THE APPLICABILITY OF THE $^{40}Ar/^{39}Ar$

STEPWISE OUTGASSING METHOD TO THE

DATING OF SLATES

by

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Abstract

Four samples ranging in composition from a pure slate to a pure meta-siltstone, collected from the same location in the Lower Paleozoic Meguma Group, are age dated by the $^{40}Ar/^{39}Ar$ stepwise outgassing technique. The discordant age spectra record two ages, interpreted as the age of regional metamorphism and a younger thermal metamorphism. The inherited argon recording the regional metamorphic event is released in the high temperature steps. Partial outgassing caused by mild thermal metamorphism, unaccompanied by mineralogical modifications, occurs from the less retentive muscovite flakes. These muscovite flakes may be in phyllosilicate rich laminations; they may have easy access to cleavage fractures or they may be of smaller size. Because the majority of the muscovite in the slate has these characteristics, the larger proportion of the gas released upon heating gives the younger age. This effect of partial outgassing could not have been realized from a conventional K-Ar age determination. It is not known whether the effect is widespread, or occurs infrequently under rare conditions. In order to obtain the apparent age of regional metamorphism, it is desirable to find rocks exhibiting no partial outgassing at all. Since metasiltstones are less easily outgassed, these samples may be better suited for dating regional metamorphism in areas of known intrusive activity of a younger age. However, their small muscovite content may produce less reliable ages.

CHAPTER 1

INTRODUCTION

1.1 General Statement

The age of regional metamorphism and deformation is of critical importance in determining the geologic history of an area. K-Ar dating of slates and phyllites from orogenic belts may provide close approximations to these ages. The $^{40}Ar/^{39}Ar$ stepwise outgassing variation of the K-Ar dating method has the power to test certain assumptions that are inherent in the conventional K-Ar method. This study will attempt to test the applicability of the $^{40}Ar/^{39}Ar$ method on slates by dating samples of different compositions and textures. The samples chosen for the study are from the Lower Paleozoic Meguma Group of Nova Scotia. The samples are all from the same location, and therefore should give the same ages. Discrepancies should be explainable by differences in composition and texture. Knowledge of the effects of rock type on the determined apparent metamorphic age will hopefully give more confidence in and acceptance of such data.

1.2 Previous Work

The only known published work on $^{40}Ar/^{39}Ar$ dating of slates is that of Reynolds and Muecke (1978). They reported results of eight $^{40}Ar/^{39}Ar$ stepwise outgassing experiments, along with conventional K-Ar measurements, for five slates, a siltstone, a cordierite slate and a hornfels. The

samples were also from the Meguma Group of Nova Scotia. The aim of this investigation was to date the regional metamorphism of southern Nova Scotia, and to see if the $^{40}Ar/^{39}Ar$ method of dating can detect certain violations of the assumptions made in conventional K-Ar dating. In conventional K-Ar dating, the date of the regional metamorphic event is obtained only if (1) the argon system became closed at a time shortly after the event, (2) closed-system conditions prevailed subsequently, and (3) all pre-existing radiogenic argon had been expelled from the system. It was intended to determine whether or not the method can detect violations of assumptions (2) and (3) above.

The data from this former study is shown in Table 1. The total gas $^{40}Ar/^{39}Ar$ ages compare well with the conventional K-Ar ages, thus indicating no significant loss of ^{39}Ar due to recoil (see section on the $^{40}Ar/^{39}Ar$ dating method). Slates collected at significant distances from the contact with a younger granitic intrusive give well-defined age plateaus, which are interpreted to record the regional metamorphic event (minimum age ~ 400-415 Ma). Two other slates which show obvious thermal metamorphic mineralogical changes resulting from the nearby intrusive, and one slate with no mineralogical changes but located close to the contact, also give well-defined age plateaus, but with ages ranging from 362 to 387 Ma. These are very close to values determined for the granitic intrusive itself (~ 377 Ma, from revised data of Reynolds et al., 1973). Thus, a well-defined age plateau in conjunction with an absence of mineralogical modifications does not necessarily mean that the closed-system condition has been fulfilled (Reynolds and Muecke, 1978).

Table 1. Data of Reynolds and Muecke, 1978

Sample No.	Description	Age (Ma) $^{40}Ar/^{39}Ar$		K-Ar
		Plateau	Total Gas	
71-16	hornfels, close to intrusion	387	382	372
71-153	slate, close to intrusion	377	374	385
71-146	cordierite slate, close to intrusion	362	361	371
71-139	slate, far from intrusion	400-420	405	397
72-31	slate, far from intrusion	415-425	405	392
72-30	slate, with detrital K-bearing minerals	Low Temp. 400-430 High Temp. ~ 460	396	408
71-140	siltstone, with detrital K-bearing minerals	400-430 ~ 460	425	412

The $^{40}Ar/^{39}Ar$ method apparently cannot detect violations of assumption (2) above. Samples which consist in part of detrital components possibly containing pre-regional metamorphic radiogenic argon reveal themselves through highly disturbed age spectra. Thus violations of assumption (3) above can be detected by the $^{40}Ar/^{39}Ar$ method.

1.3 Purpose of the Present Study

The aim of this investigation was to see what the effects of mineralogical composition and texture are on $^{40}Ar/^{39}Ar$ slate data. In order to do this, samples ranging in composition from a slate to a metasiltstone were collected from the same outcrop. The location was suggested by Dr. G. Muecke and Dr. P. Reynolds, and is within the Halifax Formation of the Meguma Group, Nova Scotia. Collecting all the samples from the same outcrop would ensure that they had all been metamorphosed, whether regionally and/or thermally, at the same time and to the same degree. Thus, any differences in the $^{40}Ar/^{39}Ar$ data should be due to differences in mineralogical composition and/or texture. Hopefully, the study would allow conclusions to be made on the usefulness of the $^{40}Ar/^{39}Ar$ dating technique as applied to low-grade metamorphic rocks.

CHAPTER 2

BACKGROUND TO THE PRESENT STUDY

2.1 Age Studies on Slates

It is widely accepted that radiometric ages determined on metamorphic
rocks and minerals from orogenic belts can give valuable information on
the geologic history of an area. If the metamorphic event is intense
enough to recrystallize the rock, then the rock may be amenable to age
dating. For metamorphosed sedimentary rocks, the complete recrystalliza-
tion of clays can be assumed to have occurred if the basal (001) peaks of
micas on an x-ray diffraction pattern are sharp and symmetrical (Kübler,
1966). The geologic significance of the data in K-Ar dating is dependant
on the assumptions given in Section 1.2.

The ability of a K-bearing mineral to retain argon produced by the
radioactive decay of potassium depends on the temperature as well as its
crystal structure. Studies by Hart (1964) and Everenden and Kistler (1970)
have shown that the trend of argon retentivity in the major K-bearing
minerals is as follows: horblende > plagioclase (undeformed) > biotite >
orthoclase. For micas from the Central Alps, Purdy and Jäger (1976) deter-
mined the following temperatures at which they would no longer retain
argon:

muscovite and phengite	$350 \pm 50°C$
biotite	$300 \pm 50°C$

(5)

Since these are within the range where greenschist facies metamorphism is believed to occur (Fyfe et al., 1958; Miyashiro, 1972), such a degree of regional metamorphism should completely reset the "argon clocks" in rocks containing these minerals. In other words, all the argon previously accumulated should be released. Radiogenic argon would not begin to accumulate again until the intensity of the metamorphism had decreased. Thus, ages determined on metamorphic rocks reflect a time of cooling rather than metamorphism. This concept was first developed in detail by Armstrong (1966), and later applied to the British Caledonides by Harper (1967). Dodson (1979) describes the concept of closure temperature (also called blocking temperature). It is assumed that while the system is near the temperature of cyrstallization, the daughter nuclei diffuses out as fast as it is produced by radioactive decay. As the system cools, some of the daughter product accumulates, some is lost. Eventually the losses are negligible, as the radiogenic daughter product is retained in the crystal lattice. The age equation corresponds to about this time. The closure temperature is defined as the temperature of the system at the time given by its apparent age. A very slow cooling rate will result in a lower closure temperature and a younger apparent age. Thus a date obtained from a metamorphic rock which meets the previously mentioned assumptions, reflects the minimum age of the metamorphic event.

From the above discussion it is clear that potassium-argon ages approaching the true age of metamorphism can only be obtained from materials which recrystallized during metamorphism under conditions which caused complete outgassing of inherent argon, but at temperatures close to that at which subsequent radiogenic argon could accumulate. Dewey et al. (1970)

have found that slate ages tend to be older than K-Ar ages determined on
more intensely metamorphosed rocks from the same terrain, and this differ-
ence has been attributed to the ability of slates to start accumulating
radiogenic argon much sooner than rocks of higher metamorphic grade. This
is because the slates have not attained as high a temperature, thus less
time is required for the temperature to decline back to the argon retention
value. Other authors have also shown this to be the case; see Harper (1964,
1967), Harper and Landis (1967), Dodson and Rex (1971) and Adams et al.
(1974). These authors have also shown that observed K-Ar dates inter-
preted as the minimum age of metamorphism correlate well with other existing
geochronological information.

Adams et al. (1974) gives two other reasons for choosing slates in
order to date the time of regional metamorphic events:

(1) They are free of coarsely crystalline detrital
     materials that might still retain a provenance
     age and their fine-grained nature ensures that
     recrystallization during cleavage formation is
     rapidly completed, thus minimizing the possi-
     bility that "detrital" argon remains.

(2) The slate mineralogy, quartz-sericite-chlorite,
     excludes (a) minerals which have a high radio-
     genic argon retentivity, such as hornblende, and
     might therefore retain a provenance age; (b)
     feldspars that are known to have very low and
     variable argon retentivities; and (c) minerals
     that are known to incorporate substantial amounts

of "excess" argon, i.e. radiogenic argon

incorporated in minerals by processes other

than the in-situ decay of potassium.

One problem in the radiometric dating of slates stems from their

very fine grain size. The separation of the potassium-bearing minerals,

namely muscovite, is very difficult, as opposed to separation of minerals

from gneisses or granites, for example. Consequently, whole rock analyses

are usually performed on slates. The dates obtained by Harper (1964) on

whole rock samples and on mica separates from the same rocks show good

agreement. Thus it seems that whole rock data may be as reliable as data

obtained from mineral separates. However, this is not always the case.

For example, in polymetamorphic terrains radiogenic argon from an earlier

metamorphic event can be liberated from the K-bearing phases during a

later event and subsequently become trapped in fluid inclusions of quartz

and/or be incorporated in other potassium bearing minerals (see Kligfield

et al., 1977). In such cases a K-Ar whole rock age is not useful. In

polymetamorphic terrains one should be sure that fluid inclusions have not

formed in the rocks.

2.2 The $^{40}Ar/^{39}Ar$ Dating Method

The $^{40}Ar/^{39}Ar$ dating technique was first described by Sigurgeirsson

(1962), and was more fully discussed in later reports by Merrihue (1965),

Merrihue and Turner (1966) and Mitchell (1968). The theory and analytical

techniques used in this method are generally similar to those for conven-

tional K-Ar dating, in which the known rate of decay of ^{40}K to ^{40}Ar, and

the measured amounts of these isotopes in a sample are used to calculate its age. The primary difference is that in $^{40}Ar/^{39}Ar$ geochronology the direct chemical analysis of potassium is not required; instead, potassium is measured in the form of ^{39}Ar which is produced from ^{39}K by neutron irradiation. The amount of ^{39}Ar produced within a sample during irradiation may be calculated from the following equation (after Dallmeyer, 1979):

$$^{39}Ar_K = {}^{39}K \; \Delta \; T \int \phi \; (\varepsilon) \; \sigma \; (\varepsilon) \; d\varepsilon \tag{1}$$

Where $\quad {}^{39}K \quad$ = number of atoms of ^{39}K in the sample

$\qquad {}^{39}Ar_K$ = number of atoms of ^{39}Ar produced from

$\qquad\qquad {}^{39}K$ in the irradiation

$\qquad \phi \; (\varepsilon)$ = fast neutron flux at energy ε

$\qquad \sigma \; (\varepsilon)$ = neutron capture cross-section of

$\qquad\qquad {}^{39}K(n, \; p) \quad {}^{39}Ar$ at energy ε

$\qquad T$ = duration of the irradiation

$\qquad$ (The integration is performed over all incident

$\qquad$ neutron energies.)

^{40}Ar is also present in the sample as a result of the spontaneous decay of ^{40}K. In practice, a number of samples of a standard mineral of known age are irradiated along with the unknowns in a single capsule. For a sample with an apparent K-Ar age of t, the amount of radiogenic argon is given by the standard K-Ar dating equattion:

$$^{40}Ar* = {}^{40}K \; \frac{\lambda_e}{\lambda_e + \lambda_\beta} \; (e^{t/\tau} - 1) \tag{2}$$

where ^{40}K = number of atoms of ^{40}K present in the sample

$^{40}Ar*$ = number of atoms of radiogenic ^{40}Ar present

in the sample

λ_e = decay constant of ^{40}K by electron capture

λ_β = decay constant of ^{40}K by β-emission

τ = mean half-life of ^{40}K = 1.885 x 10^9 yr

Combination of equations (1) and (2) gives the $^{40}Ar*/^{39}Ar$ ratio measured for an irradiated sample of age t:

$$\frac{^{40}Ar*}{^{39}Ar_K} = \frac{^{40}K}{^{39}K} \cdot \frac{\lambda}{\lambda_e + \lambda_p} \cdot \frac{1}{\Delta T} \cdot \frac{(e^{t/\tau} - 1)}{\int \phi(\varepsilon)\ \sigma(\varepsilon)\ d\varepsilon} \tag{3}$$

To make computations less cumbersome, a quantity J is defined:

$$J = \frac{^{39}K}{^{40}K} \cdot \frac{\lambda_e + \lambda_\beta}{\lambda_e}\ \Delta T \int \phi(\varepsilon)\ \sigma(\varepsilon)\ d\varepsilon$$

From equation (3),

$$J = \frac{(e^{t/\tau} - 1)}{^{40}Ar*/^{39}Ar_K}$$

Rewriting this expression,

$$t = \tau\ \ln[1 + J(^{40}Ar*/^{39}Ar_K)] \tag{4}$$

The known K-Ar age of the standard is substituted for t in equation (4). The value of J may now be calculated directly from the measurement of the $^{40}Ar*/^{39}Ar_K$ ratio of the irradiated standard. In practice, J varies over the length of the irradiation canister. The range of variation can be established by analysis of several standards, and the appropriate J value is interpolated for each unknown. Once a value of J has been determined for the sample, its age can be calculated by measurement of the $^{40}Ar*/^{39}Ar_K$ ratios.

Besides the reaction $^{39}K(n, p)$ ^{39}Ar, several thousand other nuclear reactions occur as a result of irradiation. Of importance here are those that produce isotopes of argon, in particular ^{36}Ar, ^{39}Ar, and ^{40}Ar. The production of ^{36}Ar will effect the atmospheric correction while the production of ^{39}Ar and ^{40}Ar will affect the calculation of the age directly. The reactions responsible have been described by Turner (1971) and Brereton (1970). There are three sources of interference:

1. ^{40}Ar produced by a $^{40}K(n, p)$ reaction

2. ^{36}Ar produced by a $^{40}Ca(n, n\alpha)$ reaction

3. ^{39}Ar produced by a $^{43}Ca(n, \alpha)$ reaction

Fortunately, ^{37}Ar is also produced during neutron irradiation, by the reactions $^{40}Ca(n, \alpha)$ and $^{39}K(n, nd)$. The measurement of the relative volume of ^{37}Ar liberated from a sample may be used to estimate the extent of interference of these isotopes. The corrections derived by Brereton (1970), have been used in this study.

Following irradiation, a sample may be totally fused and all liberated argon analyzed in a single experiment. The $^{40}Ar/^{39}Ar$ age calculated by Eq. (4) is termed a total fusion age and it is roughly analogous to a conventional K-Ar date (Dallmeyer, 1979). However, the greatest potential of the $^{40}Ar/^{39}Ar$ technique is realized through the incremental (step-wise) heating of irradiated samples. Here, the sample is heated in steps for fixed periods of time (usually about 1 hour). The argon released in each temperature increment can be treated as an unknown sample, from which an apparent age may be calculated from its $^{40}Ar*/^{39}Ar_K$ ratio using Eq. (4).

Thus, a series of apparent ages (each corresponding to a specific temperature) is generated for an individual sample. These incremental ages can be plotted as a function of experimental temperature (commonly expressed as an accumulative percentage of $^{39}Ar_K$ released) giving an "age spectrum". The characteristics of the age spectrum permit evaluation of the behaviour of potassium and argon within rock and mineral samples, and thus provide a more thorough understanding of the geologic significance of geochronological results than can be obtained from conventional K-Ar studies (Dallmeyer, 1979). The theoretical basis and advantages of the $^{40}Ar/^{39}Ar$ incremental heating technique were first described by Merrihue and Turner (1966), and more fully defined by Turner (1968, 1969, 1970a, b), Fitch et al. (1969), Lanphere and Dalrymple (1971), Brereton (1972), Dalrymple and Lanphere (1974), Dallmeyer (1974, 1975a, b, c), and Dallmeyer and Sutter (1976).

If a sample contains no extraneous argon and if it has behaved as a closed system with no addition or loss of potassium and argon since its crystallization (except for the internal accumulation of radiogenic argon), the ratio of $^{40}Ar*$ to neutron-produced $^{39}Ar_K$ should be the same for each incremental gas fraction. Thus the calculated ages for each increment should be mutually similar, and approximate the total gas age defined by the $^{40}Ar*/^{39}Ar_K$ ratio of the sum quantity of all the gas released through the incremental heating experiment. The resultant age spectrum will be essentially a concordant, horizontal straight line. However, if the sample does contain a component of extraneous argon, or the closed system conditions have not been met, then the apparent ages of the various

gas increments may not be the same and the age spectrum will therefore be discordant. Obviously, total-gas $^{40}Ar/^{39}Ar$ and K-Ar ages of samples with discordant age spectra have little geologic significance. However, inferences of geologic history and estimates of original age may often be possible through examination of discordant age spectra. The most promising potential is the ability to recognize thermally overprinted samples which have been partially degassed as a result of geologic re-heating (Dallmeyer, 1979).

An important phenomenon which occurs during the irradiation of samples by fast neutrons is recoil. The recoil distance is defined as the distance an atom travels from its original site in the crystal lattice as a result of radioactive decay or fast neutron-induced reactions (Tetley, 1979). Recoil is a natural consequence of the principle of conservation of momentum in that it requires that the products of radioactive decay and nuclear reactions have kinetic energy. Turner and Cadogen (1974) suggested that the recoil of nucleogenic products may result in changes to the Ar release spectra for minerals with layered strucures, because of the possibly greater range of the nuclides in particular directions in the lattice. Many anomalous age patterns observed in $^{40}Ar/^{39}Ar$ dating of lunar rocks and meteorites have been explained by recoil of Ar out of K-rich unretentive minerals into K-poor retentive minerals (Turner and Cadogen, 1974; Huneke and Smith, 1976; Huneke, 1976). Thus any interpretation of $^{40}Ar/^{39}Ar$ data must take into account any damage to the crystal lattice caused by recoil and the effect of recoil on the age spectra.

2.3 Behaviour of K and Ar in Minerals

(i) Isotopic Fractionation

Dalrymple and Lanphere (1969) cite several studies which show it is clear that although detectable variations in the isotopic composition of potassium are rare in nature, they can occur under special conditions. The present distribution of potassium isotopes, however, suggests that it is probably safe to assume that the isotopic composition of potassium is constant in most, if not all, materials used in potassium-argon dating (Dalrymple and Lanphere, 1969). Because this seems to be true today, it is reasonable to assume that it has been true throughout geologic time.

For argon, only the isotopic composition of the present atmosphere, and of course the individual rock or mineral sample, are needed for the calculations of the rock age. Because of the rapidity of atmospheric mixing, it is reasonable to assume that the atmospheric composition of argon is constant everywhere on the earth at any given time, even though ^{40}Ar probably is being released from the crust of the earth of different rates in different places as the result of decay of ^{40}K.

(ii) Gains and Losses of ^{40}K and ^{40}Ar

One of the fundamental assumptions made in the application of K-Ar dating (or the $^{40}Ar/^{39}Ar$ variation) is that the rock or mineral has been a closed system; that is, there has been no loss or gain of ^{40}K or ^{40}Ar except for that which results from the radioactive decay of ^{40}K. Whether this assumption is valid or not depends on the geologic conditions.

Potassium migration is probably not a serious problem except possibly under certain conditions of metamorphism or hydrothermal alteration, both of which would be expected to produce recognizable changes in the rock. Dalrymple and Lanphere (1969) postulate that the gain or loss of potassium from a mineral lattice probably would lead to at least a temporary disruption of the mineral structure, which, in turn, would result in argon loss. It has been observed that badly altered and weathered biotites often give reasonable potassium-argon ages. Apparently the loss of potassium is a layer-by-layer process that also results in the loss of a porportional amount of argon, such that the age determined would be the same (Dalrymple and Lanphere, 1969).

Extraneous argon in a sample will normally result in anomalously old ages. The excess gas may be incorporated into the mineral by diffusion or occlusion. The most likely explanation is occlusion at the time of mineral formation. Excess ^{40}Ar is often found in those geologic environments where a high gas pressure might be expected to have prevailed during emplacement-kimberlite pipes, high-grade metamorphic terrains, pegmatites, and so forth (Dalrymple and Lanphere, 1969). The amount of excess ^{40}Ar in minerals increases with the degree of "openness" of the mineral lattice. Cyclo-silicates, with their large tunnel-like structures, contain much more excess ^{40}Ar than the very tightly structured pyroxenes. Any ^{40}Ar that is produced within mineral grains by the decay of ^{40}K before the event being dated is called inherited ^{40}Ar (Dalrymple and Lanphere, 1969). This ^{40}Ar may have been generated during the premetamorphic history of the rock and has survived the metamorphic event, or it may be from the incorporation of older contaminating mineral grains into the dated sample.

The effect of argon loss from a rock is a lowering of its apparent age. If the event causing the loss works to completion, then the date obtained reflects the time of this event. The events causing argon loss are often episodic, and may last a relatively long time. Factors causing argon loss from minerals are:

1. inability of lattice to retain argon

2. melting

3. metamorphism

4. weathering and alteration

5. recrystallization in solution

6. physical damage

7. poor laboratory procedures.

The mechanism of transport is by diffusion. This is a process whereby material is transferred from one place to another by random molecular motion. The diffusing material gradually moves from areas of higher concentration to lower concentration. A key parameter in formulas derived to describe the phenomenon is activation energy, that is the energy required to dislodge an atom or molecule from one site to another. Actual crystal grains may vary in size, shape, and composition, and are usually anistropic. Because the argon atoms are too large to diffuse through a perfect lattice, diffusion usually proceeds through imperfections which may be irregularly spaced and have varying activation energies (Mussett, 1969). In laboratory experiments the loss of argon differs from natural loss in three ways: damage may occur in mineral extraction, the argon is lost into a vacuum and not into a rock, and the loss usually takes place at a much

higher temperature over a much shorter time interval.

Evidence collected by Mussett (1969) suggests that, because it can only diffuse through major crystal imperfections, argon may be retained completely within small regions of a crystal which, as a whole, has lost argon. Crystal imperfections include lattice vacancies, boundaries showing lack of continuity or dislocation, disorganized regions of easy diffusion due to plastic deformation, and foreign lattices. The density of imperfections and the diffusion path are temperature dependant.

Mussett (1969) found that in a perfect crystal with the mica structure there are no spaces between atoms large enough to allow an argon atom to pass through without great distortion, and consequently escape is possible only if crystal imperfections are present. For micas, diffusion at high temperatures is due to lattice alteration produced by decomposition. At lower temperatures impurity vacancies may be the mechanism (Mussett, 1969).

CHAPTER 3

THE PRESENT STUDY

3.1 General Geology

The Meguma Group comprises at least seven kilometres of sandy flysch
overlain conformably by perhaps six kilometres of shaly flysch (Schenk,
1970). It has been divided into two formations, the Goldenville and the
Halifax Formations. The Goldenville Formation consits of alternating
layers of sandstone and finer grained beds and is interpreted as a submarine
fan deposit (Harris and Schenk, 1976). The Halifax Formation consists of
slate, siltstone, minor sandstone and Fe-Mn nodules and represents a number
of environments of deposition, including distal turbidite fan, basin plain,
interchannel areas of the inner fan, continental rise and slope and outer
shelf (Harris and Schenk, 1976; Lane, 1976). The Meguma Group is remarkably
lacking in fossils; however, erosional outliers apparently of uppermost ·
Halifax Formation along the Bay of Fundy contain fossils which are earliest
Ordovician or older (Schenk, 1970). Poorly preserved graptolites assign a
probable age of Early Ordovician to what is most likely the Goldenville
Formation (Schenk, 1970). Potassium-argon dates of detrital muscovite
from the Goldenville Formation (Poole, 1970) give an age range of 457 Ma
to 516 Ma (Early Ordovician to Late Cambrian).

The Meguma Group is overlain by the conformable rocks of the White
Rock, Kentville, New Canaan and Torbrook Formations (Keppie, 1977). These
consist of volcanics, siliclastic sediments, and limestones. Fossil assem-

(18)

blages give ages of Late Ordovician (Lane, 1976) to Early Devonian (Boucot, 1960). The contact between the Meguma Group and the directly overlying White Rock Formation has been interpreted by some to be gradational and conformable (Crosby, 1962; Taylor, 1965; Schenk, 1972; Smitheringale, 1973; Lane, 1976). However, there is some evidence for an unconformable relationship (Smitheringale, 1973; Taylor, 1965; Schenk, 1976).

The Meguma Group is intruded by the South Mountain Batholith, which is a large peraluminous, granodiorite-granite complex (Clarke and Halliday, 1979). Rb-Sr isotopic studies give ages ranging from 361 to 372 Ma (Clarke and Halliday, 1979). Conventional K-Ar ages of about 377 Ma were obtained by Reynolds et al. (1973) using revised constants.

In addition to the localized thermal metamorphism caused by the above intrusion, the Meguma Group has undergone regional metamorphism. Both types of metamorphism have resulted in recrystallization and locally in orientation of newly formed minerals (Taylor and Schiller, 1966). Metasomatism and retrograde metamorphism are subordinate and only locally important. Regionally metamorphosed rocks can be divided into greenschist and almandine amphibolite facies. Rocks chosen for this study occur in the greenschist facies with quartz-muscovite-chlorite-albite-epidote assemblages.

According to Fyson (1966), at least three generations of folds and faults affect the Meguma Group and the overlying Lower Paleozoic formations. The main folds are upright and low plunging and trend northeast to east closely parallel to the strike of a steep axial-plane foliation. The granitic intrusive intersects these major folds with little disruption

KEY:

GABBROIC DYKE
UP. TRIASSIC/LOW. JURASSIC(?)

T UNDIFFERENTIATED TRIASSIC
SEDIMENTS & VOLCANICS

C UNDIFFERENTIATED CARBONIF-
EROUS SEDIMENTS

D DEVONIAN GRANITIC ROCKS

S-D UNDIFFERENTIATED SILURAN/
DEVONIAN SEDS & VOLCS.

ƐO CAMBRO-ORDOVICIAN MEGUMA
GROUP (H-HALIFAX FM., G -
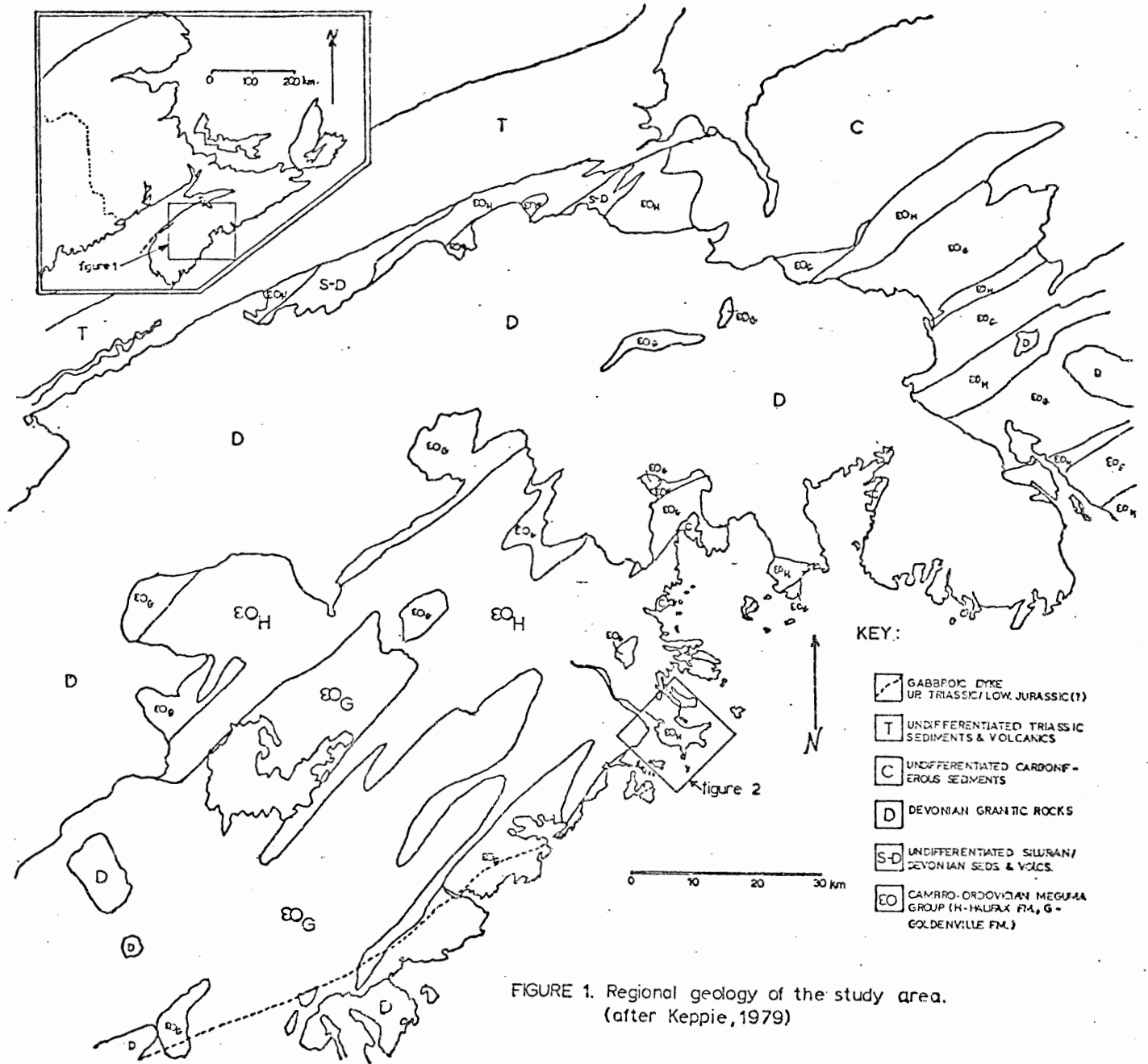GOLDENVILLE FM.)

FIGURE 1. Regional geology of the study area.
(after Keppie, 1979)

of the trends, but deformation is not present in the intrusive itself. A younger, less pervasive, set of small-scale cross-folds, which deform the bedding and axial-plane cleavage associated with the previous folding, affect the Halifax slates. In metamorphic aureoles of the granitic intrusive, quartz and feldspar grains and andalusite porphyroblasts cross the small-scale folds, an indication that the intrusive is younger (Fyson, 1966). A small-scale set of open S-shaped kinks with steep axial planes were emplaced after the granites. Fyson also postulates the existence of an even later set of kinks. Keppie (1977) believes an earlier set of folds, with an axial plane foliation, deformed the Meguma Group even before the main generation of folds described by Fyson (1966). These recombent folds are disrupted by the main folding. Supposedly they are also observed in the White Rock Formation at Yarmouth and Cape St. Mary, but not in other areas of the White Rock Formation, or in any of the other overlying formations.

The generalized geology of the area is shown in Figure 1. The geology of the study area is shown in Figure 2.

3.2 Sample Locations and Preparation

All the samples were collected from a well exposed outcrop of the Halifax formation along the shore of King's Bay, approximately 13 kilometres south of Lunenburg, Nova Scotia (Fig. 2). Samples KB-1, KB-2 and KB-3 were taken within an area of about 50 centimetres, and represent a gradation from a slate to a cross-laminated metasiltstone (Fig. 3). Sample KB-6 is a quartz-rich, phyllosilicate-poor metasiltstone collected about 100 metres away, and on strike from the above suite. A petrographic description of the
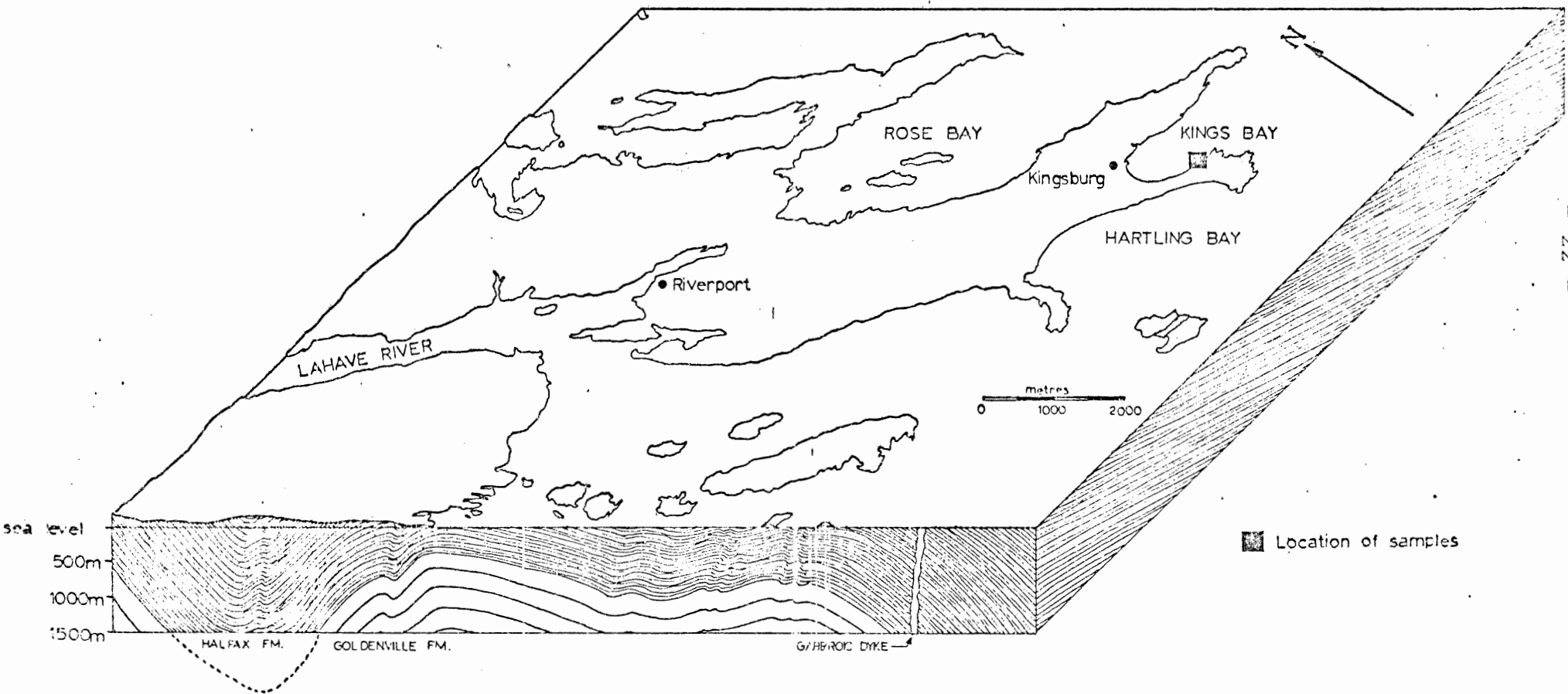
FIGURE 2. Local geology and sample location. (Geology after Faribault, 1929)

samples is given in Appendix 1, and Appendix 2 gives their semiquantitative mineralogical analyses.

Fresh samples were obtained by using a portable hand-drill. The drilling was done parallel to the plane of bedding. Approximately 30 centimetres of 2.5 centimetre diameter core was taken for each sample. Prior to crushing, the samples were sanded down with emery cloth and rinsed with water in order to remove any contamination from the drill bit. Samples were then crushed in a mechanical jaw crusher which had been thoroughly cleaned before and between samples. Pieces of the sample showing no iron-staining and no weathered surfaces were hand-picked. These "clean" pieces were further reduced in a stainless-steel shatter box, and the pulverized sample sieved to obtain the -45 to +100 mesh fraction, the finer fractions having been discarded in order to reduce the atmospheric argon contribution and to avoid possible loss of radiogenic argon (Keeling and Naughton, 1974). In order to remove any metallic contamination from the crushing process, the samples were scanned with a hand magnet. Samples were then split, one-half to be used for x-ray analysis, the other half for dating. The latter half was immersed in tetrabromoethane (S.G. 2.96) in order to remove iron sulphides and thus avoid unnecessary activities in the irradiated samples. These were then thoroughly washed in acetone and distilled water.

Schematic diagram of site
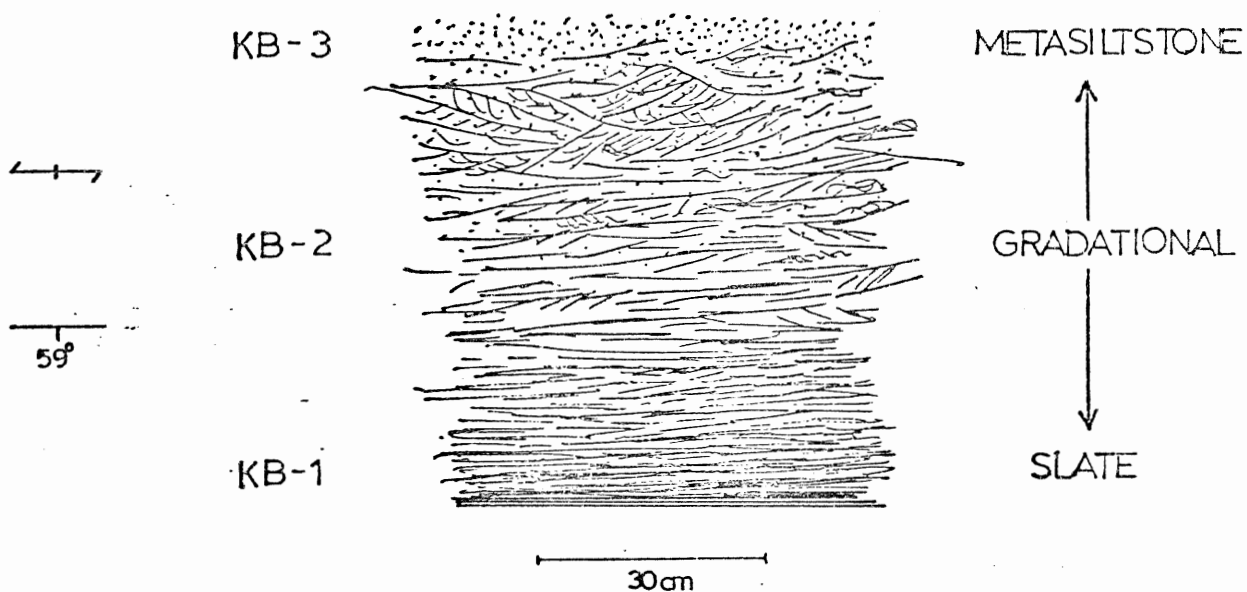chosen for drilling

KB-3 METASILTSTONE

KB-2 GRADATIONAL

KB-1 SLATE

30cm

59°

FIGURE 3. Sample locale detail.

3.3 Analytical Techniques

(i) Mineralogical Analysis

Thin-sections were cut perpendicular to the bedding plane. Petrographic analysis showed the presence of quartz, muscovite, chlorite, magnetite, apatite and tourmaline in all the samples, and the preferred orientation of the phyllosilicates parallel to cleavage and bedding. Some of the samples contained pyrite, pyrrhotite, graphite, albite and epidote. Sample descriptions are given in Appendix 1.

X-ray analyses were carried out on all the samples. The scans showed the presence of quartz, muscovite and chlorite in all the samples; two scans showed albite. A semiquantitative technique was used to determine the abundances of these minerals in the samples, a technique that was successfully applied by Liew (1979) on rocks of the Meguma Group. An explanation of the technique and the results are given in Appendix 2.

Complete recrystallization of clays is indicated by the sharp and symmetrical muscovite peaks on the x-ray scans.

(ii) Argon Analysis

An approximately 100-mg split of the fraction treated specifically for dating was used. The samples were individually wrapped in aluminum foil and arranged within the irradiation capsule together with samples of a biotite standard, the flux monitor. This sample, No. 71-231, has an age (obtained by the $^{40}Ar/^{39}Ar$ method) of 368 ± 8 Ma. The irradiation was carried out in the McMaster University nuclear reactor.

The irradiated samples were outgassed in a stepwise manner in a total of eight temperature steps (except for KB-2, which was not totally outgassed because of a leak in the extraction system after the sixth step). The previous work of Reynolds and Muecke (1978) on the $^{40}Ar/^{39}Ar$ dating of the Meguma slates was used to determine the ideal temeprature step scheme, such that the narrowest temperature steps would correspond to the steps at which the majority of outgassing occurred. This procedure would hope-- fully result in a more accurate plateau age. The sample is placed in a tantalum crucible which is in turn placed in a tantalum coil extraction furnace which is connected 'on-line' to a mass spectrometer. A 'cold finger' (a glass tube containing activated charcoal, immersed in liquid nitrogen) is attached to the furnace in order to remove the gases given off by the sample. The majority of the chemically reactive gases are removed by the use of a titanium getter. The purified argon is then analyzed by a somewhat modified AEI MS10 mass spectrometer. A more detailed account of the heating and cleaning procedure can be found in Parrott (1976), Chapter IV.

Mass spectrometrically produced peak heights corresponding to masses 40, 39, 36 (and 37 when significant) were measured in order to obtain the ratios $^{40}Ar/^{39}Ar$, $^{36}Ar/^{39}Ar$ (and $^{37}Ar/^{39}Ar$ when significant) for the age calculations. The precision of measurement of these ratios was usually better than 1%; uncertainties in mass spectrometer discrimination and fractionation corrections give rise to a probable error of ±1% (Parrott, 1976).

One of the problems encountered with mass spectrometers operated in the static mode is the "memory effect" - that is, contamination of the gas sample being analyzed by residues from previous samples. The memory effect is apparently caused by exchange with ions from previous samples that had been driven into metal or glass by the high-energy ion beam (Reynolds, 1956). The result is that the argon isotope ratios tend to change with time during the measurement period. The correct argon isotope ratios can be obtained by plotting the measured values against time and extrapolating to zero time (Dalrymple and Lanphere, 1969).

3.4 Results

The analytical results of the experiments are shown in Table 2. The corresponding age spectrum plots of apparent age versus % ^{39}Ar released are shown in Figures 4 to 7. (Note that due to a leak in the argon extraction system, the outgassing of sample KB-2 was terminated at 900°C.)

3.5 Discussion

(i) On the Interpretation of the Age Spectra

It is clear from the examination of the age spectrum plots that about the first 5% to 10% of gas released gives relatively low apparent ages. According to Reynolds and Muecke (1978) this lowering can be attributed to the diffusive loss of radiogenic ^{40}Ar from the least retentive potassium sites (for example, from lattice imperfections, crystal surfaces, cracks, non-lattice sites, etc.) say according to the models of Turner (1968) and others. Alternatively they suggest that perhaps it may indicate a

Table 2. Analytical Results of Stepwise
Outgassing Experiments

Sample (J-value)[a]	Temperature (°C)	^{39}Ar released (%)	^{40}Ar/^{36}Ar[b] (units of 10^3)	^{39}Ar/^{36}Ar[b]	Atmospheric argon correction (%)	Apparent age (Ma ± 2σ)[c]
KB-1 Slate (0.003798)	200	-	-	-.	-	-
	500	0.66	0.714	7.9	41.4	332 ± 51
	600	6.7	2.10	33.8	14.1	333 ± 3
	675	6.9	8.27	132.3	3.6	372 ± 30
	750	12.0	12.83	201.3	2.3	383 ± 1
	825	25.1	10.28	162.0	2.9	379 ± 2
	900	16.4	12.29	191.6	2.4	385 ± 3
	975	15.6	4.73	67.4	6.2	402 ± 1
	1100	16.7	2.76	38.6	10.7	392 ± 2
	Total gas					382 ± 7[d]
KB-2 Laminated slate (0.003752)	200	-	-	-	-	-
	500	2.3	0.404	2.7	73.0	255 ± 33
	600	10.1	1.23	18.1	24.1	319 ± 7
	675	14.1	3.75	55.7	7.9	377 ± 4
	750	15.0	4.79	72.89	6.2	376 ± 3
	825	32.3	5.06	76.7	5.8	378 ± 1
	900	25.9	6.01	93.4	4.9	372 ± 8

[a]Irradiation parameter J = $(e^{\lambda t}s - 1)/(^{40}Ar*/^{39}Ar)s$ where s = standard.

[b]Corrected for interfering isotope effects.

Table 2. Continued

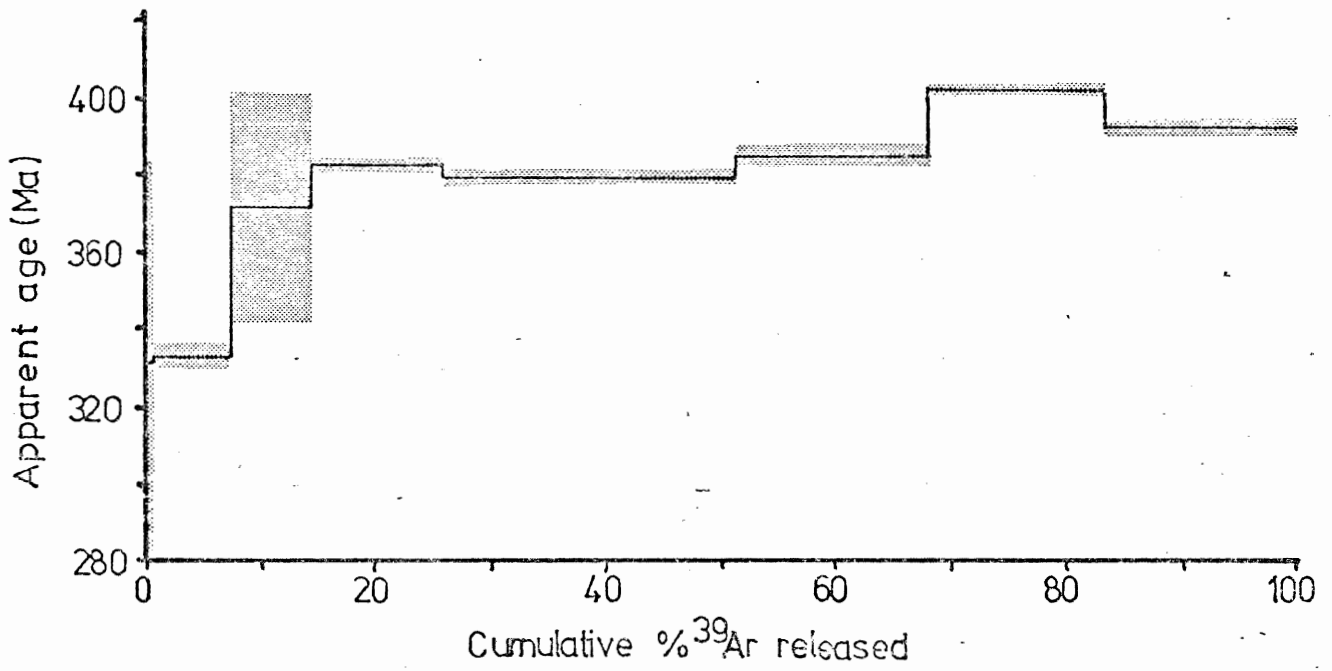| Sample (J-value)[a]	Temperature (°C)	^{39}Ar released (%)	^{40}Ar/^{36}Ar[b] (units of 10^3)	^{39}Ar/^{36}Ar[b]	Atmospheric argon correction (%)	Apparent age (Ma ± 2σ)[c]
KB-3 Laminated metasiltstone (0.003712)	200	–	–	–	–	–
	500	1.0	0.385	2.4	76.8	231 ± 13
	600	4.6	1.02	14.4	28.9	310 ± 5
	675	8.3	3.35	50.0	8.8	369 ± 5
	750	7.7	5.44	81.9	5.4	378 ± 5
	825	19.8	6.50	98.9	4.5	378 ± 2
	900	22.0	4.60	66.7	6.4	387 ± 1
	975	18.0	2.07	25.5	14.3	414 ± 2
	1100	18.6	1.56	18.6	19.0	405 ± 2
	Total gas					386 ± 6[d]
KB-6 Metasiltstone (0.003786)	200	–	–	–	–	–
	500	1.0	0.356	0.76	83.0	474 ± 166
	600	4.4	0.698	7.4	42.4	335 ± 9
	675	8.1	2.06	27.1	14.4	397 ± 21
	750	30.3	1.90	25.7	15.5	383 ± 2
	825	12.0	2.26	29.7	13.1	403 ± 17
	900	9.2	1.48	18.1	20.0	398 ± 4
	975	13.7	0.844	8.1	35.0	413 ± 10
	1100	21.2	0.687	6.0	43.0	397 ± 2
	Total gas					394 ± 11[d]

FIG. 4. Apparent age as a function of cumulative % ^{39}Ar released for sample KB-1. Error bars indicate the relative (between-steps) uncertainty at 2σ.
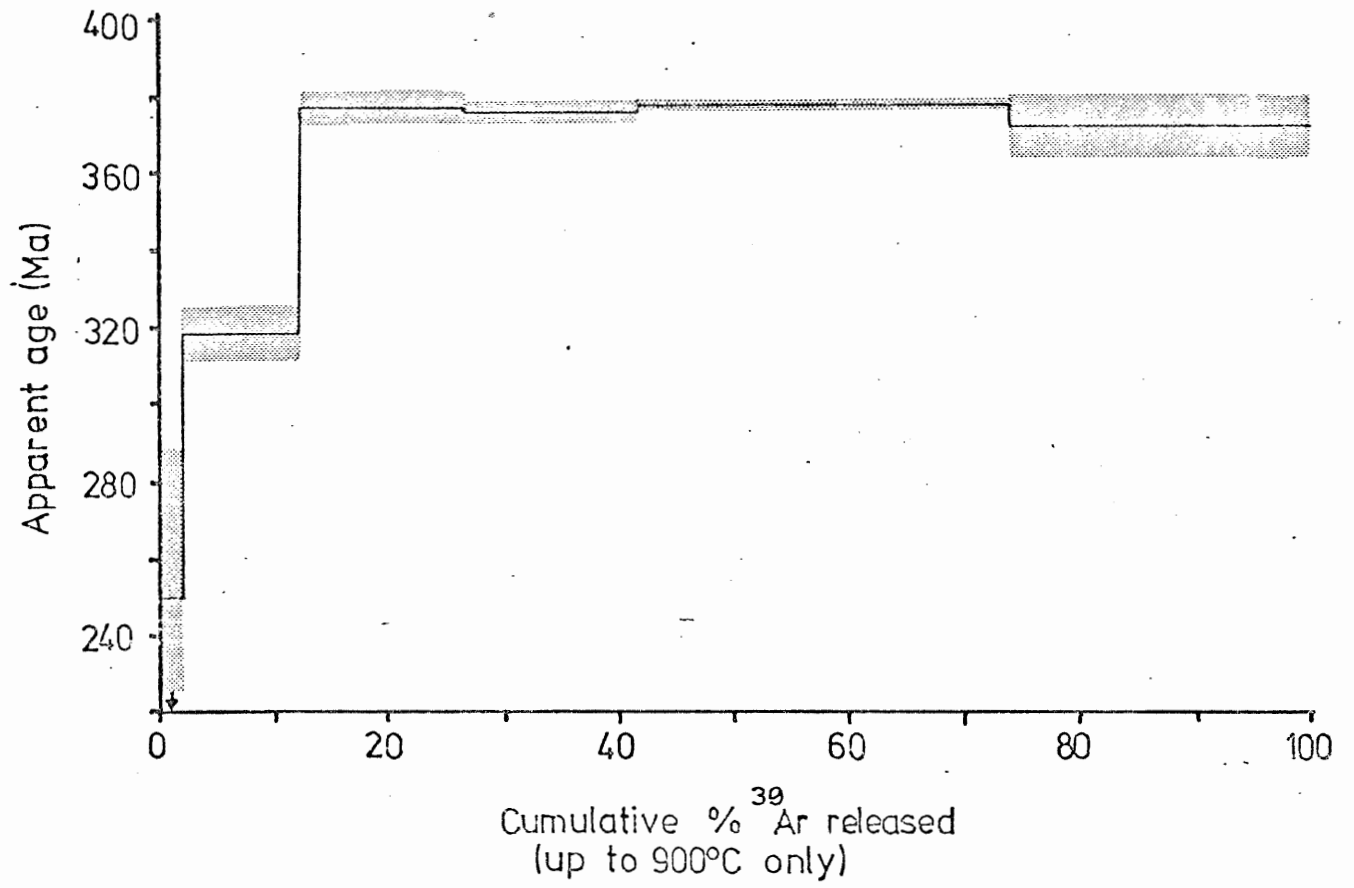
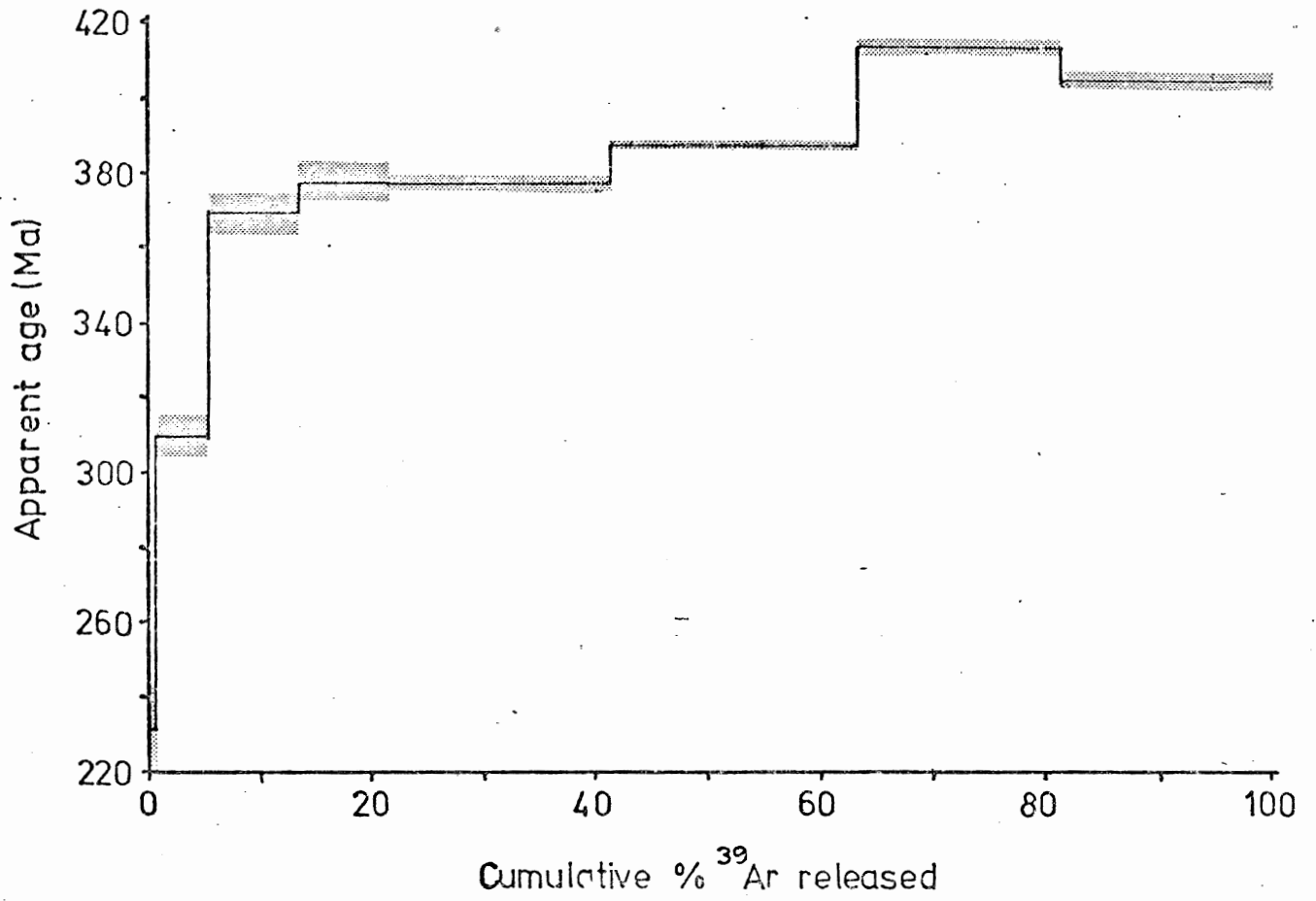FIG. 5. Apparent age spectra (as in Fig. 4) for sample KB-2.

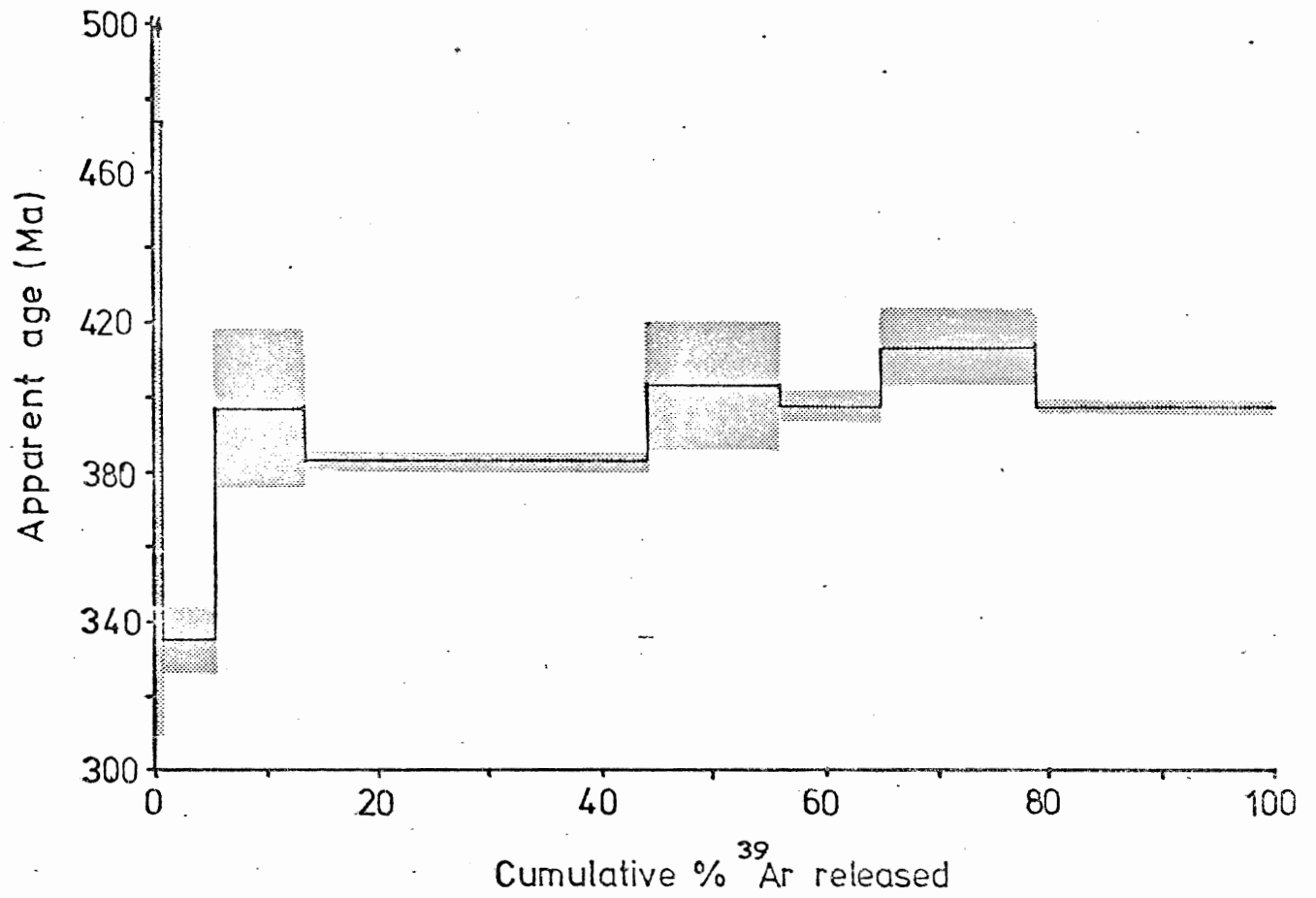FIG. 6. Apparent age spectra (as in Fig. 4) for sample KB-3.

FIG. 7. Apparent age spectra (as in Fig. 4) for sample KB-6.

closed-system irradiation-induced redistribution of argon isotopes. Examination of the age spectra for the remaining gas released reveals discordance, except for sample KB-2, which shows data only for temperatures up to 900°C. Possible explanations for the discordant spectra are:

1. errors in experimental procedure or sample contamination

2. recoil effects

3. excess argon occluded during any recrystallization

4. inherited argon due to K-bearing detrital minerals

5. inherited argon due to incomplete recrystallization of clays

6. loss of argon due to weathering and alteration

7. inherited argon due to incomplete outgassing by a later metamorphic event.

The general similarity of the results obtained for the four samples along with the close agreement between the present results and those obtained by Reynolds and Muecke (1978) indicate that (1) above does not apply. Reynolds and Muecke also concluded that significant loss of argon due to recoil did not occur under their (and the present) experimental conditions. Their evidence for this was the consistency observed between total gas $^{40}Ar/^{39}Ar$ ages and conventional K-Ar ages. Absence of hydrothermal activity as evidenced by the lack of quartz veins and fluid inclusions argues against the third explanation above. No detrital K-bearing minerals were evident in the thin section examinations or in the x-ray studies. The x-ray data also indicates complete recrystallization of the

clay minerals. The samples do not exhibit any weathering or associated alterations except for iron oxide staining, which was removed before analysis anyway. The only reasonable explanation which remains is that of inherited argon due to incomplete outgassing by a later metamorphic event.

Closer inspection of the age spectra reveals that generally there are two different ages recorded in the rocks: (i) an age in the range ~ 375-385 Ma for the lower temperature steps, and (ii) ages from about 395-415 Ma for the higher temperature steps. Not surprisingly, the two age ranges correspond to values previously determined by Reynolds and Muecke on the rocks of the Meguma Group. Their study found that slates close to the contact with a younger granitic intrusive reflected ages similar to that of the granite (~ 377 Ma, revised data from Reynolds et al., 1973). Slates at significant distances from the granite recorded ages of about 400-415 Ma which was interpreted as the minimum age of a regional metamorphic event.

It must be noted that the age spectra of the samples of Reynolds and Muecke having no detrital K-bearing phases were concordant and yielded well-defined plateaus. These spectra are shown in Figure 8. The spectra yielding ages similar to the intrusive indicate complete outgassing by the thermal event. The spectra yielding ages interpreted as the minimum age of regional metamorphism indicate no outgassing resulted from the thermal event.

It seems reasonable then to conclude that the samples of the present study have been only partially outgassed by the granitic intrusive. However,
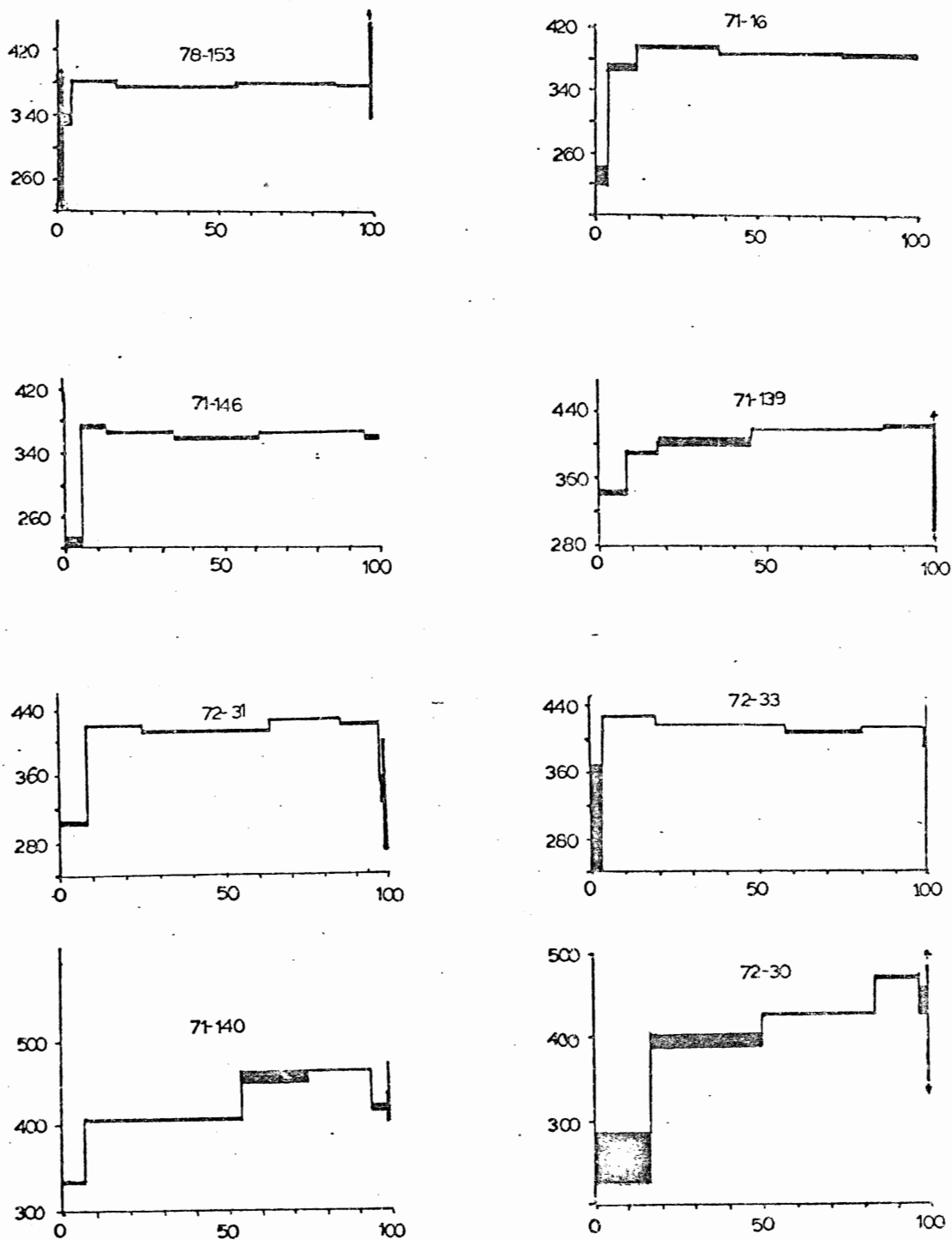
FIGURE 8. Apparent age spectra (as in Fig. 4) of the samples of Reynolds and Muecke (1978). The y-axis is the apparent age in Ma ; the x-axis is the cumulative % of ^{39}Ar released.

the nearest surface outcrop of this granitic rock is approximately 32 km
to the northeast. Also, the samples do not show any mineralogical or
textural characteristics of thermal metamorphism. The first problem can
be explained by the gravity models of Garland (1964) and O'Reilly (1975),
which postulate a sheet-like extension of the granites beneath the Meguma
Group between Shelburne and Mahone Bay, an area which includes the present
sample locality. Presumably, the heating here was just enough to partially
outgas the rocks but not enough to mineralogically or texturally alter
them. Such a presumption probably occurred to sample 71-153 of Reynolds
and Muecke. This sample records an age very close to that of the granite
and was therfore completely outgassed by it; however, no mineralogical or
textural alterations associated with thermal metamorphism are present in
it.

Two questions remain to be answered. Firstly, why should the rocks be
only partially outgassed as opposed to completely outgassed by the intru-
sion? Secondly, is there any difference between the argon retentive
capabilities of a slate versus a siltstone, and if there is, can it be
explained by mineralogical or textural differences?

A possible explanation to the first problem is as follows. There is a
gradual increase in the apparent age with increasing temperature of
release. Generally, one can find two age ranges in the spectra as was
previously mentioned. The younger age for sample KB-1 has been recorded
by the first 70% of the gas, for KB-3 the first 63%, and KB-6 the first
43% (excluding the first 5 to 10% of gas released, which records anomously
low ages for reasons previously given). The remaining gas released in

each sample gives a significantly higher age. Presumably the same effect would be seen in sample KB-2, had it been completely outgassed. For some reason the muscovite, which is the only potassium-bearing mineral in the rock, can be prevented from releasing all or any of its argon during a thermal event. There are several ways this may be explained. It can be seen from the thin sections that the slates are almost entirely composed of laminations of very fine chlorite, quartz, and muscovite, with inter-stitial graphite. The flakes of muscovite usually border and overlap other muscovite flakes. By contrast, the metasiltstone is composed mostly of quartz-rich laminations, with very little chlorite, muscovite and graphite. In these rocks most of the muscovite flakes are isolated from other musco-vite flakes. The muscovite that is within laminations of high muscovite content might be expected to release argon quite easily, since diffusion could easily continue between overlapping muscovite flakes. The argon could also possibly diffuse out of the rock through chlorite or along graphite. A muscovite flake in the quartz-rich laminations may not be able to release its argon until it reached a higher temperature, since its isolation from other muscovite flakes (and chlorite and graphite) would make diffusion possible only through the surrounding quartz grains. It can be seen that these quartz grains exhibit a granoblastic polygonal texture, and the grain contacts are well cemented. Thus, diffusion through such surroundings is probably very slow, until a temperature is reached, at which grain boundary diffusion becomes effective. What may have happened at this particular loca-tion in the Meguma is that the temperature reached by the rock, as a result of the intrusion below, was high enough only to allow the outgassing of the argon from the muscovite within the muscovite-rich laminations. The

temperature needed to allow the release of argon from the muscovite flakes completely surrounded by quartz was not reached. Thus these flakes of muscovite retained their radiogenic argon and therefore record the regional metamorphic event. In the stepwise outgassing experiment, these muscovite flakes with inherited radiogenic argon would not be outgassed until a sufficiently high temperature step. When this step was reached, there presumably would be a sudden release of argon with an anomously high ^{40}Ar content, thus accounting for the significant rise in age. Since the meta-siltstones contain more of the quartz-rich laminations than the slate, it would be expected that a larger portion of gas released from a metasiltstone would give a high apparent age (reflecting the time of regional metamor-phism). The latter effect can be seen in the age spectra. For sample KB-6, a metasiltstone, a high age is given by approximately the last 55% of gas released. Sample KB-3, which contains about one-third less quartz, records a high age for about the last 38% of gas released. The pure slate KB-1 contains even less quartz and only about the last 30% of gas released records a high age.

Another possible explanation for the partial outgassing of the samples relates to their varying phyllosilicate contents. The slates, with a high phyllosilicate content, have a very well-developed cleavage, and the rocks break very easily along these planes. The metasiltstones have very poor cleavage, and fracture only when a large force is applied, the plane of fracture being only roughly parallel to the cleavage of the slates. Thus diffusion in the slates is probably a much easier process. The gentle thermal metamorphism at this location may have resulted in outgassing of

the muscovites most accessible to the cleavage planes. Muscovites in laminations exhibiting little or no cleavage would retain their ^{40}Ar accumulated since the regional metmorphic event, because a higher temeprature would be needed to release it. Thus the samples record two different ages in the outgassing experiments. Since the slates exhibit the most well-developed cleavage, most of the muscovite could probably lose its argon quite easily. Thus for the slates, the majority of the gas released should give a low age, and vice-versa for the metasiltstones. As noted above, this to a first approximation appears to be the case.

The theoretical work of Turner (1967) provides another possible explanation for the observed spectra. Turner constructed a series of theoretical laboratory release patterns for the hypothetical case of a 4500 Ma old mineral concentrate which has a distribution of grain sizes and was partially outgassed 500 Ma ago. The resulting curves show apparent ages which gradually increase with the fraction of ^{39}Ar released. Turner concludes that the initial low age part of the curve arises mainly from the outgassing of small grains while the later high age part arises from the less readily outgassed large grains. Perhaps then the young ages of the slates and metasiltstones of this study result from the easily out-gassed smaller muscovite flakes. There is a small observed increase in the range of sizes of muscovite grains as one goes from a slate to a meta-siltstone (from about 0.01-0.05 mm to about 0.02-0.08 mm in length). This may account for the larger portion of the gas of the metasiltstones giving an older apparent age.

It is probable that the above three possible explanations for the partial outgassing of the samples and the resulting discordant spectra all operate together to some degree and all contribute to the observed data.

In the above discussion it has been assumed that the partial outgassing was the result of gentle thermal metamorphism from the intrusive at depth. However, other reasons may be envisaged. For example, it can be seen that samples 72-31 and 72-33 of Reynolds and Muecke (1978) give well-defined plateaus (Fig. 8) which record the oldest ages measured. Therefore they are assumed to record the most likely age of regional metamorphism. The locations of these samples are about 80 km north-northeast of the present study area. Sample 71-139 of the former study, located about 18 km north-northeast of the present study area, has an age spectra similar to those of the present study. That is, the apparent age gradually increases with higher temperature steps. However, the low temperature ages are not quite as young. A comparison of the sample locations and age spectra to a metamorphic map of Nova Scotia (N.S. Dept. of Mines, 1979, compiled by G.K. Muecke and J.D. Keppie) suggests that the amount of partial outgassing increases with increasing metamorphic grade, as indicated by the closeness to the next highest metamorphic zone. This may be explained by assuming that the rate of cooling was slower in areas metamorphosed to higher degrees. In these areas argon would begin to accumulate at a later time, depending on the difference in cooling rates. The rocks would thus record a younger age. The high temperature steps may give older ages because of the presence of both weak and strong argon retentive sites in

the slates, as previously discussed.

From previous discussions it is apparent that the question of whether there is any difference between the argon retentive capabilities of a slate versus a siltstone has been answered. Due to their finer grain size, higher phyllosilicate content, and excellent cleavage, slates are not as argon-retentive as siltstones. Partial outgassing may occur as a result of a gentle episode of thermal metamorphism, or from the effects of cooling rates. A slate has a larger number of less retentive sites than a siltstone, and thus a greater proportion of the gas released gives an apparently younger age. This is readily apparent from examination of the age spectra. However, had conventional K-Ar age determinations been done, this conclusion could not have been made. It can be assumed that the total gas $^{40}Ar/^{39}Ar$ would be similar to the K-Ar age had the latter been measured (see Reynolds and Muecke, 1978). The total gas ages for the present samples are: KB-1, 382 Ma; KB-3, 386 Ma; KB-6, 394 Ma. Although differences are slight, there is an increase in age with decreasing "slatey" characteristics.

Another feature of the stepwise outgassing method which can be studied is the release pattern. This shows the percent ^{39}Ar released per step as a function of temperature. Figure 9 shows the release patterns for the rocks of the present study and the study of Reynolds and Muecke. For the rocks of the previous study, most of the gas is released in one of two temperature steps. Samples yielding the older age supposedly reflecting the regional metamorphism outgas mostly between 600°C and 700°C. The samples containing detrital K-bearing phases also outgas mostly between
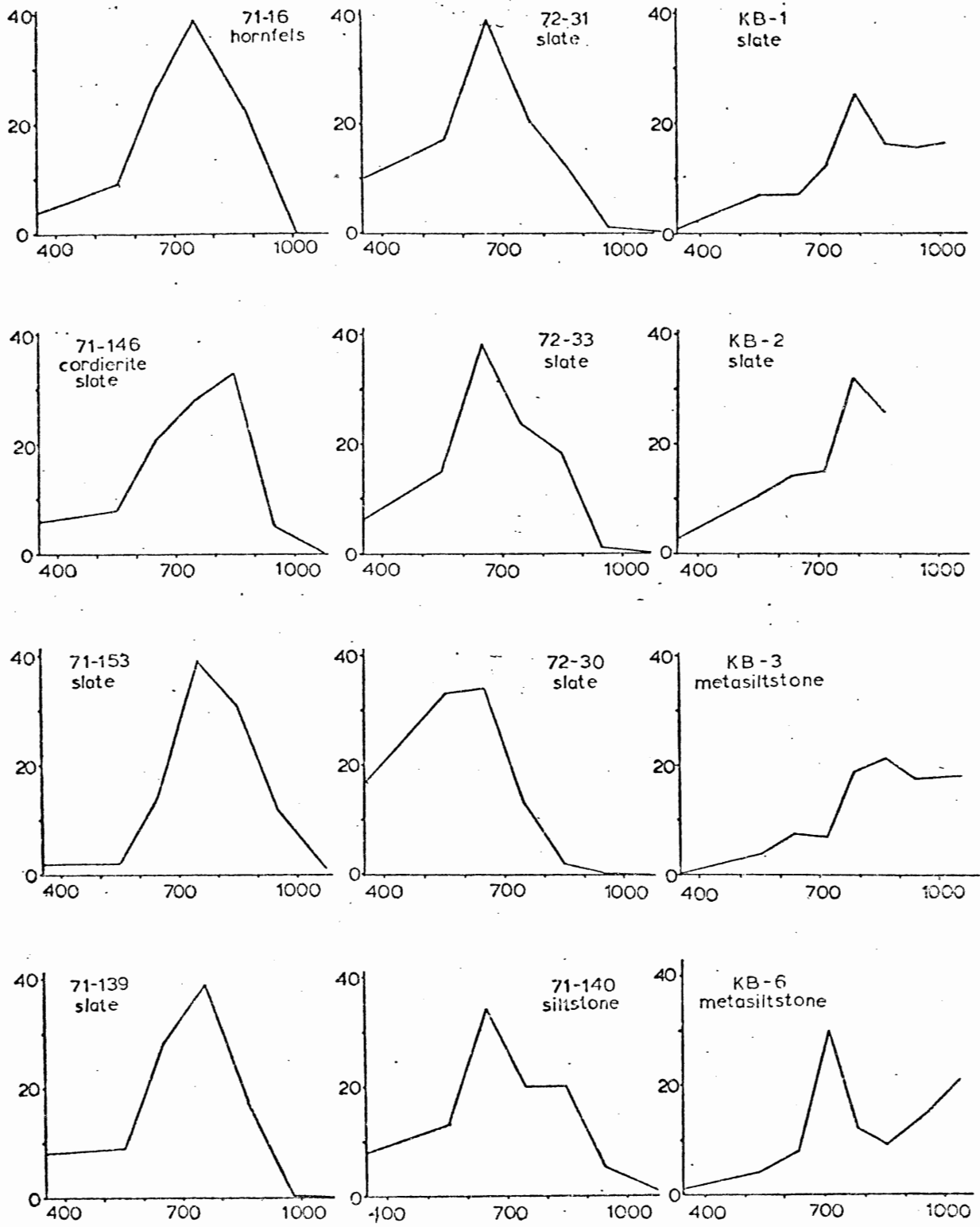
FIGURE 9. ^{39}Ar release patterns for samples of Reynolds and Muecke (1978), and the present study. The % ^{39}Ar released per step is plotted on the y-axis; the x-axis is temperature in °C.

600°C and 700°C. Perhaps the thermal metamorphism that the latter samples were subjected to resulted in certain mineralogical or textural modifications, such as increase in grain size, that would make the rock as a whole more argon-retentive. However, sample 71-153, a slate, shows no visual modifications, and still releases most of its gas during the higher temperature range. It can also be seen that sample 71-139, which has an older age, does not release the majority of its gas during the lower temperature step, but during the 700°C to 800°C step. As noted previously, this sample may have been partially outgassed to a small degree. It is possible then that some unseen modification has increased the argon retentiveness of this rock.

Samples KB-1, KB-2 and KB-3 of the present study yield most of their gas in the 750°C to 900°C range. This pattern is similar to that for the thermally metamorphosed rocks of the previous study. It suggests that the rocks of this study have also been thermally metamorphosed to some degree. The release pattern of KB-6, the quartz-rich metasiltstone, is quite different. There are two peaks at which the majority of outgassing occurs: (i) between 675°C and 750°C, and (ii) beteen 975°C and 1150°C. There is also a slight peak between 975°C and 1150°C for KB-3. It is probable that the first peak corresponds to outgassing by the least argon-retentive sites; the second peak then corresponds to the more argon-retentive sites. The effect is noticeable only in rocks with abundances of both types of argon sites.

(ii)   On the Applicability of the $^{40}Ar/^{39}Ar$ Technique to Slates

Reynolds and Muecke (1978) concluded that a well-defined age plateau
in conjunction with an absence of mineralogical modifications does not
necessarily mean that the closed-system condition has been fulfilled.
From the present study it is evident that violations of the closed-system
assumption can be revealed by discordant spectra in the absence of mineral-
ogical modifications. Only when the system has been partially outgassed
can the $^{40}Ar/^{39}Ar$ method detect violations of the closed-system assumption.
Whether or not this partial outgassing, as displayed by the rocks of this
study, is a widespread phenomenon is not known. In any case, when dating
a regional metamorphic event with slates using the $^{40}Ar/^{39}Ar$ method, it
should be known whether or not intrusive activity has occurred in the
area, and what age the intrusive event is. A sample completely outgassed
by the thermal event will give an age very close to that of the intrusive,
as shown by Reynolds and muecke (1978). A partially outgassed sample will
show a discordant age spectra, the apparent ages for each step increasing
with increasing temperature. It is possible that both the age of the
intrusive and the regional metamorphic event could be recorded by the same
rock, provided the two ages are relatively close, and the rock has the
capability of retaining argon at higher temperatures in certain sites.

It may also be possible for slates in metamorphic terrains with
differing cooling rates to show discordant spectra.

In any case, the application of the $^{40}Ar/^{39}Ar$ method to dating meta-
morphic events of slates yields more information and can thus reveal certain

errors which would have been impossible to detect using the conventional

K-Ar method.

CHAPTER 4

CONCLUSIONS

The sample location was originally chosen with the hope that the rocks in the area would not show any evidence of thermal metamorphism. However, the data has shown that the rocks may have been subject to mild thermal metamorphism, enough to partially outgas the samples.

Due to their very fine grain size, high phyllosilicate content and excellent cleavage, slates are more easily outgassed than siltstones. This effect is only noticeable if the rock has been very mildly metamorphosed. Alternatively, the same effect may be produced if the rock is subjected to a very slow rate of cooling.

Regional metamorphism of greenschist facies grade (chlorite zone) and thermal metamorphism will completely outgas both a slate or a siltstone, provided no detrital potassium-bearing minerals are present. In such cases differences in outgassing properties are unimportant. The $^{40}Ar/^{39}Ar$ method of dating can detect violations of the closed system assumption only if the secondary disturbance produces a partial outgassing of the rocks. In such cases conventional K-Ar ages would be erroneous.

The differences in outgassing properties between slates and siltstones which have been explained by textural differences might be checked by performing $^{40}Ar/^{39}Ar$ stepwise outgassing experiments on muscovite separates. Here, presumably both slates and siltstones should yield well-defined age plateaus since all the muscovite flakes, whether retaining radiogenic

(47)

argon from the regional metamorphic event or not, should outgas at the same time. However, the plateau age of the siltstones should be higher, since they contain a larger proportion of inherited argon. Effects of grain size might be determined by comparison of the results from experiments on different grain size fractions.

In this study, interpretations of discordant age spectra are somewhat ambiguous, because of the closeness in age of the regional and thermal metamorphic events. Perhaps less ambiguous data on the outgassing properties of a slate versus a siltstone could be obtained from a similar study on rocks collected at several different distances from the Great Dyke of Nova Scotia which intrudes the Meguma Group about 3 km southeast of the present study area. The age of the dyke is given as either Late Triassic or Early Jurassic (~ 195 Ma) by the 1979 version of the Geological Map of Nova Scotia (N.S. Dept. of Mines, compiled by J.D. Keppie).

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APPENDIX A

THIN-SECTION DESCRIPTIONS

KB-1   SLATE

General: Dark greyish-black, very fine-grained, with very

                         faint, thin laminations. Cleaves evenly and easily

                         at about 50° to the laminations.

Texture: Well developed preferred orientation shown by phyl-

                         losilicates and streaks of graphite, parallel to

                         cleavage and laminations.

Major Minerals: Muscovite - abund. ~ 35%, rectangular flakes 0.01 to

                         0.05 mm in length, transparent, sometimes surrounds

                         and fills cracks in chlorite.

                         Quartz - abund. ~ 30%, polygonal, diameter from 0.04

                         to 0.09 mm.

                         Chlorite - abund. ~ 25%, oval shaped, length from

                         0.1 to 0.2 mm, transparent.

                         Graphite - abund. ~ 10%, microcrystalline, black,

                         opaque, occurs as wavy streaks between grains.

Accessory Minerals: Pyrite, pyrrhotite, magnetite, apatite, tourmaline.

KB-2   LAMINATED SLATE

General: Dark greyish-black, very fine-grained, with thin, lighter, wrinkled laminations rich in quartz, sometimes cross-laminated. Poor cleavage, cleavage surfaces irregular, iron-stained.

Texture: Well developed preferred orientation shown by phyllosilicates, elongated pyrrhotite grains, and graphite streaks, parallel to laminations and cleavage.

Major Minerals: Muscovite - abund. ~ 35%, rectangular flakes, 0.01 to 0.05 mm, transparent, sometimes surrounds and fills cracks in chlorite.

Quartz - abund. ~ 30%, polygonal, diameter from 0.04 to 0.08 mm.

Chlorite - abund. ~ 25%, oval, length 0.05 to 0.1 mm, transparent.

Pyrite - abund. ~ 4%, euhedral cubes 2 to 4 mm across.

Accessory Minerals: pyrrhotite, albite, graphite, apatite, tourmaline.

KB-3   LAMINATED METASILTSTONE

General: Dark grey, with distinct lighter, wrinkled laminations rich in quartz, fine-grained, cleaves very poorly along irregular surfaces, cross-laminations prominent.

Texture: Good preferred orientation of phyllosilicates, also shown by graphite streaks and elongated pyrrhotite grains.

Major Minerals: Quartz - abund. ~ 45%, polygonal, diameter from 0.05 to 0.1 mm, abundance greater in light laminations.

Muscovite - abund. ~ 30%, rectangular flakes 0l0l to 0.06 mm, transparent.

Chlorite - abund. ~ 15%, oval shaped, length 0.05 to 0.1 mm, transparent.

Albite - abund. ~ 5%, polygonal, diameter 0.05 to 0.1 mm.

Pyrite - abund. ~ 2.5%, euhedral cubes, 2-4 mm across

Pyrrhotite - abund. ~ 2.5%, irregular oval shaped, 2 to 4 mm across.

Accessory Minerals: Epidote, apatite, tourmaline, magnetite, graphite.

KB-6   METASILTSTONE

General: Grey, faintly laminated, cross-laminations prominent, cleaves very poorly along irregular surfaces, cleavage surfaces iron-stained.

Texture: Quartz shows a granoblastic polygonal texture, with the phyllosilicates showing preferred orientation, parallel to cleavage and laminations.

Major Minerals: Quartz - abund. ~ 75%, diameter 0.05 to 0.15 mm.

Chlorite - abund. ~ 15%, oval shaped, length 0.03 to 0.3 mm, transparent.

Muscovite - abund. ~ 5%, rectangular flakes 0.02 to 0.08 mm, transparent.

Pyrite - abund. ~ 2.5%, cubic, 2 to 4 mm across.

Pyrrhotite - abund. ~ 2.5%, oval shaped with very irregular edges, elongated parallel to cleavage and laminations, 2-4 mm across.

Accessory Minerals: Apatite, tourmaline, magnetite.

APPENDIX B

SEMIQUANTITATIVE X-RAY ANALYSIS OF THE MEGUMA METASEDIMENTS

The method involves the simultaneous x-ray analysis of four major

mineral components in the meguma metasediments. The technique was

applied to the metasediments of the Goldenville Formation by Liew (1978)[1]

and is similar to that of Shaw and Weaver (1965)[2]. For semiquantitative

analysis of fine-grained sedimentary rocks, Shaw and Weaver found this

method to be far superior than standard petrographic techniques, which

tend to underestimate quantities of quartz and feldspar. Liew found

good agreement ($\pm \sim 5\%$) between this method and the normative mineralogi-

cal composition as deduced from its chemical analysis.

Most of the major minerals present in the metasediments, except

chlorite, have similar mass absorption coefficients (quartz = 35, albite = 3r,

muscovite = 44, chlorite = 54-104) for Cuk_{α} radiation (Carroll, 1969)[3]. Because

the standards used by Liew to derive the working curves (Figs. B1 and B2)

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1. Liew, M.Y. (1979); Geochemical Studies of the Goldenville Formation at

    Taylor Head, N.S. Unpublished M.Sc. Thesis, Dalhousie U., pp. A8-A12.

2. Shaw, D.B. and Weaver, C.E. (1965); The Mineralogical Composition of

    Shales. J. Sed. Pet. 35, pp. 213-222.

3. Carroll, D. (1969); Clay Minerals: A Guide to their X-ray Diffraction.

    G.S.A. sp. paper # 126, p. 47.

were prepared by mixing a pure mineral with another, both having composi-
tions close to those of the rock, the mass absorption coefficients have
been ignored. To avoid preferred orientation of some phyllosilicates,
both the standards and sample powder were analyzed using a rotating sample
holder. The operating conditions of the experiment were as follows:

Source: Cuk_α radiation

Filter: Nickel

Window: LL 350

Kv: 40

Ma: 20

Scan speed: 1/8° per minute

Chart speed: 1 cm per minute

The minerals selected for analysis were quartz, albite, muscovite,
and chlorite. The peak areas of different reflections of the minerals
were determined by the use of a planimeter. The reflections selected for
measurement were: quartz (100), albite (201), chlorite (004), and musco-
vite (004). These areas were then used to determine the reatios used to
plot the standard curves. The apparent proportions of the two minerals
in the sample can be obtained from the x-axis of the standard curves. The
proportions obtainable are:

$$\frac{chl}{qtz} = w \qquad\qquad \frac{qtz}{alb} = y$$

$$\frac{musc}{qtz} = x \qquad\qquad \frac{alb}{chl} = z$$

If it is assumed that these four minerals make up the total bulk of the
sample, then the following equation applies:

qtz + alb + musc + chl = 100%

Using the proportions obtained from the standard curves and the above equation, the percentage of quartz, albite, muscovite, and chlorite can be algebraically solved for:

$$qtz = \frac{100}{w + x + \dfrac{1}{y} + 1}$$

$$musc = qtz \cdot x$$

$$alb = \frac{100 - musc}{1 + \dfrac{1}{z} + y}$$

$$chl = \frac{100 - musc}{1 + \dfrac{1}{w} + z}$$

Due to the absorption effects from other minerals present in the sample the sum of the calculated percentages of quartz, albite, chlorite and muscovite will not quite equal 100%. The percentages obtained are recalculated into 100%.

If the sample contains an unmeasurable quantity of albite, the following equations apply:

$$\frac{chl}{qtz} = x$$

$$\frac{musc}{qtz} = y$$

qtz + musc + chl = 100%

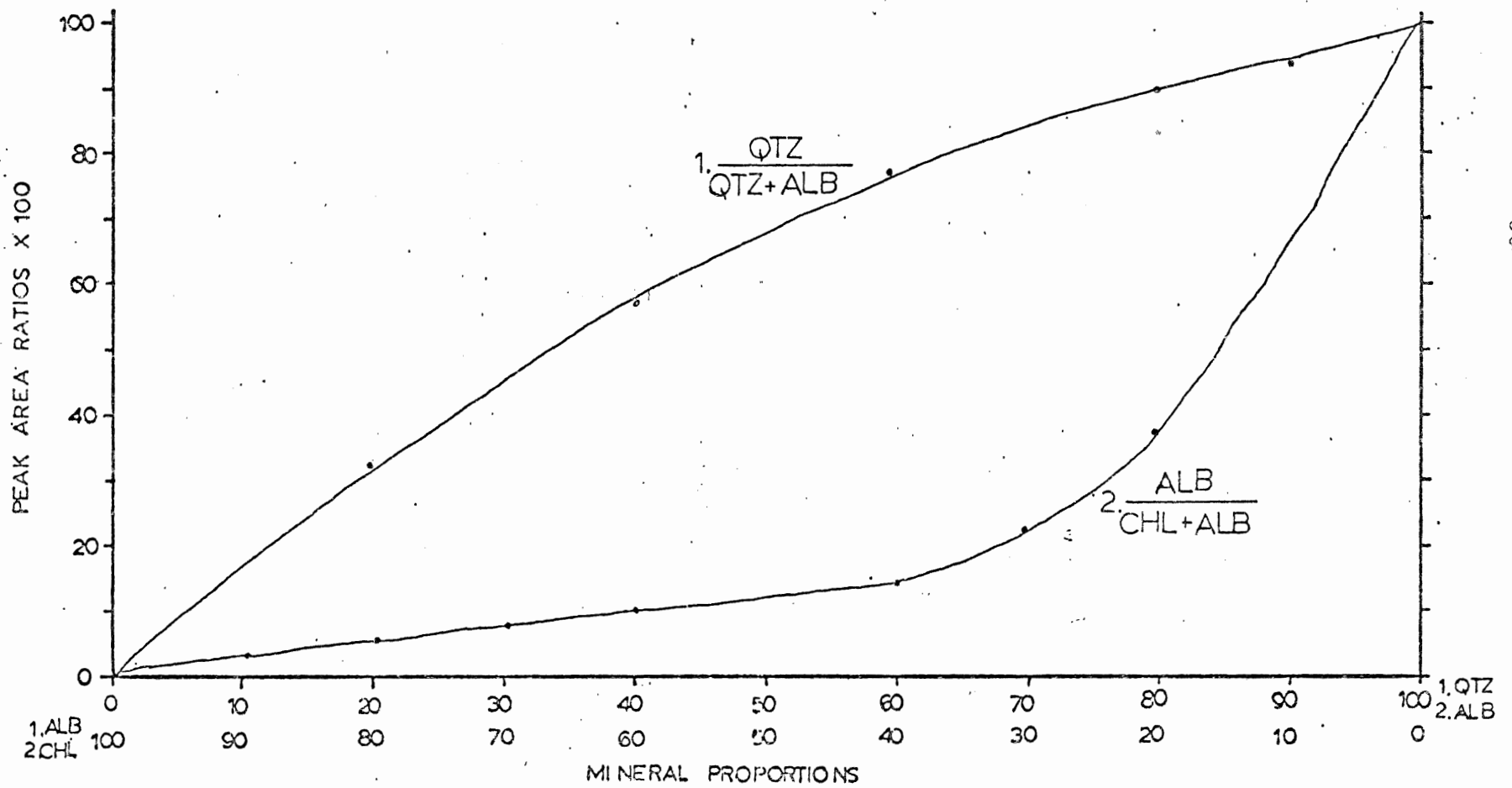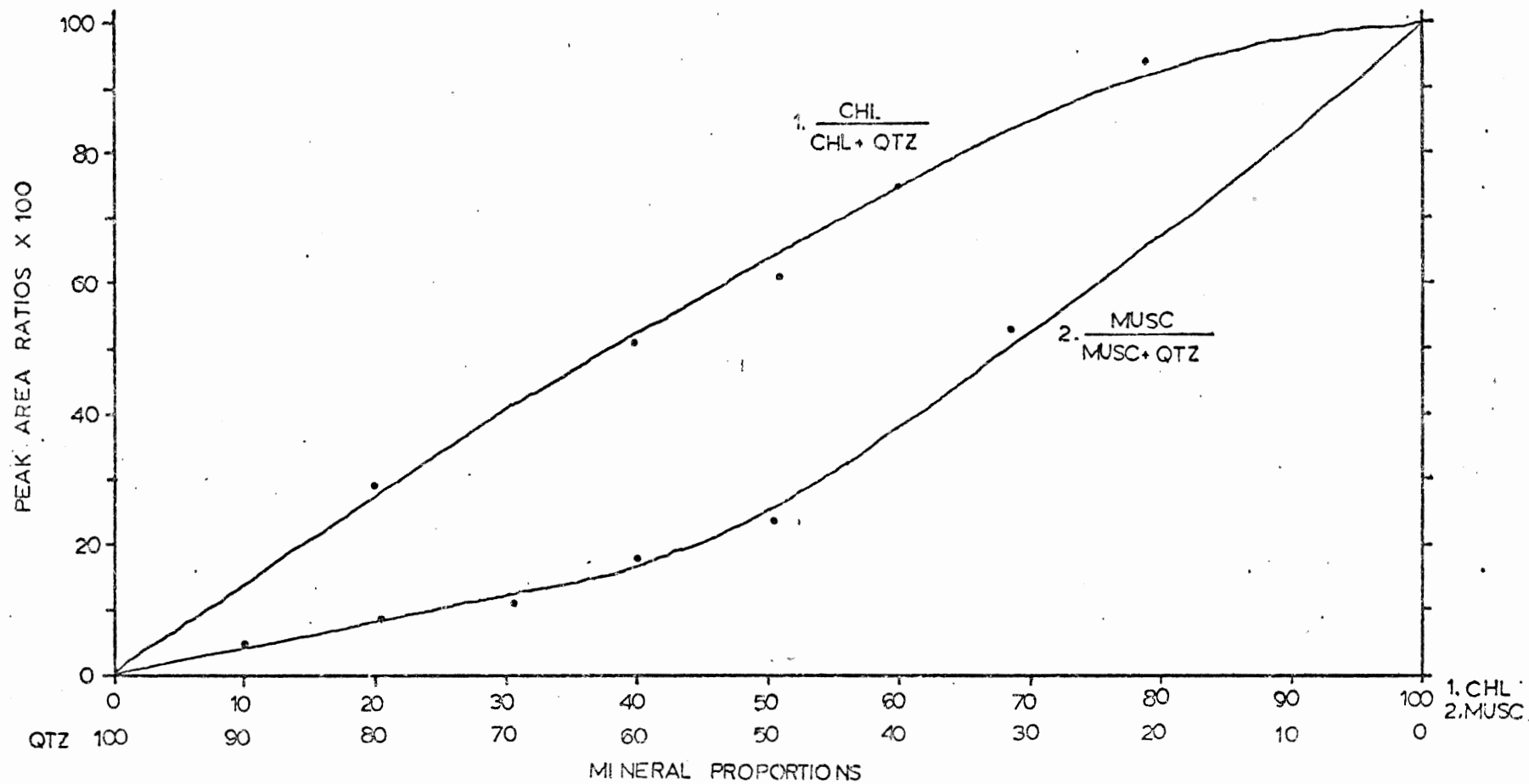FIGURE B2. Standard curves for quartz and albite. (from Liew, 1979)

- 60 -

FIGURE B1. Standard curves for chlorite and muscovite.(from Liew, 1979)

Solving for individual percentages,

$$qtz = \frac{100}{x + y + 1}$$

$$musc = qtz \cdot y$$

$$chl = qtz \cdot x$$

The results of this study are listed in Table B1.

Table B1. X-ray Data Summary

Sample No.	Peak Areas (in^3)				Abund. %			
	qtz	alb	chl	musc	qtz	alb	chl	musc
KB-1	3.41	–	4.76	1.34	34	–	28	38
KB-2	3.36	0.17	5.32	1.69	30	3	27	40
KB-3	5.91	0.30	4.18	1.09	47	5	19	29
KB-6	6.65	–	1.79	0.25	79	–	14	7