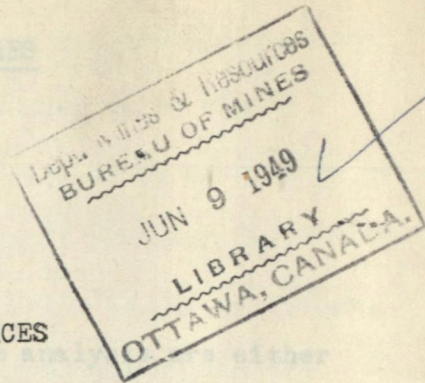


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DETERMINATION OF URANIUM IN ORES

BY FIELD ANALYSIS

by

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Radioactivity Division

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Introduction

The usual laboratory methods of uranium analysis are either tedious, time-consuming chemical procedures or elaborate physical methods requiring sensitive equipment. There is thus an urgent need for a method of estimating by approximate analysis the uranium content of ore samples in the field. A rough field analysis is eminently practicable to the mining engineer who must make on-the-spot ore assays. When a new property is being developed it is desirable to know the uranium content of channel samples in order to guide the diamond drilling and digging operations. Prospectors, too, require approximate analyses of their hand specimens to evaluate their discoveries.

To satisfy the demand for these types of analyses, several field procedures have been tried. Two general plans can be followed. One is fast but will yield only a rough estimate. The other is slightly longer but will give an assay probably within 10 per cent of the true value if done carefully.

For field operation it is necessary to strip the more accurate laboratory methods of analysis of all refinements because of the limited equipment and skilled techniques involved. Most of the corrections normally used, with the exception of that for equilibrium, will not alter the gross analysis to any extent.

When uranium atoms in pitchblende disintegrate, they break down into other radioactive elements and eventually the number of elements being formed is equal to the number breaking up. The ore is then said to be in

equilibrium. Certain geological processes, however, such as leaching, sometimes selectively remove some of these elements and hence reduce the radioactivity of the ore sample. The ore is then said to be out of equilibrium. The field analysis may then be in error by as much as 50 per cent since the apparatus is calibrated with standards that are in equilibrium. However, such a large error due to loss of equilibrium is the exception, and in most cases uncorrected field types of analysis are justified.

The analyses given by the following methods are in terms of per cent U_3O_8 equivalent. That is, the total activity of the sample may be due to thorium as well as uranium, and since the field counter reacts to both elements, the analysis is given in percentage U_3O_8 which is equivalent to the total radioactivity of the sample.

Field Estimate Method

Prospectors often obtain a rough estimate of the radioactivity content of their specimens by simply holding them under the Geiger counter. The samples can thus be graded according to their activity, but this is often very deceiving. A small piece of high grade pitchblende on a corner of the specimen may show more activity than a lower grade of ore, although the latter may actually contain more uranium. For a better estimate the procedure given below, following the comments on the counter, has been tried and is recommended.

The apparatus required is simple. For field work it is assumed that there is no source of electric power, and accordingly only portable Geiger counters are considered. The counter should be of a sensitive design and be constructed with a ratemeter. The small earphone types, although better for prospecting, are limited in their usefulness for field assay work to the weaker samples. With active samples the counts are too rapid for an accurate count of the earphone clicks.

The ratemeter type counter used to test the field estimate and field assay methods is shown in Fig. 1. It is a commercial type designed after the National Research Council type GP-19. It contains three Geiger tubes and the ratemeter is sufficiently stable to make reasonably accurate analyses. Any good commercial type with similar characteristics can be used with equal success. The carrying case should be removed and the ratemeter, (A), (see Fig. 2), mounted on a box, (B), fitted with a removable shelf, (C). The top of the box has been cut away so that the bottom of the counter is exposed to the inside. The shelf rests on two runners and can be placed either two inches or five inches from the bottom of the counter case.

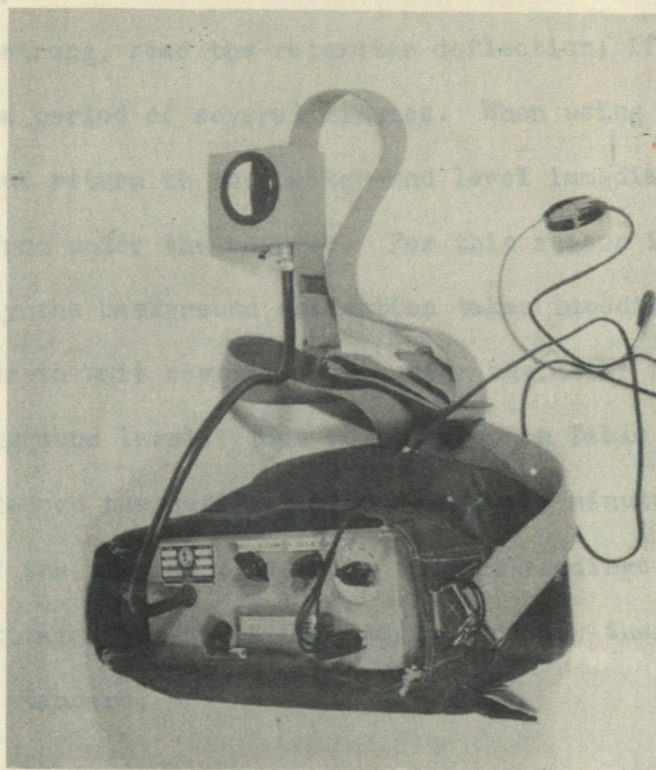


Fig. 1 Ratemeter type counter.

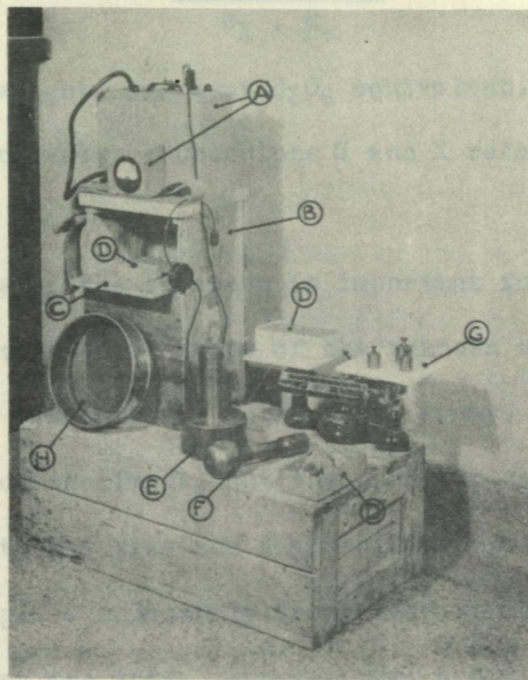


Fig. 2

- | | |
|-------------------------------|------------|
| A - Geiger ratemeter | E - Mortar |
| B - Sample box | F - Pestle |
| C - Removable shelf | G - Scale |
| D - Sample and standard trays | H - Screen |

Having set up the apparatus the experimental determinations are easy. Place the hand specimen on the shelf under the counter. If the specimen is relatively strong, read the ratemeter deflection; if weak, take an earphone count over a period of several minutes. When using a ratemeter, the meter needle does not return to the background level immediately on the removal of a specimen from under the counter. For this reason it is sometimes better to use only the background deflection taken immediately before the sample deflection or to wait several minutes for the meter needle to return to the true background level. (See method used in Table 1 at the end of this report). Record the deflection or counts per minute for each of the several sides of the specimen. Average the net readings and weigh the sample. The approximate percentage U_3O_8 equivalent may then be obtained by comparison with the standard.

Thus

$$\% U_3O_8 \text{ equivalent } (C_X) = \frac{W_S \cdot C_S \cdot N_X}{W_X \cdot N_S}$$

where W, C, and N are the weight, per cent U_3O_8 equivalent, and the net deflection or count, respectively. Subscripts S and X refer to the standard and the unknown sample.

Choice of the size of the specimen is important for the best results. Technically the volume and shape of the unknown specimen should be the same as that of the standard for an accurate assay. Obviously this is impossible for odd-shaped specimens, but the specimen can be chipped down with a hammer to approximate the size and shape of the standard.

Consider for example a relatively large hand specimen having roughly four large sides, neglecting the ends. Suppose that a quick check under the counter indicates slight activity except at one corner where the activity is considerably higher. Also, suppose that the specimen, with a slight amount of trimming, is about the same size, say about 6" x 3" x $1\frac{1}{2}$ ", and shape as

one of the larger and weaker standards described later. Readings should be taken with the appropriate backgrounds for each of the four sides as is shown in Table 1. The estimated activity will be that of the specimen as a whole. To get the U_3O_8 equivalent content of the active corner, the specimen should be broken down to the size of one of the smaller and more active standards, say $3'' \times 1\frac{1}{2}'' \times 3/4''$, and a separate estimate should be made on the active portion. It is apparent that the procedure to be followed will depend upon the specimen and can be altered somewhat to fit the circumstances.

As this method is subject to large error much care should be taken when comparison is made with the standard and in positioning the specimen similarly to the standard. Since the specimen does not have to be crushed, this method is used mostly in screening samples and drill cores where only the order of the magnitude of the U_3O_8 content is necessary.

Field Assay Method

To measure a wide range of sample activities a ratemeter can be used best for field assays, but the more simple earphone types are workable up to one and two per cent U_3O_8 equivalent without difficulty. The complete apparatus is shown in Fig. 2. The other associated necessary equipment consists of a suitable set of standards, standard ore trays, (D), an iron mortar, (E), and pestle, (F), a simple scale, (G), a screen, (H) (about 35 mesh), and a rock sledge hammer.

A suitable type of mortar consists of a heavy steel anvil $2\frac{1}{2}''$ deep and 5" diameter. A 2" diameter round recess is cut $1/4''$ deep in the centre of the upper surface to hold a $1/8''$ thick steel tube 5" long. Small chips of rock can be easily pulverized by a flat-end pestle in such a mortar.

Particular care should be exercised in choosing standards. They should be analyzed by the more accurate laboratory methods, and it is recommended that from two to four standards be used covering a range up to

about 10 per cent U_3O_8 . Standards of about 0.1, 1.0, 5.0, and 10.0 per cent U_3O_8 are suitable. Standards should be natural, not synthetic, i.e., they should not be made by diluting an active rock with some inert pulp. In such mixtures the heavier uranium particles tend to separate out over a period of time, especially with the rough handling received in the field. A single type of ore pulp, although not entirely free from this source of error, is relatively free from serious segregation. In either case standards should be checked frequently to note any increase in activity when in an up-side-down position. The specimen to be used for the standard should be ground to pass a 35-mesh screen, analyzed by a good laboratory method, and sealed in tight metal containers to prevent loss of the pulp. The two boxes used to hold the standards in these tests measured 6" x 3" x $1\frac{1}{2}$ " for standards up to 2.0 per cent, and 3" x $1\frac{1}{2}$ " x $3/4$ " for standards from 2.0 to 10.0 per cent.

A limited number of these standards will be sold by the Bureau of Mines for a fee to bona fide prospectors and mining companies.

The unknown sample must be placed in trays of the same shape and volume as that holding the standard with which it is compared. With a little experience an operator can quickly estimate the size of tray necessary for an analysis by holding the uncrushed specimen under the counter for a brief period. It is advantageous to choose a standard, and hence the corresponding tray, that will give the same order of deflection or counts as that of the unknown sample. Thus, for the greatest accuracy, a sample of 650 grams giving a deflection of 10 divisions should not be compared with a 150-gram standard giving a deflection of 50 divisions, but rather with say, a 500-gram standard that will give the same order of deflection or count.

The sample to be analysed should be roughly crushed with a field hammer and then further reduced to 35-mesh or smaller in the mortar. If the sample is known by its effect on the Geiger counter to be relatively weak, sufficient should be pulverized to fill a large tray. Smaller amounts of the

more active samples may be used with smaller trays. In all cases the sample trays should be covered with a flat piece of sheet metal of the same gauge as that in the trays to reproduce similar absorption conditions as are present in the sealed standard trays.

The measuring procedure to be followed should be similar to the field estimate method described above, but more care should be taken to insure that all readings are taken in a similar manner. It is important that the instrument be at least 50 feet from any ore bag or other active material. If possible, the Geiger counter should be shielded with 2" thick lead bricks (not shown in the picture), although this is essential only for very weak samples. Lead shielding will reduce the background count and increase the relative sensitivity of the instrument.

One serious difficulty with portable instruments of this type is the change in sensitivity with time. To obviate this difficulty it is absolutely necessary to standardize the instrument before and after each sample determination. The sequence of the determinations should be in this order -- background, standard, background, unknown sample, background, standard, and background. Since the ratemeter action depends upon the charging and discharging of a condenser, sufficient time should lapse after the standard and sample determinations to insure a true background reading. Some counters are supplied with a shorting key to discharge the condenser and thus bring the meter back to zero. This key should not be used when the greatest possible accuracy is desired.

To show the proper procedure more clearly, the results of several samples are given in Table II(at the end of this report). The final result is calculated from the formula used in the field estimate method. To obtain enough counts to operate the ratemeter with weak samples it is sometimes necessary to raise the voltage on the Geiger tubes. This effect can be seen

in Table II, column B, where the background deflection is about twice that in column A for a stronger sample.

It should be emphasized that unless the above methods are used with care, the analyses may involve a very large error. For ores containing over one per cent U_3O_8 equivalent an accuracy within 10 per cent of the true value can be expected provided the apparatus is properly standardized and the procedure regulated. For ores between one per cent and 0.1 per cent, an accuracy within 20 per cent of the true value should be obtained.

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% by weight of uranium or thorium are to be reported forthwith to the Chief of the Geological Survey, Department of Mines and Resources, 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.

TABLE I

FIELD ESTIMATE METHOD

Steps

1. Sample - Hand specimen having roughly 4 sides

Preliminary Data

- | | |
|--|------|
| 2. Sample weight, W_x grams | 248 |
| 3. Standard weight, W_s grams | 503 |
| 4. Standard strength, C_s per cent U_3O_8 equivalent | 1.37 |

Experimental Determinations *

- | | |
|---|------|
| 5. Background activity | 7.5 |
| 6. Standard activity | 27.0 |
| 7. Background activity | 6.0 |
| 8. Net Standard activity, (6 - average of 5 and 7) | 20.3 |
| 9. Side #1 of unknown specimen | 11.0 |
| 10. Side #2 of unknown specimen | 12.5 |
| 11. Background activity ** | 5.5 |
| 12. Side #3 of unknown specimen | 11.0 |
| 13. Side #4 of unknown specimen | 9.5 |
| 14. Background activity ** | 5.5 |
| 15. Standard activity | 27.5 |
| 16. Background activity ** | 6.0 |
| 17. Net Standard activity, (15 - average of 14 and 16) | 21.8 |
| 18. Average of 8 and 17, N_s | 21.0 |
| 19. Net specimen activity, N_x , (average of 9,10,12,13 - average of 7,11,14) | 5.3 |

Calculation

- | | |
|---|------|
| 20. Per cent U_3O_8 equivalent, $C_x = \frac{W_s \cdot C_s \cdot N_x}{W_x \cdot N_s} =$ | 0.70 |
|---|------|

Accuracy

- | | |
|--|------|
| 21. Laboratory assay, per cent U_3O_8 equivalent | 0.56 |
| 22. Error, per cent | 25 |

* Activity in divisions on the ratemeter at distance of 2".

** Reading taken several minutes after previous one in order to allow the ratemeter to adjust itself to true background.

TABLE II

Steps	Deflection Method		Count Method
	A	B	C
1. Sample number	#2	#4	#4
<u>Preliminary Data</u>			
2. Sample weight, W_x grams	150.0	533.0	533.0
3. Standard weight, W_s grams	108.7	503.3	503.3
4. Standard strength, C_s per cent U_3O_8 equivalent	5.96	1.37	1.37
<u>Experimental Determinations *</u>			
5. Background activity	5.0	10.3	23.0
6. Standard activity	14.3	33.3	88.0
7. Background activity	4.0	11.0	21.5
8. Net Standard activity, (6 - average of 5 and 7)	9.8	22.7	65.8
9. Unknown sample	28.5	15.4	34.5
10. Background activity	4.3	11.0	20.5
11. Net Sample activity, N_x , (9 - average of 7 and 10)	24.3	4.4	13.5
12. Standard activity	15.3	31.8	76.5
13. Background activity	5.3	11.0	18.5
14. Net Standard activity, (12 - average of 10 and 13)	10.5	20.8	57.0
15. Average of No. 8 and No. 14, N_s	10.1	21.7	61.4
<u>Calculation</u>			
16. Per cent U_3O_8 equivalent, $C_x = \frac{W_s \cdot C_s \cdot N_x}{W_x \cdot N_s} = 10.4$		0.26	0.28
<u>Accuracy</u>			
17. Laboratory Assay, per cent U_3O_8 equivalent	10.3	0.30	0.30
18. Error, per cent	1	13	6.7

* Columns A and B in ratemeter divisions; column C in counts per minute.

Column A: Standard and Sample 5" from counter

Columns B and C: Standard and Sample 2" from counter.