K$^{40}$ - Ar$^{40}$ ISOTOPIC AGE DETERMINATION
OF THE NELSON BATHOLITH, B.C.

by

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We accept this thesis as conforming
to the required standard

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ABSTRACT

The purposes of this thesis are to examine ages of the Nelson batholith and its satellites because of the wide range of biotite model ages previously published in the literature, and to test hornblende and pyroxene further for their application in K$^{40}$ - Ar$^{40}$ dating as reliable minerals.

K$^{40}$ - Ar$^{40}$ model ages obtained on biotite, hornblende and clinopyroxene from the present study indicate with certainty that the various phases of the Nelson batholith were emplaced during a short span of time, centered around 156 m.y. corresponding to the Upper-Middle Jurassic boundary of the Kulp's time scale. At least a period of hydrothermal alteration has occurred in this area since that time. It is evident that these phases cannot be distinguished on the basis of their K$^{40}$ - Ar$^{40}$ model ages. It is also evident that hornblende gives reliable model ages. Clinopyroxene contains significant excess of radiogenic argon and should not be used for K$^{40}$ - Ar$^{40}$ dating. Biotite may occasionally contain excess of radiogenic argon, particularly under high temperature and high argon pressure environmental conditions.

Analytical techniques used and descriptions of the samples analyzed are given as appendixes.
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K\textsuperscript{40} - Ar\textsuperscript{40} METHOD OF AGE DETERMINATION

1.1. Introduction

Isotope geophysics is one of the main fields of research in the Departments of Geophysics and Geology of the University of British Columbia.

In parallel with the extensive research in the variation of lead isotopes and the isotopic composition of common strontium, the potassium-argon studies are currently under progress.

The potassium-argon laboratory was constructed early in 1963 and became operational late in 1964. The dual objectives are to investigate further the techniques of the potassium-argon method and to help unravel the history of complex geologic areas.

Today, more than 200 samples have been analyzed in the laboratory. The ages obtained range from less than 10 million years to greater than 3,000 million years.

The main interest of the present writer has been to apply these facilities to solve the geologic history of a complex area.

The Nelson Batholith, a massive granite pluton between Kootenay and Slocan Lakes, is in southern British Columbia. It is exposed over an area of approximately 900 square miles.
The major portion of the Nelson Batholith lies within the area covered by the Geological Survey of Canada, map 1090A on a scale of four miles to the inch (Little, 1960), or by the Geological Survey of Canada, map 272A on a scale of one mile to the inch (Cairnes, 1934).

(a) The purposes of the present study are:

To apply the facilities provided in the potassium-argon laboratory for dating biotites, hornblendes, pyroxene and cogenetic minerals.

To test hornblende and pyroxene further for their application in potassium-argon age determination as reliable minerals.

To determine the potassium-argon ages of the Nelson Batholith with the aim of obtaining more information on geologic history.

(b) The Nelson Batholith was selected for investigation because:

Lead isotopes studies of the area have been investigated recently by P. Reynolds (1967).

In addition, Sinclair (1964), Kanasewich (1962), Leech and Wanless (1962) investigated lead isotopes on the areas around Nelson, and:

The area contains economically important ore deposits.
The stratigraphy and structure of the area is reasonably well-known (Little, 1960).

Current field works have been carried out by members of Geology Department (Sinclair & Libby, 1967).

Potassium-argon age determinations carried out by the Geological Survey of Canada have been made only on biotites. Moreover, the published data by Gabrielse and Reesor (1964) exhibit a broad range between 49 to 171 m.y.

1.2 The $K^{40} - Ar^{40}$ method of age determination.

1.2.1 Introduction

Isotopic age determination by means of radioactive decays was first suggested by Rutherford in 1904. He postulated that the ratio of uranium to helium in a mineral should be time dependent. Subsequent investigations by Rutherford and others (Thompson, 1905; Campbell and Wood, 1906), indicated that helium is usually partially lost by the mineral making the time of "geological clock" unreliable.

Thompson (1905) and Campbell and Wood (1906), first proved the radioactive decay of potassium to calcium. It was not until 1937 when Von Weizsäcker discovered the branching decay of $K^{40}$ to $Ar^{40}$. This provided a promising possibility of two "geological clocks" within a single mineral.

* m.y.: million years
Potassium consists of three naturally occurring isotopes with the mass numbers 39, 40 and 41. Only potassium 40 is radioactive. The percent atomic abundances of the isotopes of potassium are: $K^{39} = 93.08 \pm 0.04$; $K^{40} = 0.01181 \pm 0.0001$; $K^{41} = 6.91 \pm 0.04$; (Nier, 1950; Goldich et al., 1961; A.E.C. Nuclear Data Tables, 1959). The isotopic composition of air argon was also determined by Nier (1950): $Ar^{36} = 0.337\%$; $Ar^{38} = 0.063\%$; $Ar^{40} = 99.600\%$.

Potassium decays, by both beta emission and orbital electron capture, to calcium 40 and argon 40 respectively, (Fig. 1.1).

Fig. 1.1:

Decay scheme of K$^{40}$ (after Aldrich & Wetherill, 1958). The equations for the decay can be written as follows:

$$K^{40} + e^- \rightarrow Ar^{40} + \gamma + X\text{ray or Auger electron}$$

$$K^{40} \rightarrow Ca^{40} + \beta^-$$
It is known that approximately 89 percent of potassium 40 decays to calcium 40 by beta emission but the K\textsuperscript{40} - Ca\textsuperscript{40} method has been of limited use. This is due to the fact that calcium is a very common constituent in geological materials and calcium 40 forms 97 percent of common calcium, therefore the small amount of radiogenic calcium 40 is very difficult to distinguish from the larger amount of common calcium. Moreover, Polevaya et al., (1960), mentioned that the K\textsuperscript{40} - Ca\textsuperscript{40} method can only be used for routine age determination if the following problems are satisfactorily solved: 1) find a suitable method of separation for small amount of Ca from K; 2) the Ca-44 in natural calcium must be precisely determined; 3) a "powerful" mass spectrometric technique for the determination of trace Ca.

This is not the case in the K\textsuperscript{40} - Ar\textsuperscript{40} method, where the amount of common argon contained in minerals and rocks is very low and the measurement of argon, which is one of the rare gases, is much easier. Broad time ranges are measurable with the K\textsuperscript{40} - Ar\textsuperscript{40} method because potassium is a common element in geological materials and the potassium-argon half-life is very long.

1.2.2 K\textsuperscript{40} - Ar\textsuperscript{40} model age

The K\textsuperscript{40} - Ar\textsuperscript{40} method of isotopic age determination has been developed during the past fifteen years to a high degree of sophistication. With improved analytical techniques, geological materials of very young age and low potassium content can now be dated with precise and reliable results (Gale et al., 1966; Curtis, 1966).
This makes the K⁴⁰ - Ar⁴⁰ method a widely used tool in modern geochronology. However, the accuracy in analytical techniques supplying a precise and accurate determination of the Ar⁴⁰/K⁴⁰ ratio, is only a necessary but not sufficient condition. The K⁴⁰ - Ar⁴⁰ "ages" are meaningful if and only if, the following conditions are satisfied: 1) the decay constants must be accurately known; 2) the sample must not contain excess radiogenic argon - 40, i.e. no radiogenic argon was included in the sample at the time it was formed; 3) the sample must have been a closed system, i.e. no loss or gain of potassium or radiogenic argon has occurred since it was formed, other than by radioactive decay of potassium 40.

The consistency of results will be a key point to check whether or not these assumptions are fulfilled.

1.2.2.1 Decay constants

As mentioned above, K⁴⁰ is radioactive and decays by K - electron capture to Ar⁴⁰ and by beta emission to Ca⁴⁰. The determination of these decay constants is difficult. However, a knowledge of these is absolutely necessary to predict the accumulation of radiogenic argon in a potash mineral. Many workers have tried with all available means to determine these decay constants and the branching ratio, particularly during the period between 1950 and 1960. Extensive reviewing of the available data were given by Smith (1964) and Houtermans (1966). Two methods have been used for this purpose: the physical or counting method; and the geological method.
Physical determination

This method is based on direct counting measurements. As no suitable emitting standards are available, the measurement of the emission of 1.46 MeV γ radiation is quite difficult. However, in making use of Co60 and Na24 standards and by extrapolation technique Wetherill (1957), arrived to determine the specific gamma activity. A value of $3.39 \pm 0.12 \, \text{S/g sec.}$ which corresponds to a K-electron capture decay constant $\lambda_c$ of $0.585 \times 10^{-10} \, \text{yr}^{-1}$ has been obtained.

Due to the maximum of the high beta energy of 1.34 MeV, the determination of the beta decay constant is less difficult than the previous case. A specific beta activity of $27.6 \, \text{S/g sec.}$, corresponding to a beta-decay constant $\lambda_\beta$ of $4.72 \times 10^{-10} \, \text{yr}^{-1}$ has been obtained. Endt and Kuyver (1954), Aldrich and Wetherill (1958), reviewed the available physical measurements and suggested values of $0.585 \times 10^{-10} \, \text{yr}^{-1}$ for partial electron capture decay constant $\lambda_c$ and $4.72 \times 10^{-10} \, \text{yr}^{-1}$ for $\lambda_\beta$, the partial beta decay constant.

It should mention that a new determination of the specific beta activity has been made by Glendenin (1961). By using a liquid scintillator counting technique, Glendenin obtained a value of $28.2 \pm 0.3 \, \text{S/g sec.}$ However, most laboratories have continued to adopt the values suggested by Aldrich and Wetherill.
Geological determination

By determining the production of radiogenic Ar$^{40}$ in minerals of known age, the geological method may yield satisfactory values for the decay constants. The best values obtained by this method are in quite good agreement with physical counting determinations (Wetherill et al., 1956). The work of Wetherill et al. (1956) yielded a specific gamma activity of 3.24 ± 0.15 $\gamma$/g sec. and a specific beta activity of 27.4 $\beta$/g sec., corresponding to a beta decay constant $\lambda_\beta$ of 4.70 x 10$^{-10}$ yr$^{-1}$, and K-electron capture decay constant $\lambda_e$ of 0.557 ± 0.026 x 10$^{-10}$ yr$^{-1}$. Earlier investigations using geological methods included the work of Russell et al. (1953), Wasserburg and Hayden (1954), Shillibeer et al. (1954). However, their determinations yielded a too low branching ratio. The reason for this was found out later to be due to the incomplete extraction of argon or the closed system condition was not satisfied.

1.2.2.2 Excess of radiogenic argon

It is known that in natural environments, particularly at great depth within the crust or where high argon pressure might develop, excess radiogenic argon is present. Mineral phases which crystallize in these environments are more or less subject to this argon "contamination".

The excess of radiogenic argon, occluded by the minerals at the time of crystallization is probably held in the crystal
imperfections such as dislocations or structural holes. The excess of radiogenic argon in pyroxene has been observed by many investigators. Hart and Dodd (1962) found surprisingly high "ages" for pyroxenes from a pyroxene gneiss and an amphibolite. They concluded that these pyroxenes have incorporated excess argon either during their initial crystallization or during a subsequent recrystallization. McDougall and Green (1964) dating pyroxenes from the Norwegian eclogites found also the occurrence of excess argon in these samples. A similar result has been reported by Allsopp (1965) for pyroxene from the great dyke of Southern Rhodesia. However, McDougall (1963), and Stern et al. (1965) reported that pyroxene from shallowly intruded basic igneous rocks yielded satisfactory dates. More complete degassing might be expected in low pressure environments so that these observations are not in conflict with the occurrence of excess of radiogenic argon in pyroxene, from deep-seated environments. Damon and Kulp (1958) observed the excess of argon in beryl and cordierite. They suggested further that an excess of radiogenic argon in amphiboles is possible. Funkhouser et al. (1965) reported also excess argon in hornblende from young Hawaiian rocks. The excess of radiogenic argon in fluid inclusions occurring generally in most minerals, except possibly micas, has been concluded by Rama et al. (1965).

However, Evernden and Richards (1962), Hart (1960, 1961) and Gerling et al (1965) did not prove any evidence of excess argon in amphiboles. They concluded that amphiboles may give
reliable $K^{40} - Ar^{40}$ "ages".

In praising amphibole as a reliable mineral for $K^{40} - Ar^{40}$ dating, Hart (1964), Aldrich et al (1969) and Gerling et al (1965) showed that amphiboles are generally more resistant to argon loss, through diffusion than micas because the former have a higher radiogenic argon diffusion threshold temperature (fig.1-3).

The most unfortunate is the possibility of excess of radiogenic argon in hornblende and mica, the two most commonly used for $K^{40} - Ar^{40}$ dating (Hunt, 1962; Leech et al., 1963). This raises the question whether, in certain cases, the higher argon retentivity in hornblende represents in fact a compensating effect between the excess and loss of argon. In general, one favours the idea that micas do not contain excess of radiogenic argon. However, geological and experimental evidences show that in some cases they do (Stockwell, 1963; Pepin et al., 1964). Richards and Pageon (1963) reported that some biotite samples of Broken Hill district, Australia, gave ages which were nearly twice the $Rb^{87}Sr^{87}$ ages of the same phase. Excess radiogenic argon in phlogopite and biotite from young Hawaiian rocks has been reported by Funkhouser et al. (1965).

The error in $K^{40} - Ar^{40}$ dating, caused by the excess argon, will be very serious if low potassium content minerals such as pyroxene, quartz, fluorite etc. are used for dating.

The situation is still worse if these minerals contain in addition, fluid inclusions or vacancy defects.
Possible occurrence of excess of radiogenic argon in datable minerals suggest that the interpretation of $K^40 - Ar^40$ data must be done with as much caution as possible. Usually, the presence of excess helium may be used as a good indication of excess radiogenic argon (Damon and Green, 1963). The most reliable results are believed to be obtained when the stability of the $Ar^40/K^40$ ratio in minerals possessing different potassium content is assured. This suggests the use of cogenetic minerals, for example; biotite - hornblende pairs, to test the reliability and the usefulness of the dating materials. The use of other radiometric methods, for example; the $Rb^87/Sr^87$ or lead - uranium methods would be another alternative.

In cases where cogenetic minerals are not available, the reliability must be assessed from internal consistency between the results and the known geology.

1.2.2.3 Argon loss by diffusion

Due to its great importance in the $K^40 - Ar^40$ method, the problem of diffusion of radiogenic argon will be discussed in a greater extent in the following part of this chapter.

One knows that the argon loss is dependent on the mineral and rock type. While micas are the most suitable minerals for dating, feldspars commonly show argon loss, Folinsbee et al. (1956); Zartman (1964). Dates obtained from whole rock samples in which an appreciable amount of potassium is in feldspars are therefore suspect. Erickson and Kulp (1961) applied the whole
rock technique to the basic rocks from Palisades sill and showed that the argon retentivity is much better in the finer than in the coarser grained facies.

In general, the discordance in $K^{40} - Ar^{40}$ isotopic "ages" can most often and most easily be explained by daughter product diffusion. Diffusion is defined by the American Geological Institute, "A process of spreading out of molecules, atoms or ions into a vacuum, a fluid or a porous medium, in a direction tending to equalize concentrations in all parts of a system".

The problem of argon leakage by diffusion is of such importance in $K^{40} - Ar^{40}$ dating that numerous papers dealing with both experimental and theoretical aspect of the subject have been published. Argon diffusion from minerals has been studied under laboratory conditions by Gerling and Morozova (1958, 1962); Evernden et al. (1960); Hart (1960); Baadgaard et al. (1961); Brandt (1962) and under geologic field conditions by Hurley et al. (1962); Hart (1964) and Hanson and Gast (1967). Recent reviews of argon diffusion have been made by Fechtig and Kalbitzer (1966).

**Fundamental Laws of Diffusion**

The first and second laws postulated by Adolf Fick, are given as follows:

1. The diffusion flux across a given plane is proportional to the concentration gradient across that plane:

   \[ J = - D \nabla C \]
where \( J \) is the diffusion flux, \( D \) is the diffusion constant and \( \nabla c \) is the gradient concentration.

2. The divergence of the diffusion flux is equal to the change of concentration with time:

\[
\frac{\partial c}{\partial t} = - \text{div} \ J = \text{div} \ (D \nabla c)
\]

The diffusion constant \( D \) is temperature dependent, as shown by the Arrhenius relation:

\[
D = D_0 e^{-\frac{E}{RT}}
\]

where \( E \) is the activation energy, \( D_0 \) is the characteristic constant, \( R \) is the gas constant and \( T \) is the absolute temperature.

The experimental determination of the activation energy is based on the kinetics study of argon released from minerals at different temperatures. With an initial homogeneous concentration of argon and zero concentration of argon at the surface, the process of gas release from minerals may be given in the following equations (Crank, 1956; Jost, 1960):

\[
F = 1 - \frac{6}{\pi^2} \sum_{n=1}^{\infty} \left( \frac{1}{n^2} e^{-\frac{n^2 \pi^2 D t}{a^2}} \right)
\]

where \( F \) is the fraction of total argon released at any time \( t \). This has been obtained from solution of Fick's law for a spherical grain of radius \( a \). Reichenberg (1953), by using a Fourier integral transform, obtained an approximation for \( 0 < F < 0.85 \), as follows:

\[
F = \frac{6}{\pi^2} \left( \frac{Dt}{a^2} \right)^{\frac{1}{2}} - 3 \frac{Dt}{a^2}
\]
In the case of an infinite slab; the fraction $F$ is given as follows:

$$F = 1 - \frac{8}{\pi^2} \sum_{n=0}^{\infty} \left( \frac{1}{(2n+1)^2} \right) e^{-\frac{(2n+1)^2\pi^2D}{4h^2}}$$

where $h$ is the half-thickness of the slab.

The fraction of total argon released at a given temperature for a given time interval is commonly the data received from diffusion measurements.

In general, argon in the mineral occupies several different structural positions with a different activation energy associated with each position. This arises as the function $\log \frac{D}{a^2}$ versus $\frac{1}{T}$ is generally non-linear for the entire temperature range between $25^\circ C$ and $1,000^\circ C$. It is often observed that the general result is several straight line segments (Hart, 1960; Gerling and Morozova, 1962; Gerling et al., 1969) corresponding to different activation energies, as presented schematically in figure 1-2.

![Schematic plot of log D versus 1/T](image-url)

Figure 1-2: Schematic plot of $\log \frac{D}{a^2}$ versus $\frac{1}{T}$.
In general, good preservation of the mineral itself is considered as a sufficient condition of complete retention of radiogenic argon. The activation energy of minerals can also serve as criterion of the retention of radiogenic argon in minerals. Sardarov (1963) carried out an experimental investigation on the retentivity of radiogenic argon. He concluded that argon is quantitatively preserved in the glauconites up to 400°C and the escape of argon is due to structural decomposition of the mineral at this temperature. With an activation energy of \( E = 60 \) Kcal/mole glauconite should retain argon completely at a lower temperature.

The works of Gerling and Morozova (1958, 1962), Gerling et al. (1963) showed that three different activation energies are characteristic of argon in muscovite, and they found that the argon in micas was held in three types of sites: a few percent in the first, 60 - 70 percent in the second and the remainder in the third. However, the work of Brandt (1962) indicated only one single activation energy for muscovite. Gerling and Morozova (1962) observed also that the argon was held in microcline-perthite in five different sites, corresponding to five different activation energies; the first three phases accounted for 20 percent of the argon, the fourth contained about 15 percent of the argon and the fifth contained the remainder of the argon, 65 percent.

Amirkhanov et al. (1959) has made some argon diffusion
measurements on various potash minerals and observed three
different activation energies in each of two feldspar samples.

Fechtig et al. (1961) investigated the argon diffusion
from a number of minerals and rocks. They found that the argon
diffusion from phonolite, at temperatures up to 600°C did not
show a straight line on the log $D$ versus $\frac{1}{T}$ plot. However, in
the case of augite, two straight line segments were obtained.

It is found that the results of diffusion measurements
made by various investigators on the same minerals but different
samples vary significantly. Many authors have correlated this
variation to structural dislocations or lattice changes in
minerals (Baadsgaard et al., 1961).

In any case, most of the above authors proved essentially
that micas and hornblendes are suitable for K$^{40}$ - Ar$^{40}$ dating.
Hurley et al. (1962), Hart. (1964) and Hanson and Gast (1967)
showed that the apparent ages obtained by K$^{40}$ - Ar$^{40}$ method or
Rb-Sr method are a function of distance from a contact zone
as shown in figure 1-3. The higher retentivity of hornblende
over biotite may also be seen from this diagram.

Other possible loss of radiogenic argon has been accoun-
ted to chemical alteration (Kulp and Engels, 1963; Manka et al.,
1965 and Goldfisch et al., 1966). and thermal effect due to
mechanical abrasion (Gerling et al., 1961).
Figure 1-3:

Variation of apparent ages of biotite, hornblende and feldspar as a function of distance from an intrusive contact (After Hart, 1964).

1.2.3 Materials Used for K⁴⁰ - Ar⁴⁰ Dating

Depending on the type of materials analyzed, crystallization metamorphic events in some cases and local intrusion or origin of rock formation in others, may be events recorded by K⁴⁰ - Ar⁴⁰ method.

The laboratory geochronologist must therefore be constantly aware of the sufficient conditions for the K⁴⁰ - Ar⁴⁰ model age, particularly the second and third conditions.
In general, micas, sanidine, glauconite are the most widely used minerals for $K^40 - Ar^40$ dating because of their large range of occurrence, high potassium content and high argon retentivity. However, with improved analytical techniques, geological materials other than micas can also be used. All igneous metamorphic and sedimentary rocks have been used as geological materials for dating.

The $K^40 - Ar^40$ "age" obtained on minerals of an igneous rock gives the time of emplacement, provided that argon has not been lost by diffusion process and the rock has not been subjected to a later metamorphism (sufficient conditions). Similarly, the age yielded from minerals of a metamorphic rock will indicate the time of the metamorphic event as previously accumulated argon is completely lost by this metamorphic event.

An excellent description of dating of sediments by $K^40 - Ar^40$ method has been made by Hurley (1966) and Hower et al. (1963). The essential things are, they must retain well radiogenic argon and must not contain fragments of detrital minerals. The reason for this is based on the fact that these fragments may contain relict argon.

Using fossil bones, limestones and fluorite as dating material for $K^40 - Ar^40$ method, an acceptable total fossil bone age has been obtained and reported by Lippolt and Gentner (1963).
It should be noted that, from their three dated fluorites, contained about 50 ppm of potassium; only one sample yielded an acceptable date. The other two gave high ages implying probably the sufficient condition (2) was not fulfilled. Therefore, in order to obtain reliable results, careful selection of samples is of primary importance. Weathered samples should be discarded, fresh materials are highly desirable for K\textsuperscript{40} - \textsuperscript{40}Ar dating.
CHAPTER II

GENERAL GEOLOGY OF THE NELSON BATHOLITH

2.1 Introduction

2.1.1 History of Literature

After seventy years of mining activity the first geologic report of the West Kootenay District, British Columbia was published by G.M. Dawson (1889).

The stimulation of metallic mineral deposits particularly gold and silver, spurred others to look enquiringly at the Nelson region. Field investigations and geological studies have therefore been carried out by prospectors and members of the Geological Survey of Canada, (Cairnes, 1934; Mulligan, 1952 etc.).

Most of the previous works, as well as his own are summarized by H.W. Little, (1960). Recent geological investigation of the northern part of the Nelson batholith has been carried out by members of the Geology Department (Sinclair and Libby, 1967). Much of the geological informations in the present study has been based mainly on the above-mentioned authors' work: , particularly on Little's memoir.

2.1.2 Composite Nature

Hamilton and Myers (1967), in their paper entitled "The Nature of Batholiths" indicate a composite petrography is characteristic of batholiths. The Nelson batholith can be
Figure 2-1: Location Map of K-Ar* Samples of Nelson Batholith and Related Rocks

New Denver - Kaslo, B.C.

Base Map, H.W. Little, Map 1090A, Nelson Quad (w)
reasonably described in terms of four phases of intrusion. These are, Nelson rocks, Nelson satellites including the Mount Carlyle stock, (Sinclair and Libby, 1967), Valhalla rocks (Little, 1960) and Lamprophyre dykes that cut the Nelson batholith. Each of these may be further subdivided either in terms of field or laboratory criteria.

2.2 Different Phases of the Nelson Batholith

2.2.1 Nelson Rocks

The main part of the area of the present study is underlain by Nelson rocks. Compared with many other batholiths, (Hamilton and Myers, 1967), the Nelson batholith is in the main granite rather than falling in the broad "granitic" classification of the Sierra Nevada and others. Toward the contact borders with the country rocks the content of ferromagnesian minerals tends to increase.

In many sections the Nelson rocks may be described as belonging to either a porphyritic or a non-porphyritic member. The porphyritic member is characterized by considerably large crystals of sodic orthoclase. In a few localities, these phenocrysts may contribute up to 50 percent of the rock and range in size with an average of about two inches. In areas exhibiting deformation, these are replaced by microcline. The groundmass of the porphyritic member is characterized by an abundance of hornblende over the biotite. Partial chloritic alteration is common in these ferromagnesian minerals.
Figure 2-2: Setting of the Nelson Batholith and some satellites (After Little, 1960)
The porphyritic member is cut by, or sometimes grades into, the non-porphyritic member. The latter tends to be granodiorite as the acidic phenocrysts are missing and the other mineral constituents are the same as in the porphyritic member. The npn-porphyritic member of the Nelson batholith is considered to be slightly younger than the porphyritic member. This is based on the fact that the former crosscuts, in places, the latter.

2.2.2 Nelson Satellites: Rocks

The Nelson satellites comprise apophyses and stocks of the Nelson batholith, including the Mount Carlyle stock. The Slocan series south of the Mount Carlyle stock forms a septum several hundred feet wide separating the stock from the northern edges of the batholith. (Sinclair and Libby, 1967). With some exceptions, the composition of the satellitic rocks, on the whole, resembles very much that of the Nelson rocks. Both porphyritic and non-porphyritic members are present. Most of these satellites have been observed to intrude the Triassic Slocan series and an important number of them intersect other formations. Mulligan (1952), believed that the stocks are of early phases of the Nelson intrusion. However, Cairnes (1934), suggested that the satellites represent a later stage of the intrusion of the Nelson batholith. Little (1960), concluded that these satellites are if not contemporaneous, slightly younger than the Nelson batholith.
2.2.3 Valhalla Rocks

Chemically and mineralogically, the Valhalla rocks are essentially equivalent to the Nelson rocks. The differences are predominately textural, finer grained and allotriomorphic instead of hypidiomorphic texture as in the case of the Nelson rocks. The contacts between the Valhalla rocks and the Nelson rocks are commonly gradational but sharp contacts occur where the former intrudes the latter. The similarity between these phases led Cairnes (1934), to drop the name Valhalla, but Little (1960), preserves the distinction.

The Valhalla rocks outcrop toward the west, but a hat-shaped mass of Valhalla is exposed over five square miles about the headwaters of Maurier, Fennell and Silverton creeks. The rarity of Valhalla rocks in the eastern Slocan river and Slocan lake and "the Nelson rocks comprise a large proportion of this part of the batholith, possibly owing to mobilization of the porphyritic material which may have flowed out from the autochtonous core, pushing the eastern peripheral rocks into their present arc-like position", has been suggested by Little (1960).

As the contacts between the Nelson rocks and Valhalla rocks are most often indistinct, Little (1960), assumed contemporaneity of these two phases.

2.2.4 Lamprophyre Dykes

Dykes have been observed to cut the Nelson plutonic rocks.
Figure 2-3: Previous age determinations by field geology, Nelson batholith.
Their composition, on the whole, resembles very much that of the Nelson rocks. They are considered to represent a late stage in the intrusion of the Nelson batholith, (Cairnes, 1934).

2.3 Origin Due to Metasomatism of Paleozoic and Early Mesozoic

From observations of its components, (the paragneiss, the Nelson satellites and Valhalla rocks) and its structure, the origin of the Nelson batholith may be deduced.

The paragneiss exposed in the Nelson batholith core and elsewhere has been correlated with the Hall formation of Middle (?) and Upper Jurassic age. The paragneiss, and Valhalla rocks form successive layers with gradational borders. Lying above the paragneiss and grading laterally into it are the Nelson rocks which, in turn, are overlain and with gradational contact by Valhalla non-porphyritic rocks. The gradational contacts between the Valhalla and the Nelson are often observed. However in some places the dykes of the Valhalla rocks cut the Nelson, normally without exhibiting dilation.

It is concluded by Little (1960), that the core of the Nelson batholith exhibits evidence of a metasomatism origin, i.e., the rocks were formed by metasomatism of pre-existing rocks such as sedimentary rocks. Little showed also evidence to suggest that the region now occupied by the Nelson batholith had been deeply buried. Moreover, the autochthonous core of the Nelson batholith is typical of Read's deep-seated batholith, (Read, 1951). This has been used by Little to ensure his hypothesis.
However, Little (1960), pointed out that the rocks about the periphery, unlike those of the core, have been mobilized and injected into older rocks.

2.4 Age of Metasomatism and Intrusion by Stratigraphy and Structure

The Nelson batholith seems to be the oldest major batholithic mass formed during the Mesozoic orogeny in the Arc. Cairnes (1934), reported that the emplacement of the Nelson batholith followed intense folding of the Sloe an series. The orogenic disturbances have caused the fracturing and brecciation exhibited by the Nelson rocks. Based on the chain-relation between the orogenic disturbance and the mineralization which is, in turn, closely related in origin to the Nelson rocks, the age of the Nelson batholith may be deduced if the date of the orogenic disturbance is known. This orogenic disturbance was believed to occur at about the same time in the Selkirk as in the Rocky Mountains. Cairnes (1934), assigned consequently, the invasion of the Nelson batholith to late Cretaceous.

Earlier investigators, based on the similarity of the Nelson rocks and the Coast rocks, suggested the same age for both Coast Range and Nelson rocks. The Coast Range intrusives have been observed to cut Jurassic rocks, (Middle and Lower Jurassic fossils were observed) and in places, the batholithic rocks intrude sediments of lower Cretaceous age. From those evidences, it was suggested that the Coast plutonic rocks represent a long
period of intrusion, from Upper Jurassic to Cretaceous. Based on the fossils of late Upper Jurassic age observed from the basal Kootenay bed and on the assumption that the Kootenay was chiefly derived from the geanticline batholith, Newmarch (1953) suggested the Upper Jurassic as the lower limit time of emplacement of the Nelson batholith. However, the existence of the fossils of Lower Cretaceous age are also observed, because of these inconclusive evidence available, Newmarch stated that the Upper Jurassic or Lower Cretaceous as the lower limit time of emplacement of the Nelson batholith.

In observing the Nelson rocks to contain a heavy mineral suite which is identical with that of the Blairmore formation, igneous pebbles from McDeugall-Segur conglomerate, Beveridge and Folinsbee (1956), proposed that the Nelson rock was unroofed just after the deposition of the basal Blairmore conglomerate. The age of this conglomerate has been assigned to Lower Cretaceous, (Albian) by Bell (1956).

Little (1960), concluded that "the lower limit of the age of the Nelson plutonic rocks, with some confidence, is post Middle Jurassic", and, "the upper limit of the age of the Nelson rocks can be set, with rather less confidence, as pre-Upper Cretaceous". The lower limit was based on the fact that the Nelson rocks cut sedimentary and volcanic rocks overlying the Hall formation whose youngest fossils are of early Middle Jurassic age.
The upper limit was based on the fact that the plant fossils from the Sophie Mountain formation are probably of Upper Cretaceous age and the Sophie Mountain conglomerate contains granitic pebbles derived from Nelson. On the other hand, the Sophie Mountain formation rests unconformably upon the Rossland formation and is cut by the Nelson, (figure 2-2).

In any case, the surest limits of the emplacement of the Nelson batholith and its satellites can be set as following: the Nelson rocks cut the Triassic or Lower Jurassic Slocan series, this indicates that they are younger than the Slocan series and of course they must be older than the mineralization period.

2.5 Post-Crystallization History

The Nelson batholith is considered to be a massive granitic pluton, however areas of strong foliations may be observed within the pluton. A deep break which has been a centre of deformation and later intrusion, occurs in the western part of the Nelson batholith, along the Slocan Lake (Little, 1960). The eastern part of the batholith has been subjected to significant post-crystalline deformation which affected the enclosing sedimentary and metamorphic rocks. The period of deformation was marked by the fissuring and shearing whose direction is controlled by well developed joint fractures. As mineral deposits occur, in general, in these fractures, it is believed that the mineralization took place after most of these fractures occurred.

In effect, the age of the Nelson batholith determined by geological investigation indicates a somewhat complex intrusion
history. The geological relationships are clearcut as there
are different opinions concerning the age of the batholith,
as described above. (figure 2-3). The only definite result
is that the igneous event of the Lamprophyre dykes which are
considered to be slightly younger or contemporaneous with the
main batholith. (Little, 1960). Even so, geological investiga-
gations could not determine an "absolute age" of the formation.
The situation is resolvable only within the last decade with
the method of physical determination.
CHAPTER III

PHYSICAL DETERMINATION

3.1 Previous Works

As the "age" of the Nelson Batholith could not be solved definitely by geological means, some workers have attempted to solve this complex history batholith by means of physical determination.

By using lead alpha activity ratios on accessory minerals zircons, monazite and xenotime, Larson et al. (1934), arrived to determined the age of rocks from the Southern California, Sierra Nevada and Idaho batholiths. Five age determinations on Idaho batholith average 103 m.y. Beveridge and Folinsbee (1956), in using the radioactive method have evaluated the age of zircon from the gneissic phase of the Nelson batholith, near Whatshan Lake, B.C.. A date of 105 m.y. resulted. Based on the good agreement between this result and those from southern batholiths, Beveridge and Folinsbee suggested that the emplacement of the Nelson batholith was in late lower Cretaceous time.

A K\textsuperscript{40} - Ar\textsuperscript{40} age determination on biotites separated from granodiorite near the city of Nelson, yielded a value of 86 m.y. as reported by Little (1960). However, Little stated that "this figure, which of course appears to be too small, may be incorrect because of recrystallization of the biotite by metamorphism long after emplacement of the batholith."

Following this, the Geological Survey of Canada conducted a series of age determinations by K\textsuperscript{40} Ar\textsuperscript{40} method on biotite. The
samples have been collected and interpreted by Reesor and reported by Lowdon et al. (1961, 1963), Leech et al. (1965) and Gabrielse and Reesor (1964).

A total of 10 age determinations have been resulted as follows: GSC 60-21 (49 m.y.), GSC 60-22 (55 m.y.), GSC 61-17 (131 m.y.), GSC 62-26 (165 m.y.), GSC 62-27 (159 m.y.), GSC 62-28 (171 m.y.), GSC 62-29 (163 m.y.), GSC 62-30 (171 m.y.), GSC 62-31 (105 m.y.), GSC 62-32 (63 m.y.). The widespread in ages led Reesor to propose that the emplacement of Nelson batholith has evolved through a very long time. It developed in three main stages distinguished by after three groups of ages:

(a) The older group (from 159 to 171 m.y.) represents the emplacement and consolidation of hornblende, biotite and granodiorite before 171 m.y..

(b) The intermediate group (from 86 to 131 m.y.) indicates a mobilization and reintrusion, due to an intense structural episode occurring about the earliest Cretaceous, of a relatively incompetent mass.

(c) Finally, the younger group (from 49 to 63 m.y.) represents further emplacement of leucocratic quartz monzonite.

Reesor then concluded that if this hypothesis is accepted, there is thus no conflict between the age data and stratigraphic-structural data. However, the considerable spreading in ages even, within a very short distance between localities, which generally
assumed to be of nearly the same age, appears to be unsatisfactorily justified.

Although, Reesor has carefully formulated his hypothesis within three possible major factors that could affect the age measurement. These factors are: the complex succession of structural events within the mesozoic cycle of deformation, the associated complex evolution of rocks throughout the development of the mobile belt during the Mesozoic and finally the complex gradual cooling of emplaced mass during uplift and uproofing.

One can therefore see that Reesor's views of the Nelson batholith are somewhat different or it could be said directly that the required a new definition for the batholith to warrant his hypothesis.

3.2 Current Project

3.2.1 Introduction

To test the validity of the "Reesor hypothesis" of the evolution of the Nelson batholith, it is considered necessary to extend further structural, petrologic studies as well as to make additional isotopic age determinations. Geological study of the northern part of the Nelson batholith has been carried out by Sinclair and Libby of the Department of Geology, who have kindly provided a total of eleven specimens for the present study.

Four plutonic rock units are considered in this study:
1. The northern part of the Nelson batholith.


3. Little's "Valhalla plutonic rocks" centered on the headwaters of the Fennel Creek.

4. Lamprophyre dykes that cut the Nelson batholith.

All samples used for K\textsuperscript{40} - Ar\textsuperscript{40} isotopic age determination were chosen after careful examination of thin sections of specimens. Sample locations shown in figure 2.1 are listed in the Appendix II with the petrographic descriptions. Experimental procedures used are listed in Appendix I.

Three major steps involved in the K\textsuperscript{40} - Ar\textsuperscript{40} isotopic age determination of the present study are:

1. The separation of the minerals from rocks.

2. The determination of potassium content in minerals by flame photometry (Dean, 1960; Cooper, 1963; Cooper et al., 1966 and Dirom, 1965).

3. The determination of argon content in minerals by mass spectrometry static isotope dilution method (Reynolds, 1956; Farrar et al., 1964) using a highly enriched Ar\textsuperscript{38} tracer. The argon analytical system used is shown in figure 3-1.
Figure 3-1: UBC Argon Extraction and Analytical System
3.2.2 The K\(^{40}\) - Ar\(^{40}\) Results

The results obtained by K\(^{40}\) - Ar\(^{40}\) age determinations on biotite, hornblende and pyroxene from the various phases of the Nelson batholith are presented in Table 3.1. Sample numbers correspond to those given in figure 2-1. The data is conveniently presented in a graphical form in figure 3-2, a plot of log Ar\(^{40}\) versus log K. The 156 m.y. isochron, a straight line containing all the points of the same age, is shown. It is accompanied by two other isochrons of 100 m.y. and 200 m.y. respectively. All plotted points located on the upper side of the straight line are of older ages and those on the lower side are of younger ages. The sign of excess radiogenic argon or argon loss of the samples can therefore be checked with this kind of graphical presentation. The data obtained by Reesor, (Gabrielse and Reesor, 1964) are also plotted in the same figure 3-2.

A histogram of K\(^{40}\) - Ar\(^{40}\) dates is shown in figure 3-3, where frequency versus log ages are presented. The plotting interval for each date is equal to the estimated analytical error.

Eleven age determinations reported by Gabrielse and Reesor (1964) are concurrently presented. An error of \(\pm 8\%\) (Lowdon et al. 1963) has been attached to each date before plotting. The advantage of this kind of presentation has been discussed by Ross (1966).

Table 3.2 gives U.B.C. results obtained on interlaboratory
Figure 3-2: Plot of log Ar$^{40}$ vs. log K isochrons.
FIGURE 3.3: A plot of frequency versus log apparent ages


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<th>Sample Number</th>
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<th>K (%)</th>
<th>$\text{Ar}^{40}$* Total $\text{Ar}^{40}$</th>
<th>$\text{Ar}^{40}$* Total $\text{Ar}^{40}$ x 10^-10 moles/g</th>
<th>$\text{Ar}^{40}$/K</th>
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$\lambda_e = 0.585 \times 10^{-10}$ yr^{-1}; $\lambda_e = 4.72 \times 10^{-10}$ yr^{-1}; $K^{40}/K = 0.01181\%$

$\text{Ar}^{40}$*: radiogenic argon 40
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<td></td>
<td>Hornblende</td>
<td>1.208</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Quartz Monzonite</td>
<td>1.209</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1.209</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>$\text{Av} = 1.210 \pm 0.005$</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample Number</td>
<td>Mineral Analyzed</td>
<td>K (%)</td>
<td>Ar(^{40})(^*)/(Total Ar(^{40}))</td>
<td>Ar(^{40})(^*)-10(^{-10}) moles/g</td>
<td>Ar(^{40})(^*)/K(^{40})</td>
<td>Apparentage (m.y.)</td>
<td></td>
</tr>
<tr>
<td>---------------</td>
<td>------------------</td>
<td>-------</td>
<td>-----------------------------------</td>
<td>---------------------------------</td>
<td>-----------------</td>
<td>------------------</td>
<td></td>
</tr>
<tr>
<td>C-66</td>
<td>Hornblende</td>
<td>1.104</td>
<td>0.56</td>
<td>3.342</td>
<td>0.010032</td>
<td>164±6</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Hornblende</td>
<td>1.106</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Quartz Monzonite</td>
<td>1.099</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.104</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td><strong>Av= 1.103±0.005</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SILV-66</td>
<td>Hornblende</td>
<td>0.886</td>
<td>0.69</td>
<td>2.442</td>
<td>0.009123</td>
<td>150±5</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Hornblende</td>
<td>0.888</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Quartz Monzonite</td>
<td>0.884</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.886</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td><strong>Av= 0.886±0.003</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>257-66</td>
<td>Biotite</td>
<td>6.85</td>
<td>0.90</td>
<td>19.005</td>
<td>0.009159</td>
<td>150±5</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Nelson Batholith</td>
<td>6.86</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Quartz Diorite</td>
<td>6.87</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Porphyry</td>
<td>6.89</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td><strong>Av= 6.87±0.03</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>R-12</td>
<td>Clinopyroxene</td>
<td>0.555</td>
<td>0.71</td>
<td>2.731</td>
<td>0.016207</td>
<td>258±12</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Quartz Diorite</td>
<td>0.552</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.559</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.557</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.555</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.559</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.562</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td><strong>Av= 0.559±0.002</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
standard minerals,\(^1\) including U.B.C. standard biotite GD-12.

A comparison of analyses carried out at U.B.C. isotope geophysics laboratory with others made in various laboratories on the same mineral samples (Muscovite standard rP-207; biotite standards GE-3203 and GE-2060) is shown in Table 3.3.

Table 3.4 gives the "ages" of the various rock units on the Nelson batholith.

\[ \text{3.2.3 Analytical Precision and Accuracy} \]

The interference effect is one of the most controversial problems in the accuracy and precision of the potassium determination by flame photometry. The interference effects of elements involved in the solutions used for K\(^40\) - Ar\(^40\) dating have been investigated by many authors, (Dean, 1960; Cooper, 1963; and Cooper et al., 1966). Various types of buffering techniques have been proposed. The main purpose of the buffering action is to make both the standard and the sample solutions close to the same physical, chemical and ionic condition within the flame. This involves the addition of the same amount of an interfering element to the standard solutions as is present in the sample solutions. Cooper (1963) indicated that the interference of other ions depends considerably on the characteristics of the flame photometer type used (for example, burner, fuel). Dirom (1965) reported that with the instrument and the buffering technique used for the present study except H\(_2\)SO\(_4\), the interference effects of the--other elements,\(^\gamma\) are negligible.

\(^{1}\text{Analyzed by J. Harakal}\)
TABLE 3.2

RESULTS OF U.B.C. LABORATORY ON STANDARD MINERALS

<table>
<thead>
<tr>
<th>Sample Mineral</th>
<th>K (%)</th>
<th>$\text{Ar}^{40}$ Total Ar$^{40}$ (x10$^{-9}$ moles/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Biotite GD-12</td>
<td>$\bar{x} = 5.56$</td>
<td>0.83 2.081</td>
</tr>
<tr>
<td>Biotite GE-2060</td>
<td>$\bar{x} = 6.83$</td>
<td>0.88 2.459</td>
</tr>
<tr>
<td>Biotite B-3203</td>
<td>$\bar{x} = 8.58$</td>
<td>0.72 17.121</td>
</tr>
</tbody>
</table>

$\bar{x} =$ Arithmetic mean

$\sigma =$ Standard deviation or mean deviation
**TABLE 3.3**

**COMPARISON OF ANALYSES CARRIED OUT IN**

**U.B.C. WITH OTHERS MADE IN VARIOUS**

**LABORATORIES ON STANDARD MINERALS.**

<table>
<thead>
<tr>
<th>Sample Mineral</th>
<th>Data Source (Date)</th>
<th>K (%)</th>
<th>$\text{Ar}^{40}$* (x10^-9 moles/g)</th>
<th>UBC Difference in $\text{Ar}^{40}$* Analyses (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Columbia Biota (1963)</td>
<td>$\bar{x} = 6.87$</td>
<td>$\bar{x} = 2.500$</td>
<td>$\pm 0.05$</td>
<td>$\pm 0.011$</td>
</tr>
<tr>
<td>Yale Biota (1963)</td>
<td>$\bar{x} = 6.83$</td>
<td>$\bar{x} = 2.472$</td>
<td>$\pm 0.06$</td>
<td>$\pm 0.034$</td>
</tr>
<tr>
<td>UBC (1965-1967)</td>
<td>$\bar{x} = 7.58$</td>
<td>$\bar{x} = 17.30$</td>
<td>$\pm 0.12$</td>
<td>$\pm 0.06$</td>
</tr>
<tr>
<td>Lanphere and Dalrymple (1965)</td>
<td>$\bar{x} = 7.42$</td>
<td>$\bar{x} = 17.33$</td>
<td>$\pm 0.02$</td>
<td>$\pm 0.06$</td>
</tr>
<tr>
<td>UBC (1965)</td>
<td>$\bar{x} = 8.58$</td>
<td>$\bar{x} = 1.260$</td>
<td>$\pm 0.12$</td>
<td>$\pm 0.024$</td>
</tr>
<tr>
<td>Summary (Lanphere and Dalrymple 1967)</td>
<td>$\bar{x} = 8.58$</td>
<td>$\bar{x} = 1.244$</td>
<td>$\pm 0.05$</td>
<td>$\pm 0.017$</td>
</tr>
</tbody>
</table>

$\bar{x}$ = Arithmetic mean  
$\sigma$ = Standard deviation or mean deviation
Moreover, the interference effect of $\text{H}_2\text{SO}_4$ was only significant for low potassium content amphibole and pyroxene solutions. For this reason, the standard solutions used for biotite analysis did not require any addition of $\text{H}_2\text{SO}_4$ for buffering purpose.

In the present study, for those samples which resisted complete dissolution, further additional heating or nitric acid was used to replace perchloric acid. This was to avoid the possible interference effects of perchloric acid which may cause some depression of potassium emission.

The potassium analysis was carried out at least in quadruplicate for each sample. The obtained average values of potassium content were then used in the calculation of the apparent age. The results obtained on thirty-five analyses in duplicate or quadruplicate of the L.G.O. biotite standard GE-2060, U.S.G.S. muscovite standard P-207 and Nelson project biotite samples indicate a precision of about 0.7 percent (Tables 3.1 and 3.2). From Table 3.3, the absolute agreement obtained on the potassium determination of the standard P-207, between the U.B.C. results ($8.58 \pm 0.05$) and the averaged results of twelve different laboratories ($8.58 \pm 0.12$) is noteworthy. From the standard GE-2060, a difference of about 0.6 percent resulted. The potassium analysis is therefore believed to be accurate within 0.6 percent or better.

For hornblende and pyroxene, the results obtained from four samples, in at least quadruplicate, on the Nelson project show a
precision of about 0.4 percent (Table 3.1). Unfortunately no hornblende or pyroxene standard is available at present. However, with careful work, on the preparation of samples, sample and standard solutions, it is believed that the accuracy is better than two percent.

One of the most serious problems in the argon analysis by mass spectrometry static method is the contamination of the previous sample on the sample to be processed. This causes a change in the isotopic ratios measured with time. However, with the mass spectrometer MS-10 used in the present study, no "memory effect" has been detected. The standard deviation of the average isotopic ratios was always less than 0.2 percent. Farrar et al., (1964) ascribed this to the low accelerating voltage, about 100 volts, used in the mass spectrometer MS-10. The resolving power of the MS-10 is approximately 50, this permits to resolve perfectly all mass peaks of argon isotopes 36, 38 and 40. A sample run for atmospheric argon, given in figure 3-4, shows the resolution in the mass 36 - 40 range. Based on the high precision and accuracy obtained previously in the laboratory, (White et al., 1967), only a single run was carried out for each sample, (except the clinopyroxene sample R-12 which was run in duplicate). Seventeen analysis on biotite standards indicate a precision of approximately one percent (Table 3.2). It may be seen that the U.B.C. argon analysis results are systematically lower than those reported by other laboratories. The difference is approximately one percent (Table 3.3.).

It may be deduced from Table 3.2 that the results obtained in the present study are internally consistent within two percent
Figure 3-4: Typical atmospheric argon run shows the resolution in the mass 36-40 range.
### TABLE 3.4

**Ages of the Various Rock Units on the Nelson Batholith**

<table>
<thead>
<tr>
<th>Rock Unit</th>
<th>Sample Number</th>
<th>Rock Type</th>
<th>Mineral Dated</th>
<th>Model Age (m.y.)</th>
<th>Remark on Sample Analyzed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nelson batholith</td>
<td>R-11</td>
<td>Granodiorite</td>
<td>Biotite</td>
<td>120</td>
<td>Chloritized</td>
</tr>
<tr>
<td></td>
<td>R-13</td>
<td>Granodiorite</td>
<td>Biotite</td>
<td>130</td>
<td>Chloritized</td>
</tr>
<tr>
<td></td>
<td>R-12</td>
<td>Quartz diorite</td>
<td>Biotite</td>
<td>146</td>
<td>Fresh</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Clinopyroxene</td>
<td>265</td>
<td>Fresh</td>
</tr>
<tr>
<td></td>
<td>C-66</td>
<td>Quartz monzonite</td>
<td>Biotite</td>
<td>151</td>
<td>Very fresh</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Hornblende</td>
<td>164</td>
<td>Very fresh</td>
</tr>
<tr>
<td></td>
<td>SILV-66</td>
<td>Quartz monzonite</td>
<td>Hornblende</td>
<td>150</td>
<td>Biotite completely chloritized but hornblende is fresh in appearance. About 15% of Biotite has ragged edges.</td>
</tr>
<tr>
<td></td>
<td>L-236</td>
<td>Hornfels</td>
<td>Biotite</td>
<td>135</td>
<td></td>
</tr>
<tr>
<td>Nelson satellites</td>
<td>L-257</td>
<td>Quartz diorite</td>
<td>Biotite</td>
<td>150</td>
<td>Altered</td>
</tr>
<tr>
<td>(including Mont Carlyle stock)</td>
<td>K-66</td>
<td>Quartz monzonite</td>
<td>Hornblende</td>
<td>161</td>
<td>Very fresh</td>
</tr>
<tr>
<td>Valhalla Plutonic Rocks</td>
<td>A-66</td>
<td>Granodiorite</td>
<td>Biotite</td>
<td>149</td>
<td>Slightly altered</td>
</tr>
<tr>
<td>Post-Nelson batholith</td>
<td>D-66</td>
<td>Lamprophyre dyke</td>
<td>Biotite</td>
<td>169</td>
<td>Fresh</td>
</tr>
</tbody>
</table>
of the apparent age. However, due to the discrepancy met when comparing the results obtained by various laboratories, larger error limits are adopted. The apparent ages are assigned error limits ranging from three to six percent. The lower precision was assigned to the analysis which indicated less than 50 percent radiogenic Ar$^{40}$ as suggested by Lipson (1958).

### TABLE 3.5

RELIABLE MODEL AGES SELECTED FOR DATA INTERPRETATION

<table>
<thead>
<tr>
<th>Mineral dated</th>
<th>Sample number</th>
<th>Model age (m.y.)</th>
<th>Standard deviation (m.y.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Biotite</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>R-12</td>
<td>146</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td>A-66</td>
<td>149</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>C-66</td>
<td>151</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>D-66</td>
<td>169</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>Hornblende</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SILV-66</td>
<td>150</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td>C-66</td>
<td>164</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>K-66</td>
<td>161</td>
<td>6</td>
<td></td>
</tr>
</tbody>
</table>
3.3 Discussion

The K$_{40}$ - Ar$_{40}$ isotopic age determination of the Nelson batholith, in the present study, yielded values which agree reasonably well with the result obtained from geological evidences.

On the whole, the values range from 120 m.y. to 169 m.y., with the exception of the clinopyroxene sample R-12 which yielded an anomalous age of 265 m.y.. A large group of ages fall around 150 m.y. Without further considerations, the values indicate that the various phases of the Nelson batholith were emplaced during a considerable interval of time, from Middle Jurassic to Lower Cretaceous. The age results fit therefore the sequences of emplacement of the granitic rocks as established by contact relationships observed in the field. Cairnes (1934), in his memoir stated that, "the sequence commenced with the batholithic intrusion which was followed by the invasion of stocks and minor intrusives and concluded with the mafic dykes".

However, a more detailed interpretation of the data needs further refinement. From twelve age determinations obtained, of which three have been obtained on hornblendes, one on clinopyroxene and the remainder on biotites, Age determinations were carried out for two cogenetic minerals pairs. Five age determinations have been considered unreliable, based on the fact that the prerequisite sufficient conditions posed previously were not fulfilled.
First of all, the clinopyroxene sample R-12 yielded an age of 265 m.y. which is unquestionably anomalous, regarding the results obtained on the other samples. The results were checked analytically for both potassium and argon determinations. On the flame photometry determination, an average of 0.5585 percent with a standard deviation from the mean of less than 0.5 percent has been obtained from eight different aliquots. Its potassium content is relatively low. However, based on the precision and accuracy obtained from previous analyses on very low potassium bearing materials (White et al., 1967), it is believed that the potassium determination is accurate within ±2 percent, if not better. The argon analyses were carried out in duplicate. The experimental data are therefore considered to be reliable. The accuracy and precision are believed to be within the error limits assigned. On the other hand, the highly discordant apparent ages obtained from the pair of cogenetic minerals sample R-12, 146 m.y. from biotite against 265 m.y. from the clinopyroxene sample, suggest to the writer that clinopyroxene contains excess radiogenic argon. The use of the clinopyroxene for K\(^{40}\) - Ar\(^{40}\) dating should therefore be avoided. Hart (1961), investigating pyroxene for K\(^{40}\) - Ar\(^{40}\) dating stated that "pyroxenes are unlikely hosts for excess argon as they do not have structural vacancies or holes as amphiboles". However, further works of Hart and Dodd (1962) and Lovering and Richards (1964), proved the presence of excess radiogenic argon in pyroxene. This "age" may be disregarded on the basis of the prerequisite conditions for a K\(^{40}\) - Ar\(^{40}\)
model age are not fulfilled. Therefore, highly anomalous clinopyroxene "age" must not be included in the data interpretation.

The other four samples, L-236, R-11, R-13 and L-257, do not satisfy the third sufficient condition of the $K^{40} - Ar^{40}$ model age. In particular, the sample L-257 is so heavily chloritized and altered that even the yielded value of 150 m.y. which is fairly concordant with the "age" of the Nelson plutonic rocks. This date is still considered unreliable. Moreover, the sample was collected from an intrusion in rocks of the Slocan series, north of the Nelson batholith. It was found that the sample, petrographically, is very distinct from the Nelson plutonic rocks. The date is believed to indicate only a late stage of hydrothermal alteration. Samples R-11, R-13 and L-236 yielded 120 m.y., 130 m.y. and 135 m.y. respectively. The low ages may be explained by the argon loss through diffusion. The deleterious effects of alteration on the $K^{40} - Ar^{40}$ ages of biotites and hornblendes have been discussed by Goldich & Gast (1966). They showed that alteration has reduced approximately 25 percent the $K^{40} - Ar^{40}$ age of biotites from Morton gneiss of southwestern Minnesota. Kulp and Engels (1963), Martin et al. (1965), also discussed the possible effects of alteration on $K^{40} - Ar^{40}$ ages of biotites. These effects are generally more drastic on $Rb^{87} - Sr^{87}$ ages than on $K^{40} - Ar^{40}$ ages mainly because of the large exponential effect in the $A^{40*} / K^{40}$ ratio age relation. In any case, those samples did not
fulfill the prerequisite conditions for the $K^{40} - Ar^{40}$ model age. They may be disregarded in the interpretation of the emplacement history of the Nelson batholith.

Finally, a remark should be made on samples D-66 and SILV-66. The sample D-66 yielded an age of 169 m.y. which appears high in light of geologic evidence. From field geology, it was observed that the lamprophyre dyke definitely cut the Nelson rocks (A.J. Sinclair, personal communication). This anomalous biotite age could again be explained by the fact that the prerequisite conditions might not be completely fulfilled. An excess of radiogenic argon in the sample, is not improbable. Richards and Pidgeon (1963), reported that some biotite samples of Broken Hill of New South Wales, Australia, gave ages which were nearly twice the Rb-Sr age of the same phases. A similar excess of radiogenic argon in micas have also been reported by many others (Stockwell, 1963; Pepin et al., 1964).

It is known that the measured radiogenic $Ar^{40}$ content of mineral depends not only on the argon retentivity and the temperature history of the phase, but also on partial argon pressure of the geological environment where the mineral was formed. It seems therefore possible, that the high $K^{40} - Ar^{40}$ model ages appear to occur in rocks formed under high pressure and temperature conditions. This implies that relatively high argon pressure could possibly develop and consequently
excess radiogenic argon could be trapped into the existing or newly formed minerals. Nevertheless, the idea that argon content of mica may be affected by external argon pressure has been shown by Karpinskaya et al. (1961). They proved experimentally that a considerable excess of argon can be forced into muscovite under high argon partial pressures and at relatively high temperature. This has been supported by Hart (1961). Thus the slightly high model age of 169 m.y. yielded by the biotite sample D-66, may indicate locally high argon partial pressures operating at some time in the history of biotite. This supports further the hypothesis of deep-seated origin of the Nelson batholith proposed by Little (1960).

However, in the case of the present study the excess argon is quite insignificant. The error involved is still within experimental limits, and the date obtained is therefore considered to be reliable. Sample SILV-66 has been collected from the eastern edge of a major fracture zone forming the western contact of the Nelson batholith. Extensive chloritization is recognized along this zone (A.J. Sinclair, personal communication). The rock has undergone considerable cataclasis and biotite has been completely replaced by pseudomorphs of chlorite. However, hornblende is undeformed and appears fresh. The apparent age of 150 m.y. yielded by hornblende sample SILV-66 should therefore be included in data interpretation. In any case, the date seems
to be very interesting; the high argon retentivity of hornblende is worthy for praising. Its usefulness in the $\text{K}^{40} - \text{Ar}^{40}$ dating is unquestionably significant. The high reliability of the hornblende dates has been affirmed by Hart (1964) and Gerling et al. (1965).

After the above-mentioned "restrictions", the remaining data are four fresh biotite model ages, which yield a mean value of 154±10 m.y. and three hornblende model ages which yield a mean value of 158±7 m.y. (Table 3.5). These values are believed to be reliable within limits of experimental error. It may be seen that the pair of cogenetic biotite-hornblende from the specimen C-66 gives fairly concordant isotopic ages; 151 m.y. for biotite against 164 m.y. for hornblende. The low biotite age may be explained by the fact that loss of argon in biotite is more significant than that in hornblende. In other words, hornblende has had higher retentivity of radiogenic argon than biotite (Hart, 1964; Gerling et al., 1965; Lovering & Richards, 1964). The low apparent age from biotite sample C-66 may be caused by heat produced along the contact zone, between Valhalla and Nelson plutonic rocks, which would result in argon loss from the relatively older Nelson rocks. As the $\text{K}^{40} - \text{Ar}^{40}$ model ages obtained from rocks and minerals may vary with the distance from an intrusive contact (Aldrich et al., 1960; Hart, 1964; Hanson and Gast, 1967), biotite may lose up to 50 percent of
its argon within one mile whereas within a very small distance even as near as 1,000 feet from the contact, hornblende may retain well all its argon (figure 1-2). Some possibility of error may occur in these results which were based on two-dimension studies instead of three. Further geophysical investigations should be carried out for a more accurate result. However, quantitatively, it may be concluded that the hornblende model age of 164 m.y. is more reliable and more closer to the true age than its cogenetic biotite model age of 151 m.y.

From the results obtained in this investigation, one may see that it is impossible to distinguish the various phases of the Nelson batholith by means of K\textsuperscript{40} - Ar\textsuperscript{40} dating. The variations in the model ages obtained were within the analytical limits of error. This reaffirms the importance of field geology in the investigation of phase relationships of an area. Many laboratory geochronologists have often the tendency of over-estimating the role of K\textsuperscript{40} - Ar\textsuperscript{40} dating in geochronology investigations. However, on the basis of the present study and the work of K. Northcote of the Geology Department, on the Guichon batholith, it is clearly realized that without field geology controls, misinterpretation may arise. In the case of the Nelson and Guichon batholiths, the K\textsuperscript{40} - Ar\textsuperscript{40} results indicate very little about the distinction of various phases. The time at which minerals begin to retain the radiogenic argon is nearly the same for each phase. Conversely, field geologists attempt to think of geological processes expanding at time intervals of tens of millions of years, whereas in reality, the periods
From geological field study the sequence of events which occurred in the Nelson batholith may be summarized in rough geochronologic order as follows: The Nelson rocks, the Nelson satellites, the Valhalla rocks and the Lamprophyre dyke.

If the age of the Nelson batholith is defined as the time at which the minerals of the batholith start to retain radiogenic argon, then the model age of 154±10 m.y. is the representing figure. However it is believed that the model age of 158±7 m.y. obtained on hornblende is closer to the true age of the Nelson batholith. The higher retentivity of argon of hornblende over biotite has been recognized by many workers in the field of geochronology (Hart, 1964; Gerling et al., 1965).

If the model age for the lamprophyre dyke is accepted, then on the basis of geological evidence, an assumption that all the other samples have lost argon must be made; the age of the Nelson batholith is therefore older than 170 m.y. Structural relations require that the sample D-66 be younger than the other samples. The model age obtained of 169±6 m.y. is slightly higher than one would expect on structural evidence. Possible excess of radiogenic argon in this sample has been discussed above. X-ray diffraction was also used to check whether or not the sample has been contaminated by other minerals such as clino-pyroxene. The result from X-ray diffraction affirmed further the "purity" of the sample. In any case, the "excess age" is so small, within the limits of experimental error, that
the date does not cause any significant conflict to the model age obtained on hornblende samples.

From the above discussion, it is preferable to accept a true "age" for the Nelson batholith within the limits of control of data on biotite, hornblende and geological (lamprophyre dyke) model ages.

Therefore the average $\text{K}^{40} - \text{Ar}^{40}$ model age of 156±9 m.y. may be considered to be a close approximation to the true age of the batholith.
CONCLUSION

The $^{K_40} - ^{Ar_{40}}$ isotopic age determinations on biotite, hornblende and pyroxene of the Nelson batholith in the present study indicate that the various phases of the batholith (at least in the northern part), were emplaced during a short interval of time, not more than 18 m.y., centered around 156 m.y., corresponding to the Upper-Middle Jurassic boundary of the Kulp's time scale. At some later time, less than 120 m.y., ago, the batholith was subjected to at least one period of hydrothermal alteration, resulting in local chloritization and therefore variable loss of radiogenic argon from biotites, thus accounting for the scatter of model ages between 120 - 146 m.y..

It is also concluded that the various phases of the Nelson batholith cannot be distinguished on the basis of the $^{K_40} - ^{Ar_{40}}$ isotopic dating; the variations in "ages" of these phases were within experimental error limits, of the analytical techniques used.

On the basis of the results obtained on two pairs of cogenetic samples, biotite-hornblende and biotite-clinopyroxene, it is evident that: Clinopyroxene contains significant excess of radiogenic argon and therefore it should not be used for $^{K_40} - ^{Ar_{40}}$ dating.

Hornblendes give reliable $^{K_40} - ^{Ar_{40}}$ model ages. Their high retentivity of radiogenic argon is worthy for praise. Biotites may contain excess of radiogenic argon, particularly under high temperature and high argon pressure environmental conditions. It is therefore evident that $^{K_40} - ^{Ar_{40}}$ dates are...
particularly suitable, especially in an initial reconnaissance, for identifying hitherto unsuspected complexities.

However, it is necessary to consider the $K^{40} - Ar^{40}$ dates along with all available petrographic and field geology evidences to be able to give a clearcut conclusion.

Further investigations on the Nelson batholith, especially in the southern part, should involve further detailed mapping in conjunction with isotopic age determinations, particularly by $Rb^{87} - Sr^{87}$ method. This might unravel the metamorphic, structural and intrusive history of the whole area.
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APPENDIX I

EXPERIMENTAL PROCEDURE

A-I-1 Mineral Separation

The samples were extracted to detain concentrate pyroxene, hornblende and biotite.

The equipment was carefully cleaned and decontaminated from the previous samples with a split of the sample to be processed.

Rocks were crushed, ground and sieved into size fractions ranging from 35 mesh to 100 mesh (in the case of hornblende, it was often necessary to reach a finer size).

Using in various combinations hydraulic classification, heavy liquid, electrostatic and magnetic separation techniques.

The procedures may be summarized in the following flow sheet in figure A-I-1.

To obtain pure minerals, great care was taken. As a final step of clean-up of the sample, it was always necessary to use "hand picking" to remove any impurity, the surest way to obtain a clean sample of not less than 99% pure hornblende or biotite.
Fig. A-I-1: Flowsheet showing various steps of mineral separation.

Hand size rock

\[ \downarrow \]

jaw crusher

\[ \downarrow \]

gyratory-crusher

\[ \downarrow \]

cone crusher

\[ \downarrow \]

screen

14 mesh \[ \rightarrow \] 14 mesh

Pulverizer

Screen

\[ \downarrow \]

35 mesh \[ \rightarrow \] -35 +50 mesh \[ \rightarrow \] -50 +70 mesh \[ \rightarrow \] -70 +100 mesh \[ \rightarrow \] -100 mesh

Wash in \( H_2O \) column

\[ \downarrow \]

Dry (65° - 100°C)

\[ \downarrow \]

Ding electrostatic separator

A: BIOTITE CONCENTRATE

B: REJECT

A: Biotite Concentrate

\[ \downarrow \]

tetrabromoethane

\[ \downarrow \]

sink \[ \rightarrow \] float

(biotite concentrated) \[ \downarrow \] (reject)

Plastic sheet

\[ \downarrow \]

biotite concentrated \[ \rightarrow \] reject

handpicking under binocular microscope for complete cleaning

\[ \downarrow \]

biotite \[ > \] 99% pure
Fig. A-I-1 (Cont'd...)

**B: Reject**

- Remove strongly magnetic particle with hand magnet
- Frantz isodynamic separator (vertical position)

  - reject, quartz, feldspar
  - hornblende concentrate

  - tetrabromoethane
    - float (reject, quartz, feldspar)
    - sink
      - hornblende concentrate

  - Diiodomethane
    - float (hornblende concentrate)
    - sink (epidote)

  Frantz inclined feed (20° forward, 15° side, ≈ 0.425 amp.)
  - reject
  - hornblende concentrate

  - plastic sheet (remove biotite)
  - handpicking under binocular microscope for further cleaning

  - hornblende ≈ 99% purity
In the separation of the minerals from rocks, the only difficulty met was the removal of all epidote grains (the common impurity) from amphibole concentrates.

To achieve an optimum separation it was found necessary to use a high-power binocular microscope, which was also employed to monitor each step of separation, as an aid for hand picking of impurities. However, the presence of a small amount of epidote in the amphibole concentrates introduces insignificant error in potassium content as the content of potassium in epidote is negligible (Dirom, 1965).

The purified pyroxene, hornblende and biotite concentrates were washed several times in acetone or methyl alcohol to remove the heavy liquid before being used for analysis.

The Determination of Potassium Content in Mineral by Flame Photometry

The potassium content in mineral was determined by flame photometric techniques involving two main steps:

1. Acid digestion of mineral sample by chemical procedures.

2. Determination of potassium content by flame photometer.
Chemical Procedures

1. About four aliquots of approximately 0.25 gm of biotite sample were extracted by microsplitting (in the case of muscovite, aliquots of about 0.18 gm should be reasonably adequate).

2. Weigh samples into teflon dishes.

3. Add 1.5 ml concentrated H$_2$SO$_4$ and 25 ml 49% HF.

4. Place on hot plate and evaporate down to about 5 ml at a temperature of approximately 250°C (using asbestos mats on the hot plate to protect teflon dishes).

5. Add 10 ml HF and evaporate down again to about 5 ml.

6. Add 2 ml concentrated HNO$_3$, the temperature was then increased until the first white fumes of H$_2$SO$_4$ appear.

7. The samples were allowed to cool, about 50 ml of pure water were added and evaporate down again.

8. Add 50 ml of pure H$_2$O, warm and transfer solution to a 250 ml beaker.

9. Place on hot plate and bring to a boil, stop and cool.

10. To each aliquot, add 50 ml of stock solution containing 5,000 ppm Na and 2,000 ppm Li.
11. Transfer each aliquot to a 500 ml volumetric flask and dilute with pure water to a final volume of 500 ml.

12. Store in clean, dry and tight looking cap polyethylene bottles for flame photometric analysis.

In the case of hornblende or pyroxene, the above-described procedure was used. Due to the low potassium contents in hornblende and pyroxene, samples of 0.75 gram were decomposed in a total of 3 ml of concentrated H$_2$SO$_4$, 35 ml and 49% HF and 2 ml concentrated HNO$_3$. However, if the samples resist complete dissolution, further additional heating and if necessary, an addition of approximately 1 ml of concentrated HNO$_3$ was found to yield satisfactory digestion. The use of other reagents such as perchloric acid was therefore avoided. The solution was transferred to a 250 ml volumetric flask to which was added 25 ml of the stock solution containing 5,000 ppm Na and 2,000 ppm Li. The solution was then made to volume with pure water and stored in polyethylene bottles for flame photometric analysis.

**Flame Photometric Measurement**

The sample solutions were run on a Baird Atomic model KY-1 flame photometer, bracketed by standard solutions ranging from 0 ppm to 50 ppm in approximately 4 ppm increments. These standard solutions contain the same
concentration of Li, Na and H₂SO₄ as the sample solutions. However, H₂SO₄ was not necessarily added to the standard solutions for biotite solutions because the interference effect of H₂SO₄ was negligible in this case (Dirom, 1965).

The results were then recorded on a 2-second response, 10 millivolt zero centre WESTON strip chart recorded connected in series with the photometer output. After standardizing the flame photometer as recommended by the maker's instructions, four runs for each analysis were made in alternative direction. The results of the four runs were then averaged. The concentration interval between the higher and lower standard solutions was reasonably kept small so that the relationship between concentration of potassium in the standard and sample solutions may be considered linear. The concentration of potassium in the sample solution was then calculated by using the following equation.

\[ x = \frac{y - y_1}{y_2 - y_1} (x_2 - x_1) + x_1 \]

where

- \( x \) = concentration of potassium in the sample solution
- \( x_1 \) = concentration of potassium in the lower standard solution
- \( x_2 \) = concentration of potassium in the upper standard solution
\[ y = \text{dial reading for the sample solution} \]
\[ y_1 = \text{dial reading for the lower standard solution} \]
\[ y_2 = \text{dial reading for the higher standard solution} \]

The Determination of Argon Content in Minerals

The radiogenic argon content in minerals was determined by mass spectrometric isotope dilution techniques.

The argon analytical system used for the present study consists of 5 main sections, as shown in figure 3.1.

- the fusion and first purification section
- the second purification section
- the mass spectrometer section
- the argon 38 spike metering section
- the air calibration section.

With a system of ultra high vacuum metal valves, any of these sections can be isolated or connected properly. Except for the mass spectrometer section where an iron pump was used, the other four sections were evacuated by mercury diffusion pumps, backed by high vacuum rotary pumps. Five pirani gauges were also added into the system for the measurement of the pressure of the different sections.

The Radiogenic Argon Extraction and Purifications

A sample charge of from 0.5 to 0.8 gram biotite (in the case of hornblende and pyroxene, the charges used were from 2 to 6 grams), contained in an alundum en-
closed molybdenum crucible, was placed in the fusion jar (water jacketed pyrex jar), which, by a copper gasketed flange coupling, was connected to the argon line. It was outgassed by overnight baking at a temperature of approximately 200°C, which was done only in the case of the hornblende and pyroxene sample. In the case of the biotite sample, overnight baking at room temperature was found sufficiently adequate.

The sample was heated by means of a 6 KW radio frequency generator and induction coil fit around the fusion jar. Cold trap and U trap containing activated charcoal were used and kept at temperature of nitrogen liquid. Hydrogen and carbon monoxide are oxidized to become water and carbon dioxide which together with the other impurities were removed by the cold finger trap which prevents the water from reaching the charcoal trap; nitrogen was removed by allowing the gas to react with the hot titanium sponge. When the temperature of the sample attained approximately 1,000°C an Argon 38 spike was admitted into the fusion and first purification section by means of two high-vacuum metal valves. The temperature of the sample was then raised gradually to its final value of approximately 1,500°C, and at this temperature the fusion of the sample may be considered as complete.
However, it was maintained at this temperature for almost 15 minutes to ensure the complete removal of argon from the sample. All the argon was then absorbed by activated charcoal at low temperature, (the temperature of the nitrogen liquid).

To ensure that the purification was complete, the sample was repurified, particularly in the case of amphibole, by means of separate titanium sponges and pumping system in the fusion and first-purification and second purification sections. The system was so designed that any number of purification cycles could be conveniently realized by allowing the gas back and forth between the purification sections. However, a single cleanup cycle is found to be adequate, especially in the case of the biotite sample.

After the purification steps have been completed, argon was absorbed on charcoal trap at nitrogen liquid temperature and the remaining gas was pumped away.

Mass Spectrometric Measurement

Before each analysis, leak testing was carried out to check the build-up of the mass 40 signal. This was done simply by opening each section in turn to
the mass spectrometer. A measure of the relative abundance of argon 40 and argon 36 contained in the sample enabled one to make a correction for any contamination of the sample by atmospheric argon.

The purified gas from the second purification section was then admitted to the mass spectrometer section of the system to measure its isotopic composition.

The mass spectrometer used for the present study is a clinical model MS10, made by Associated Electric Industries Ltd., using a two inch radius of curvature with 180° deflection and a permanent magnet producing a magnetic field of approximately 1830 gauss.

Some slight modifications have been made, for example a more sensitive vibrating reed electrometer amplifier with a 1011 ohm input resistor to replace the maker's electrometer. The mechanical scanner was also replaced by an electrostatic scanner which causes the appropriate variations in accelerating voltage. Therefore any mass peak may be reached immediately, the recording time becomes shorter and the signal decay becomes consequently negligible. Other detailed descriptions may be found in the maker's instructions catalogue. A Honeywell -
Brown potentiometer-type recorder of ½ second response, 10 millivolt full scale, to record the results.

Mass spectrometry static method was used. A small fraction of the total gas sample was admitted to the mass spectrometer tube which was then isolated by an ultra high vacuum metal valve and analyzed.

As the mass spectrometer is maintained continuously at a pressure of less than $10^{-8}$ mm Hg, by means of an ion pump, the vacuum inside the mass spectrometer tube was proved significantly sufficient for the statical analysis to be carried on.

Before each run, the background was checked; it was observed that the argon 36 and argon 38 background was reduced to minimum so that they were nearly undetectable. Argon 40 background was also reduced to minimum so that it was only detectable on the most sensitive range. Only mass 28 signal which is supposed to be due to nitrogen and carbon monoxide was significant. Mass 44 signal which is supposed to be due to carbon dioxide was also reduced to minimum.

It was found that an amount of between 5 and 10 volts of argon 40 signal admitted to the mass spectrometer is effectively adequate for the analysis to be carried out.
Figure A-I-2: Fractionation correction vs \( \% \) admitted to the spectrometer tune.
Isotopic ratios $\text{Ar}_{40}/\text{Ar}_{38}$ and $\text{Ar}_{40}/\text{Ar}_{36}$, at least 10 times for each sample, were measured and averaged.

Before being used in the age calculation these ratios had to be subjected to two corrections: discrimination correction and fractionation correction.

Discrimination in the mass spectrometer has been evaluated by analyzing atmospheric argon.

According to Graham's Law, isotopic fractionation is caused by the slower passage of the heavier isotope into the leak valve relative to the lighter isotope. In order to have the fractionation at the inlet leak valve evaluated, a check of the total quantity of argon sample admitted to the mass spectrometer was always made. With the designed system used in the present study, this requires no difficulties. A portion of gas sample of approximately 9% in volume of the second purification section, could be measured accurately with mass spectrometer.

A graph showing fractionation correction against the percentage of quantity of gas admitted to the mass spectrometer for analysis is given in figure A-I-2.
APPENDIX II

NELSON PLUTONIC ROCKS

DESCRIPTIONS OF SPECIMENS FOR K\(^{40}\)\(^{40}\)DATING
(PREPARED BY DR. A.J. SINCLAIR)

Specimen A-66:

The rock is a massive, medium-grained, leucocratic granodiorite of the Valhalla plutonic suite that appears fresh in hand specimen. A thin section mode is: plagioclase (50%), quartz (24%), K-feldspar-perthitic microcline (15%), biotite (5%), hornblende (1%), sphene (1%), opaque minerals (1%), muscovite and sericite (1%), and trace amounts of epidote, chlorite, apatite, carbonate and zircon. The rock has undergone very slight deformation as indicated by shadowy extinction of quartz and gently curved cleavage traces of some biotite grains. Some alteration effects are apparent but these are not pronounced. Biotite is fresh in appearance. A small amount of chlorite is present as monomineralic grains. Locally, plagioclase has been altered to epidote, calcite and sericite, although most of the epidote in the rock occurs as fairly large, anhedral grains associated with hornblende.

Specimen C-66:

The specimen of Nelson plutonic rock is a medium-to coarse-grained, massive hornblende quartz monzonite containing 10% mafic minerals (mainly hornblende) and 90% felsic constituents (mainly feldspar) with a few percent subhedral, K-feldspar phenocrysts up to 15 mm. in length. A mode determined from thin section is: plagioclase-An\(_{20}\) (47%), K-feldspar-microcline (30%), quartz (15%), hornblende (5%), biotite (3%), and trace amounts of myrmekite,
apatite, sphene and opaque minerals. There is no evidence of superimposed alteration effects.

Specimen D-66:

The rock is a fine-grained, grey dyke that cuts Nelson plutonic rock. Rock-forming minerals could not be identified in hand specimen although a minor amount of chalcopyrite was observed. A mode determined from thin section is as follows: plagioclase-An\(_{31}\) (56%), clinopyroxene (30%), biotite (12%), olivine and iddingsite (2%) and trace amounts of apatite and opaque minerals. Plagioclase occurs mainly as lath-shaped crystals showing vague pilotaxitic texture. Olivine and iddingsite occur as relict anhedral cores texture. The specimen shows no effects of alteration apart from what can be ascribed to deuteric processes.

Specimen K-66:

The specimen is a medium-grained, massive, porphyritic hornblende quartz monzonite from Mount Carlyle stock containing abundant pale pink phenocrysts of K-feldspar up to 2.5 cm. in length. A medium-grained matrix consists predominantly of plagioclase, quartz and about 10% hornblende as narrow prisms about 2 mm. long. A mode estimated from thin section is: K-feldspar-perthitic microcline (45%), plagioclase-An\(_{20}\) (37%), quartz (10%), hornblende (5%), epidote-including allanite (1%), sphene (1%), biotite (0.5%) and trace amounts of myrmekite, chlorite, carbonate, apatite, zircon and opaque minerals. Alteration effects are minor. Minerals are fresh in appearance, although very small amounts of chlorite are present, apparently pseudomorphous after biotite.
Specimen Silv-66:

The rock from the northwest corner of the Nelson Batholith is a chloritized hornblende quartz monzonite, medium-grained, containing about 30% mafic constituents. Rare phenocrysts exist up to 1 cm. in maximum dimension. Trace amounts of pyrite can be seen in the hand specimen. The specimen locality is on the eastern edge of a major fracture zone that forms the western contact of the northern part of the Nelson Batholith. Extensive chloritization is recognized along this zone and south of the sample locality quartz-microcline pegmatites have been introduced locally within the zone. The rock has undergone considerable cataclasis—about 20 percent of the specimen is very fine-grained cataclastic material that has been recrystallized. A mode estimated from thin section is as follows: plagioclase-An36 (30%), K-feldspar-microcline (30%), hornblende (20%), quartz (15%), chlorite (3%), sphene (1%) and trace amounts of epidote, apatite and opaque minerals. Alteration is extensive in that biotite has been completely replaced by pseudomorphs of chlorite. No other effects of chemical alterations are apparent. Hornblende is undeformed and fresh in appearance.

Specimen R-11:

The specimen is a medium-grained, melanocratic granodiorite with subparallel alignment of mafic minerals and tabular feldspar crystals. It contains rare phenocrysts of K-feldspar up to 1.5 cm long. A mode estimated from thin section is: plagioclase (50%), K-feldspar (15%), hornblende (15%), biotite (10%), quartz (10%),
and trace amounts of epidote, sphene, apatite, myrmekite, chlorite and opaque minerals. Alteration effects are slight with only trace amounts of chlorite occurring as interleaves with some biotite crystals.

Specimen R-12:

The rock is a medium - to coarse-grained melanocratic quartz diorite with vague foliation of mafic minerals (biotite and hornblende). The specimen is fresh in appearance. A relatively uncommon feature compared with most Nelson plutonic rocks is the absence of K-feldspar phenocrysts. A mode determined from thin section is: plagioclase (60%), clinopyroxene (12%), biotite (10%), quartz (10%), K-feldspar (5%), apatite (2%), hornblende (1%), and trace amounts of sphene. The rock is generally fresh in appearance. A slight dust-like, dark alteration of plagioclase is distributed sporadically but is not extensive.

Specimen R-13:

The rock, a medium-grained granodiorite from the Nelson Batholith, has rare K-feldspar phenocrysts up to 2 cm. long. A faint foliation is evident due to alignment of mafic minerals and tabular K-feldspar phenocrysts. Mafic minerals (biotite and hornblende) account for about 20% of rock volume. The specimen has an overall grey caste and appears fresh. A mode estimated from thin section examination is: plagioclase-An25 (40%), quartz (25%), biotite (15%), K-feldspar (12%), hornblende (5%), epidote (2%) and trace amounts of chlorite, clinopyroxene, myrmekite, sphene, apatite, sericite and opaque minerals. The only evidence of alteration is limited formation of sericite and epidote in cores of some plagioclase grains, and some interleaves of chlorite in biotite crystals.
Specimen L-236:

The specimen is a fine-grained, grey hornfels taken from an inclusion within Nelson plutonic rocks. Original character of the rock is uncertain although it may represent metamorphosed and metasomatized rock of the Slocan Series. A vague subparallel alignment of biotite crystals can be seen in thin section. Examination of a thin section revealed the following mode: plagioclase (64%), biotite (20%), quartz (10%), K-feldspar-microcline (2%), epidote (2%), apatite (2%), and trace amounts of sphene, sericite and opaque minerals. The specimen is fairly fresh in appearance although a significant proportion (about 15%) of the biotite has ranged edges and appears almost opaque along some cleavage planes suggesting that it is in the initial stages of alteration.

Specimen L-257:

The specimen is a quartz diorite porphyry from a dyke-like intrusion in rocks of the Slocan Series and is considered a "satellite" of the Nelson batholith to the south. It consists of about 85% medium - to coarse-grained phenocrysts in a fine grained matrix. Plagioclase is the only identifiable felsic mineral in hand specimen. Scattered anhedral blebs of pyrite can be seen and the rock effervesces on treatment with dilute HCl indicating the presence of calcite. About 5% mafic minerals can be seen, only rarely identifiable as biotite. A mode determined from thin section investigation is plagioclase-An$_2$g (67%), quartz (20%), biotite (3%), calcite (3%), chlorite (2%), muscovite and sericite (2%), apatite (1%), K-feldspar-microcline (1%) and trace amounts of sphene, horn-
blende, epidote and opaque minerals. The rock has been extensively altered with chlorite having formed partial and complete pseudomorphs after biotite, and plagioclase having been altered extensively to calcite, epidote and sericite.