

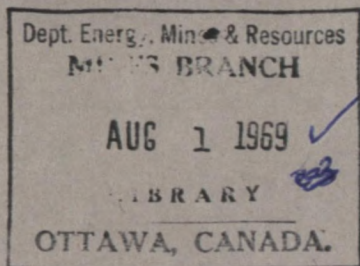
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CANADA
DEPARTMENT OF MINES AND TECHNICAL SURVEYS
MINES BRANCH

RADIOASSAY OF URANIUM ORE WITH THE GEIGER TYPE EQUILIBRIUM COUNTER

by

R. D. Wilmot and C. McMahon
RADIOACTIVITY DIVISION



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1951

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RADIOASSAY OF URANIUM ORE WITH THE GEIGER TYPE EQUILIBRIUM COUNTER

by

R. D. Wilmot and C. McMahon

Radioactivity Division

INTRODUCTION

Over the past few years Federal Government policy with regard to price and an assured market has stimulated a widespread search for uranium deposits. As many of the important finds are distant from established assay centres, information on the physical assay of radioactivity has been compiled for those interested in providing their own facilities. Although not an absolute means for determining uranium contents, radioassay is simpler and faster than chemical methods.

To meet typical field conditions a counter should be capable of determining radioactive contents up to 60% with an average accuracy of from 5 to 10%.

A basic theory, procedures in assaying, and apparatus required are discussed. A glossary of the more commonly used terms in radioassay is included.

APPLICATIONS AND LIMITATIONS

Once calibrated with chemically determined standards the tray-type beta-gamma counter can be used to determine the beta and gamma activities of dry pulverized ore. Mill products, including sink, float, and certain leach residues, crushed drill cores, and spot samples can be assayed with the counter described. The dry pulverized sample prepared for assay should never be less than 200 gm.

The beta-gamma counter was especially designed for the assay of ores out of equilibrium* as in these cases the beta and gamma assays will not agree with each other nor with the chemical determinations. The absolute accuracy of the beta-gamma counter is limited principally by the time allocated for each determination and is normally better than 5%. However, as the order of uncertainty of chemical assays is from 3 to 5%, assays by the two methods on the same ore have generally agreed to within 10%. Greater discrepancies** are caused usually by the presence of thorium or potassium in the uranium ore. These effects are discussed under "Interpretation of Results".

PRINCIPLES OF OPERATION

Radiations from Radioactive Sources

As the design of radioassay equipment is based on certain properties of the radioactive source, these are discussed first. Some elements, including thorium and uranium, possess natural radioactive properties. In other words the atoms of these elements are unstable and decompose progressively until they reach a stable element lower in mass. In this decay process parts of the atom are flung out in the form of alpha (α), beta (β), and gamma (γ) rays. Alpha and beta rays may be thought of as solid particles and have relatively limited powers of penetration. Gamma rays are fundamentally similar to heat or to light radiation but may penetrate several inches of lead. Alpha particles have limited powers of penetration and are not utilized in this apparatus.

* The meaning of this term is explained in the section on Definition of Equilibrium.

** These have seldom exceeded 30% at the Mines Branch.

The beta and gamma radiations from the sample are measured by the dual Geiger counter and are compared with the activity of chemically analysed standards.

Equipment Arrangement and Operation

Thin aluminum-walled Geiger tubes are used for detecting both beta and gamma rays, as this type of tube* is inexpensive, reliable, and at present easy to obtain.

The arrangement of sample, beta and gamma tubes, scalers, timers, and recorder is shown in Figure 1 on page 5 .

Beta particles emitted from the upper surface of the sample, B, penetrate the thin-walled beta tube, A, directly above, producing a discharge which is fed to the scaler through the shielded cable, D. Simultaneously, gamma rays from the entire sample mass actuate the lower gamma tube, C. Beta particles are prevented from reaching the gamma tube by the tray holding the pan. Individual counts from each Geiger tube are fed to the scalers where electronic circuits operate to reduce the input count by a known factor so that it can be registered on its respective beta or gamma recorder.

The timer, which is usually driven by a synchronous motor operating from the power mains, freezes the count on both scalers after a pre-set time has elapsed. If each scaler has its own built-in timer both should be released simultaneously at the start of the run. The beta and gamma determinations are made simultaneously to minimize changes in counter sensitivity and possible variations in line frequency. The latter effect would be important only at high counting rates.

* Type 1B85 manufactured by Victoreen Instrument Company, Cleveland, Ohio.

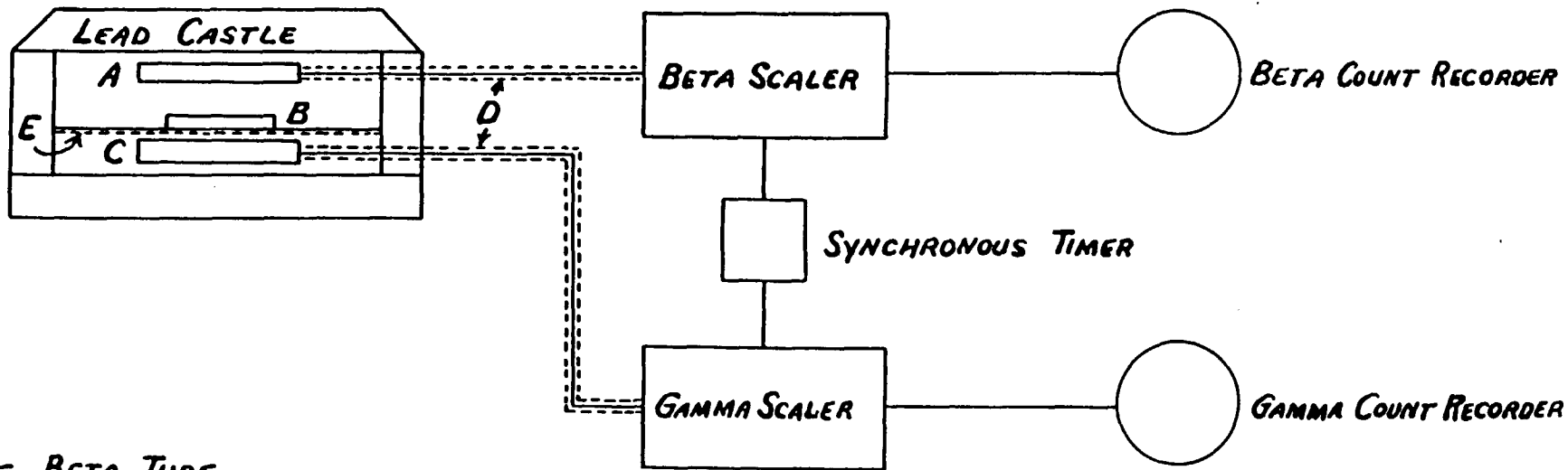
In the pre-set time method the total counts recorded in the set time interval are directly proportional to the beta or gamma activity of the sample. Although this is the method described in this manual other systems are used, including pre-set count which measures the time interval required for collecting a given number of counts. This has the advantage of obtaining the same statistical accuracy in all determinations but is time consuming at low counting rates.

Necessity for a Beta-Gamma System

Measurement of the beta or gamma sample activity alone will give the true U_3O_8 content only when the uranium family is in equilibrium and is free from other naturally radioactive elements such as thorium and potassium. As milling temporarily affects the equilibrium of radioactive ores (the beta assay may be up to 30% low for a particle size about -30 mesh if measured immediately after milling), and as thorium may be present in uranium-bearing ore, a more reliable radioassay method is desirable. The beta-gamma system, giving two independent assays on the same sample, presents enough data to calculate the U_3O_8 with an accuracy of 10% or better.

Definition of Equilibrium

The heavier radioactive elements such as uranium and thorium pass through several stages before reaching their final inactive states, radium and thorium lead. Uranium and thorium require millions of years before half of the original uranium and thorium atoms have been converted into lead, provided that none of the intermediate elements has been removed. Thus, all the transitory stages must be radioactive, and each contributes toward the total radiation from the series.



- A - BETA TUBE
- B - SAMPLE IN TRAY HOLDER
- C - GAMMA TUBE
- D - SHIELDED LEADS
- E - LEAD ABSORBER

Figure 1 - Block diagram of tray type equilibrium counter

Loss of Equilibrium

As one of the intermediate products is a gas in both the uranium and thorium series, porous rocks are not likely to contain these elements in equilibrium and will indicate a lower uranium or thorium content than is actually present when measured on either the beta or gamma counter. The escape of radioactive gases, radon and actinon, in the uranium series, and thoron in the thorium series, is more likely the more finely the sample is pulverized. Intermediate members may be removed also by weathering or by milling, as all elements are at least slightly soluble in water.

Effect of Equilibrium on Beta and Gamma Assays

The uranium (and thorium) series in equilibrium emits alpha, beta, and gamma rays (Appendix 1) and the ratio of beta to gamma radiation becomes constant and independent of the U_3O_8 content only when all members of the series are present in their equilibrium proportions. For convenience the uranium series may be divided into 2 parts. The first, or uranium family, includes the first 5 members, and the second group, or radium part, the remaining members. Deficiencies in the uranium part will reduce the beta assay but will not affect the gamma. On the other hand, loss of members in the radium family will reduce both the beta and gamma activities. Deficiencies of members in either group will thus diminish the total beta or gamma radiation. As the ratio of gamma to beta specific activity, expressed as a counting rate per % U_3O_8 per unit mass, is independent of the U_3O_8 content for ores in equilibrium, any change in the gamma-beta ratio indicates that the sample is not in equilibrium.

Calculation of U₃O₈ Content

The original method for determining the true U₃O₈ content of ores out of equilibrium* applied a correction to the gamma assay based on the ratio between the specific beta and gamma counting rates. To establish the relation between the correction factor and the gamma to beta ratio it was necessary to calibrate the counter with a set of synthetic equilibrium standards.

In the method described herein, U₃O₈ standards, in equilibrium and thorium free, are used to calibrate both beta and gamma counters. The U₃O₈ content may then be calculated directly from the following simple equation whose derivation is outlined in Appendix 3:

$$\% \text{ U}_3\text{O}_8 = \text{Twice beta assay} - \text{Gamma assay} \quad (1)$$

Significance of Discrepancies Between Beta and Gamma Assays

A higher beta than gamma assay usually indicates that the uranium series is out of equilibrium due to a deficiency of gamma emitters, but it could be caused by the presence of potassium. When both assays agree to within a few per cent, it can be assumed that the ore is in equilibrium and free from other radioactive sources. When the gamma assay exceeds the beta determination, either the presence of thorium, or loss of equilibrium due to deficiency of the uranium group, or both, is indicated.

It should be stressed that the foregoing are merely the most likely interpretations. The presence of thorium or potassium should be checked chemically and established quantitatively.

* Equilibrium Corrections in Geiger Analysis, by C. Lapointe, Canadian Mining and Metallurgical Bulletin, August, 1950, p. 465.

EQUIPMENT

Lead Shield

A lead shield is necessary to reduce the background counts to permit more accurate assays on low grade material. One cast with walls 2" thick has proved quite satisfactory and is shown in Figures 2 and 3 on pages 9 and 11 respectively. It is quite feasible to build the lead castle from lead slabs appropriately dimensioned.

The assay chamber is $1\frac{7}{8}$ " wide by $4\frac{1}{2}$ " high by $12\frac{3}{8}$ " long and is divided into two compartments by a $\frac{1}{8}$ " thick strip of lead. The lower section houses the gamma tube, and the upper the sample and beta tube. The walls and ceiling around the beta tube in the upper compartment should be lined with 30 mil aluminum sheeting to reduce background.

Samples are inserted from the front by moving the lead door sideways in its slot. Geiger-tube replacement is facilitated by a removable wall which can be slid out from the rear as shown in Figure 3 on page 11.

Geiger Tubes

The Victoreen Type 1B85 with a useful cathode length of about $2\frac{1}{2}$ " has proven to be quite suitable as both beta and gamma tube. These self-quenched tubes have a life of 10^9 counts, corresponding to from 1 to 3 months of operation, and an operating temperature range from -10 to $+100^{\circ}$ C. Their long plateaus with slopes of better than 5% per 100v, their dependability, and their low cost make them ideal.

The Geiger tubes should be mounted as close as possible to the floor and ceiling of the castle. Brackets are attached to the non-movable wall for holding the tray above the gamma tube. The spacing between sample and gamma tube

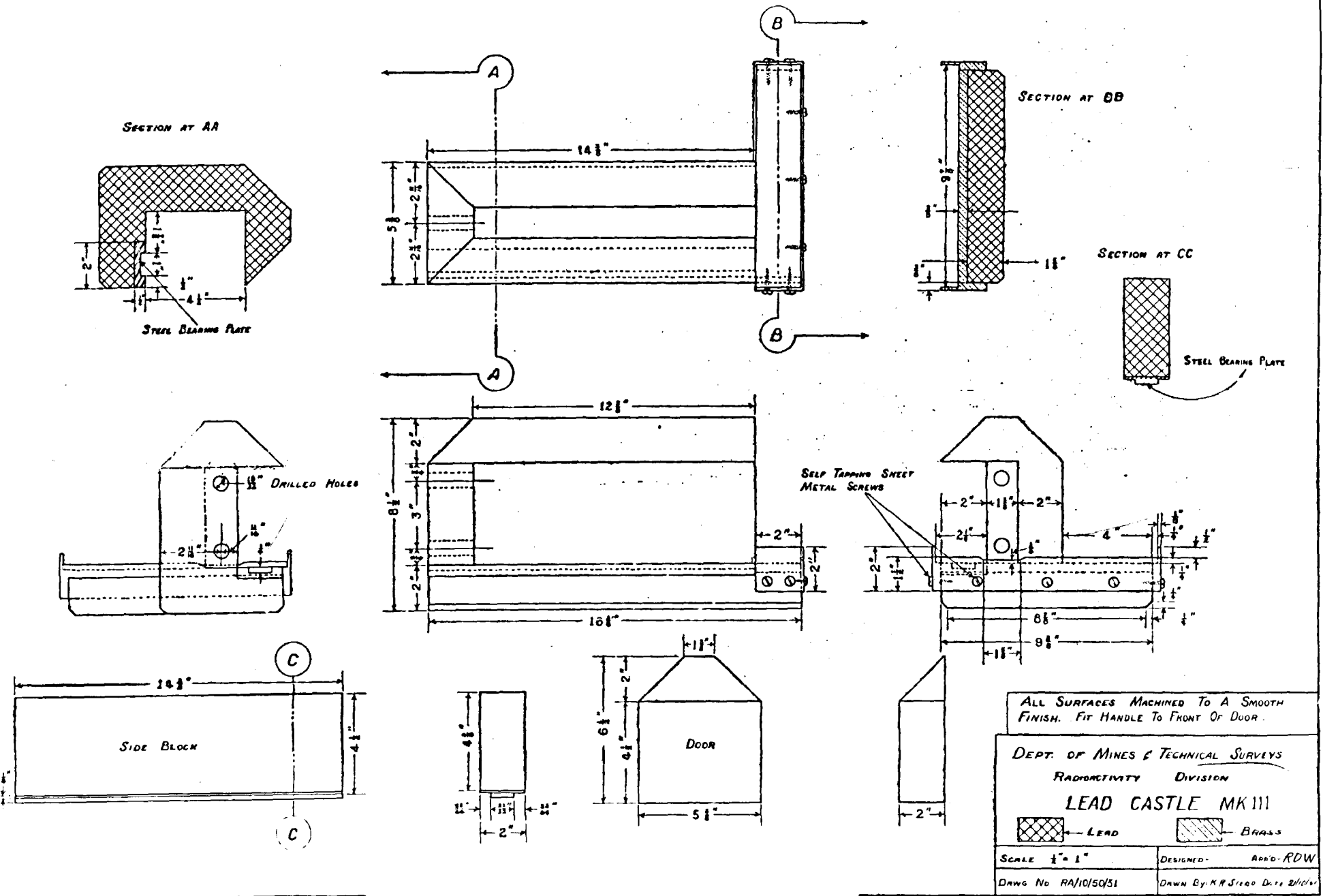


FIGURE 2. WORKING DRAWING FOR LEAD SHIELD

should be kept to a minimum to increase the effective geometry. A strip of lead 1/8" thick, 12 3/8" long, and 1 1/4" wide is supported above the gamma tube by wall brackets, and is used to absorb beta rays and weak gamma radiation from the samples.

Cable

Cables for connecting the beta and gamma tubes to their respective scalers should be shielded, have low inner-to-outer conductor capacity, and possess a voltage rating of at least 1,500 v. Shielded low-capacity cable, such as the Amphenol RG-59/U with a nominal capacity of 21 mmfds per foot, with Type 80M cable plugs to fit 80-F locknut receptacles fitted to the lead castle, has proved satisfactory. Cable lengths should be kept to a minimum and should not exceed 5'.

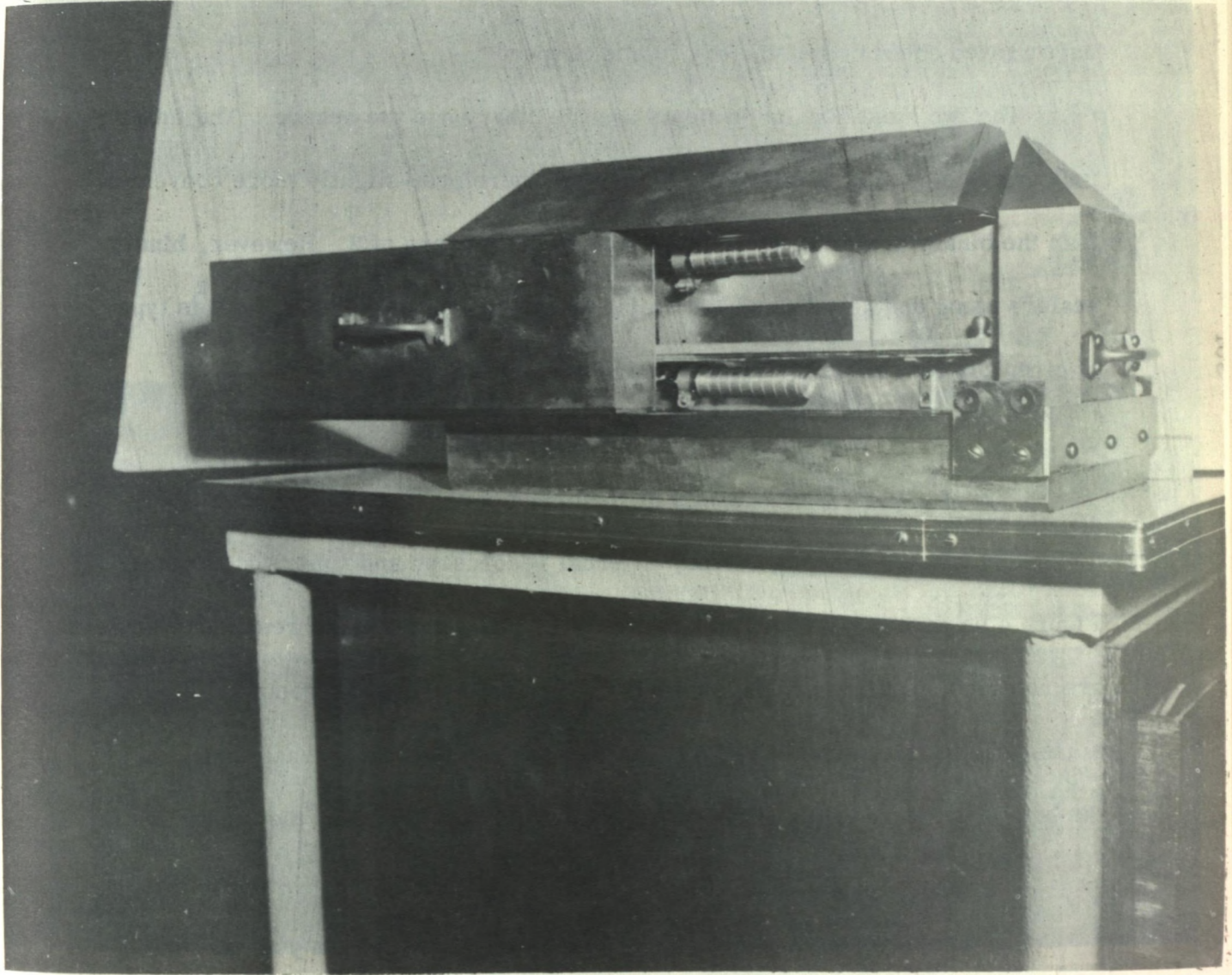
Scaling Circuits

Manufacturers of GM counting systems usually combine the Geiger tube, high voltage supply, and pulse amplifier with the scaling circuit and register drive. The high voltage supply should be capable of delivering up to 1,500 v with 0.5% regulation for a $\pm 10\%$ change in line voltage. The mechanical register and timer may be supplied as accessories or may be built into the scaling unit. The former arrangement facilitates their maintenance.

The input sensitivity of this type of instrument is about 0.25 v and the detector element has to give an output pulse in excess of this to overcome the shunt effect of the cable.

Reliability over long (6 month) periods with a minimum of servicing is a

prerequisite for the sealing circuit. Due to limited tube life or to minor



circuit operation for an operating schedule of 8 hours a day. At least 3 Geiger

Figure 4 - Typical seal and circuit

Figure 3 - Side view of lead shield with side partly removed to show relative positions of beta tube, sample, tray, lead absorber, and gamma tube in lower compartment

the individual tube. In common with most Geiger Muller tubes a few instances of

shell deterioration have been observed.

Reliability over long (6 month) periods with a minimum of servicing is a prerequisite for the scaling circuit. Due to limited tube life or to minor imperfections in circuit design very few of the commercially available instruments have proved entirely satisfactory in this respect.

The two most popular systems are the binary and the decade. The latter indicates the count in multiples of 10 and is, therefore, slightly more convenient than the binary scaler whose scaling factors are powers of 2. However, binary scalers using double triodes have so far proved the more reliable, as this type of circuit is less sensitive to decreasing tube emission.

Scaling Circuit Accessories

The arrangement of lead shield, combined amplifier-scaling circuit-high voltage supply for the Geiger tube, external recorders, and timers is shown in Figure 4 on page 13 . When the external timers on top of their respective scalers are released simultaneously, counts from the gamma tube are recorded by the scaler and register, shown beside the timer, to the left of the lead castle, while the beta count is recorded at the same time by the equipment to the right.

Spare Electronic Equipment

One complete tube complement should be on hand for each year of scaling-circuit operation for an operating schedule of 8 hours a day. At least 3 Geiger tubes should be kept in reserve for each 1 in operation, as the life of the 1B85 Geiger tube varies from 1 to 3 months depending upon the grades assayed and on the individual tube. In common with most Geiger Muller tubes a few instances of shelf deterioration have been observed.

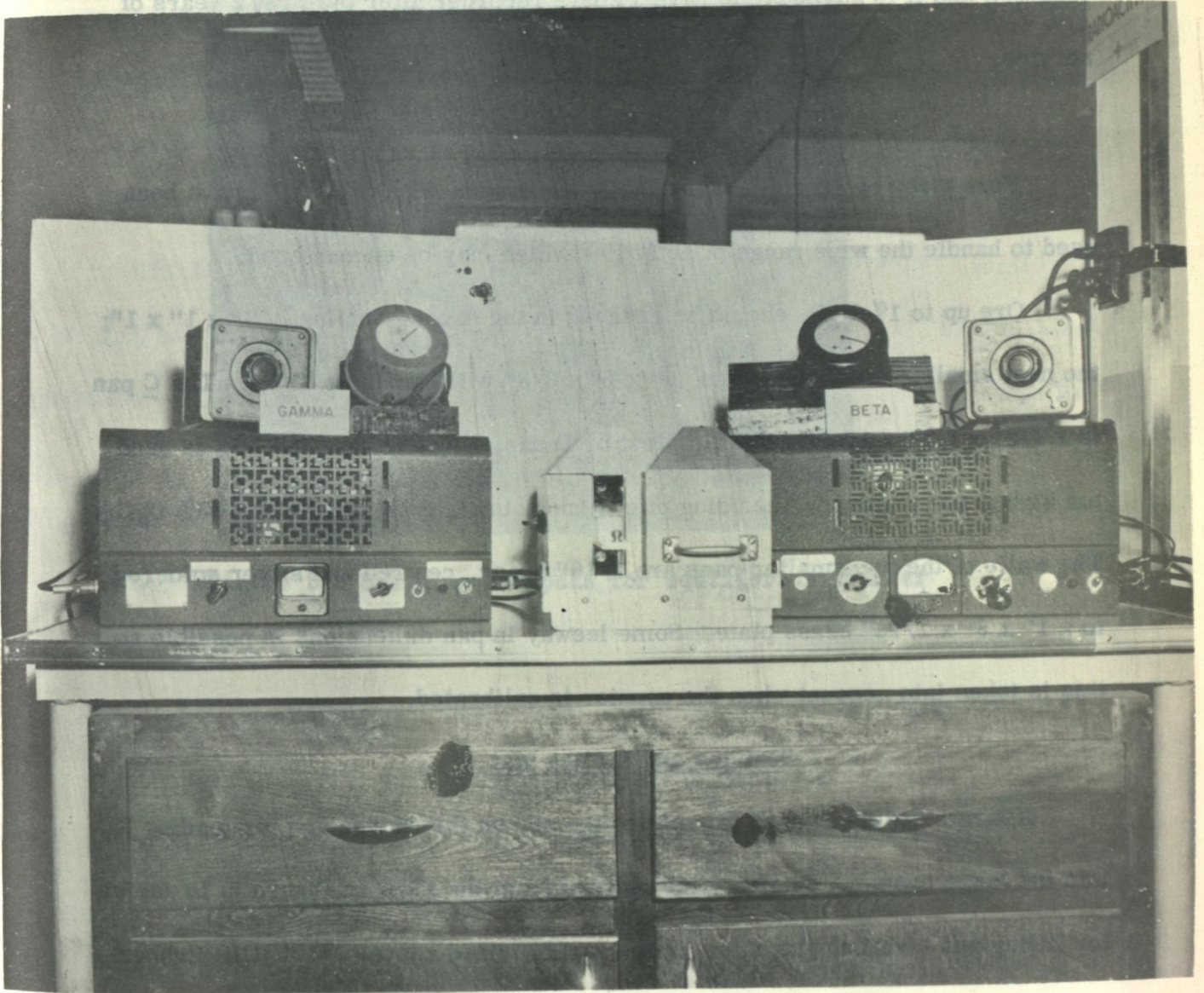


Figure 4 - Typical beta gamma assay system

lined sheet), about 2' x 2' provides a convenient mixing and rolling mat. These operations are described under "Tray".

Tray

When filled, the pan is placed in a shallow tray and slid into the lead shield. The tray is shown at the left in Figure 8 on page 17.

Spare circuit fuses and neon lamps for indicating residual counts are essential and it is advisable to have a spare recorder after the first 2 years of service.

Pans

Four sizes of pans, shown to the left in Figure 5 on page 15, have been used to handle the wide range of activities which may be encountered.

Ore up to 1% U_3O_8 should be assayed in the A pan, nominally 6" x 1" x 1"; the next smaller size, the B pan, 6" x 1" x 3/8", will handle up to 5%. The C pan is 1 1/2" x 1/2" x 1", and the smallest or D pan, 1 1/2" x 1/8" x 3/8", is used for high grade material, including pitchblende, usually assaying about 63% U_3O_8 . The walls of the two smaller pans are 1/16" brass, centred and silver soldered to a 1" x 6" x 1/16" brass plate. Some leeway in pan dimensions is possible as this is taken into account when the counter is calibrated.

Spatulas and Rolling Mat

A large spatula is used for mixing the ore on the canvas and for loading it into the large pans. A smaller size (second from the right in Figure 5) is useful for filling and levelling the other pans. A rubberized canvas sheet (CIL Rubber-lined Sheet), about 2' x 2' provides a convenient mixing and rolling mat. These operations are described under "Sample Preparation".

Tray

When filled, the pan is placed in a shallow tray and slid into the lead shield. The tray is shown at the left in Figure 6 on page 17.

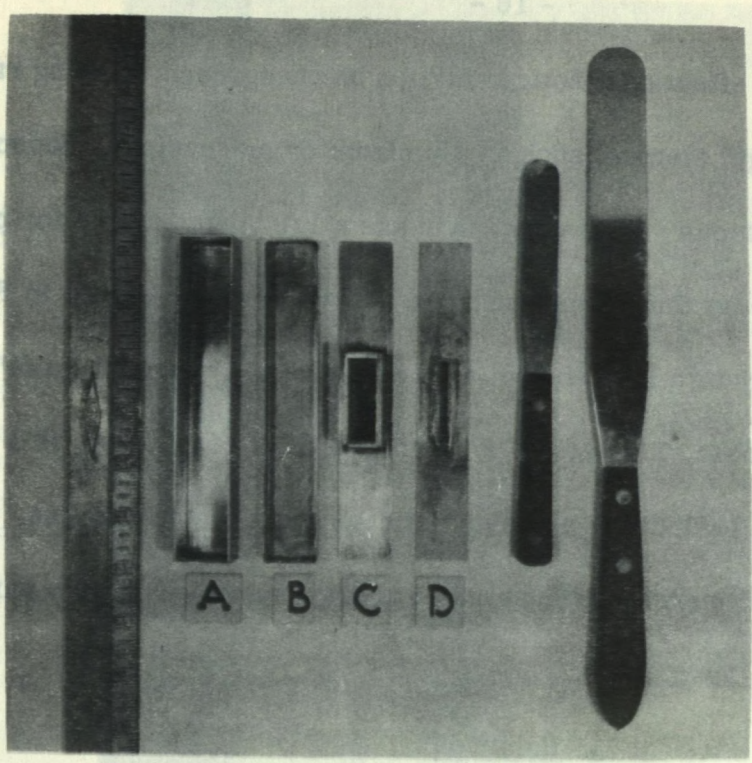


Figure 5 - Spatulas and pans for equilibrium counter

ats the
 and wood
 postting
 wire &
 wide by
 1/18"
 The tray
 row chamber

To reduce the
 tray is made of a
 should also be
 each pan directly
 For use with the
 1/4" thick. The
 wide, 8 1/18" long
 is fitted with a
 in the lead cast

A set of

for weighing the pans empty and loaded. The balance should be accurate to 1/10 gm

Figure 5 - (A) for (B) and should handle up to 500 gm. (A) is 5 cm long and 1.5 cm wide. (B) is 1.5 cm long and 1.5 cm wide.

Operating Log and Assay Sheets

Details of daily operation, including sensitivity runs, backgrounds, plateau checks, and records of each assay, should be maintained, preferably in a still-covered book. Records should identify the Geiger tubes, their normal operating voltage, the duration of the sample run, the sample weight, the size of pan, the scale factor, and final count for both beta and gamma scintels.

At the Mines Branch, the uranium-oxide content of each sample is indicated on a form which is returned to the engineer submitting the sample. Identification of previously assayed samples is facilitated by assigning to each sample to be assayed a reference number indicating the month and year.

To reduce the effect of cosmic radiation on background counting rate the tray is made of a light element such as aluminum or magnesium. Seasoned wood should also be satisfactory. Its upper face has a 1/8" deep groove for positioning each pan directly above the gamma tube and below the beta tube, as in Figure 4. For use with the standard lead shield, the tray is 12 3/8" long by 1 1/4" wide by 1/4" thick. The groove, located 3 3/8" from the front of the tray, is 1 1/16" wide, 6 1/16" long, 1/8" deep, and symmetrical about the centre line. The tray is fitted with a small knob at its front end for easy removal from its narrow chamber in the lead castle.

Balances

A set of balances, such as the Cenco single-beam trip scale, is necessary for weighing the pans empty and loaded. The balances should be accurate to 1/10 gm and should handle up to 500 gm.

Operating Log and Assay Sheets

Details of daily operation, including sensitivity runs, backgrounds, plateau checks, and records of each assay, should be maintained, preferably in a stiff-covered book. Records should identify the Geiger tubes, their normal operating voltage, the duration of the sample run, the sample weight, the size of pan, the scale factor, and final count for both beta and gamma scalers.

At the Mines Branch, the uranium-oxide content of each sample is indicated on a form which is returned to the engineer submitting the sample. Identification of previously assayed samples is facilitated by assigning to each sample to be assayed a reference number indicating the month and year.

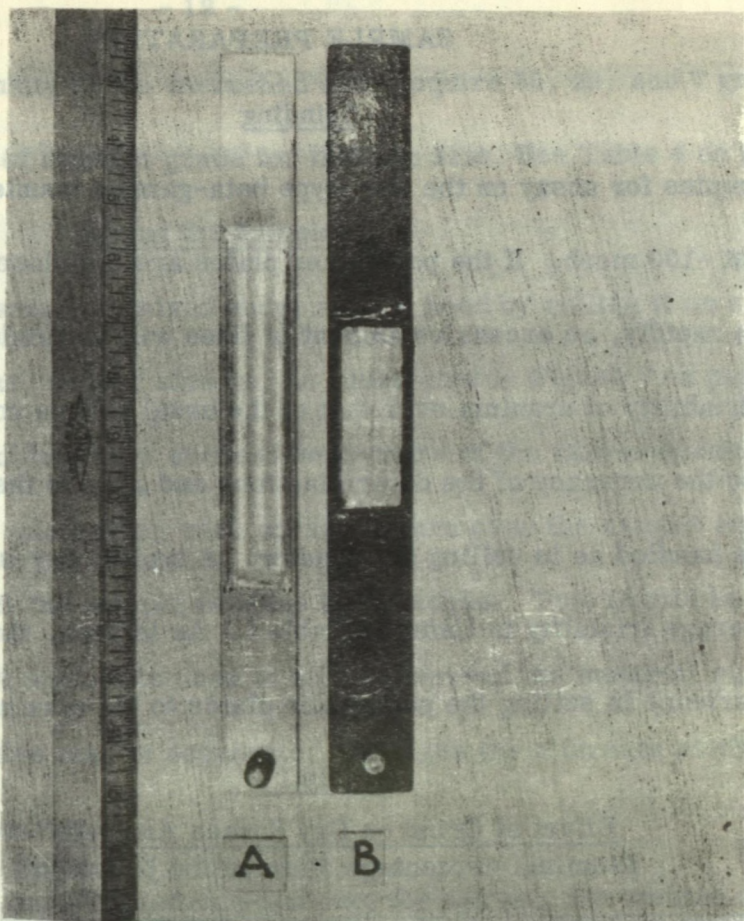


Figure 6 - Tray (A) for holding sample pans and sensitivity standard (B)

STANDARDS AND CALIBRATION

The large pan holds approximately 250 gm of

powdered sample. The smaller pan holds approximately 25 gm of

powdered sample. The degree of pulverization of the sample and

* Brass pulverizers have been used for this work.

SAMPLE PREPARATION

Grinding

Samples for assay on the tray-type beta-gamma counter should be ground* to at least -100 mesh. If the pulverizer plates are too close or if the pulverizer is fed too rapidly, an excessive amount of fines will be produced due to the greater friability of uranium over that of the usual gangue constituents. This influences the accuracy of the determinations and adds to the dust hazard when the sample is handled as in rolling and loading. A typical dry-screen analysis for an ore of average friability is listed in Table I. As is seen, the results depend largely upon experience in setting the pulverizer plates to the maximum nominal size.

Table I

Effect of Grind on Dry Screen Analysis
(Nominal percentage distribution by weight)

| Mesh | -48 | -65 | -100 | -150 | -200 | | |
|------|-----|------|------|------|------|------|------|
| Size | +48 | +65 | +100 | +150 | +200 | +325 | -325 |
| -48 | 4.7 | 16.9 | 15.4 | 11.0 | 9.2 | 9.5 | 33.4 |
| -100 | | | | 4.3 | 8.7 | 10.6 | 76.4 |
| -150 | | | 0.5 | 1.0 | 10.7 | 13.5 | 74.4 |
| -200 | | | | | | 2.3 | 97.7 |

As the sample was not screened during pulverization most of its weight is concentrated in the fines.

Weight of Sample Required

The large pan holds approximately 120 cc of sample so that 250 gm of pulverized sample is ample. The smaller pans hold approximately 32, 12, and 1 cc and, depending chiefly upon the degree of pulverization of the sample and to a

* Braun pulverizers have been used for this work.

lesser extent on the density of the uncrushed ore, require 50, 25, and 7 gm respectively of sample of suitable grade for that pan size. See Table 4 on page 28 .

Mixing the Sample

The pulverized sample is mixed under a fume hood by rolling it on a piece of rubber-backed canvas, rubber side up, as illustrated in Figure 7 on page 21 .

This is done by folding over successive corners of the canvas sheeting, normally about 2' x 2', and pulling each corner in turn over the sample and past its opposite corner until the sample is close to the edges. This should be repeated, in rotation, 8 times. A spatula is used to mix and spread the material since the heavier uranium particles tend to segregate. Continue the alternate rolling and mixing for at least 2 minutes.

After it has been assayed and emptied onto the canvas, the sample can be returned to its original container using the large spatula as a ladle. Last traces of the sample can be transferred to the sample envelope by lifting up the mat and utilizing it as a funnel. These operations must be done under cover, such as a fume cupboard with exhaust fan, to eliminate the possibility of dust inhalation. The rubber-lined canvas should then be washed with water or rubbed with a damp cloth and dried before using again. Depending upon the usage and quality of the canvas, mats should last about a month before cracks appear.

STANDARDS AND CALIBRATION

Desirable Characteristics of a Standard

Desirable features of a radioactive standard include constancy (a long parent half life, 10^9 years for uranium) and freedom from foreign radioactive constituents.

In the case of uranium standards, all members of the uranium series should be present and in equilibrium with each other and the ore should be free from potassium* and thorium. The last two are estimated to be three times as abundant in the earth's surface as uranium. These conditions are fulfilled by the pitchblende ore found at Port Radium, Northwest Territories, and this ore was used in preparing the various grades of radioactive standards used at the Mines Branch.

Porosity of the ore also influences its suitability as a standard because the more porous ores, such as the carnotite deposits of the Colorado Plateau, permit the ready escape of the gaseous member, radon, of the uranium series. This increases the background during a determination to an extent dependent upon the time required for the determination, the activity, the emanating power, and the degree of pulverization of the sample. Sintering may be used to seal in the radon but it is time consuming. Eldorado standards are suitable in this respect as their emanating power is very small.

Sources of Supply

Pulverized standards ranging in activity from 0.026 to 48.0% U_3O_8 may be obtained at a nominal charge of \$7.50 per 100 gm from the Radioactivity Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa. These standards are thorium-free in equilibrium and have negligible emanating power.

* Potassium has a slight beta and gamma activity. Approximate beta and gamma equivalents of 1% potassium are 0.0007% and 0.0003% U_3O_8 respectively.

Once the beta and gamma activities of the sample are related with Mines Branch standards by determining the ratio between the beta and gamma assay

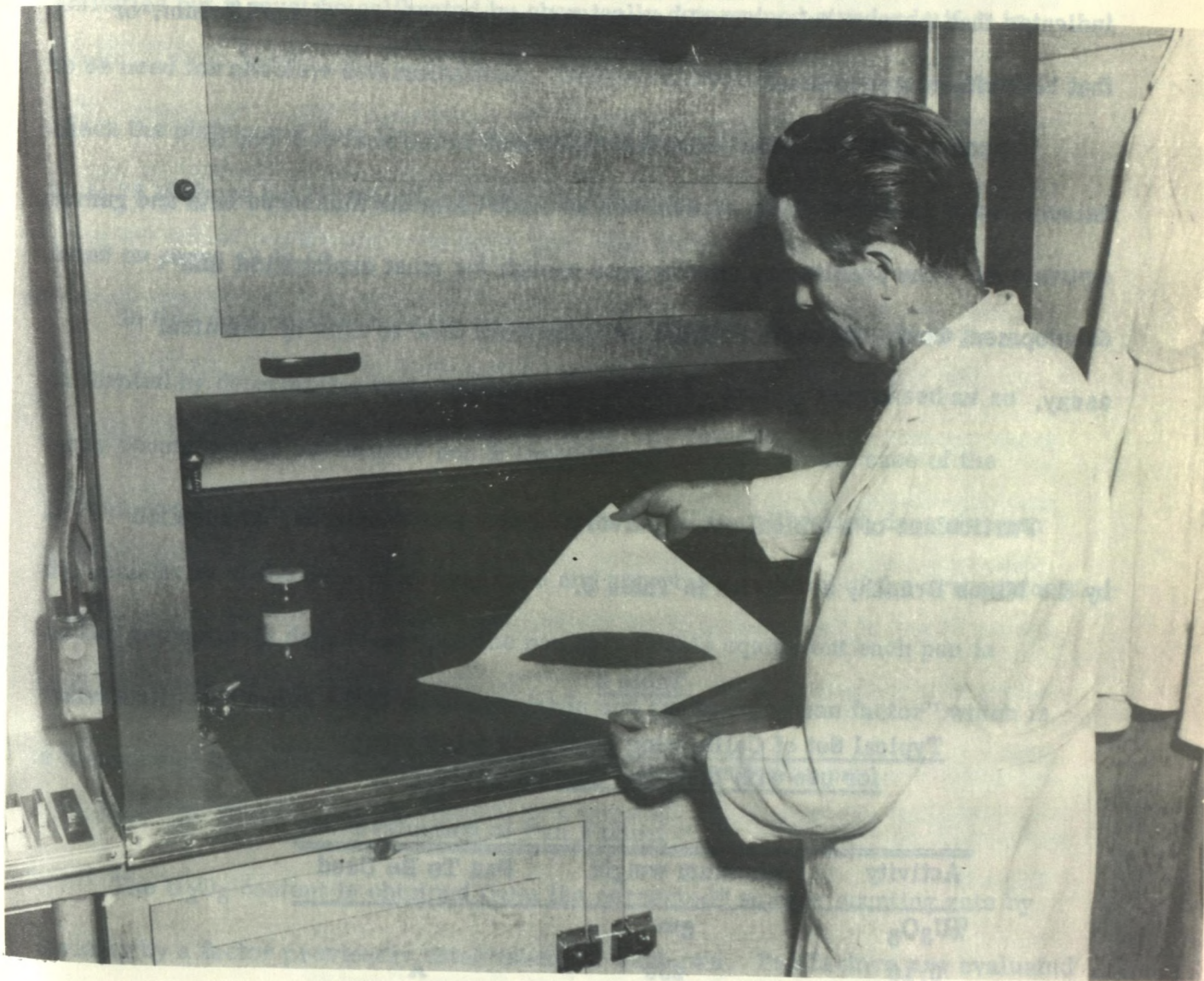


Figure 7 - Mixing pulverized sample under a fume hood

by running a standard sample and dividing the corrected counting rate by the U_3O_8 content of the standard. The beta pan factor is calculated at the same time, as in the following equations:

$$\text{Beta pan factor} = \frac{\text{Corrected counting rate}}{\%U_3O_8 \text{ of standard}} \quad (2)$$

$$\text{Gamma pan factor} = \frac{\text{Corrected counting rate per gm}}{\%U_3O_8 \text{ of standard}} \quad (3)$$

* For background and sensitivity, counting rate is expressed in counts per minute (c/min).

Custom Analyses

Once the beta and gamma scalers have been calibrated with Mines Branch standards, a discrepancy greater than 30% between the beta and gamma assay indicates that the particular sample contains thorium or is out of equilibrium, or that both effects are present.

In such cases, using the procedure described in the next section, a calculation of the actual U_3O_8 content can be made from the measured beta and gamma equivalents. The results are usually good enough for most exploration and development work, but checks should be made from time to time by chemical assay.

Range and Weight

Particulars of a typical set of pulverized uranium standards, as supplied by the Mines Branch, are listed in Table 2.

Table 2

Typical Set of Calibration Standards (-100 mesh)
for use with the Beta-Gamma Counter

| Activity | Minimum weight | Pan To Be Used |
|------------|----------------|----------------|
| % U_3O_8 | gm | |
| 0.10 | 250 | A |
| 0.55 | 250 | A or B |
| 1.37 | 100 | B or C |
| 8.76 | 50 | C |
| 18.67 | 50 | D |
| 35.47 | 50 | D |
| 48.0 | 50 | D |

Determination of Specific Sensitivity

The beta-gamma counter offers a convenient means of comparing radio-activities but it must be calibrated by chemically determined standards if it is to be used for absolute determinations. Before calibration, the operator should check the plateaus of both Geiger tubes and set the individual voltages at from 50 to 75 V above the threshold. Further information on plateau plotting may be found on pages 42 to 44.

In this dual counter both the beta and the gamma sections should be calibrated by determining their specific sensitivities, usually expressed as so many counts counts per minute per % U_3O_8 (c/min/%), and in the case of the gamma counter, per gram as well (c/min/gm/%). The calibration depends upon the sensitivity of the beta or gamma tube and upon the geometry of the arrangement. As the geometry is dependent upon the pan size in this equipment each pan is individually calibrated with known standards and assigned a "pan factor" which is a measure of the overall counter sensitivity for that particular pan.

Evaluation of Pan Factor

The U_3O_8 content is obtained from the corrected* sample counting rate by dividing by a factor previously established for each pan. Pan factors are evaluated by running a standard sample and dividing the corrected counting rate by the U_3O_8 content of the standard. Both beta and gamma pan factors are established at the same time, as in the following equations:

$$\text{Beta pan factor} = \frac{\text{Corrected counting rate}}{\%U_3O_8 \text{ of standard}} \quad (2)$$

$$\text{Gamma pan factor} = \frac{\text{Corrected counting rate per gm}}{\%U_3O_8 \text{ of standard}} \quad (3)$$

* For background and sensitivity. Counting rate is expressed in counts per minute (c/min).

Two grades of standards should be used with each pan to determine its beta and gamma pan factors. Thus, the 0.30% and the 1.37% standards would be used to find the average factor of the large pan.

With the gamma counter, the pan factor would be determined by dividing the net counting rate per gram of standard by its percentage U_3O_8 . The number of runs to be made in establishing the pan factor depends upon the stability of the assay unit. Refills should agree with each other to within 5% and 1% for the gamma and beta counts respectively. A minimum of 2 runs with each standard is desirable.

Pan factors for the beta counter are determined in a similar manner, but it is not necessary to correct for the variation of sample weight by adjusting to a nominal value unless an accuracy of better than 2% is required. Due to the limited power of penetration of beta particles, the sample is infinitely thick when filled to the top of any of the standard sizes of pan. Additional mass causes only a small increase in the beta count owing chiefly to the slight gamma sensitivity of the beta tube.

Typical pan factors for the apparatus described in this manual are listed in Table 3.

Table 3
Typical Pan Factors for Beta-Gamma Counter

| Pan | Beta Pan Factor C/min/% U_3O_8 | Gamma Pan Factor C/min/gm/% U_3O_8 |
|-----|-------------------------------------|---|
| A | 3,624 | 2.28 |
| B | 2,824 | 3.42 |
| C | 1,072 | 3.35 |
| D | 170 | 5.20 |

As these values are dependent upon several factors, including Geiger tube sensitivities, pan size, thickness of lead absorber above the gamma tube, and sample distance from the beta and gamma tube, actual pan factors may vary somewhat from those listed in Table 3. They are presented to illustrate their relative values.

ASSAY PROCEDURE

Theory

Assuming normal operation of the assay equipment, including correct voltages on the beta and gamma tubes, the beta counting rate is proportional to the surface activity and the gamma counting rate is dependent upon the total uranium content of the sample, and hence on the sample weight. The lower the grade, the more important it is to correct for the fluctuating background which is measured before and after each run. The average background is then subtracted from the sample count to give the net sample count. This, when divided by the pan factor and by the sample weight in the case of the gamma count, and corrected for sensitivity, gives the percentage U_3O_8 , by weight, of the sample as determined by both the beta and gamma counters.

If the beta and gamma determinations are identical, the sample is in equilibrium and is free from thorium. When the gamma assay value is higher than the beta assay either the presence of thorium or the loss of one or more of the first four members of the uranium series* is indicated. If the gamma count is lower than the beta, either potassium is present or one or more gamma emitters

* Properties of the members of the uranium, thorium and actinium series are listed in Appendix 1.

in the uranium series are missing. The true U_3O_8 content can be found in the absence of potassium by substituting the beta and gamma assays into the following equation. The derivation of this equation is found in Appendix 3.

$$C = \beta + Y (\beta - \gamma) \quad \text{when } \beta > \gamma \quad (4)$$

$$\text{or } C = \beta - Y (\gamma - \beta) \quad \text{when } \gamma > \beta$$

where C is the true U_3O_8 content, β is the beta assay, γ is the gamma assay, and Y is the ratio of the relative beta activities of the uranium and radium parts of the uranium family. If the value of Y be taken as unity (Appendix 3), Equation 4 reduces to:

$$C = 2\beta - \gamma \quad (1)$$

Background

Even with no sample in the lead castle, a count (varying from 20 to 50 c/min* for the Victoreen 1B85 Geiger Tube) will be recorded by the equipment. This so-called background is due to energetic radiation, including cosmic radiation and rays from certain members in the uranium and thorium series, penetrating the 2" lead shielding and actuating the Geiger tubes. High grade ore and standards should be stored at least 30' from the lead chamber to keep the background below 30 c/min.

As the lowest grade that can be detected is limited by the magnitude of the background this should be kept to a minimum. It can be reduced but not eliminated by thicker shielding. A shield thickness of 2" was chosen as a compromise between theoretical and practical considerations of additional weight and cost. Backgrounds registered by the beta and gamma tubes should agree to within 20% in

* The lower limit is obtained with an extra 1 1/2" of lead around the castle. The upper limit depends on the proximity of large U_3O_8 content.

normal operation, if the background is measured over a 10-minute period.

Although the tray and empty pan are usually left in the lead shield while the background is being determined, it is quite feasible to do this without the pan if one is sure that it is not contaminated. Such a condition would be indicated by a higher background count with the pan than without it. Contaminated pans may be cleaned by rinsing in a grease remover such as carbon tetrachloride or by chemical solvents. It is a good plan to clean the walls and floor of the beta tube compartment once a month with a rag soaked in a grease solvent to remove any radioactive dust.

Choice of Pan

The criterion in selecting a pan is to obtain as high a counting rate as possible providing it does not exceed the limits of reliability of the mechanical recorder and scaling circuit. On the scale of 64 and a recorder limit of 3 c/sec this would correspond to a limit of 192 c/sec. As a trial the largest pan should be used unless the uranium content of the sample is known to exceed 1%. A preliminary run of a few moments on the highest scaling range will indicate whether a smaller pan or a lower scale should be used. The former is indicated by a recording rate in excess of 3 c/sec, and the latter if the rate is less than 1 c/sec. Extremely low grade samples should be run in the largest pan and on the lowest scale whereas more active samples should be run in smaller trays and at higher scaling factors as indicated in Table 4.

Table 4

Maximum Grades for the Various Pans

| Maximum U ₃ O ₈ Content | Pan Size | Nominal Sample Weight |
|--|-------------|-----------------------|
| % | | gm |
| 2 | A | 115 |
| 3 | B | 50 |
| 7 | C | 25 |
| 45 | D | 7 |

The indicated uranium content is the maximum grade that can be used in the corresponding pan size with the beta scale at 64 and a recorder speed of 2 c/sec. The D pan may be used up to 67% U₃O₈ corresponding to a recorder speed of 3 c/sec.

Pan Filling, Levelling, and Weighing

Due to the large percentage of fines in ore pulverized to -100 mesh such operations as rolling (Figure 7), loading, and emptying the pan should be done with extreme care and preferably under cover (such as a fume hood) to reduce danger of inhalation of radioactive dust particles.

The pans are most conveniently filled above the rubber canvas using the large spatula for loading the pulverized sample into the two larger pans and the smaller spatula for the other sizes. The latter is useful for packing in the sample and for levelling its surface flush with the edges of the pan. This is done by drawing the inclined spatula across the top of the pan in a manner similar to that of smoothing mortar with a trowel. Excess sample may be removed from the flat parts of the smaller pans with a 3/8" paint brush.

The filled pan is then weighed to the nearest 1/10 gm and the weight of the pan is subtracted from this to ascertain the net sample weight.

Inserting Sample into Chamber

The aluminum or magnesium tray (Figure 6) provides a convenient means for inserting the sample into the narrow lead chamber and of centering each pan with respect to the beta and gamma tubes. All sizes of sample pans fit snugly into a 1 1/16" wide groove in the upper face of the tray.

Background and Sample Determinations

As background is always present during a sample run, due allowance has to be made. Thus it is apparent that the lower the activity of the unknown the greater the need for accuracy in determining the background. From statistical considerations applicable to the random nature of radioactive disintegrations it is necessary to record 180 counts to reduce the per cent probable error* to 5 (Figure 8). At an average background of 35 c/min this would take 5 minutes and 43 seconds. Thus 5 minutes would be the minimum time and 10 minutes the usual time for background determinations. These are made before and after the sample run, which is then corrected for background by subtracting the average of the two runs.

After a trial run to determine the largest pan that can be used without overloading the mechanical register, the duration of the sample run is chosen from 3 to 10 minutes, depending upon the activity. For example, samples containing less than 0.01% should be assayed in the large pan for at least 5 minutes. Longer sample runs should have correspondingly longer background determinations.

* Per cent probable error = $\frac{67.4}{\sqrt{N}}$ where N is the total number of counts.

On completion of the sample run, the total (and residual) count is noted from the mechanical register and when multiplied by the scale factor and divided by the duration of the run in seconds gives the gross average counting rate.

The pan is then removed, emptied, and depending upon the accuracy desired, may be refilled, weighed, and assayed again or cleaned directly and placed in the castle for the background run. This should be made as soon as possible after the sample run.

As mentioned earlier, it is quite feasible to make the background determination without the pan but it is necessary to ensure that the latter is not contaminated.

Allowance for Background

The average of the background runs immediately preceding and following the sample determination should be subtracted from the sample counting rate to obtain the net counting rate.

Correction for Sensitivity

Fluctuations in the overall sensitivity of the assay unit, including increased threshold voltage of the Geiger tube* and changes in the scaling circuit, may be detected and compensated for by periodically measuring the activity of a fixed source, the "Mariner's Compass" of the operator. This may be made by filling a 1/4" deep pan of about one-third the area of the A pan with high-grade ore of about 50% U_3O_8 , sealing with molten paraffin, and covering with thin aluminum foil to prevent overloading the beta counter by reducing the beta count.

* Due to decreased life with use, or to changes in temperature.

A sensitivity standard of this type (Figure 6) has beta and gamma activities of approximately 7,050 and 4,160 c/min respectively. It is physically similar to the tray but is not used with the latter. The sensitivity standard should not be confused with ore standards, which have to be loaded into pans for each determination.

Ten-minute sensitivity checks should be run at the start, middle, and end of the day, or more often if the readings are not reasonably consistent or if the line voltage fluctuations exceed a few per cent. Sensitivity runs should be taken before and after the calibration tests on each pan. The average of the sensitivity runs made while determining the pan factors is used as the reference sensitivity.

The net sample counting rate is corrected for possible changes in equipment sensitivity by multiplying it by the factor,

$$\frac{\text{reference sensitivity (a constant)}}{\text{average sensitivity}}$$

For example, if the gamma reference sensitivity is 4,160 c/min and the average of two bordering sensitivity runs is 4,200 c/min, the net sample counting rate should be multiplied by $\frac{4,160}{4,200}$. It should be noted that the reference sensitivity always appears in the numerator of the correction factor. Its application is illustrated in the following section.

Sample Calculations

Pulverized ore is brought in for beta-gamma assay. Make a 5-minute sensitivity run followed by a 10-minute background run. Then load the ore into the largest (A) pan, which is inserted for a trial run on the scale of 64. If it is found that the beta counts are too rapid for reliable operation of the mechanical

recorder, the sample should be re-loaded into the next smaller pan. If the resulting beta register counting rate is less than 1 c/sec, the scale selector switch should be rotated to a lower scaling factor until the beta mechanical register is operating at about 2 c/sec. Owing to the lower efficiency of the gamma tube, the gamma register rate will be considerably less* than the beta register. Set the timer manually to 5 minutes, note the reading on the mechanical register, and begin the run (at the end of the timed interval the scaler is automatically shut off). To ascertain the gross counting rate, subtract the old register reading from the new one, multiply by the scale factor, and divide by the time for the run. Then, to find the net counting rate, subtract the average of the backgrounds immediately preceding and following the sample run.

$$\frac{(\text{Gross counts} \times \text{scale factor}) - \text{Av. background}}{\text{Interval (minutes)}} = \text{Net c/min} \quad (5)$$

Correct for possible changes in sensitivity by multiplying the net counting rate by the reference sensitivity over the average or nearest sensitivity run. Then divide by the previously established pan factor for that particular pan. Thus the corrected beta assay is given by the following equation:

$$\%U_3O_8 = \text{Net sample counting rate} \times \frac{\text{Reference sensitivity}}{\text{Av. sensitivity}} \times \frac{1}{\text{Beta pan factor}} \quad (6)$$

The corrected gamma assay for the same sample is equal to:

$$\%U_3O_8 = \frac{\text{Net sample counting rate}}{\text{Sample wt (gm)}} \times \frac{\text{Reference sensitivity}}{\text{Av. sensitivity}} \times \frac{1}{\text{Gamma pan factor}} \quad (7)$$

* About 1/6 of the beta tube counting rate using a single 1B85 and a 1/8" thick lead absorber below the sample.

As mentioned on page 7 there are three possible combinations of beta and gamma determinations:

- Beta assay higher than gamma.
- Beta and gamma assay agree to within $\pm 10\%$.
- Beta assay lower than the gamma.

In all three possibilities the true U_3O_8 content may be found with an accuracy to within 10% for most samples, by substituting in Equation 1*:

$$\text{True } U_3O_8 \text{ content} = \text{Twice beta assay} - \text{Gamma assay} \quad (1)$$

The following examples are typical of samples falling into the three groups.

Example 1

Beta assay higher than gamma assay.
 Sample weight -124.2 gm
 Run in A pan for 10 minutes

| | <u>Gamma Counter</u> | <u>Beta Counter</u> |
|-------------------------------------|---------------------------------|-----------------------------------|
| Pan factor (A pan) | 2.28 c/min/gm/% | 3,624 c/min/% |
| Reference sensitivity (c/min) | $650 \times \frac{64^{**}}{10}$ | $1,100 \times \frac{64^{**}}{10}$ |
| 10-min background before (c/min) | 27.1 | 31.5 |
| Sample run (total counts) | 1,330 | 17,262 |
| 10-min background after (c/min) | 25.0 | 30.6 |
| Sensitivity (c/min) | $663 \times \frac{64}{10}$ | $1,092 \times \frac{64}{10}$ |

* See Appendix 3 for derivation of this equation.

** The scaler here was operating on a scale of 64, that is, for every 64 counts the scaler causes the number shown by the mechanical register to increase by one. The gamma and beta registers in this example showed a net reading of 650 and 1,100 respectively over a 10-minute counting period. Thus the reference sensitivity was $650 \times \frac{64}{10}$ c/min.

Calculation of average background:

| | <u>Gamma</u> c/min | <u>Beta</u> c/min |
|---------|-----------------------|----------------------|
| Before | 27.1 | 31.5 |
| After | 25.0 | 30.6 |
| Average | 26.0 | 31.0 |

Net counting rate:

$$\begin{aligned} \text{Beta} &= \frac{(\text{Recorder count} \times \text{Scale factor})}{\text{Interval (min)}} - \text{Av background} \\ &= \frac{17,262}{10} - 31 = 1,695 \text{ c/min} \end{aligned}$$

$$\begin{aligned} \text{Gamma} &= \frac{(\text{Recorder count} \times \text{Scale factor})}{\text{Interval (min)}} - \text{Av background} \\ &\times \frac{1}{\text{Sample wt (gm)}} \\ &= \frac{(1,330 - 26)}{10} \times \frac{1}{124.2} = \frac{107}{124.2} = 0.862 \text{ c/min/gm} \end{aligned}$$

Correcting for sensitivity:

$$\text{Beta net counting rate} = 1,695 \times \frac{1,100}{1,092} = 1,708$$

$$\text{Gamma net counting rate per gm} = 0.862 \times \frac{650}{623} = 0.845$$

Calculation of beta and gamma assay:

$$\begin{aligned} \%U_3O_8 \text{ (Beta assay)} &= \frac{\text{Net beta counting rate}}{\text{Beta pan factor}} \\ &= \frac{1,708}{3,624} = 0.473 \%U_3O_8 \end{aligned}$$

$$\begin{aligned} \%U_3O_8 \text{ (Gamma assay)} &= \frac{\text{Net gamma counting rate per gm}}{\text{Gamma pan factor}} \\ &= \frac{0.845}{2.28} = 0.371 \%U_3O_8 \end{aligned}$$

$$\begin{aligned}
 \text{True U}_3\text{O}_8 \text{ content} &= \text{Twice beta assay} - \text{Gamma assay} \\
 &= 2 \times 0.473 - 0.371 \\
 &= 0.57 \% \text{U}_3\text{O}_8
 \end{aligned}$$

(1)

which agrees with the chemical assay of 0.52% to within 10%.

Example 2

Beta and gamma assays equal.
 Sample weight-114.2 gm.
 Run in A pan for 10 minutes.

| | <u>Gamma Counter</u> | <u>Beta Counter</u> |
|-------------------------------------|---|---|
| Pan factor (A pan) | 2.28 c/min/gm/% | 3,624 c/min/% |
| Reference sensitivity (c/min) | $650 \times \frac{64}{10}$ | $1,100 \times \frac{64}{10}$ |
| 10-min background before (c/min) | 21.3 | 19.4 |
| Sample run (total counts) | 2,994 | 36,624 |
| 10-min background after (c/min) | 22.7 | 22.2 |
| Sensitivity (c/min) | $666 \times \frac{64}{10}$ | $1,079 \times \frac{64}{10}$ |
| Average background | 22.0 | 20.8 |
| Net counting rate (c/min) | $\frac{2,994}{10} - 22 = 277$ | $\frac{36,624}{10} - 20.8 = 3,642$ |
| Correcting for sensitivity | $277 \times \frac{650}{666} = 270$ | $3,642 \times \frac{1,100}{1,079} = 3,714$ |
| Assay | $\frac{270}{114.2} \times \frac{1}{2.28} = 1.04\% \text{U}_3\text{O}_8$ | $\frac{3,714}{3,624} = 1.02\% \text{U}_3\text{O}_8$ |

$$\begin{aligned} \text{True } U_3O_8 \text{ content} &= 2\beta - \gamma \\ &= 2.04 - 1.04 = 1.00 \%U_3O_8 \end{aligned}$$

which agrees with the chemical assay of 1.00 %U₃O₈.

This particular example is well within the usual agreement of 10% with chemical determinations whose accuracy varies from 3 to 5%.

Example 3

Beta assay lower than the gamma assay.
Sample Weight-20.48 gm.
Run in C pan for 10 minutes.

| | <u>Gamma Counter</u> | <u>Beta Counter</u> |
|--|-------------------------------------|--|
| Pan factor (C pan) | 3.35 c/min/gm/% | 1,072 c/min/% |
| Reference sensitivity (c/min) | $650 \times \frac{64}{10}$ | $1,100 \times \frac{64}{10}$ |
| 10-min background before (c/min) | 19.9 | 22.2 |
| Sample run (total counts) | 88.52×64 | $1,032.82 \times 64$ |
| 10-min background after (c/min) | 21.8 | 26.7 |
| Sensitivity (av of run before and after) | $677 \times \frac{64}{10}$ | $1,082 \times \frac{64}{10}$ |
| Average background (c/min) | 20.8 | 24.4 |
| Net counting rate (c/min) | $\frac{88.52 \times 64}{10} - 20.8$ | $\frac{1,032.82 \times 64}{10} - 24.4$ |
| | = 545 | = 6,505 |
| Correcting for sensitivity | $545 \times \frac{650}{677}$ | $6,585 \times \frac{1,100}{1,082}$ |
| | = 523 | = 6,700 |

$$\text{Assay} \quad \frac{523}{20.5} \times \frac{1}{3.35} = 7.62 \% \text{U}_3\text{O}_8 \quad \frac{6700}{1072} = 6.25 \% \text{U}_3\text{O}_8$$

$$\begin{aligned} \text{True U}_3\text{O}_8 \text{ content} &= 2 \beta - \gamma \\ &= 2 \times 6.25 - 7.62 = 4.9 \% \text{U}_3\text{O}_8 \end{aligned}$$

which agrees with the chemical assay of 5.1% U₃O₈ to within 10%.

FACTORS AFFECTING ACCURACY

Apart from possible fluctuations in overall counter sensitivity and assuming reliable scaling circuit operation within the limits claimed by the manufacturer, reliable assays are improbable unless the following factors are considered.

Errors at Low Counting Rates

Since the instantaneous rate of radioactive decay fluctuates greatly, the longer the interval over which this is measured, the greater will be the accuracy. This may be expressed mathematically by stating that the probable error* of a random process obeying the Poisson Distribution Law is equal to $0.674 \sqrt{m}$ where m is the total counts recorded in a given period. Hence the probable error increases but the percentage probable error decreases with the total number of counts as shown in Figure 8 on page 39. The standard deviation is that within which there is a 68.27% chance of the true result being found. There is also a 19 to 1 (95.45%) chance of the true result being within twice the standard deviation of the observed value.

Thus, to limit the probable error to 2%, 1,015 counts would have to be recorded. As the average background is only 30 or so c/min, a 34-minute run

* Probable error is the value of the most likely deviation of a series of readings from their average. Half of the deviations will exceed and half will be within the probable error.

would be necessary to achieve this accuracy. Unless the sample activity is of the same order as the background, 10-minute backgrounds corresponding to a probable error of 3.8% are usually sufficient. It is interesting to note in the latter case that although the time has been reduced to one third, the per cent probable error has not even doubled.

Losses at High Counting Rate

Accurate measurement of high counting rates is limited by:

1. Dead time of mechanical register (1/16 sec approx.)
2. Resolution of scaler and recorder (about 40,000 c/sec)
3. Geiger-tube dead time (100 - 200 μ sec)

Hence on a scale of 64 counter the limiting factor for reliable counting is

the dead time of the mechanical recorder. Assuming reliable register operation at 16 c/sec this would correspond to a scaler input of 1,020 c/sec.

For a particle arrival rate of p per second suppose that r c/sec are recorded. If the minimum resolving time of the counter is $1/q$ sec, the counter is unable to operate for r/q sec, during which time pr particles arrive and are not recorded.

$$\text{Hence, } p = r + \frac{pr}{q} \text{ or } p = r / (1 - r/q) \tag{8}$$

$$\text{If } r = 1,020 \text{ and } q = 40,000, \text{ then } p = \frac{1,020}{1 - \frac{1,020}{40,000}} = \frac{1,020}{0.97} = 1,049 \text{ c/sec}$$

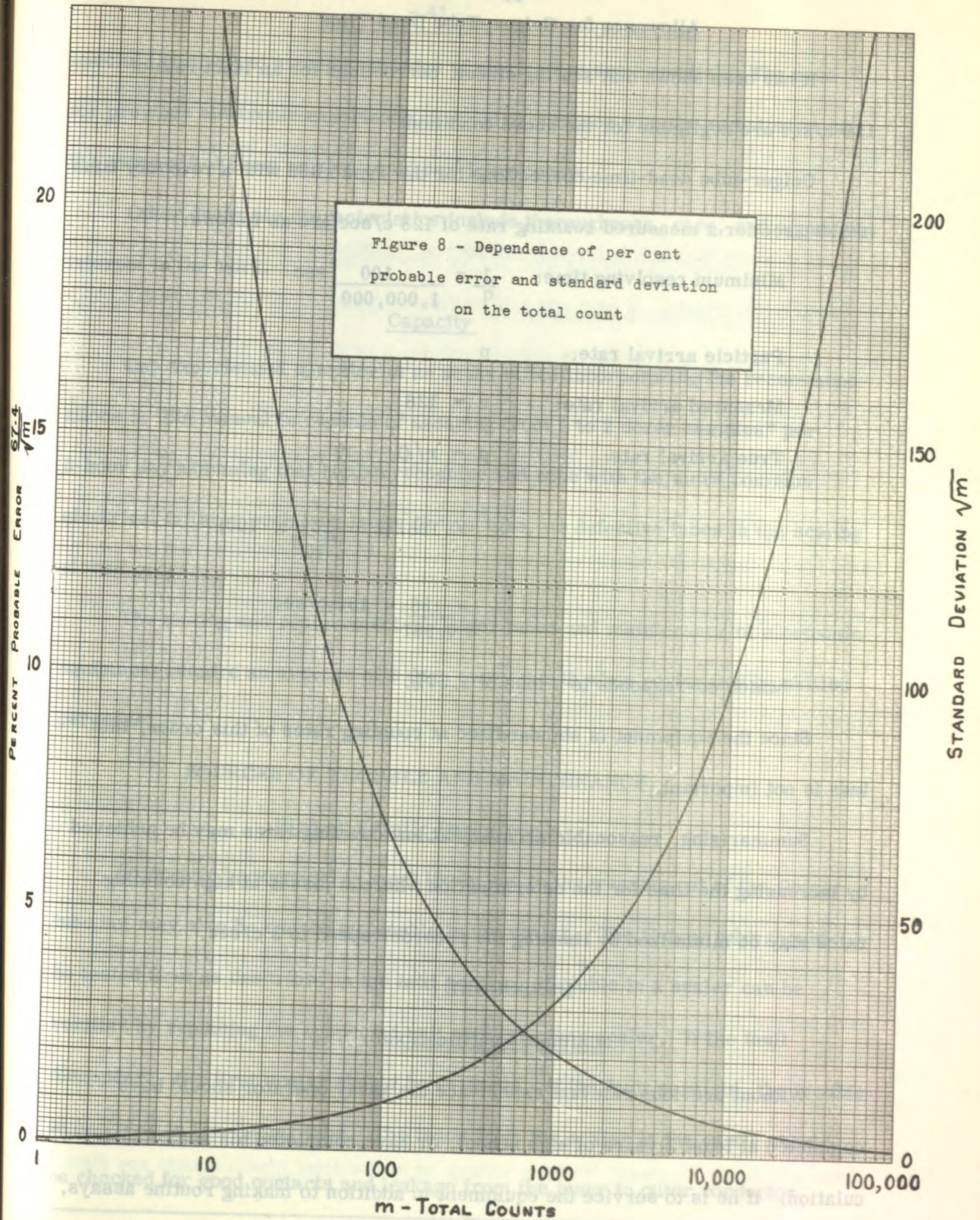
viz., 97.6% of the particles are recorded. If the register speed is limited to 2 c/sec, corresponding to an input rate of 128 c/sec on the scale of 64, the true arrival rate is equal to:

$$p = \frac{128}{1 - \frac{128}{40,000}} = \frac{128}{0.99} = 128.5 \text{ c/sec, or a loss of } 0.32\%.$$

PERCENT PROBABLE ERROR

5

0



* Dependent upon the grade (as low grade samples take longer to assay) and on the number of re-runs.

Allowance for Geiger-Tube Dead Time

It has been shown that scaling-circuit corrections for an input rate of 128 c/sec are negligible for the above conditions.

Geiger-tube dead-time corrections for the Type 1B85 with a recovery time of 100 μ sec for a measured counting rate of 128 c/sec are as follows:

$$\begin{aligned} \text{Minimum resolving time: } \frac{1}{q} &= \frac{100}{1,000,000} \text{ sec} \\ \text{Particle arrival rate: } &p \\ \text{Measured arrival rate: } &r = 128 \\ \text{True arrival rate: } &p = r / \left(1 - \frac{r}{q}\right) \\ &= 128 / \left(1 - \frac{128 \times 100}{10^6}\right) \\ &= \frac{128}{0.987} = 130 \text{ c/sec} \end{aligned}$$

which corresponds to a loss of 1.54%.

Since the equipment is standardized at counting rates of this order, this loss is not important.

Summarizing, reasonable accuracy at low counting rates may be achieved by increasing the time for the determination whereas losses at high counting rates may be minimized by limiting the recorder speed to 3 c/sec.

PERSONNEL

Minimum Qualifications

Without previous relevant experience the assay operator should have completed at least 2 years of high school, or have completed his senior matriculation. If he is to service the equipment in addition to making routine assays,

special training and a good knowledge of electronics are essential. The need for previous electronic experience obviously increases with the remoteness of the assay office from an electronic servicing centre.

Other desirable characteristics include thoroughness, care, and a genuine interest in the work.

Capacity

One experienced operator in an assay office incorporating the recommendations in this manual is capable of assaying from 1 to 2 dozen samples* per 8-hour day assuming that he can recognize and cope with the more common electrical failures such as a faulty Geiger tube, or defective tubes in the scaling circuit proper.

The testing and replacement of Geiger tubes and maintenance of electronic equipment require an average of 2 days every month when performed by skilled personnel.

SOURCES OF TROUBLE AND MAINTENANCE

High or Erratic Backgrounds

An erratic background is most likely due to the wearing out of the Geiger tube but may also be caused by trouble in the scaler. The Geiger tube should be tested first as described in the next section. Trouble in a scaler can be verified by replacing the suspected unit with the other scaler. If the fault disappears, the first scaler should be serviced. If the fault persists, the trouble is probably in the Geiger tube. The leads from the scaler to the castle should be checked for good contacts and leakage from the inner to outer conductor.

* Dependent upon the grade (as low grade samples take longer to assay) and on the number of re-runs.

Daily fluctuations in background rate up to 20% may be expected. If abnormally high (greater than 40 c/min) backgrounds are registered from both the beta and the gamma tube, the proximity of a strong gamma emitter is indicated.

If it is not feasible to remove the source, additional lead shielding should be added to the castle. If only one tube registers a high count either the tube or its scaling circuit is at fault. A contaminated pan or chamber might also be indicated.

Electrical interference from adjacent devices including electric computing machines, high voltage devices, commutating machinery and occasionally faulty fluorescent lights will cause erratic backgrounds, and these devices should be provided with shields or suppressors. With the electric computing machine, an 0.5 μ fd paper condenser across the line is usually effective. The scaling circuits should be totally enclosed and the metal cabinet grounded properly.

Geiger Tube

The most common source of trouble in the assay unit is the gradual deterioration of the Geiger tube due to its limited life of 10^9 counts. This is evidenced by an increase in threshold voltage, and a shorter, steeper plateau. As the tube is normally operated from 50 to 70 v above threshold, an increase in the latter may reduce the pulse amplitude until it no longer triggers the scaling circuit.

On installation and whenever Geiger-tube trouble is suspected, a plateau check should be made as follows: with the sensitivity source inserted and the timer on, slowly increase the high voltage up to the point where counts are first

recorded. This is the threshold voltage, normally in the region between 700 and 750 v for the 1B85. Two-minute runs are taken at 50 v intervals to a maximum of 200 v above the threshold or to where the count has doubled, whichever occurs first.

Table 5 illustrates plateau runs on the beta and gamma tubes to establish threshold, plateau, and operating point. It may be noted that the plateau of V16 is only half of that for V20 and that probably the former will have to be replaced before the latter. Due to the higher beta than gamma activity of samples, the beta Geiger tube will have to be replaced more frequently than the gamma tube.

Table 5
Variation of Counting Rate With Geiger Tube Voltage

| β Tube No. V 16 | | γ Tube No. V 20 | |
|-------------------------|---------------|-------------------------|---------------|
| Voltage | Counts x 1/64 | Voltage | Counts x 1/64 |
| 815 | 67 | 850 | 5 |
| 825 | 98 | 900 | 54 |
| 875 | 108 | 950 | 68 |
| 925 | 113 | 1000 | 70 |
| 975 | 228 | 1050 | 78 |
| Operating voltage - 860 | | Operating voltage - 920 | |

Scaling Unit

The operating manual supplied with each scaler will usually contain further particulars on the more common circuit failures and the operator would be well advised to familiarize himself with them.

In a properly designed scaler, loss of tube emission in the first 2 or 3 scaling stages, in the regulated high voltage supply, or in the input amplifier are the principal sources of trouble.

Table 6

Common Failures and Remedies

1. Scaling Circuit

| <u>Symptoms</u> | <u>Cause</u> | <u>Remedy</u> |
|--|---|---|
| Twice the number of normal counts on background, sensitivity, etc. | Failure of early scaling stage | Replace input stage(s) with new tube |
| Neon light in one stage remains on - neon lights after this stage are unaffected by counts | Failure of counter to scale at the stage corresponding to the steadily glowing neon | This tube can be identified by the corresponding neon light and replaced |
| Weak or intermittent register operation | 1. Weak register drive tube 2. Register out of adjustment or needs cleaning and oiling | Check register with other scaler and repair if faulty. Test output tube in other scaler and replace if necessary |
| Extra counts registered when timer shuts off | Poor circuit design | Depends on circuit. Augment paper by-pass shunt condenser(s) to screen of tube cut off by the timer switch with 0.01 μ fd mica by-pass condenser(s) |

| <u>Symptoms</u> | <u>Cause</u> | <u>Remedy</u> |
|---|---|---|
| Jitter in regulated high voltage supply | <ol style="list-style-type: none">1. Loss of emission in one or more of the following tubes: high voltage rectifier, series regulator, or voltage amplifier2. Unstable voltage reference tube in hv supply | Test suspected tubes one by one in the corresponding positions in the other scaler, or replace suspected tubes with new ones |
| Slow voltage variations in hv supply | <ol style="list-style-type: none">1. Most likely due to loss of emission in voltage amplifier tube2. May be due to other causes of jitter listed above3. Gradual increase in resistance of potentiometer network in hv supply | As above As above Check values with ohmmeter and compare with those indicated in schematic in operating manual |
| 2. <u>Timer</u> | | |
| Circuit continues scaling after timer shuts off | <ol style="list-style-type: none">1. Cam in timer is not throwing lever of micro-switch far enough2. Worn out micro-switch(possible but not commonly encountered)3. Broken or improperly connected lead to micro-switch | Repair obvious mechanical defects by inspection Check microswitch with testmeter for proper operation and replace if necessary Check leads with testmeter |
| Timer fails to shut off. | Slipping clutch in timer | Clean with grease remover and adjust to take up any unnecessary slack |

3. Register

| <u>Symptoms</u> | <u>Cause</u> | <u>Remedy</u> |
|--|---|---|
| Weak or intermittent register operation | 1. Weak register drive tube 2. Register out of adjustment or needs cleaning and oiling | Refer to previous section |
| Register only operates consistently on alternate pulses, i. e. tries to register twice as many counts as it should for a known input counting rate | 1. Improper circuit design | Reduce grid resistor to register drive tube |

4. Geiger Tubes

| | | |
|---|----------------------|--|
| Apparent gradual decrease in activity of sensitivity standard. Counts are not reproducible to within the limits of probability. Decrease in length of Geiger-tube plateau | Worn out Geiger tube | Check plateau of Geiger tube observing any increase in threshold voltage and any decrease in length of plateau. If same effects present when checked by other scaler, replace the suspected Geiger tube. Operate tube about 60 v above its threshold |
|---|----------------------|--|

General Note

These symptoms, if not apparent when the tubes are new, may develop as the tubes age and with mechanical wear of moving parts. In the event of more stubborn troubles, the manufacturer concerned should be contacted.

GLOSSARY OF TERMS USED IN RADIOASSAY

Average sensitivity - the average activity expressed in counts per minute (c/min) of the source used in determining the reference sensitivity (see below).

Sensitivity runs are made before and after the sample determination from which the average sensitivity can be calculated.

Background - the number of counts recorded per minute with no source or sample in the chamber.

Count - a terminated discharge produced by an ionizing event in a counter tube.

Counting rate (gross) - the number of counts per minute (c/min) due to the background and to any other sources of radioactivity in the counter chamber.

The gross counting rate is the product of the net counts on the mechanical recorder times the scaling factor on the counter divided by the duration of the sample run in minutes.

Counting rate (net) - Subtract the average of the backgrounds before and after the sample run from the gross counting rate to obtain the net counting rate.

Dead time - the time interval after recording a count during which the counter tube is insensitive to further ionizing events.

Geiger counter - a system of electrodes, usually a cylinder and concentric wire in a gas. The centre wire is at a positive potential with respect to the cylinder. Under suitable conditions an ionizing event will produce a discharge in the gas creating a uniform voltage pulse between the electrodes independent of the size of the initial ionizing agent.

Geiger region of a counter tube - the voltage interval over which the charge transferred per isolated count is independent of the ionizing event and the applied voltage.

Geiger threshold - the lowest voltage applied to the counter tube for which the output pulse is independent of the ionizing event.

Half-life of an isotope - the time required for half of its radioactive atoms to decay. This time is characteristic of the isotope and is independent of its age.

Ionizing event - For the equipment described all beta particles and about 1% of the gamma rays penetrating the walls of the Geiger tube are effective ionizing agents.

Overvoltage - the difference in voltage between the Geiger threshold and the actual operating voltage of the counter tube. For reasonable tube life this should not exceed 70 v.

Pan Factor (beta) of a sample pan - the net counting rate of a standard sample divided by the %U₃O₈ of the standard.

Pan Factor (gamma) of a sample pan - the net counting rate of a standard sample divided by the product of its %U₃O₈ content and its weight in grams.

Plateau (length) - the portion subtended on the voltage axis (abscissa) by the linear portion of the counting rate voltage characteristic (ordinate) of the Geiger tube.

Plateau (slope) - usually expressed as the change in counting rate per 100 v of plateau divided by the average counting rate. With good Geiger tubes this should not exceed 5% per 100 v.

Recovery time after an ionizing event - that time necessary for the Geiger counter to deliver a successive pulse of normal size.

Scaling circuit or scaler - an electronic apparatus for dividing the input counting rate by the scaling factor to permit registration of the pulses by mechanical means.

Scaling factor of a scaling circuit - the ratio of input to output pulses operating the mechanical recorder. Means may be provided on the scaler for selecting one of several factors, the lower factors for small counting rates.

Sensitivity standard or reference sensitivity - the average activity expressed in counts per minute (c/min) of a specially constructed standard source. The reference sensitivity is restricted to the average of sensitivity runs made during calibration.

Threshold voltage - See Geiger threshold.

APPENDIX I

Uranium, Actinium, and Thorium Series*

I. Uranium Series

| Element | Isotope | Atomic No. | Half-life | Emission Energies MeV. | |
|-----------------|--------------|------------|----------------------------|------------------------|----------|
| | | | | Beta | Gamma |
| U ₁ | U 238 | 92 | 4.51 x 10 ¹⁰ yr | | |
| UX ₁ | Th 234 | 90 | 24.5 days | 0.21 | X |
| UX ₂ | 99.7% Pa 234 | 91 | 1.14 min | 2.3 | 0.8, 0.9 |
| UZ | 0.3% Pa 234 | 91 | 6.7 hr | 0.5, 1.2 | 0.8 |
| U ₁₁ | U 234 | 92 | 2.6 x 10 ⁵ yr | | |
| Io | Th 230 | 90 | 8.3 x 10 ⁴ yr | | X |
| Ra | Ra 226 | 88 | 1,590 yr | | X |
| Rn | Rn 222 | 86 | 3.83 days | | |
| RaA | Po 218 | 84 | 3.05 min | | |
| RaB | Pb 214 | 82 | 26.8 min | 0.65 | X |
| RaC | Bi 214 | 83 | 19.7 min | 3.15 | 2.2, 1.8 |
| RaC' | Po 214 | 84 | 150 μ sec | | |
| RaC'' | 0.04% Tl 210 | 81 | 1.32 min | 1.8 | 4 |
| RaD | Pb 210 | 82 | 22 yr | X | X |
| RaE | Bi 210 | 83 | 5.0 day | 1.17 | |
| Po, RaF | Po 210 | 84 | 1.38 day | | |
| Pb | Pb 208 | 82 | stable | | |

X - Energy less than 0.2 million electron volts (MeV)

Relative abundance of uranium isotopes:

$$U\ 238, U\ 235, U\ 234 = 100/0.725/0.00522$$

* Where no beta emission is shown the isotope emits alpha particles only.

II. Actinium Series*

| Element | Isotope | Atomic No. | Half-life | Emission Energies MeV | |
|---------|---------|------------|---------------------------|-----------------------|------------|
| | | | | Beta | Gamma |
| AcU | U 235 | 92 | 8.9×10^8 yr | | X |
| UY | Th 231 | 90 | 25.5 hr | 0.21 | X |
| Pa | Pa 231 | 91 | 3.4×10^4 yr | | 0.32, 0.29 |
| Ac | Ac 227 | 89 | 22 yr | 0.22 | |
| RdAc | Th 227 | 90 | 18.6 day | | 0.64 |
| AcK | Fr 223 | 87 | 21 min | 1.2 | X |
| AcX | Ra 223 | 88 | 11.2 day | | 0.44 |
| An | Em 219 | 86 | 3.92 sec | | |
| AcA | Po 215 | 84 | 1.83×10^{-3} sec | | |
| AcB | Pb 211 | 82 | 36.1 min | 0.5, 1.4 | 0.83, 0.76 |
| AcC | Bi 211 | 83 | 2.16 min | | 0.35 |
| AcC' | Po 211 | 84 | 5×10^{-3} sec | | |
| AcC'' | Tl 207 | 81 | 4.76 min | 1.45 | 0.87 |
| Pb | Pb 207 | 82 | stable | | |

X - Energy less than 0.2 MeV

* As the parent member AcU of this series is present to less than 1% by weight of the more common U 238, the actinium series has negligible effect on the beta and gamma assays.

III. Thorium Series

| Element | Isotope | Atomic No. | Half-life | Emission Energies MeV | |
|-----------|---------|------------|--------------------------|-----------------------|----------|
| | | | | Beta | Gamma |
| Th | Th 232 | 90 | 1.39×10^{10} yr | | |
| MTh 1 | Ra 228 | 88 | 6.7 yr | X | |
| MTh 2 | Ac 228 | 89 | 6.13 hr | 1.55 | X |
| RTh | Th 228 | 90 | 1.90 yr | | X |
| ThX | Ra 224 | 88 | 3.64 day | | 0.25 |
| Th | Em 220 | 86 | 54.5 sec | | |
| ThA | Po 216 | 84 | 0.16 sec | X | |
| ThB | Pb 212 | 82 | 10.6 hr | 0.36 | 0.24 |
| ThC | Bi 212 | 83 | 60.5 min | 2.20 | 1.8 |
| ThC' 65% | Po 212 | 84 | 0.3 μ sec | | |
| ThC" 35% | Tl 208 | 81 | 3.1 min | 1.8, 1.5, 1.3 | 2.6, 3.2 |
| Pb | Pb 208 | 82 | stable | | |
| Potassium | K 40 | 19 | 4×10^8 yr | 1.4 | 1.5 |

X-Energy less than 0.2 MeV

APPENDIX 2

Prices and Manufacturers of Assay Equipment

A - Typical prices of assay equipment as of June, 1951

| <u>Item</u> | <u>Description</u> | <u>Approx. Price</u> |
|--------------------------------|---|---|
| <u>Beta Assay Unit</u> | | |
| Scaler | Built-in hv supply. Range Selector switch. Upper scale limit of 64 or 128. Preferably binary system. Built-in register, but less timer. | \$450 |
| Automatic Timer | 1 sec to 55 min in 1 sec steps. | 75 |
| Geiger Tube | Thin aluminum-wall beta tube, 3" active length or equivalent. | 8 |
| Cable and connectors | From lead castle to scaler | (Probably included in scaler price.) |
| | Total price | 533 |
| <u>Gamma Assay Unit</u> | | |
| | Same as beta unit | |
| | Total price | 533 |
| <u>Accessories</u> | | |
| Spare tubes for scalers | | 50 |
| Spare Geiger Tubes | 6-1B85's or equivalent | 48 |
| Lead Castle | Slabs for a castle as illustrated in Figure 3, commercially available at | 250 |
| Rolling mat, spatulas, etc. | | 15 |
| Balance | Such as the Cenco Single Beam Trip scales - to permit weighings accurate to 1/10 gm | 14 |
| | Total price of assay equipment | <u>1,443</u> |

B - Better known Manufacturers of Scaling Equipment

Canadian

Canadian Marconi Company Limited, 2442 Trenton Ave., Montreal.

- * Electronic Associates Limited, 2760 Yonge Street, Toronto 12, Ontario.

American

- * Atomic Instrument Company, 84 Massachusetts Ave., Cambridge 39, Mass.

Berkeley Scientific Company, 6th & Nevin Ave., Richmond, Calif.

- * El-Tronics Incorporated, 2649 N. Howard Street, Philadelphia, 33, Pa.

- * General Electric Company, Apparatus Department, Schenectady 5, N. Y.

Kelley-Koett Instrument Company, 936 York Street, Cincinnati 14, Ohio.
(now amalgamated with Tracerlab Incorporated.)

- * Nuclear Instrument & Chemical Corporation, 223 West Erie St., Chicago 10, Ill.

- * Radiation Counter Laboratories Incorporated, 1844 West 21st Street, Chicago 8, Ill.

Technical Associates Incorporated, 3730 San Fernando Rd., Glendale 4, Calif.

- * Tracerlab Incorporated, 130 High Street, Boston 10, Mass.

British

Cinema Television Limited, Worsley Bridge Rd., London, S.E. 26.

Dynatron Radio Limited, Maidenhead, Berks.

- * E.K. Cole Limited, Malmesbury, Wilts.
(Canadian Rep. - Ahearn & Soper, P. O. Box 794, Ottawa)

Labgear Limited, Fitzroy Lane, Cambridge.

Marconi Limited, St. Albans, Herts.

* These firms have a complete line of assay equipment available including timers, recorders, Geiger tubes, and lead. The status of the other British firms in this respect is unknown to the authors.

APPENDIX 3

Calculation of U_3O_8 Content from Beta and Gamma Assays

The formula used in calculating true U_3O_8 content can be derived on the basis of certain simplifying assumptions:

1. The uranium series is arbitrarily divided into the uranium family and radium family - the radium family including radium and its descendants.

2. Of a mineral sample which is out of equilibrium, it is assumed that the radium family is present in an amount which is either greater than or less than the amount which would be in radioactive equilibrium with the uranium ($U\ 238$) present.

The state of equilibrium is then expressed by a factor E such that when $E = 0$, the radium family is missing, and when $E = 1$ the radium family is present in equilibrium proportion to the uranium.

Let $k\beta$ = fraction of the total measurable equilibrium beta activity due to the uranium family.

Let $k\gamma$ = fraction of the total measurable equilibrium gamma activity due to the uranium family.

Let β and γ = measured beta and gamma activities of a sample whose state of equilibrium is not known, expressed as the $\%U_3O_8$ content which would correspond to this activity of a sample in radioactive equilibrium. In short, let β and γ be called the beta equivalent and gamma equivalent respectively.

Let $C =$ the true $\%U_3O_8$ content.

When $E = 1$, that is, when a sample is in radioactive equilibrium (and contains no thorium and no large amounts of potassium), $C = \beta = \gamma$.

When E has any value other than 1, then

Activity due to Uranium Family

$$\gamma = k_{\gamma} C +$$

Activity due to Radium Family

$$(1 - k_{\gamma}) EC \quad (9)$$

$$\beta = k_{\beta} C +$$

$$(1 - k_{\beta}) EC \quad (10)$$

Combining these equations, E disappears and

$$\begin{aligned} (\beta - C) / (\gamma - C) &= (1 - k_{\beta}) / (1 - k_{\gamma}) \\ &= \text{a constant} \end{aligned} \quad (11)$$

For arithmetic convenience let

$$(1 - k_{\beta}) / (1 - k_{\gamma}) = Y / (Y + 1)$$

$$\text{then } (\beta - C) / (\gamma - C) = Y / (Y + 1)$$

$$\text{Whence } C = \beta + Y(\beta - \gamma) \quad (4)$$

$$\text{or } C = \beta - Y(\gamma - \beta)$$

$$\text{now } Y = \frac{1 - k_{\beta}}{(1 - k_{\gamma}) - (1 - k_{\beta})} \quad (12)$$

In Appendix 1 it is noted that in the uranium series at equilibrium, the radium family is the source of close to 50% of the measurable beta activity and practically all of the measurable gamma activity.

Therefore, the fractions of the total equilibrium beta and gamma activities due to the uranium family are:

$$k_{\gamma} = 0, \text{ and } k_{\beta} = 0.5, \text{ i.e., } Y = 1$$

$$\text{and (11) becomes: } \frac{\beta - C}{\gamma - C} = \frac{0.5}{1.0} \text{ and } \gamma - C = 2(\beta - C)$$

$$\text{Thus } C = 2\beta - \gamma \tag{1}$$

Results given by (1) have generally agreed with the chemical determinations; corroborating the values of k_{β} and k_{γ}

Calculation of U_3O_8 in Presence of Thorium

The presence of thorium in a sample affects the activity measurements in the same way as lack of equilibrium in the uranium series, and the actual U_3O_8 content may be estimated by the same formula.

Each 1% ThO_2 gives rise to beta activity equivalent to the presence of approximately 0.25% U_3O_8 , and gamma activity equivalent to the presence of approximately 0.5% U_3O_8 . Therefore, using symbols previously defined:

$$\text{the net gamma activity is: } \gamma = C + 1/2T \tag{13}$$

$$\text{and the net beta activity is: } \beta = C + 1/4T \tag{14}$$

where T is the % ThO_2

Combining (13) and (14):

$$C = 2\beta - \gamma \tag{1}$$

$$\text{and } T = 4(\gamma - \beta) \tag{15}$$

APPENDIX 4

Reporting of Uranium or Thorium Determinations

Under the Atomic Energy Regulations of Canada, the results of an assay or analysis of a mineral that indicates a content of more than 0.05 per cent by weight of uranium or thorium are to be reported forthwith to the Director of the Geological Survey, Department of Mines and Technical Surveys, Ottawa, for the Atomic Energy Control Board, together with full particulars relating to the material assayed or analysed, including the name and address of the person from whom such material was received, the purpose of the assay or analysis, and the origin of the material so far as known to the person making the report. This requirement does not apply to assays or analyses made for persons operating under orders of the Board which provide for periodical reports.

Copies of the Regulations may be obtained on application to the Secretary, Atomic Energy Control Board, Ottawa.