AGE DETERMINATION OF ACCESSORY

ZIRCON FROM GRANITIC ROCKS OF

THE KENORA AREA, ONTARIO

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ABSTRACT

A method of recovering accessory zircon from large granite samples is described. The major equipment includes a disc-pulverizer, Haultain Superpanner, and Franz isodynamic separator.

The zircon ages were determined by the lead:alpha method. A description of scintillation alpha counting of zircon samples is given along with a brief outline of the method of colorimetric trace lead analysis. Results are given for an X-ray:alpha method of age determination of zircon which proved unsuccessful.

The lead:alpha ages of three accessory zircons were found to be 771 million years, 869 million years, and 1707 million years, indicating at least two widely separated periods of granite intrusion in the area.

CONTENTS

LIST OF TABLES

LIST OF ILLUSTRATIONS

INTRODUCTION

Age determination of radioactive minerals has achieved a position of some respect and promises to provide solutions to many geological problems, particularly problems of the Precambrian.

The lead:alpha method has been used with success to date zircon. The purpose of the present work was to improve methods of recovering accessory zircon from granitic rocks and to determine the lead:alpha ages of the zircons collected.

A Haultain Superpanner was used to concentrate the heavy accessory minerals. Following this, a Franz isodynamic separator, heavy liquids, and acid treatment were used to recover the zircon.

A scintillation counter was used to determine the alpha activity of the zircon samples. Lead analyses were done by a chemical colorimetric method. Ages were also determined by an X-ray:alpha method to find how they compared to the lead: alpha ages.

Acknowledgements

Assistance and advice on various aspects of the work given by Dr. H.D.B. Wilson, Dr. G.M. Brownell, and Dr. R.B. Ferguson of the Geology department, are gratefully acknowledged. Dr. K.I. Roulston was helpful in correcting electronic difficulties. The Assay Laboratory of the Manitoba Mines Branch kindly permitted the use of their grinding equipment.

DATING IGNEOUS ROCKS FROM THEIR ACCESSORY MINERALS

According to Larsen and Phair (1954) the minerals of
moderate to high radioactivity in order of decreasing abund-
ance in igneous rocks are: zircon, allanite, monazite, xeno-
time, thorite, pyrochlore, and uraninite. Zircon, sphene,
and apatite have been used by Larsen, Keevil, and Harrison,
(1952) to date granitic rocks with the lead:alpha method.
Zircon gave the most consistently good results. Sphene and
apatite gave erratic values with sphene ages tending to be
very high, probably due to primary lead content. Apatite
has very low radioactivity.

Zircon averages less than 100 ppm. in most igneous rocks
but occasionally it is much more abundant. It is more common
in granites than in basic rocks and also contains proportion-
ately more radioactive elements. The average zircon contains
about equal amounts by weight of uranium and thorium, but in
some specimens either uranium or thorium is abnormally high.

The lead method is one of the earliest to be applied to
age determination of minerals. It is based on the natural
disintegration of U^{238}, U^{235}, and Th^{232}, into the stable lead
isotopes Pb^{206}, Pb^{207}, and Pb^{208}. If nothing has been added
or removed from a system in equilibrium and the amount of
parent and daughter elements is known together with the rate
of decay of the parent, the age of the system can be calculated.

Innumerable age determinations have been made with the

lead method but many of them have been shown to be valueless due to selective leaching by ground waters or primary lead content in the mineral. Zircon is highly resistant to chemical change unless badly metamict. Measurements on young, inactive zircons have indicated that primary lead is negligible. Abnormally high ages have not been obtained from zircon, which is further evidence of low primary lead content. Apparently the closely-packed zircon structure prohibits the entrance of lead ions during crystallization. Thus the basic requirements of the method seem to be satisfied by the properties of zircon.

The lead:alpha method used by Larsen et al (1952) is a variation of the lead method. It is rapid and relatively simple, requiring only an alpha count and a lead analysis on the zircon. The age is given by:

$$t = \frac{c\ Pb}{\propto}$$

where t = age in millions of years

c = a constant varying from 2660 (U only) to 1990 (Th only)

Pb = lead content in ppm.

$\propto$ = alpha activity in alphas / mg. / hr.

Most zircons have been found to have their radioactivity due largely to uranium. Larsen gives c = 2480 as an average value for zircon. Even a large error in the assumed Th/U ratio however, results in a relatively small error in the calculated age.

RECOVERY OF ACCESSORY ZIRCON

One of the major problems of dating rocks from their accessory mineral content is that of recovering a sufficient quantity of material with which to work. The task is laborious because comparatively large rock samples are required. The method outlined below was designed to speed recovery of heavy accessory minerals and differs somewhat from standard procedures.

The samples described were all from the Kenora area of northwestern Ontario (fig. 1). They consisted of granites and granodiorites ranging in size from 20 to 50 lbs. nominal weight.

Crushing and Grinding

A laboratory jaw crusher was used to reduce the sample to about 1/4 inch maximum diameter. The sample was then passed through a disc-grinder (Braun Pulverizer) set to reduce the material to 100 percent minus 65 mesh. One pass through the disc-grinder was sufficient. Further reduction resulted in too much fine material ie. minus 325 mesh, which was difficult to work with in the final stages of separation.

Generally, material coarser than plus 150 mesh contains little free zircon and grinding would be more effective with a 150 mesh screen in closed circuit with the pulverizer, the coarse material being returned for further grinding.

Heavy Mineral Concentration

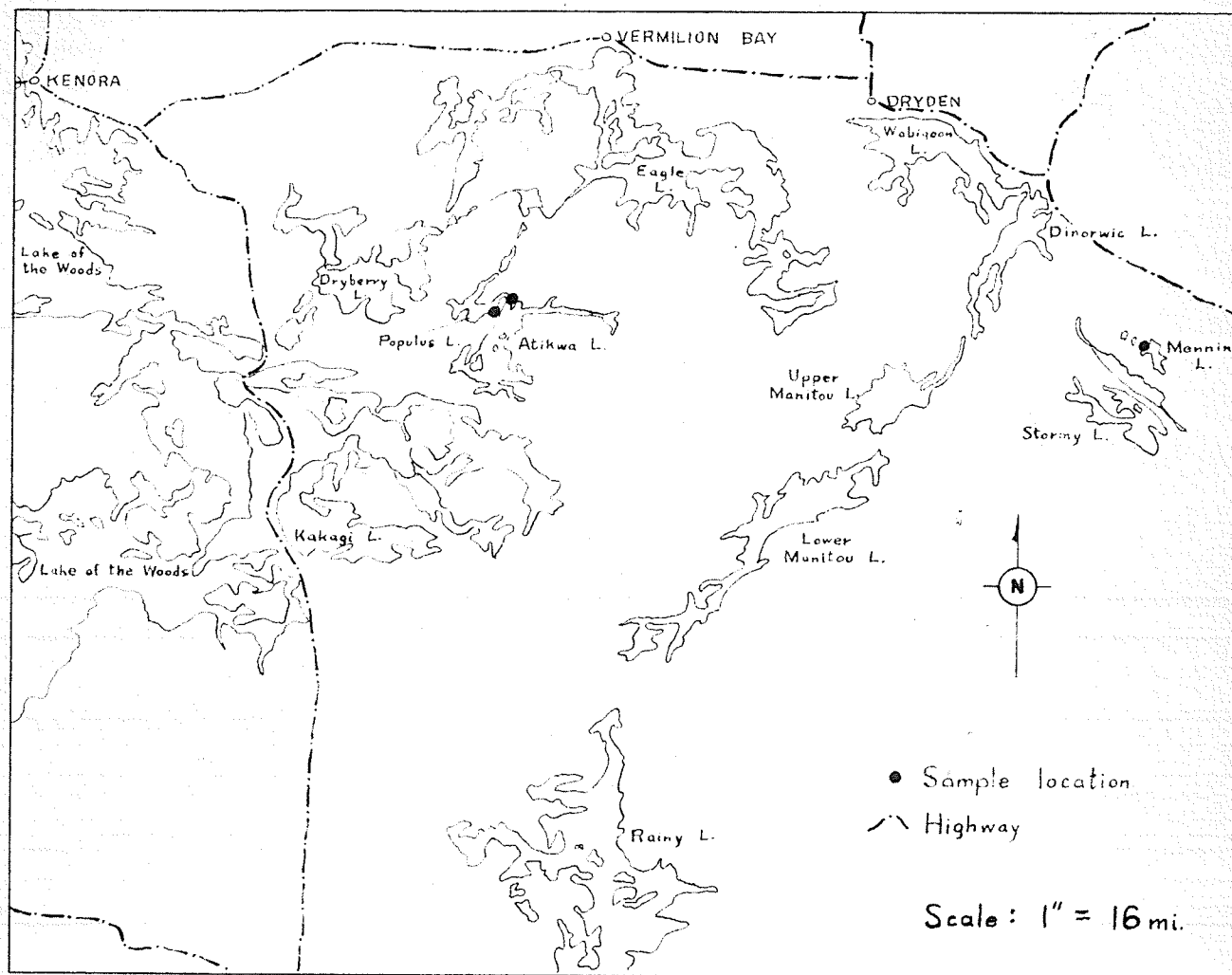Heavy liquids and to some extent flotation are usually

Fig. 1

Map showing sample locations in northwestern Ontario.

used to separate the large bulk of waste material from the relatively minor amount of heavy constituents. However, the use of heavy liquids in quantity is slow, expensive, and not particularly efficient. The method used largely eliminates heavy liquid separations.

The Haultain Superpanner was developed by Prof. H.E.T. Haultain of the University of Toronto for laboratory research on gold ores. This device provides a rapid means of recovering the heavy mineral fraction from large rock samples. With a little practice more than 99 percent of the original sample can be discarded in one operation.

The ground sample was screened through a nest of Tyler standard sieves to obtain +100, +150, +200, and -200 mesh fractions. These screen fractions are kept separate through the entire concentrating procedure. Each screen fraction was panned separately starting with +200 mesh which was found to be the coarsest size that may be expected to contain a significant amount of zircon. If zircon is abundant in this fraction the +150 mesh material is panned next. The coarser the zircons recovered the better, because in the final stages a certain amount of hand picking is usually necessary. If examination of the +200 fraction shows a scarcity of zircon the -200 mesh size must be panned.

It was usually convenient to take two separate fractions from the Superpanner "tip" concentrate: one "high grade"

fraction (No.1 concentrate) at the extreme tip, and a second (No.2 concentrate) immediately behind this. Depending on the heavy mineral constituents of the individual rock, zircon is more abundant in one or other of the concentrates. For example, usually the zircon is abundant in the No.1 concentrate but if the granite is high in sulphides this concentrate will be largely sulphides.

Magnetite was readily removed during the panning operation. A strong permanent magnet covered with a sheet of paper was passed over the tip just before removing the concentrates. If magnetite is not removed here it causes trouble when the concentrates are dried, coating the grains with rusty oxide and sticking them together. Magnetite will also clog the Franz isodynamic separator if it is too abundant.

Magnetic Separation

After drying, the concentrates were passed through a Franz separator. With the inclined feed attachment the separator was set with a forward tilt of 30°, a side tilt of 7°, and a current of 0.2 amps. in the electromagnet. The sample was run through twice at this setting and the procedure repeated, increasing the current in 0.2 amp. increments up to 1.0 amp. A large part of the magnetic minerals including most of the sphene was removed at this point and the current was then raised to 1.8 amps. (full current). Side tilt was reduced to 5° and then to 3°. All magnetic fractions had to be examined under the microscope. At 3° the zircon usually began to appear

in quantity in the magnetic fraction. Zircon apparently
becomes more magnetic with increasing uranium content (Webber,
Hurley, and Fairairn, 1956).

Heavy Liquids

The No.2 concentrates were put through a bromoform sep-
aration to eliminate light minerals which usually contaminate
this fraction. The "sink" was examined carefully and if zircon
was present the fraction was added to the corresponding No.1
concentrate. If the "sink" did not contain zircon it was dis-
carded.

Clerici solution was used for the final separation. The
specific gravity of zircon ranges from 3.9 to 5.0, due partly
to composition but mainly to the degree of metamictness (Holland
and Gottfried, 1955). The sink consisted mainly of zircon and
sulphides; apatite floated. The -200 mesh concentrate was
screened through a 325 mesh sieve and only the +325 fraction
separated with Clerici solution.

The zircon-sulphide concentrates were treated with a hot,
strong solution of nitric acid. This dissolved the sulphides.
The few impurities left were hand-picked under the micro-
scope. The acid treatment also served to remove surface lead
contamination. Pyrite becomes magnetic after heating for a
few minutes above 450°C and may be removed with a hand magnet.
This method was not used however, because the zircon was to
be X-rayed and there was the possibility of thermal annealing

of the zircon structure. It is important that the zircon concentrate be as pure as possible. Larsen notes that pyrite may contain as much as 100 ppm. of lead.

Separation of grains finer than +325 mesh by settling in Clerici solution is not practical. The viscosity of the solution makes the process extremely slow and the resulting separations are poor. Zircon is often abundant in this fine material and would be well worth recovering. Acid treatment appears to be the most feasible method. However, hot, strong acids must be used for considerable periods and the effect on zircon is not known. A sample of Ceylon gem zircon treated with 1:1 aqua regia at 80°C for 1/2 hour showed a negligible change in determined age (Gottfried, Senftle, and Waring, 1956). Probably only badly metamict zircon would be much affected.

Zircon Concentrates

Table 1 describes briefly the rocks from which the zircon came and the amount of zircon recovered from each.

The final concentrates were largely whole zircon crystals. It appears that the crystals were small enough so that even the violent grinding action of the pulverizer merely "popped" them free from enclosing mineral grains.

Most zircons collected were brown to reddish brown. Birefringence was considerably lower than normal probably because of their metamict condition which was substantiated by X-ray photographs. Zoning was noted in crystals from each sample

and was not characteristic of any one sample.

Table 1

Sample Description and Amount of Zircon Recovered

Locality and Description	Wt. of Sample	Wt. of Zircon
Mennin Lake, Ont.- medium grained biotite granite. Slightly altered texture.	50 lb.	650 mg.
Tail Bay, Atikwa Lake, Ont. - coarse grained biotite granite. Fresh texture.	48 lb.	trace
Populus Lake, Ont.- coarse grained biotite granite. Fresh texture.	35 lb.	90 mg.
*Dryberry Lake, Ont.(older) - fine to medium grained gneissic biotite grano- diorite. Altered texture.	20 lb.	65 mg.
*Dryberry Lake, Ont.(younger) - coarse, pink, pegmatitic granite. Fresh texture.	20 lb.	trace

*Supplied by Mr. S.N. Watowich, Falconbridge Nickel Mines Ltd., Kenora, Ontario.

The Dryberry Lake granodiorite yielded two distinct var-
ieties of zircon, one brown and cloudy, the other clear and
colorless. They were about equally abundant but separation was
not attempted due to limited time and the small amount of
material available.

Thin sections from several of the rock samples were exam-
ined. A single thin section is not a reliable indicator of the
amount of zircon occurring in a rock. Brief descriptions of
the thin sections are given below.
Mennin Lake, Ont. - Biotite granite

Orthoclase, the predominant feldspar, has strongly sericitized
and kaolinized cores with fresher rims. A small amount of plagio-
clase and fresh-looking microcline are present. Quartz is fractured

and shows wavy extinction. Grain boundaries of feldspars and quartz are not sharp. A few small patches of ragged biotite, muscovite, and finely recrystallized quartz and feldspar occur. Accessory minerals include apatite, epidote, clinozoisite, sphene, and magnetite. Pleochroic haloes are common in biotite and most are caused by sphene. Only one zircon crystal was identified.

Tail Bay, Atikwa Lake, Ont. - Biotite granite

Orthoclase has kaolinized cores with fresh rims. Plagioclase is altered similarly but is less abundant. Microcline is fresh. Quartz is abundant in large clear grains commonly showing wavy extinction. Accessory minerals include biotite, muscovite, epidote, sphene, apatite, magnetite, and hematite. Pleochroic haloes occur in biotite but the mineral causing them was not identified. Zircon was not seen.

Populus Lake, Ont. - Biotite granite

Orthoclase and plagioclase are strongly sericitized and kaolinized. Quartz is abundant, occurring in large grains and granular masses. Accessory minerals include biotite, muscovite, epidote, magnetite, and hematite. Three tiny zircon crystals enclosed by biotite were noted.

RADIOACTIVITY MEASUREMENTS

The alpha activity of the zircon samples was measured with a scintillation counter. The method was similar to that proposed by Kulp, Holland, and Volchok (1952) for a thin source.

The electronic equipment has been described by Turnock (1956). The method of mounting the sample is shown in fig.2. The sample tray was a sheet of plastic with a circle inscribed on it about one inch larger in diameter than the sensitive face of the photomultiplier tube. The inscribed area was smeared with a very light film of silicone fluid, the sample tray weighed, and the finely ground sample (-325 mesh) spread on with the aid of a camels hair brush. Excess material was shaken off, the little powder which had fallen outside the inscribed circle was trimmed, and the sample plus tray was weighed. Samples ground in preparation for counting were allowed to stand for at least a week before the alpha count was taken to allow the sample to approach equilibrium with respect to radon and thoron.

A remarkably even distribution of grains can be obtained with concentrations as low as 0.3 mg./cm^2. The work of Kulp et al (1952) shows that for samples well down in the thin source range ($<$1.0 mg./cm^2), the counting rate is proportional to sample thickness ie. essentially all alpha particles are able to escape from within the source.

Ag activated ZnS was used as a scintillator. The face of the photomultiplier tube was smeared with a light film of sil-

photomultiplier
tube

$\frac{1}{4}''$ plywood

plastic sample tray

Sample Holder

inscribed
circle

7.0 cm.
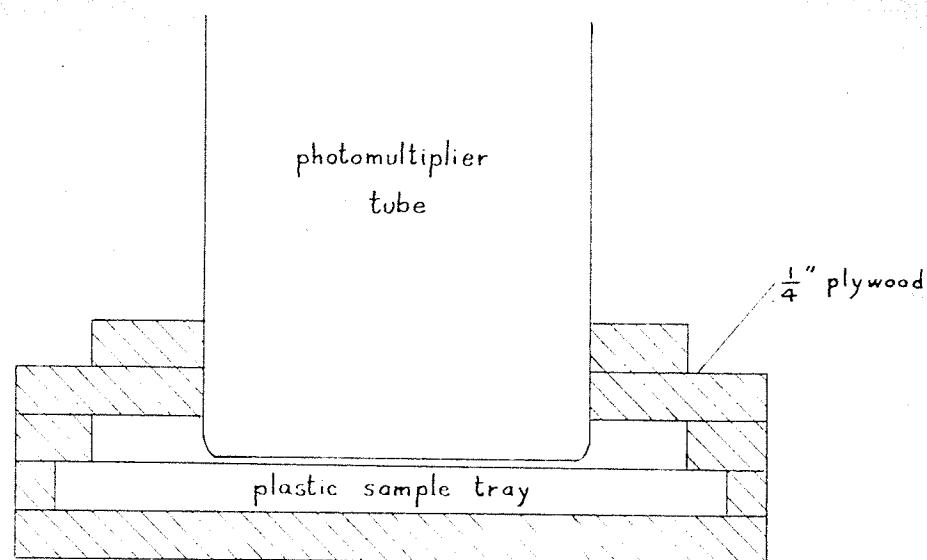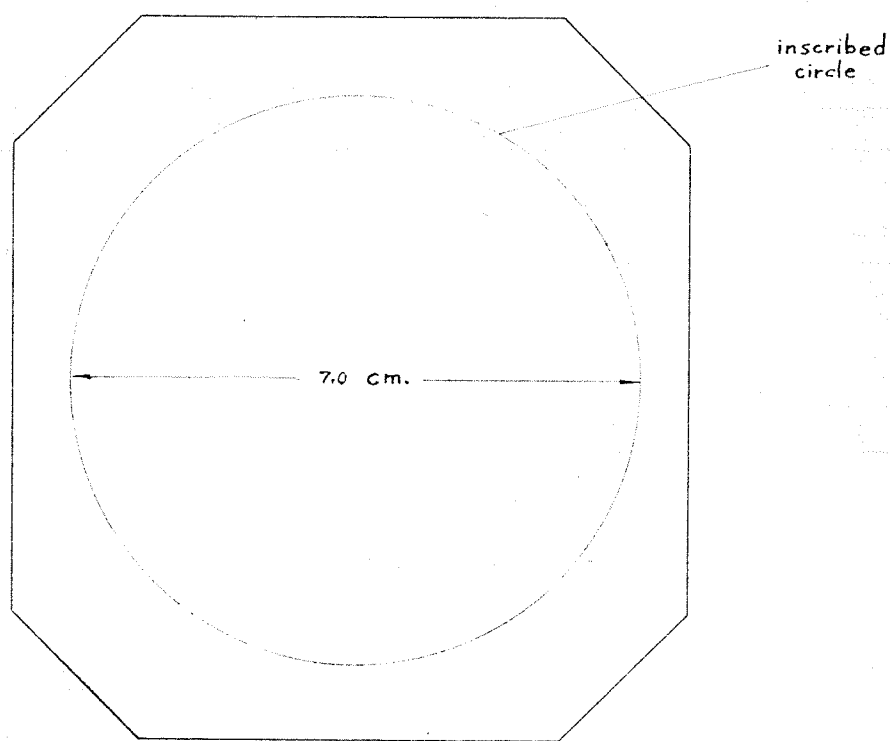
Sample Tray

Fig. 2

SAMPLE MOUNT FOR THIN SOURCE
ALPHA COUNTING

icone fluid, the powdered ZnS(Ag) sprinkled on, and the excess
shaken off. The photomultiplier tube and sample holder are en-
closed in a light-tight container and the whole placed in a
lead-block castle. Sample changing was done in darkness because
ZnS(Ag) has a certain amount of phosphorescence after exposure
to strong light. After about 8 hours in the dark this phosphor-
escence becomes negligible (Kulp et al 1952).

Voltage on the photomultiplier tube was kept low so that
tube noise would be at a minimum. The variation in counting rate
and background with tube voltage is shown in fig.3. With the
present equipment a $757\frac{1}{2}$ volt potential was used on the tube
with an amplification of about 40. A trigger level of 20 volts
was used to minimize low energy background noise and still keep
the counting rate relatively high. This level was above some of
the lower energy alpha particles and thus they would not count.
However, because all samples are considered to be in equilibrium
and the trigger level remained the same for both the standard
and the unknown samples, this is not important. The background
was about 20 counts per hour and remained fairly constant.

The source was considered to be an "infinite" plane. The
photomultiplier tube was as close as possible to the source ie.
less than 1 mm.; thus the number of alpha particles impinging on
the tube face from the outer edge of the sample was negligible.
Calculation of the alpha emission of a sample from the source
geometry was not practical so the instrument was calibrated with
an analyzed sample of the same grain size. The strength of the
calibrating source is not of first-order importance but should
be kept near the range expected in the unknown samples. With
the sample arrangement used, the same proportion of the total
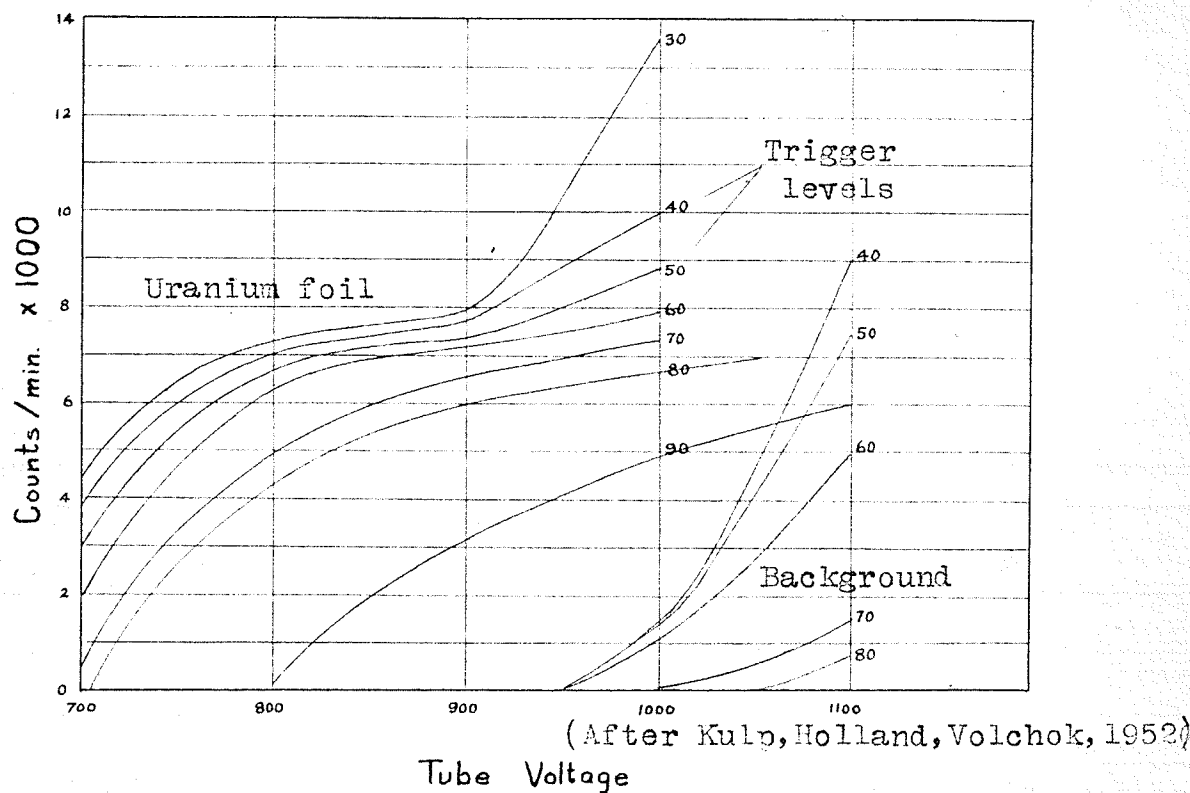
(After Kulp, Holland, Volchok, 1952)

Fig. 3

Variation of counting rate and background with
tube voltage. This is modified by characteristics of
individual photomultiplier tubes.

particles emitted from the sample will be counted regardless
of its strength. Because the alpha particle energies of both
the U and Th series are similar, the source may contain either
one or both of these elements.

The analyzed sample supplied by the Department of Mines,
Ottawa, was a monazite containing 6.12% Th, and 0.12% U. The
total alpha activity of this source may be calculated as follows:

$$T = \frac{\ln 2}{\lambda} = \frac{0.693}{\lambda} \qquad \text{where} \quad T = \text{half-life of parent}$$

$$\lambda = \text{decay constant of parent}$$

for U^{238} $\lambda = \frac{0.693}{T} = \frac{0.693}{4.498 \times 10^9 \text{years}}$

$$= 1.54 \times 10^{-10} \text{years}^{-1}$$

for Th^{232} $\lambda = \frac{0.693}{T} = \frac{0.693}{1.39 \times 10^{10} \text{years}}$

$$= 4.98 \times 10^{-11} \text{years}^{-1}$$

Thus U^{238} decays $\frac{1.54 \times 10^{-10}}{4.98 \times 10^{-11}} = 3.09$ times as fast as Th^{232}.

There are 6 alpha emissions in the Th^{232} series and 8
alpha emissions in the U^{238} series. Therefore, for alpha act-
ivity the Th^{232} equivalent of U^{238} is,

$$\frac{6}{8} \times \frac{1}{3.09} = 0.243 \, Th^{232}$$

For alpha activity, the equivalent U^{238} in the standard
sample is,

$$0.243 \times 6.12 + 0.12$$

$$= 1.65\%$$

$eU\% = 2.75 \times 10^{-4} \, I$ (Gottfried, Senftle, and Waring, 1956)

where eU = equivalent uranium
       I = alpha activity in alphas/mg./hr.

For the analyzed sample:

$$I = \frac{eU\%}{2.75 \times 10^{-4}}$$

$$= \frac{1.65}{2.75 \times 10^{-4}}$$

$$= 6000 \text{ alphas/mg./hr.}$$

The sample was prepared for counting as described above.

Wt. of sample tray + silicone film = 38.70144 gm.

Wt. of sample tray + silicone film + sample = 38.74150 gm.

Wt. of sample = 38.74150 - 38.70144 = 40.06 mg.

Area of Sample = 38.52 cm^2.

Wt. of sample/cm^2 = $\frac{40.06}{38.52}$ = 1.040 mg./cm^2.

24 hour count on sample = 628670 counts

24 hour count on background = 495 counts

Corrected count = 628670 - 495 = 628175 counts

Counts/hr. = $\frac{628175}{24}$ = 26173 c/hr.

Sensitive area of phototube = 17.95 cm^2.

Counts/cm^2 of tube area = $\frac{26173}{17.95}$ = 1458 c/cm^2/hr.

1 cm^2 of sample weighs 1.040 mg.

Counts/mg./hr. = $\frac{1458}{1.040}$ = 1402 c/mg./hr.

Total alpha emission from the sample = 6000 alphas/mg./hr.

$$6000 = k \times 1402$$

$$k = \frac{6000}{1402} = 4.28$$

The factor k can now be used to correct the observed counts/ mg./hr. from an unknown sample to total alpha emission in alphas/

mg./hr.

Counting data are given in table 2.

Table 2

Alpha Activity of the Zircon Samples

Locality	Amount (mg./cm.^{2})	Observed (c/mg./hr.)	Corrected Value (alphas/mg./hr.)
Mennin Lake, Ont.	1.034	39.84	170.5
Populus Lake, Ont.	0.312	88.49	378.7
Dryberry Lake, Ont.	0.801	42.50	181.9
*Renfrew Co. Ont.	0.295	6.76	28.9
*"Straw" zircon	1.047	46.48	198.9

*Additional samples from the Mineralogy Museum, University of Man.

The probable statistical counting error was about 2% for these counts. Counting time should be extended to reduce this to less than 1%. Counts on separately mounted samples of the same material agreed within 3.5%. The percentage error in the standard sample used for calibration is not known but should be very small.
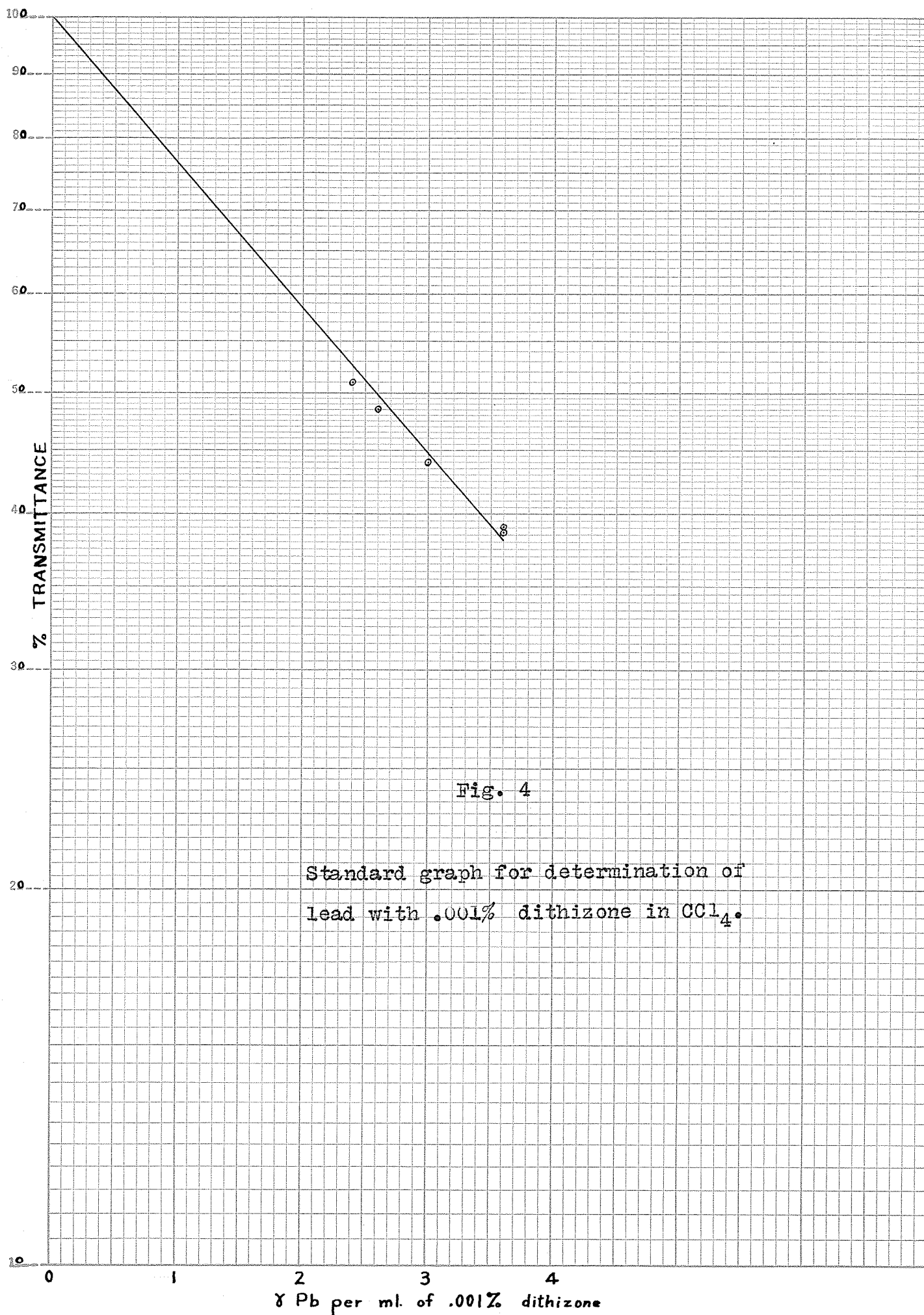
LEAD ANALYSIS

A colorimetric method described by Sandell (1950) was used for trace analysis for lead in the zircons. The accuracy of the method is claimed to be comparable to that of spectro-chemical analysis.

Zircon, being highly refractory and chemically resistant, was difficult to disintegrate. Borax was the only common flux active enough to break up zircon completely with one fusing. About 1.5 grams of borax was required for each 50 milligrams of zircon. The borax was fused in platinum, cooled, and the finely powdered zircon placed on top. Fusion was continued until the mass was transparent which took about 1/2 hour. The fused mass was stirred occasionally with a platinum wire to hasten disint-egration.

The melt was dissolved by warming with 1:5 HCl and lead was extracted from the solution at pH 9 with .01% (weight/volume) dithizone in CCl_4.

Final determination of lead was done with .001% dithizone in CCl_4, the transmittance was measured with a Coleman Spectro-photometer at a wavelength of 525 millimicrons. The reading was compared to a standard curve obtained under the same cond-itions (fig.4). If conditions are duplicated precisely and the transmittance measured in the proper range (40 to 70%), reproducibility is excellent. A blank must be run through the entire procedure to find the amount of lead contributed by

Fig. 4

Standard graph for determination of
lead with .001% dithizone in CCl$_4$.

% TRANSMITTANCE

γ Pb per ml. of .001% dithizone

reagents and apparatus. Table 3 lists the results of the lead analyses.

Table 3

Lead Analyses of the Zircon Samples

Locality	Wt. of Sample (gm.)	γ Pb found - blank corr'n	Pb content (ppm.)
Mennin Lake, Ont.	0.0619	4.0 ± 0.3	64.6 ± 4.9
Populus Lake, Ont.	0.0405	5.1 ± 0.3	125.9 ± 7.4
Dryberry Lake, Ont.	0.0362	5.4 ± 0.3	149.2 ± 8.3
Renfrew Co. Ont.	0.1416	1.41 ± 0.3	9.96 ± 2.1
"Straw" zircon	0.1534	3.0 ± 0.3	19.6 ± 2.0

CALCULATION OF AGE

Ages greater than 500 million years obtained from the equation $t = \dfrac{c\ Pb}{\alpha}$ should be corrected for U^{235} content. U^{235} makes up 1/139 of the total uranium and because it decays more rapidly than U^{238} the age will be reduced by the correction.

The corrected age is given by: $t = t_1 - \frac{1}{2}kt_1^2$ (Keevil, 1939) where t_1 is the uncorrected lead:alpha age. k is calculated from the following relation:

$$k = \frac{1.953 \times 10^{-10} + 1.624 \times 10^{-11}\ Th/U}{1 + 0.334\ Th/U}$$

For a Th/U ratio of 1, $k = 1.58 \times 10^{-10}$. This value of k was used in correcting the ages shown in table 4.

Table 4

Ages of the Zircon Samples

Locality	alpha activity (α's/mg./hr.)	Pb content (ppm.)	Pb:α age (yrs.)	Corrected (yrs.)
Mennin Lake, Ont.	170.5	64.6	940×10^6	869×10^6
Populus Lake, Ont.	378.7	125.9	825×10^6	771×10^6
Dryberry Lake, Ont.	181.9	149.2	2034×10^6	1707×10^6
Renfrew Co. Ont.	28.9	9.96	855×10^6	797×10^6
"Straw" zircon	198.9	19.6	244×10^6	-

ZIRCON AGES FROM RADIATION DAMAGE TO THE STRUCTURE

The methods and data of Holland and Gottfried (1955) were used to find how the age values compared to those obtained with the lead:alpha method.

Briefly, the basis of the method is that if the total radiation dosage received by the zircon can be determined, and the present rate of irradiation is known, then the age can be calculated. Holland's work relates the radiation dosage received to the unit cell dimensions which are changed by displacement of the atoms. A variation of this method gives the radiation dosage as a function of the change in reflection angle of the (112) planes in the zircon (Hurley and Fairbairn 1953). An important requirement is that the rate of annealing of the structure must have been zero or otherwise constant. Indications are that only alpha irradiation is important, β and γ activity contributing negligibly to disordering the atoms.

The total radiation dosage is related to the present alpha activity and to the time elapsed since irradiation began by:

$$\mathcal{f} = A \left[\frac{8 (e^{\lambda_u t} - 1) + \frac{7}{139} (e^{\lambda_{AcU} t} - 1) + 6 \phi (e^{\lambda_{Th} t} - 1)}{8\lambda_u + \frac{7}{139} \lambda_{AcU} + 6 \phi \lambda_{Th}} \right]$$

where $\mathcal{f}$ = total number of alpha disintegrations

A = present alpha activity in alphas/mg./year

$\lambda_u, \lambda_{AcU}, \lambda_{Th}$ = decay constants of U^{238}, U^{235}, and Th^{232}

ϕ = present ratio of Th to U in the sample

t = age of sample in years

Theoretically then, an alpha count on a zircon sample plus determination of the total radiation dosage from the unit cell dimensions allows solution of the above equation for t, the age of the sample. Holland considers that with his graphs c_0 gives a more accurate value of δ than the a_0 dimension. He suspected that in the suite of Ceylon gem zircons from which his data were obtained, the a_0 dimension had been slightly affected by thermal annealing.

Using the curves of fig.5, the value for t can be found from the ratio of δ/A for several Th/U ratios. It is apparent that even with ages of the order of 2 billion years, assuming Th/U = 1 when the true value is 10 results in an error of less than 10% in the determined age. Table 5 gives the ages obtained from the zircons studied, assuming a Th/U ratio of 1.
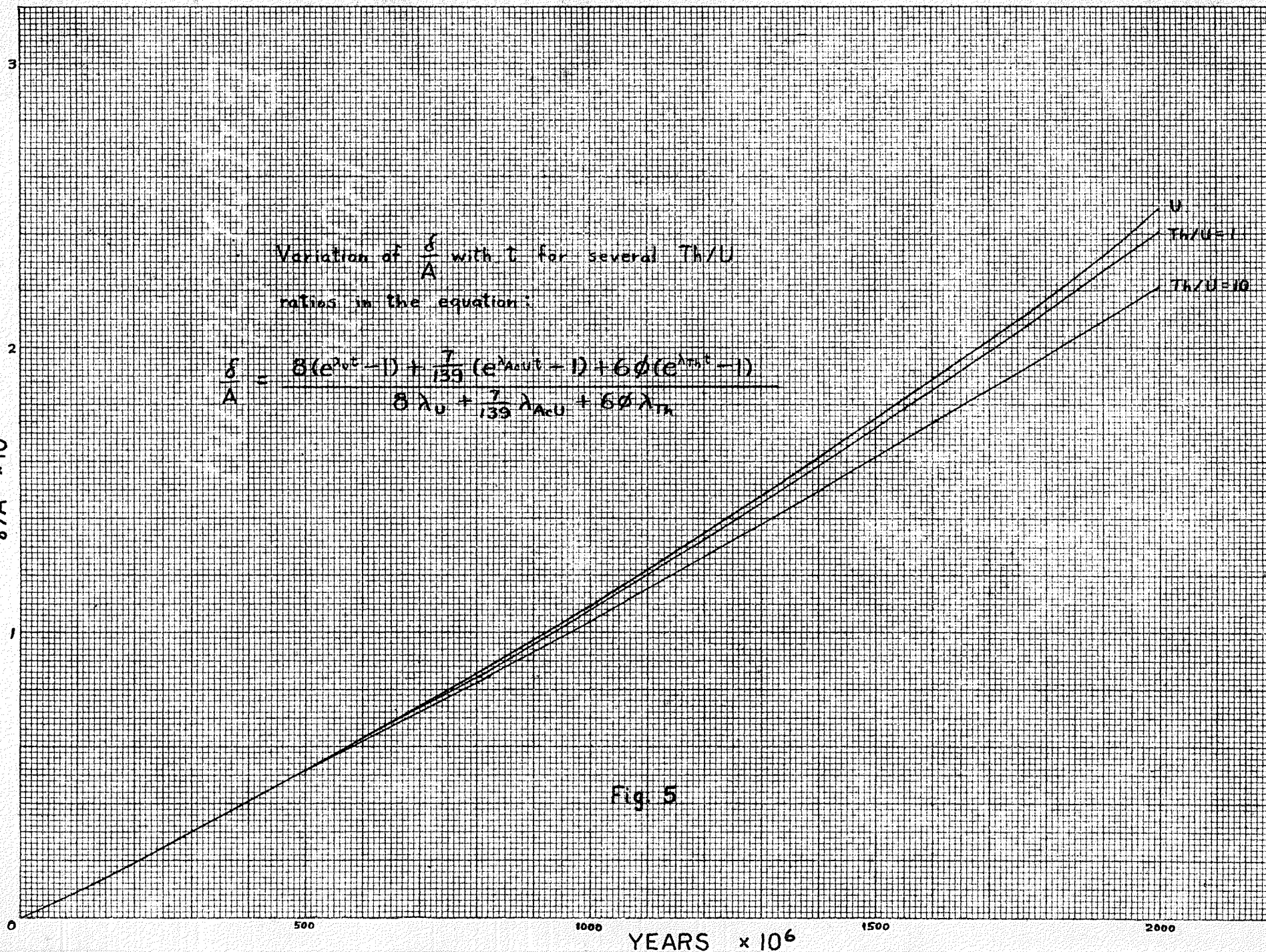
Table 5

Ages of the Zircon Samples from X-ray Data

Locality	c_0 (Å)	t from c_0	a_0 (Å)	t from a_0	Corrected Pb:α age
Mennin Lake, Ont.	6.011	660×10^6	6.604	200×10^6	869×10^6
Populus Lake, Ont.	5.992	100×10^6	6.621	390×10^6	771×10^6
Dryberry Lake, Ont.	5.992	200×10^6	6.631	1040×10^6	1707×10^6
Renfrew Co., Ont.	5.9895	570×10^6	6.6113	1280×10^6	797×10^6
"Straw" zircon	5.992	160×10^6	6.609	410×10^6	244×10^6

Except for the Mennin Lake zircon the ages calculated from c_0 are lower than those from the corresponding a_0 dimension, but the results are erratic.

The three accessory zircons gave values far below the

THIS MARGIN RESERVED FOR BINDING.

IF SHEET IS READ THIS WAY (HORIZONTALLY), THIS MUST BE TOP.

IF SHEET IS READ THE OTHER WAY (VERTICALLY), THIS MUST BE LEFT-HAND SIDE.

Variation of $\frac{\delta}{A}$ with t for several Th/U ratios in the equation:

$$\frac{\delta}{A} = \frac{8(e^{\lambda_U t}-1) + \frac{7}{139}(e^{\lambda_{AcU} t}-1) + 6\phi(e^{\lambda_{Th} t}-1)}{8\lambda_U + \frac{7}{139}\lambda_{AcU} + 6\phi\lambda_{Th}}$$

U

Th/U = 1

Th/U = 10

$\delta/A \times 10^9$

3

2

1

0

500

1000

1500

2000

YEARS $\times 10^6$

Fig. 5

lead:alpha ages. There is little doubt that they have suffered some thermal annealing. This difficulty would be expected with most Precambrian zircons and so limits the usefulness of the method. However, even though the values are incompatible with the lead:alpha ages, they do suggest the occurrence of thermal effects as late as the Mesozoic which were great enough to completely anneal the zircon structure.

Dating zircon by radiation damage to its structure is not yet considered to yield reliable ages but work is continuing and Holland believes the results are promising.

CONCLUSIONS

For age determination of accessory zircon, a 50 pound rock sample should be collected if feasible, but there was a reasonable possibility of obtaining enough zircon from as little as 20 pounds of granite. With practice, recovery of the zircon took less than 3 man-days.

Approximately 50 to 100 milligrams of zircon was necessary for an age determination using the methods described. About 30 milligrams was needed for an alpha count but the controlling factor was the lead content of the zircon. The sample should contain at least 4 micrograms of lead for accurate colorimetric determination.

The age of the Renfrew Co., Ont. zircon (855 million years uncorrected for U^{235}) compares favorably with the 900 million years (also uncorrected) obtained by Larsen et al (1952) for zircon from the same locality.

The lead:alpha ages of accessory zircons from the Kenora area indicates at least two widely separated periods of granitic intrusion. Ages as young as 771 million years and 869 million years were previously unsuspected but other age determinations are not recorded for the area. The nearest localities for which published data are available are the Huron claim pegmatites, Winnipeg River, Manitoba, with an average Pb^{207}/Pb^{206} age of 2550 million years, and a pegmatite at the Silverleaf Mine, Manitoba, with an A^{40}/K^{40} age of 2550 million years (Wilson, Russell, and Farquhar, 1956).

The lead:alpha age of 1707 million years obtained for the zircon from the Dryberry Lake granodiorite unfortunately probably represents a composite age due to the two varieties of zircon which were present.

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