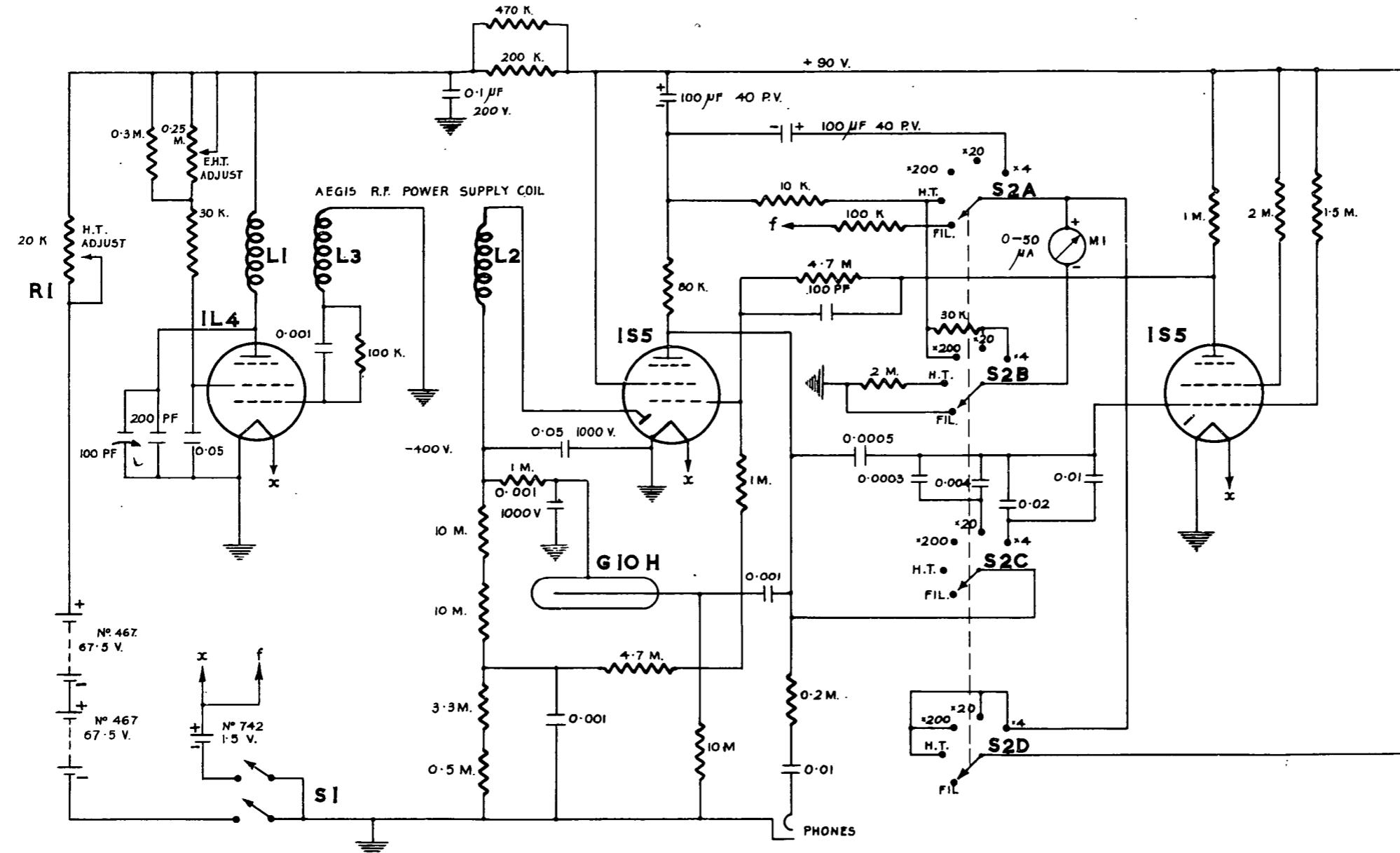
GEIGER COUNTER — HEADPHONE TYPE



GEIGER COUNTER - LOUDSPEAKER TYPE



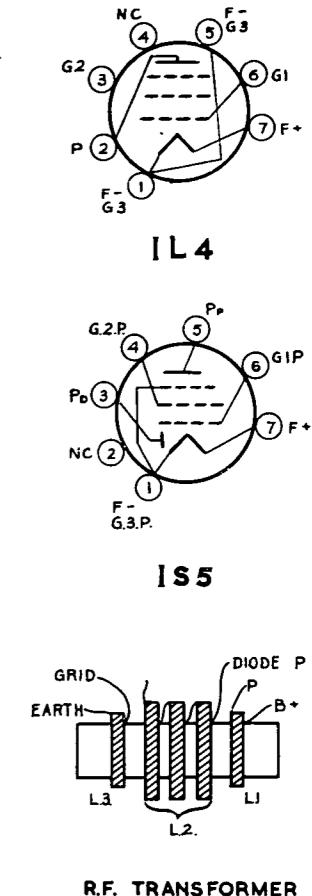
BATTERY OPERATED RATEMETER FOR FIELD ASSAYING

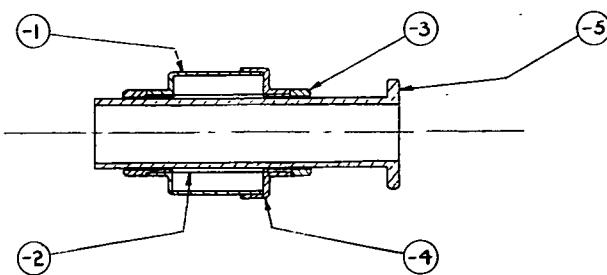


M.I. TO READ 0-5 VOLTS IN "FIL" POSITION
 M.I. " " 0-100 VOLTS IN H.T. "
 M.I. " " 0-10,000 COUNTS PER MINUTE IN "x200" POSITION.
 M.I. " " 0-1,000 " " "x20" "
 M.I. " " 0-200 " " "x4" "
 SET "H.T." TO 90 VOLTS BY MEANS OF "H.T. ADJUST"
 ALL RESISTORS $\frac{1}{2}$ WATT.

S² BREAK BEFORE MAKE, 4 POLE, 5 POSITION, WAFER SWITCH

PORTABLE GEIGER-MÜLLER COUNTER WITH HEADPHONE AND RATEMETER INDICATION

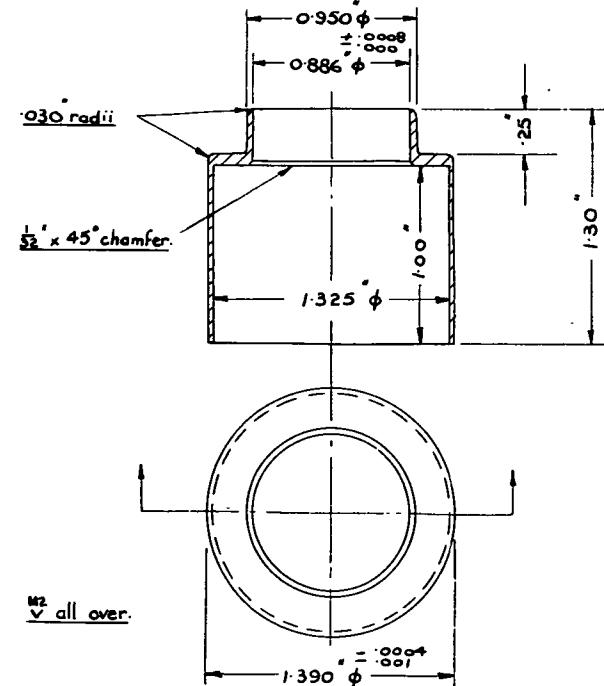




Annular Sample Holder Assembly

Scale 1:2 (approx.)

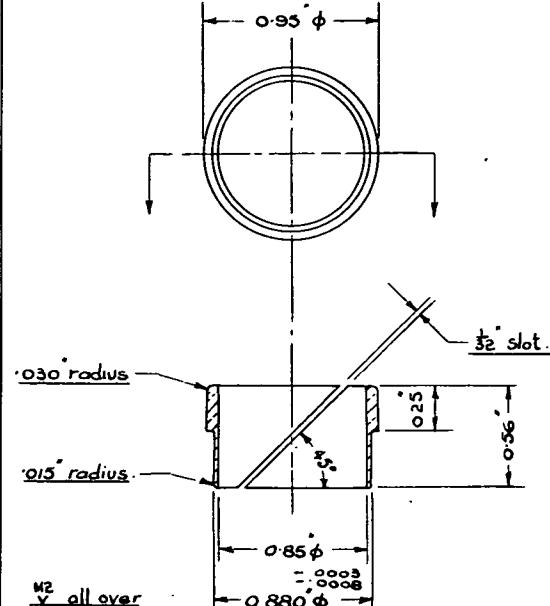
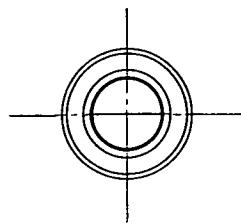
Note Tolerances to be $\pm \frac{1}{64}$ " for fractional sizes and
 $\pm .005$ " for decimal sizes unless otherwise specified.



Body
Matl. Dural.

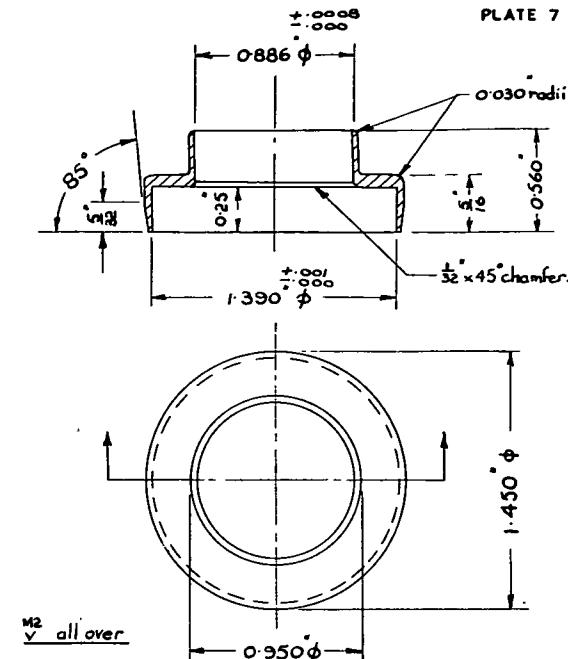
Reqd. One per.
Scale 1:1 (approx.)

UO1-1



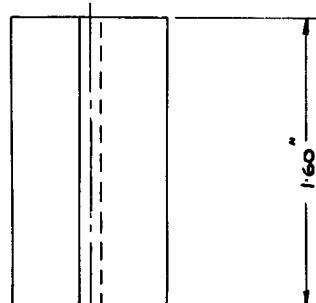
Split Sleeve
Matl. Hard brass

Reqd. Two per
Scale 1:1 (approx.) UO1-3

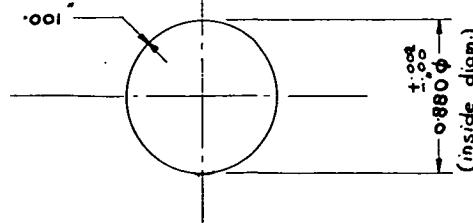


Lid
Matl. Dural.

Reqd. One per.
Scale 1:1 (approx.) UO1-4

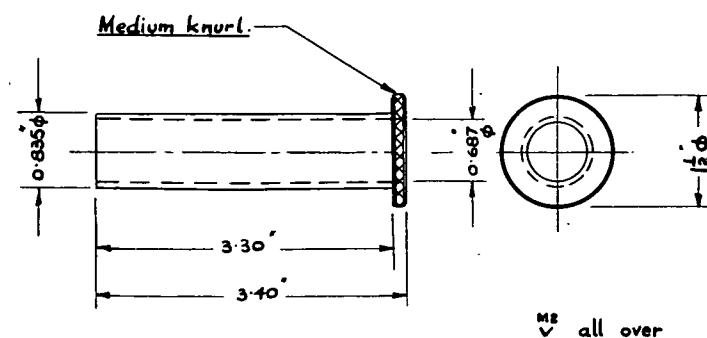


Developed length 2.86"
Lap over for $\frac{3}{32}$ " soft solder.



Inner Sleeve
Matl. .001" shim copper.

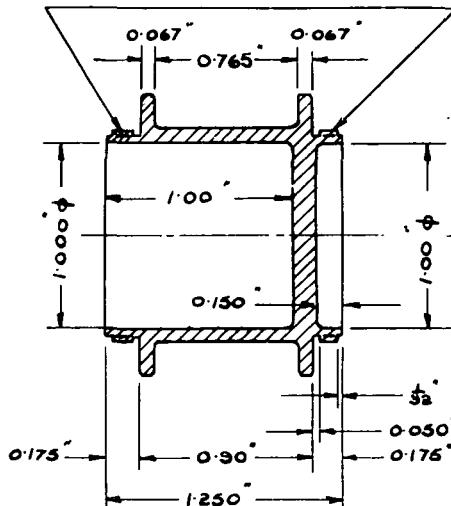
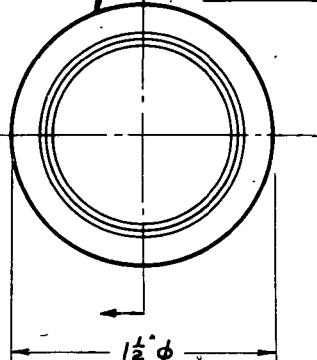
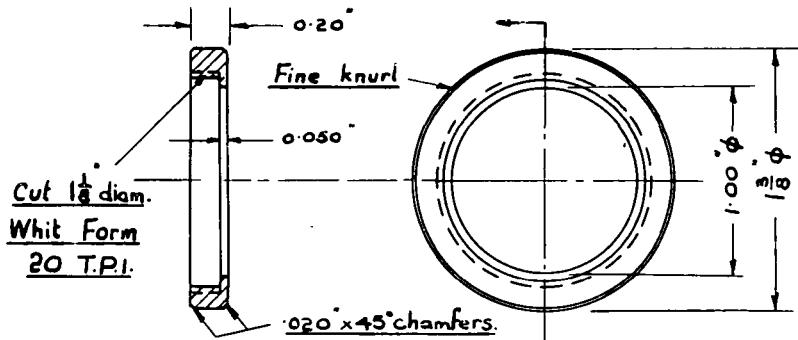
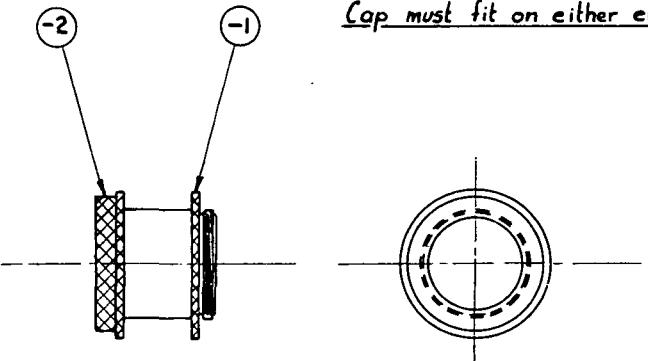
Reqd. One per.
Scale 1:1 (approx.) UO1-2



Beta Absorber
Matl. Brass.

Reqd. One per.
Scale 1:2 (approx.) UO1-5

U.O.I. 13755 F.H.A.
ANNULAR SAMPLE HOLDER.

Cut 1:125 ϕ 20 T.P.I.Whit form.Fine knurl.v all overNote. Cut recesses to clear root diam. of thread.Reqd. One per.Matl. Alum. - Grey Anodize.Scale 1:1 (approx) UO2-1CapReqd. One per.Matl. Alum. - Grey Anodize. Scale. 1:1 (approx) UO2-2Sample Holder Assembly.Scale 1:2Note. Tolerances to be $\pm \frac{1}{64}$ " on fractional sizes +
 $\pm .005$ " on decimal sizes, unless otherwise specified.

U.O2 157.55 FWD

END WINDOW SAMPLE HOLDER.