

THE UNIVERSITY OF MANITOBA

RUBIDIUM-STRONTIUM AGE DETERMINATIONS  
FROM THE FILE-MORTON-WOOSEY LAKES AREA  
OF THE FLIN FLON VOLCANIC BELT, WEST CENTRAL MANITOBA

BY

GENIC RAYMOND JOSSE

A THESIS

SUBMITTED TO THE FACULTY OF GRADUATE STUDIES  
IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE  
OF  
MASTER OF SCIENCE  
DEPARTMENT OF EARTH SCIENCES

WINNIPEG, MANITOBA

OCTOBER, 1974

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A dissertation submitted to the Faculty of Graduate Studies of  
the University of Manitoba in partial fulfillment of the requirements  
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## ABSTRACT

Rubidium-strontium whole rock isochron ages have been determined on rock units from the File-Morton-Woosey Lakes area of the Flin Flon volcanic belt. A minimum age of emplacement is given by the radiometric age of  $1860 \pm 112$  m.y. and an  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratio of  $0.7019 \pm 0.0005$  for the Ham pluton, a syn- to late kinematic 'granitic' intrusion ( $^{87}\text{Rb}$  decay constant of  $1.39 \times 10^{-11} \text{yr}^{-1}$ ). This age also provides a minimum age for the Amisk Group volcanic strata, the Missi Group sedimentary strata, and all other rocks, such as the Morton porphyry, and early deformation that predate the Ham pluton in the study area. The approximate age of  $1737 \pm 257$  m.y. and an initial ratio of  $0.7020 \pm 0.0002$  obtained for the Elmes Island Formation dacite flows, (an Amisk Group felsic volcanic), and the radiometric age of  $1715 \pm 83$  m.y. and an initial ratio of  $0.7027 \pm 0.0001$  for the Morton porphyry, (a pre- to early kinematic hypabyssal intrusion), are considered to be metamorphic ages and not emplacement ages, and that these units were updated during regional metamorphism.

The age of the regional metamorphism that affected the study area is given by the radiometric age of  $1760 \pm 43$  m.y. obtained for the File gneiss dome, a granitoid paragneiss complex from the Kisseynew sedimentary gneiss belt.

The  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratio for this unit is  $0.7038 \pm 0.0008$ .

The late pegmatite dykes are post-kinematic intrusions that postdate all other rocks in the study area. The calculated mineral ages of  $1741 \pm 55$  m.y. and  $1763 \pm 55$  m.y. (assumed initial ratio of 0.710), give the best approximation of the age of the youngest intrusive event in the study area.

The late Aphebian age of emplacement for the Ham pluton is consistent with the interpretation of Mukherjee (1971) and Sangster (1972) that the Flin Flon volcanic belt is late Aphebian. The area was subjected to regional metamorphism approximately 1760 m.y. ago, a late Aphebian event that may be representative of the Hudsonian orogeny.

## ACKNOWLEDGEMENTS

The writer is indebted to Dr. G. S. Clark for suggesting and supervising this study, and for the criticism, suggestions, and patience extended during the preparation of the manuscript.

Financial assistance for the support of this study was provided by the National Research Council and the Faculty of Graduate Studies in the form of graduate assistantships.

Special thanks are extended to A. H. Bailes, who collected the samples used in this study during the course of a geological survey for the Manitoba Mines Branch, and who gave full access to geological information contained in preliminary draughts of a Manitoba Mines Branch report and a PhD thesis on the study area. His advice on geological aspects of this study have been very helpful during the preparation of this report.

Special thanks are also extended to the following members of the Mass Spectrometry Laboratory of the Department of Physics for their interest, assistance, and instruction provided during mass spectrometric analyses: Dr. R. C. Barber, Dr. P. Williams, Dr. J. Meredith, J. Barnard, and K. Kozier.

P. Beaudoin ably assisted in the chemical preparation of many of the samples analyzed.

## TABLE OF CONTENTS

|  | <u>Page</u> |
|--|-------------|
| ABSTRACT.....  | i           |
| ACKNOWLEDGEMENTS.....  | iii         |
| TABLE OF CONTENTS.....   | iv          |
| LIST OF TABLES.....  | viii        |
| LIST OF FIGURES.....   | ix          |
| CHAPTER 1. INTRODUCTION.....                                     | 1           |
| 1.1. General Statement.....                                      | 1           |
| 1.2. Object of study.....  | 3           |
| CHAPTER 2. GEOLOGY OF THE FILE-MORTON-<br>WOOSEY LAKES AREA..... | 5           |
| 2.1. Regional setting.....                                       | 5           |
| 2.1.1. Flin Flon volcanic belt.....                              | 5           |
| 2.1.2. Kisseynew sedimentary gneiss belt.....                    | 6           |
| 2.1.3. Metamorphism.....   | 6           |
| 2.1.4. Stratigraphic correlation.....                            | 9           |
| 2.2. Geology of the study area.....                              | 10          |
| 2.2.1. The Amisk Group.....                                      | 10          |
| 2.2.2. The Missi Group.....                                      | 11          |
| 2.2.3. Intrusive Rocks.....                                      | 12          |
| CHAPTER 3. PREVIOUS AGE DETERMINATIONS.....                      | 13          |
| CHAPTER 4. DISCUSSION OF ROCK UNITS DATED.....                   | 23          |
| 4.1. Elmes Island Formation.....                                 | 23          |
| 4.2. Morton Porphyry.....  | 24          |

|   |    |
|---|----|
| 4.3. Ham Pluton.....  | 24 |
| 4.4. File Gneiss Dome.....  | 25 |
| 4.5. Late Pegmatite Dykes.....  | 26 |
| CHAPTER 5. EXPERIMENTAL TECHNIQUES.....                                 | 27 |
| 5.1. Sample collection, preparation, and selection..                    | 27 |
| 5.2. Isotope dilution analysis.....                                     | 28 |
| 5.3. Chemical procedure.....  | 28 |
| 5.4. Mass spectrometry.....   | 29 |
| 5.5. Analytical precision.....  | 29 |
| CHAPTER 6. ANALYTICAL RESULTS.....                                      | 31 |
| 6.1. Rb and Sr standards.....   | 31 |
| 6.2. Rb and Sr blank analyses.....                                      | 34 |
| 6.3. Composition and concentration of<br>spike solutions.....           | 34 |
| 6.4. Duplicate Analyses.....  | 35 |
| 6.5. Analytical results from the File-Morton-<br>Woosey Lakes area..... | 37 |
| 6.5.1. Elmes Island Formation.....                                      | 37 |
| 6.5.2. Morton porphyry.....   | 40 |
| 6.5.3. Ham pluton.....  | 40 |
| 6.5.4. File gneiss dome.....  | 45 |
| 6.5.5. Late pegmatite dykes.....  | 45 |
| CHAPTER 7. INTERPRETATION OF ISOTOPIIC DATA.....                        | 49 |
| 7.1. Discussion of isotopic data and age relations.                     | 49 |
| 7.1.1. Elmes Island Formation.....                                      | 49 |
| 7.1.2. Morton porphyry.....   | 52 |
| 7.1.3. Ham pluton.....  | 54 |
| 7.1.4. File gneiss dome.....  | 55 |

|  |    |
|--|----|
| 7.1.5. Late pegmatite dykes.....   | 56 |
| 7.2. $^{87}\text{Sr}/^{86}\text{Sr}$ initial ratios from the<br>Flin Flon volcanic belt.....   | 56 |
| CHAPTER 8. SUMMARY AND CONCLUSIONS.....  | 60 |
| REFERENCES CITED.....  | 63 |
| APPENDIX I. SAMPLE DESCRIPTION.....  | 70 |
| I.1. Elmes Island Formation.....   | 70 |
| I.2. Morton porphyry.....  | 71 |
| I.3. Ham pluton.....   | 72 |
| I.4. File gneiss dome.....   | 72 |
| I.5. Late pegmatite dykes.....   | 73 |
| APPENDIX II. DETAILED DISCUSSION OF<br>CHEMICAL PROCEDURES.....                                | 75 |
| II.1. Sample preparation and selection.....  | 75 |
| II.2. Chemical procedure.....  | 76 |
| II.2.1. Dissolution and digestion.....   | 76 |
| II.2.2. Spiking procedures.....  | 77 |
| II.2.3. Preparation of the Rb sample.....  | 77 |
| II.2.4. Preparation of the Sr sample.....  | 78 |
| II.3. Ion exchange columns.....  | 80 |
| II.4. Preparation of the potassium-feldspar<br>mineral concentrates from pegmatite samples.... | 82 |
| II.5. Preparation of spike solutions.....  | 83 |
| APPENDIX III. ISOTOPE DILUTION ANALYSES.....   | 85 |
| III.1. Isotope dilution calculation for rubidium.....  | 85 |
| III.2. Isotope dilution calculation for strontium....  | 86 |
| III.3. Calculation of the radiogenic $^{87}\text{Sr}$ content....                              | 92 |
| APPENDIX IV. MASS SPECTROMETRY.....  | 95 |
| IV.1. Description of the mass spectrometer.....  | 95 |



|                                   |     |
|-----------------------------------|-----|
| IV.2. Operating procedure.....    | 96  |
| IV.3. Reproducible analyses.....  | 98  |
| IV.4. Rubidium contamination..... | 100 |

## LIST OF TABLES

|  | <u>Page</u> |
|--|-------------|
| TABLE 1. Table of Formations.....  | 8           |
| TABLE 2. Previous age determinations;<br>Flin Flon Volcanic Belt.....      | 17          |
| TABLE 3. Analytical results for the NBS 70a-II<br>K-feldspar standard..... | 32          |
| TABLE 4. Rb and Sr blank analyses.....                                     | 33          |
| TABLE 5. Composition and concentration of<br>spike solutions.....          | 36          |
| TABLE 6. Analytical results from the Elmes<br>Island Formation.....        | 38          |
| TABLE 7. Analytical results from the<br>Morton porphyry.....               | 41          |
| TABLE 8. Analytical results from the Ham pluton.....                       | 43          |
| TABLE 9. Analytical results from the File<br>gneiss dome.....              | 46          |
| TABLE 10. Analytical results from the Late<br>pegmatite dykes.....         | 48          |
| TABLE 11. Rb-Sr age determinations: File-Morton-<br>Woosey Lakes area..... | 50          |

## LIST OF FIGURES

|  | <u>Page</u> |
|--|-------------|
| FIGURE 1. Location map of the File-Morton-Woosey Lakes study area.....                               | 2           |
| FIGURE 2. Generalized geological map of the File-Morton-Woosey Lakes area, with sample stations..... | 7           |
| FIGURE 3. Partial isochron fitting of the analytical data from the Elmes Island Formation.....       | 39          |
| FIGURE 4. Rb-Sr whole rock isochron for the Morton porphyry.....                                     | 42          |
| FIGURE 5. Rb-Sr whole rock isochron for the Ham pluton.....  | 44          |
| FIGURE 6. Rb-Sr whole rock isochron for the File gneiss dome.....                                    | 47          |
| FIGURE 7. Sr isotope evolution diagram for the Flin Flon volcanic belt.....                          | 57          |

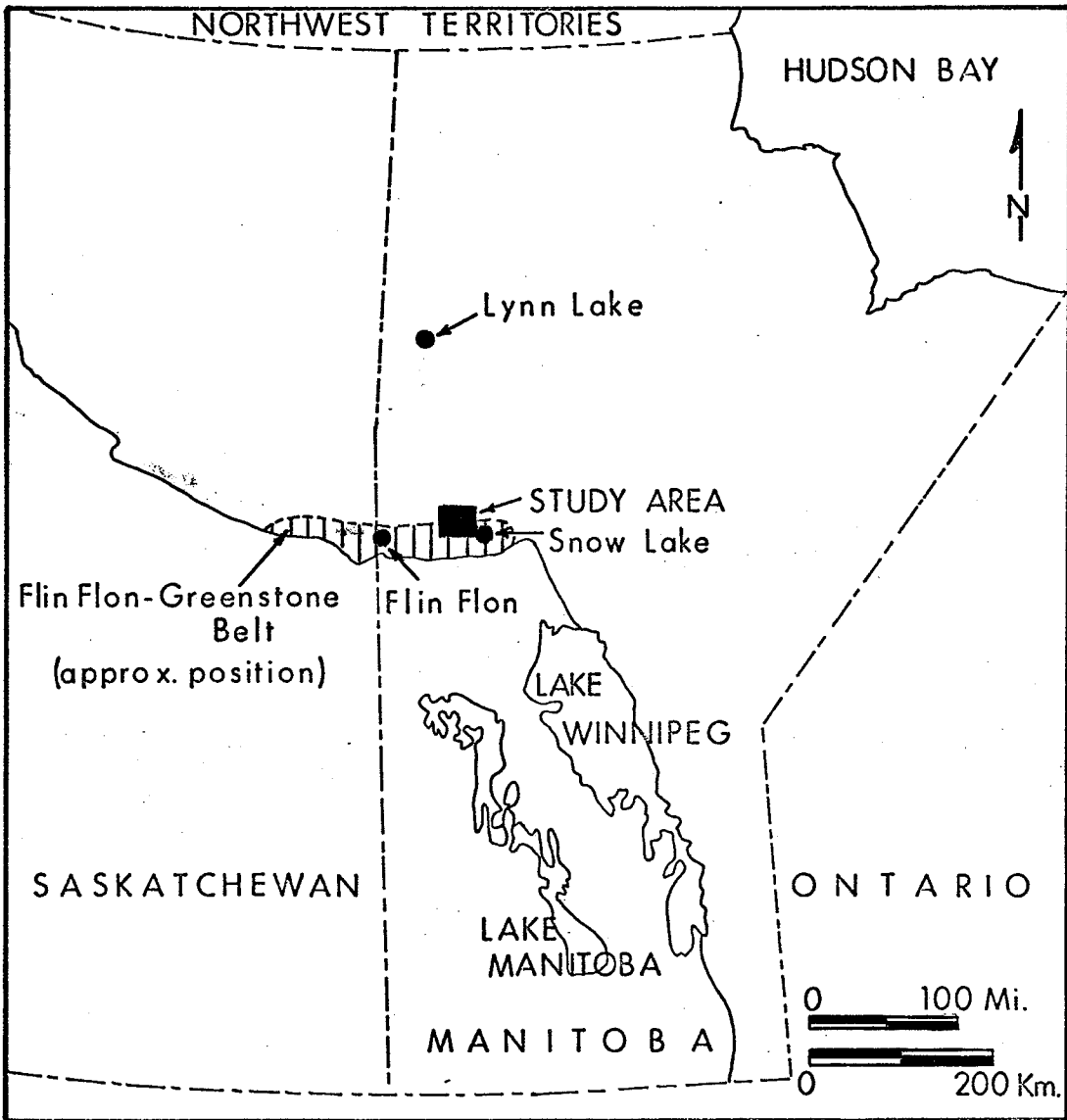
## CHAPTER 1. INTRODUCTION

### 1.1. General Statement

The File-Morton-Woosey Lakes area is located within the southeastern portion of the Churchill geological province of the Canadian Shield in west central Manitoba. The area includes portions of two major easterly trending lithological belts: the Flin Flon volcanic belt to the south, and the Kisseynew metasedimentary gneiss belt to the north (Bailes, 1971b). The study area is located 20 kilometers (12 miles) west of the town of Snow Lake, and is bounded by latitudes  $54^{\circ}45'$  and  $55^{\circ}00'$  north, and longitudes  $100^{\circ}10'$  and  $100^{\circ}30'$  west (see Figure 1).

Traditionally, the Flin Flon volcanic belt (also called the Flin Flon-Snow Lake greenstone belt) has been considered Archean in age (e.g. Stockwell, 1963, 1964; McGlynn, 1970; Bailes, 1971b; Tectonic Map of Canada, G.S.C. Map 1251A). K-Ar age determinations of micas from rocks of this belt by the Geological Survey of Canada gave Hudsonian ages (e.g. Lowdon, 1961; Lowdon *et al.*, 1963; Wanless *et al.*, 1965, 1967). These ages generally have been attributed to updating of Archean rocks by the Hudsonian orogeny (e.g. Stockwell, 1963, 1964). Archean Rb-Sr whole rock isochron ages obtained by Coleman (1970)

**Figure 1. Location map of the File-Morton-  
Woosey Lakes study area.**



on volcanic and plutonic rocks at Hanson Lake, an area that may be the western extension of the Flin Flon volcanic belt, appear to support this Archean premise. However, more recently, Aphebian Rb-Sr whole rock isochron ages have been obtained by Mukherjee et al. (1971) on volcanic and plutonic rocks near Flin Flon, and Aphebian common lead isotope ages have been obtained by Sangster (1972) on galena from sulfide deposits that are considered by him to be coeval with volcanism and to consist of single stage leads. Both authors have interpreted these Aphebian dates as the age of the Flin Flon volcanic belt. However, Slawson and Russell (1973) have questioned the validity of Sangster's lead ages, suggesting that they are multistaged leads and that the secondary U/Pb system formed at least 2450 m.y. (million years) ago.

## 1.2. Object of Study

The object of this study was to test the validity of Mukherjee's and Sangster's conclusion that the Flin Flon volcanic belt is Aphebian in age by preparing rubidium-strontium whole rock isochron ages on rock samples from the eastern portion of the belt. A paragneiss unit from the Kisseynew sedimentary gneiss belt, located in the north of the study area, was dated to obtain the age of regional metamorphism. K-feldspar mineral concentrates from late pegmatite dykes, that postdate the regional metamorphic event, were dated to obtain the youngest intrusive

event(s) in the study area.  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratios obtained for igneous rocks have been interpreted to indicate the petrogenesis of the igneous rocks, and by inference, the petrogenesis of the rest of the Flin Flon volcanic belt.



## CHAPTER 2. GEOLOGY OF THE FILE-MORTON-WOOSEY LAKES AREA

### 2.1. Regional Setting

The Precambrian rocks of the File-Morton-Woosey Lakes area comprise rocks of two broad easterly trending belts of differing lithology, degree of deformation and grade of metamorphism: the Flin Flon volcanic belt to the south and the Kisseynew metasedimentary belt to the north (Bailes, 1971b). The geology of the File-Morton-Woosey Lakes area is shown in Figure 2, and the corresponding Table of Formations is given in Table 1.

The following discussion of the regional geology of the Flin Flon volcanic belt and Kisseynew metasedimentary gneiss belt is based on a regional compilation prepared by Bailes (1971b). Other reports of related interest are: Davies *et al.* (1962), Harrison (1949, 1951), McGlynn (1959, 1970), Robertson (1953), Williams (1966), Russell (1957), Kalliokoski (1952, 1953), Kornik (1968), Hunt (1970), Rousell (1970), Mukherjee (1971), Byers *et al.* (1954, 1965), and Moore and Froese (1971, 1972).

#### 2.1.1. Flin Flon Volcanic Belt

The Flin Flon volcanic belt comprises Amisk Group volcanic and sedimentary rocks, Missi Group sedimentary rocks, and intrusive rocks that outcrop over a distance of

240 km (150 miles), extending from 32 km (20 miles) east of Snow Lake to 48 km (30 miles) west of Flin Flon. The average exposed width of the Flin Flon volcanic belt is 48 km (30 miles). The volcanic belt is bounded to the north by the Kisseynew sedimentary gneiss belt and is overlain to the south by flat-lying Paleozoic sedimentary rocks.

In the study area, Bailes (in preparation) has subdivided the Amisk Group into Lower Amisk and Upper Amisk Subgroups, consisting of volcanic and sedimentary strata, respectively (see Table 1).

#### 2.1.2. Kisseynew Metasedimentary Gneiss Belt

The Kisseynew sedimentary gneiss belt consists of two major metasedimentary sequences: a lower meta-greywacke unit and an upper unit of meta-arkose. Robertson (1953) termed these units the Nokomis Group and the Sherridon Group, respectively. In the study area, Bailes (in preparation) has termed these units the Upper Amisk Subgroup and the Missi Group, respectively, due to the probable equivalence of these strata to those of the Flin Flon volcanic belt. The dimensions of the Kisseynew gneiss belt are approximately 200 km (120 miles) in width and 500 km (300 miles) in length.

#### 2.1.3. Metamorphism

The major metamorphic and tectonic event(s) post-date deposition of all volcanic and sedimentary strata. The

Figure 2. Generalized geological map of the  
File-Morton-Woosey Lakes area, with  
sample stations (after Bailes, 1971a).

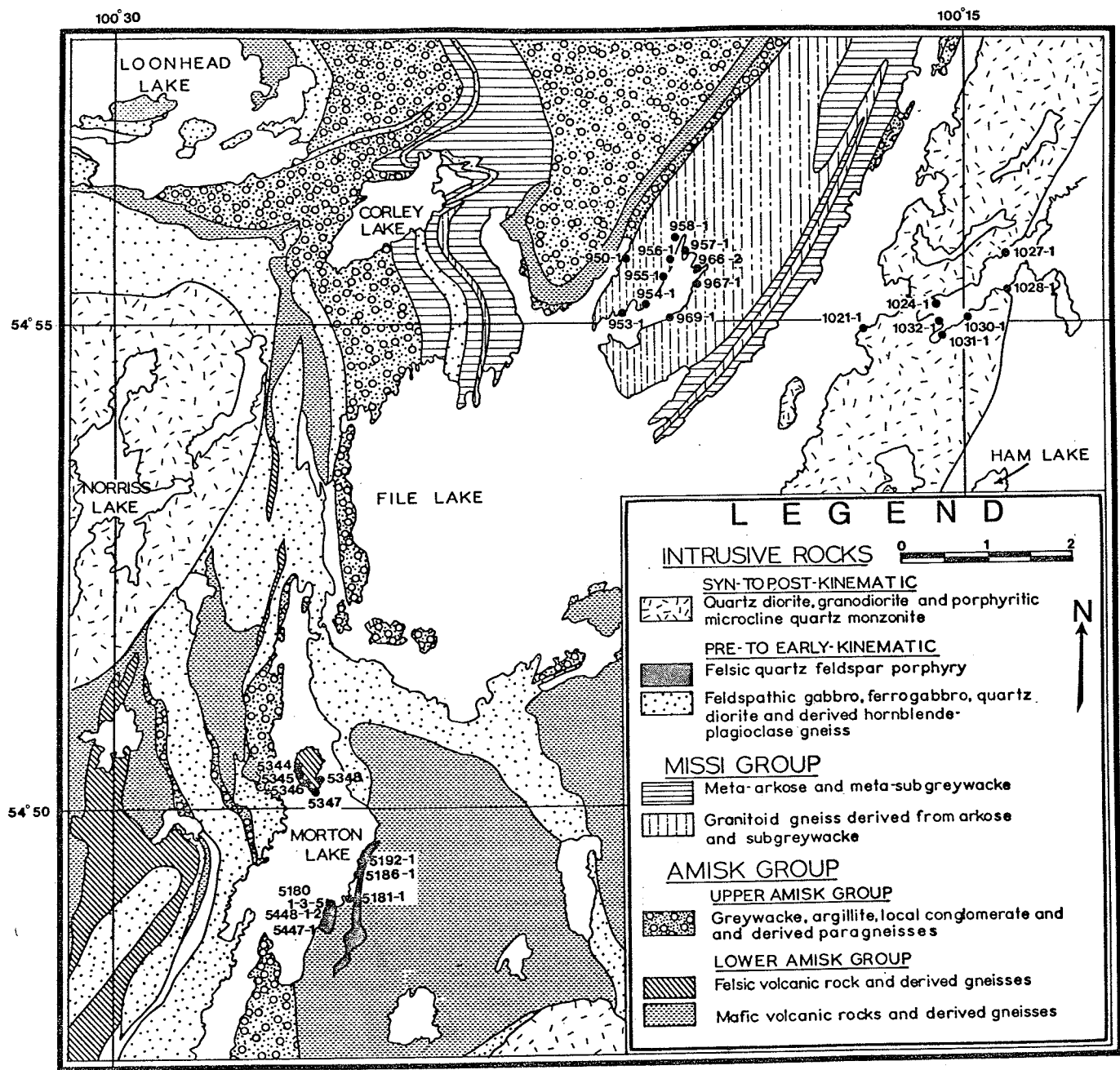


TABLE 1. TABLE OF FORMATIONS [after Bailes (in preparation)]

|                          |                   |                        |  |   |
|--------------------------|-------------------|------------------------|--|---|
| Pleistocene & Recent     |                   | Great Unconformity     |  |   |
| PROTEROZOIC<br>ALBERTIAN | Intrusive Rocks   | Post-kinematic         | +Pegmatite Dykes   |   |
|                          |                   | Intrusive Contact      |  |   |
|                          |                   | Syn- to late-kinematic | Porphyritic microcline granite<br>●Granodiorite, tonalite, quartz diorite  | Meta-Complexes<br>Anatectic Complex:<br>Pegmatitic granodiorite and granite<br>File Gneiss Dome Complex:<br>■ Granitoid gneiss derived from the Missi Group |
|                          |                   | Intrusive Contact      |  |   |
|                          |                   | Pre-to early-kinematic | +Felsic quartz feldspar porphyry<br>Josland Formation: Differentiated gabbro sills   |   |
|                          | Intrusive Contact |                        |  |   |
|                          | Missi Group       | Meta-arkose            |  |   |
|                          | ?Disconformity?   |                        |  |   |
|                          | Amisk Group       | Upper Amisk Subgroup   | ▲File Lake Formations: Interbedded greywacke and shale and derived paragneisses.<br>Yakimiw Formation: Interbedded greywacke and shale.  |   |
|                          |                   | Lower Amisk Subgroup   | Elmes Island Formation: Dacite Flows.<br>Storozuk Formation: Basalt and andesite flows.<br>Dickstone Formation: Rhyolite and dacite flows and tuff<br>Preston Formation: Basalt and andesite flows<br>Undifferentiated basalt and andesite flows | } "2nd" volcanic cycle<br>} "1st" volcanic cycle  |

Units dated:

▲Elmes Island Formation of dacite flows.  
+Morton porphyry.

●Ham pluton.  
■File gneiss dome.  
‡Late pegmatite dykes.

intensity of deformation and grade of metamorphism is highest in the Kisseynew sedimentary gneiss belt and decreases into the volcanic belt. Middle to upper almandine-amphibolite facies assemblages and widespread migmatization and granitization, dominate the Kisseynew belt. It is highly deformed.

The Flin Flon volcanic belt is less deformed and metamorphosed than the Kisseynew belt. It is composed of rocks metamorphosed under greenschist to lower almandine-amphibolite facies conditions. The metamorphic grade increases northward towards the boundary contact zone with the Kisseynew belt. Much of the deformation in the volcanic belt was caused by emplacement of large granitic plutons. The plutons have thermal aureoles in which hornblende hornfels assemblages have been developed.

#### 2.1.4. Stratigraphic Correlation

The Amisk and Missi sedimentary rocks have been correlated with rocks of the Kisseynew sedimentary gneiss belt by Bailes (1971b, p.13-14) north of Wekusko Lake, Manitoba, and by Byers and Dahlstrom (1954, p.50) north of Amisk Lake, Saskatchewan. In the study area, Bailes (in preparation) has shown that rocks of both belts are part of the same stratigraphic succession. The Nokomis Group is considered equivalent to the "flyschoid" greywacke-shale sequences of the Upper Amisk Subgroup, and the Sherridon Group to the arkosic rocks of the Missi Group.

## 2.2. Geology of the Study Area

The File-Morton-Woosey Lakes area straddles the boundary zone of the Flin Flon volcanic belt and the Kisseynew sedimentary gneiss belt. This boundary is considered to follow the south shore-line of File Lake in the study area (Figure 2). Geological mapping by Bailes (in preparation) has shown that rock units of both belts are part of the same stratigraphic succession. In the accompanying table of formations (Table 1), rocks belonging to the Nokomis and Sherridon Groups of the Kisseynew belt have been included by him with the Upper Amisk Subgroup and Missi Group of the Flin Flon belt, respectively. The rocks of the study area have been subdivided into three main groups (from oldest to youngest): the Amisk Group, the Missi Group, and the Intrusive Rocks.

The following description of the geology for the study area is based on information provided by Bailes from a geological report in preparation by him for the Manitoba Mines Branch.

### 2.2.1. The Amisk Group

The oldest rocks of the study area are the Amisk Group, which consist of volcanic strata (Lower Amisk Subgroup) and overlying sedimentary strata (Upper Amisk Subgroup). There are believed to be at least two mafic to felsic volcanic cycles in the Lower Amisk Subgroup. The volcanic

rocks comprise primarily subaqueous flows of calc-alkaline composition. Mafic volcanism consists largely of flows, whereas felsic volcanism is a mixture of flows and pyroclastic material.

Sedimentary rocks of the Upper Amisk Subgroup comprise immature deposits of interbedded greywacke and shale. The deposits consist of the Yakimiw and File Lake formations, which are composed primarily of volcanic detritus and likely were derived by erosion of loose debris from contemporaneous volcanic deposits. The formations were deposited rapidly in deep water by turbidity currents. The rocks north of File Lake have been regionally metamorphosed to garnet-staurolite-sillimanite bearing paragneisses (i.e., lower to middle almandine-amphibolite facies). The rocks south of File Lake have been regionally metamorphosed to greenschist facies mineral assemblages.

#### 2.2.2. The Missi Group

The Upper Amisk metasedimentary rocks are overlain conformably by a meta-arkose sequence known as the Missi Group. The rocks are commonly crossbedded and are likely shallow-water deposits. They consist of arkose and subgreywacke deposits that have been metamorphosed to middle and upper almandine-amphibolite facies. North of File Lake, portions have been intensely recrystallized and homogenized to produce granitoid rocks, including the rocks of the File gneiss dome. The rocks of this domal complex are believed



to have been formed during the main regional metamorphism.

### 2.2.3. Intrusive Rocks

Several periods of metamorphism, deformation and intrusive rocks postdate the Missi Group. In the study area, the intrusive rocks have been subdivided according to their relation to the main culmination of deformation and metamorphism. The pre- to early kinematic intrusive rocks are large differentiated gabbroic sills. Chemical analyses of chilled margins from these gabbroic sills indicate that the original composition was basaltic (Bailes, personal communication). Also included in this group are plugs and dykes of tonalitic quartz-feldspar porphyry exposed east of Morton Lake.

Syn- to late kinematic intrusive rocks consist of large "granitic" plutons varying from quartz diorite to granite in composition. They have been emplaced along the entire length of the Flin Flon volcanic belt but not in the adjacent Kisseynew sedimentary gneiss belt, suggesting some major tectonic crustal control on their formation. They are calc-alkaline in composition and are true magmatic "granites" as they contain numerous angular stoped inclusions having thermal contact aureoles.

The post kinematic intrusive rocks consist of granitic pegmatite dykes that locally intruded and fractured rocks of the area, such as the Ham pluton and the File gneiss dome.

### CHAPTER 3. PREVIOUS AGE DETERMINATIONS

The Churchill geological province, comprising rocks of predominantly Aphebian age, is largely underlain by gneissic and granitoid rocks of great structural and petrological complexity (Davidson, 1972, p.382). The geological complexity is reflected by radiometric determinations from the Churchill geological province. K-Ar mica ages, determined by the Geological Survey of Canada, generally have given a mean age of  $1735 \pm 95$  m.y. which is presumed to reflect a major orogenic period, the Hudsonian (Stockwell, 1964, p.5; 1970, p.49). However, both Archean and Aphebian ages have also been determined on volcanic and plutonic rocks from the Churchill Province using the Rb-Sr whole rock isochron dating technique (e.g. Money, 1968; Money et al., 1970; Coleman, 1970; Davidson, 1972; Anderson 1974). Archean Rb-Sr whole rock isochron ages have been determined from granitic rocks believed to be basement to the metasedimentary strata of the Wollaston Lake fold belt (Money et al., 1970; Anderson, 1974).

Traditionally, the Precambrian rocks of the Flin Flon volcanic belt have been considered Archean in age, and were later deformed and updated by the Hudsonian orogeny (e.g. Stockwell, 1963, 1964; Tectonic Map of Canada, Map 1251A; McGlynn, 1970; Bailes, 1971b; Davidson, 1972).

Geologically, the assignment of an Archean age has been based on: 1) the lithological similarity of the Flin Flon volcanic belt and the Archean greenstone belts of the neighbouring Superior Province; and 2) the gross overall easterly trend of structural and aeromagnetic patterns of the volcanic belt parallels those of the adjacent Superior Province (the Churchill trends generally are considered to be north-easterly (Bailes, 1971b, p.19)).

K-Ar age determinations of micas from rocks of the Flin Flon volcanic belt by the Geological Survey of Canada gave Hudsonian ages (e.g., Lowdon, 1961; Lowdon et al., 1963; Wanless et al., 1965, 1967). No date older than  $1960 \pm 60$  m.y. has been determined (GSC 63-104, Wanless et al., 1965). These K-Ar mica ages have been attributed to updating of the Archean greenstone rocks by the Hudsonian orogeny (Stockwell, 1963, 1964).

Coleman (1970) obtained Archean ages on volcanic and plutonic rocks from the Hanson Lake area, which probably lies on the western extension of the Flin Flon volcanic belt. His dates appear to support the premise that the Flin Flon volcanic belt is Archean in age, and that the previously obtained K-Ar mica ages indicate updating of the volcanic belt by the Hudsonian orogeny. However, more recently, Aphebian ages have been obtained for the Flin Flon volcanic belt by Mukherjee et al. (1971) and Sangster (1972). Mukherjee et al. (1971) obtained two Rb-Sr whole rock isochron ages on a volcanic and plutonic suite,

respectively. Implicit in their interpretation is that these dates represent "emplacement" ages for the volcanic and plutonic rocks. Mukherjee et al. (1971, p.946) state that "although an Archean age for the Amisk Group in the Flin Flon-Amisk Lake area cannot be disproven there is no direct evidence to suggest that it is older than Aphebian. In any case, the main metamorphic and intrusive events are early Hudsonian and no evidence of the Kenoran orogeny has been observed in this region." Common lead isotope ages by Sangster (1972) and Sinha (1970) on galena from sulfide deposits from across the length of the volcanic belt are considered by them to give the age of the host volcanic strata. Their interpretation is based on the assumption that: 1) the sulfide deposits are volcanogenic (i.e. coeval with the volcanism); and 2) the lead is single-stage. They feel that the geological and chemical data are consistent with these assumptions. Aphebian ages were obtained by this method for the sulfide deposits and their host volcanic rocks. However, Slawson and Russell (1973) and Ozard et al. (1973) have suggested that the Flin Flon leads are multistaged (anomalous) leads. Slawson and Russell (1973) indicate that the minimum age for the formation of the secondary U/Pb system (and maximum age for mineralization), that is compatible with their anomalous lead line, is approximately 2450 m.y. Ozard et al. (1973) have proposed a modified three-stage model for the Flin Flon leads to have occurred at 3040 m.y., 2630 m.y., and

1500 m.y. ago. The geological significance of these multi-stage leads and their corresponding model ages are reviewed by Thorpe and Sangster (1973).

Table 2 is a list of previously determined ages from the Flin Flon volcanic belt. Only Coleman's (1970) Rb-Sr whole rock isochron ages are Archean. The remaining age determinations are Aphebian. To the author's knowledge, no U-Th-Pb concordia ages nor K-Ar hornblende ages have been reported for rocks of the Flin Flon volcanic belt.

TABLE 2. PREVIOUS AGE DETERMINATIONS: FLIN FLON VOLCANIC BELT

A) K-Ar Mineral Ages

| <u>Reference</u>                               | <u>Location</u>   | <u>Sample Description</u>   | <u>Material</u> | <u>Method</u> | <u>Age(m.y.)</u> |
|--|---|---|-----------------|---------------|------------------|
| Lowdon <u>et al.</u><br>(1963)<br>(GSC 61-112) | Reynard Lake<br>Lat: 54°41'N<br>Long: 101°59'W                      | Quartz monzonite, intrudes the Amisk Group volcanics. (syn-kinematic intrusive rock)  | Biotite         | K-Ar          | 1705 ± 85        |
| Lowdon (1961)<br>(GSC 60-74)                   | Reed Lake<br>Lat: 54°39'N<br>Long: 100°15'W                         | Muscovite-biotite granite, from granite dyke related to granite pluton that intrudes Amisk Group volcanics. (Syn-kinematic) | Biotite         | K-Ar          | 1745 ± 85        |
| Lowdon <u>et al.</u><br>(1963)<br>(GSC 61-118) | Reed Lake<br>Lat: 54°39'N<br>Long: 100°15'W                         | Same as GSC 60-74   | Muscovite       | K-Ar          | 1775 ± 90        |
| Lowdon <u>et al.</u><br>(1963)<br>(GSC 61-119) | Hat Lake<br>Lat: 54°46'W<br>Long: 99°32'W                           | Medium-grained impure meta-quartzite belonging to the Miss Group  | Biotite         | K-Ar          | 1770 ± 90        |
| Lowdon <u>et al.</u><br>(1963)<br>(GSC 61-120) | Hat Lake<br>Lat: 54°46'W<br>Long: 99°32'W                           | Same as GSC 61-119.   | Muscovite       | K-Ar          | 1620 ± 80        |
| Wanless <u>et al.</u><br>(1965)<br>(GSC 63-96) | Coronation Mine<br>(1,050' level)<br>Lat: 54°35'N<br>Long: 102°00'W | Anthophyllite-garnet-biotite skarn, comprises metamorphosed and fragmental volcanic rock. (Amisk)                           | Biotite         | K-Ar          | 1930 ± 100       |

|   |  |  |           |      |           |
|---|--|--|-----------|------|-----------|
| Wanless <u>et al.</u><br>(1965)<br>(GSC 63-99)  | Crowduck Bay,<br>Wekusko Lake<br>Lat: 54°52'N<br>Long: 99°43'W | A post-Missi quartz-microcline mica pegmatite sill that intruded metamorphosed Missi sediments. Postdates main event of regional metamorphism. | Muscovite | K-Ar | 1790 ± 60 |
| Wanless <u>et al.</u><br>(1965)<br>(GSC 63-100) | Crowduck Bay,<br>Wekusko Lake<br>Lat: 54°52'N<br>Long: 99°43'W | Same as GSC 63-99  | Biotite   | K-Ar | 1610 ± 50 |
| Wanless <u>et al.</u><br>(1965)<br>(GSC 63-104) | Goose Bay<br>Wekusko Lake<br>Lat: 54°43'N<br>Long: 99°59'W     | Massive biotite-quartz monzonite stock of variable composition; intrudes Missi sediments. (Syn-kinematic?)                                     | Biotite   | K-Ar | 1960 ± 60 |
| Wanless <u>et al.</u><br>(1965)<br>(GSC 63-106) | Cliff Lake<br>Lat: 54°48'N<br>Long: 101°50'W                   | Medium-grained altered granitic rock with eyes of quartz; shows evidence of crushing.  | Muscovite | K-Ar | 1620 ± 80 |
| Wanless <u>et al.</u><br>(1965)<br>(GSC 63-108) | Phantom Lake<br>Lat: 54°42'N<br>Long: 101°52'W                 | Massive porphyritic granodiorite with phenocrysts of feldspar; youngest granite in Flin Flon area (late-to post kinematic?).                   | Biotite   | K-Ar | 1805 ± 65 |
| Wanless <u>et al.</u><br>(1967)                 | Crowduck Bay,<br>Wekusko Lake<br>Lat: 54°51'N<br>Long: 99°38'W | Lithium-bearing pegmatite  | Muscovite | K-Ar | 1735 ± 55 |
| Moore <u>et al.</u><br>(1960)                   | Stall Lake<br>Lat: 54°51'N<br>Long: 99°56'W                    | Quartz-biotite gneiss, thought to be of sedimentary origin by Russell (1957), and an altered volcanic rock by Harrison (1949).                 | Biotite   | K-Ar | 1760      |

|                              |  |   |   |      |  |
|------------------------------|--|---|---|------|--|
| Lowdon (1961)<br>(GSC 60-73) | Kisseynew Lake<br>Lat: 54°57'N<br>Long: 101°41'W     | Medium-grained paragneiss, type area for Kisseynew metasedimentary gneiss belt.   | Biotite   | K-Ar | 1735 ± 85  |
| Hunt & Dennison<br>(1971)    | Iskwasum Lake Area<br>Lat: 54°40'N<br>Long: 100°51'W | Biotite fractions from post-Mississippian granitic plutons; whole rock sample from metamorphosed Amisk sediments. (syn-to late kinematic) | 4 Biotite Fractions<br>whole rock<br>biotite<br>amphibolite | K-Ar | Average age<br>1749 ± 24<br>(ranging from 1710-1785) |

B) Rb-Sr Ages

| Reference                     | Location   | Sample Description   | Material      | Method            | Age(m.y.) | ( <sup>87</sup> Sr/ <sup>86</sup> Sr) <sub>i</sub> |
|-------------------------------|--|--|---------------|-------------------|-----------|--|
| Mukherjee<br>et al.<br>(1971) | Flin Flon Area<br>Saskatchewan<br>Lat: 54°45'N<br>Long: 101°55'W   | Amisk Group meta-volcanics.  | Whole<br>Rock | Rb-Sr<br>Isochron | 1775 ± 89 | 0.7039<br>± 0.0013                                 |
| Mukherjee<br>et al.<br>(1971) | Annabel Lake<br>pluton<br>Lat: 54°48'N<br>Long: 102°10'W           | Large, concordant, medium to coarse-grained granitic pluton that intrudes Amisk volcanics. (Post Mississippian and syn-kinematic). | Whole<br>Rock | Rb-Sr<br>Isochron | 1805 ± 14 | 0.7023<br>± 0.0007                                 |
| Coleman<br>(1970)             | Hanson Lake area<br>Saskatchewan<br>Lat: 54°41'N<br>Long: 102°54'W | Amisk-type metavolcanics.  | Whole<br>Rock | Rb-Sr<br>Isochron | 2521 ± 60 | 0.7013<br>± 0.0006                                 |
| Coleman<br>(1970)             | Hanson Lake area<br>Saskatchewan<br>Lat: 54°45'N<br>Long: 102°54'W | Granitic rocks that intrude Amisk-type metavolcanics.  | Whole<br>Rock | Rb-Sr<br>Isochron | 2446 ± 16 | 0.7008<br>± 0.0005                                 |



|                              |  |   |  |                   |   |                           |
|------------------------------|--|---|--|-------------------|---|---------------------------|
| Coleman<br>(1970)            | Hanson Lake area<br>Saskatchewan<br>Lat: 54°45'N<br>Long: 102°54'W | Late pegmatites   | Whole<br>Rock  | Rb-Sr<br>Isochron | 1799 ± 2                                    | 0.6980<br>± 0.0015        |
| Hunt &<br>Dennison<br>(1971) | Iskwasum Lake<br>area<br>Lat: 54°40'N<br>Long: 100°51'W            | Post-Missi granitic<br>plutons (syn-to late<br>kinematic).  | Biotite  | Rb-Sr<br>Mineral  | Average<br>1760 ± 35<br>range:<br>1630-1830 | 0.702<br>assumed<br>ratio |
| Hunt &<br>Dennison<br>(1971) | Iskwasum Lake<br>area<br>Lat: 54°40'N<br>Long: 100°51'W            | Same as above   | Feldspar<br>concen-<br>trates,<br>plus one<br>whole rock | Rb-Sr<br>Isochron | 1904 ± 37                                   | 0.702                     |
| Sinha (1970)                 | Hanson Lake area<br>Saskatchewan                                   | Biotite granodiorite,<br>included as a cogene-<br>tic sample with four<br>K-feldspar concentra-<br>tes and five whole<br>rock samples from the<br>"Eastern Granite" of<br>the Wollaston Lake<br>fold belt. This<br>Hanson Lake grano-<br>diorite intrudes<br>Coleman's (1970)<br>Archean granite<br>listed above. | Potassium<br>Feldspar<br>Concen-<br>trate                | Rb-Sr<br>Isochron | 1846 ± 60                                   | 0.7040<br>± 0.0005        |

C) Model Lead Ages

| Reference                                  | Location   | Sample Description   | Material   | Method                             | Age(m.v.)   |
|--|--|--|--|------------------------------------|---|
| Sangster<br>(1972)                         | Western Nuclear<br>Mine, Hanson Lake<br>area, Saskatchewan<br>Lat: 54°42'N<br>Long: 102°50'W | Massive economic sulfide<br>deposit, considered coeval<br>with host Amisk-type meta-<br>volcanics. Contains visi-<br>ble galena. | Galena   | Single-<br>Stage<br>Model<br>Pb-Pb | 1780 ± 44   |
| Sangster<br>(1972)                         | Flin Flon Mine<br>Lat: 54°45'N<br>Long: 101°52'W   | Sulfide deposit coeval with<br>Amisk metavolcanic host<br>rocks; no visible galena.  | Lead ex-<br>tract from<br>massive<br>sulfide ore | Single-<br>stage<br>model<br>Pb-Pb | 1850 ± 44   |
| Sangster<br>(1972)                         | Schist Lake Mine<br>Lat: 54°44'N<br>Long: 101°50'W   | Same as for Flin Flon<br>mine.   | Lead extract<br>from massive<br>sulfide ore      | Single-<br>stage<br>model<br>Pb-Pb | 1800 ± 44   |
| Sangster<br>(1972)                         | Osborne Lake Mine<br>Lat: 54°58'N<br>Long: 99°42'W   | Same as above; contains<br>visible galena.   | Galena   | Single-<br>stage<br>model<br>Pb-Pb | 1900 ± 44   |
| Stacey <i>et al.</i><br>(1969)             | Flin Flon Mine<br>Lat: 54°45'N<br>Long: 101°52'W   | Massive sulfide deposit  | Galena   | Single-<br>stage<br>model Pb-Pb    | 1880  |
| Doe (1967)                                 | Flin Flon Mine   | Massive sulfide deposit  | Galena   | Single-<br>stage<br>model<br>Pb-Pb | 1750<br>1920 <sup>a</sup><br><sup>a</sup> Sinha(1970) |
| Sinha(1969,70)<br>Sinha & Tilton<br>(1973) | Flin Flon Mine   | Massive sulfide deposit,<br>considered coeval with<br>host rock volcanisms.  | Galena   | Single-<br>stage<br>model<br>Pb-Pb | 1920  |

|   |  |  |                          |                                    |      |
|---|--|--|--------------------------|------------------------------------|------|
| Sinha<br>(1970)   | Phantom Lake<br>Lat: 54°42'N<br>Long: 101°52'W | Biotite granodiorite from<br>the Phantom Lake pluton<br>that is considered to be<br>youngest intrusive in area.<br>(late to post kinematic?) | Feldspar<br>concentrates | Single-<br>stage<br>model<br>Pb-Pb | 1620 |
| Sinha (1970)  | Western Nuclear<br>(54°42'N;<br>102°50'W)      | Disseminated galena, con-<br>sidered coeval with host<br>rock volcanism.   | Galena                   | Single-<br>stage<br>model<br>Pb-Pb | 1850 |
| (The listed<br>ages were<br>obtained<br>from<br>Sangster<br>(1972),<br>Fig. 4,<br>p.508). | Hanson Lake<br>(54°45'N;<br>102°45'W)          | Massive sulfide deposit,<br>considered coeval with<br>host rock volcanism.   |                          |                                    | 1880 |
|   | Flin Flon Mine<br>(54°45'N;<br>101°52'W)       | Massive sulfide deposit,<br>considered coeval with<br>host rock volcanism.   |                          |                                    | 1880 |
|   | Stall Lake<br>(54°52'N;<br>99°57'W)            | Disseminated galena, con-<br>sidered coeval with host<br>rock volcanism.   |                          |                                    | 1920 |
|   | Chisel Lake<br>(54°50'N;<br>100°08'W)          | Disseminated galena, con-<br>sidered coeval with host<br>rock volcanism.   |                          |                                    | 1870 |
|   | Osborne Lake<br>(54°58'N;<br>99°45'W)          | Disseminated galena, con-<br>sidered coeval with host<br>rock volcanism.   |                          |                                    | 1920 |
| Sinha and<br>Tilton<br>(1973)   |  | Average galena sample,<br>Churchill Province   | Galena                   | Single-<br>stage<br>model<br>Pb-Pb | 1950 |
| Russell and<br>Farquhar<br>(1960)<br>p.139  | Snow Lake<br>54°54'N;<br>100°01'W              | Obtained from vein on<br>south shore of Snow Lake<br>on south side of Lake<br>Fault.   | Galena                   | Single-<br>stage<br>model<br>Pb-Pb | 1860 |

## CHAPTER 4. DISCUSSION OF ROCK UNITS DATED

Four rock units from the File-Morton Woosey Lakes study area were dated. These units are: 1) Elmes Island Formation dacite flows (Lower Amisk Subgroup); 2) Morton porphyry (a pre- to early kinematic intrusion); 3) Ham pluton (a syn- to late kinematic intrusion); and, 4) File gneiss dome complex (a post-Missi granitoid complex of arkosic composition). In addition, two K-feldspar mineral concentrates from late pegmatite dykes, (post-kinematic intrusions), were also dated. Descriptions of these units are summarized from Bailes (in preparation).

### 4.1. Elmes Island Formation

Based on field evidence, the oldest geological unit dated was the Elmes Island Formation, which is the felsic member of the "second" volcanic cycle of the Lower Amisk Subgroup (see Table of Formations, Table 1). It consists of a monotonous sequence of calc-alkaline subaqueous dacite flows. Metamorphism of these strata was slight, producing upper greenschist facies mineral assemblages. The purpose in dating this unit was to obtain the age of Amisk volcanism.

#### 4.2. Morton Porphyry

The Morton porphyry is a tonalitic quartz-feldspar porphyry that is exposed on the east shore of Morton Lake. It is one of the pre- to early kinematic intrusions that occur in small dykes and plugs and crosscut volcanic rocks. The unit is characterized by large subhedral to euhedral phenocrysts of blue quartz and delicately zoned plagioclase, up to 10 mm in size, that is set in a fine-grained felsic matrix. The porphyry bodies are probably high level intrusions, resembling hypabyssal or subvolcanic plugs. Their age relative to the other intrusive rocks is uncertain but they are considered to be pre- to early kinematic intrusions because: 1) they cross cut and are mutually crosscut by small subvolcanic gabbroic dykes; and, 2) they have been foliated and regionally metamorphosed to upper greenschist facies, thus predating the main periods of deformation and metamorphism in the study area. The purpose of dating this unit was to determine its age of emplacement, and a minimum age for the volcanic and sedimentary rocks of the study area.

#### 4.3. Ham Pluton

The Ham pluton is an elongate north-northwesterly trending batholith that outcrops on the east side of File Lake. It is composed of medium to coarse-grained tonalite and granodiorite. The pluton intrudes the Amisk Group, but postdates the early folds that deformed the Missi strata

and the pre- to early kinematic intrusive rocks. The pluton contains a north-northwesterly trending foliation defined by flattening and crushing of quartz and plagioclase grains. The northern portion of the pluton has been subjected to high grade regional metamorphism. In areas sampled for age determinations, the pluton generally has been metamorphosed to lower almandine-amphibolite facies and in many instances to upper almandine-amphibolite facies. The Ham pluton is considered to be one of the "older" plutons in the study area because it predates the main metamorphic event and some of the associated deformation. The pluton is also more deformed than the other plutons in the area. The objective of dating the Ham pluton was to obtain an emplacement age on one of the older "granitic" plutons in the study area, a minimum age for the deposition of the volcanic and sedimentary strata, and a minimum age for some of the deformation.

#### 4.4. File Gneiss Dome

The File gneiss dome is a complex of granitoid gneisses exposed in a domal structure at the north end of File Lake. These rocks consist of fine to medium-grained foliated gneisses that have been derived by recrystallization and metasomatism of arkosic gneisses of the Missi Group. They were initially described as granites by Harrison (1949) but are believed by Bailes (in preparation) to have formed by metasomatism and recrystallization during

regional metamorphism. The objective of dating this unit was to obtain its age of metamorphism, which is the age of the main regional metamorphic event that has affected the Kisseynew sedimentary gneiss belt.

#### 4.5. Late Pegmatite Dykes

The youngest unit dated consists of post-kinematic pegmatite dykes, which postdate all other rocks in the study area. They are randomly distributed and show no spatial association with igneous bodies, such as the Ham pluton, or metamorphic complexes, such as the File gneiss dome. Both bodies are intruded by these pegmatite dykes. The pegmatite dykes are composed mainly of perthitic microcline with minor quartz, plagioclase, and biotite. The age of the pegmatite dykes gives the age(s) of the youngest observed intrusive event in the study area.

## CHAPTER 5. EXPERIMENTAL TECHNIQUES

### 5.1. Sample Collection, Preparation, and Selection

The samples analyzed in this study were collected by A. H. Bailes while mapping the File-Morton-Woosey Lakes area for the Manitoba Mines Branch during the summers of 1970 and 1971. Sample stations are shown in Figure 2. Individual sample descriptions are given in Appendix I.

Weathered surfaces were trimmed from the individual samples prior to sample preparation. Each sample was crushed by a jaw crusher and pulverized by a Bleuler grinder to approximately 100 to 200 mesh size. A representative aliquot of approximately 100 g. was obtained by "cone and quartering" the bulk pulverized rock powder, and stored in a two ounce sample bottle.

Sample selection for mass spectrometric analysis was based on: 1) the value of the sample Rb/Sr ratio that was calculated from preliminary x-ray fluorescence analysis of rubidium and strontium concentrations; and, 2) the degree of sample weathering.

All samples prepared for mass spectrometric analyses were for whole rock analysis, with the exception of the late pegmatite dykes. K-feldspar mineral concentrates were prepared from the pegmatite samples.



## 5.2. Isotope Dilution Analysis

The method of stable isotope dilution analysis was used to determine the concentration of strontium and the  $^{87}\text{Sr}/^{86}\text{Sr}$  ratio from spiked strontium analysis, and to determine the concentration of rubidium from rubidium analysis. A detailed discussion of the method is given in Appendix III.

## 5.3. Chemical Procedure

A detailed description of the chemical procedure used in this study is outlined in Appendix II.

Briefly, three accurately weighed aliquots of a sample were dissolved and digested separately in teflon beakers: one portion was used for spiked strontium analysis, the second for unspiked strontium analysis, and the third for rubidium analysis. Dissolution and digestion was effected by refluxing each aliquot with hydrofluoric acid and perchloric acid. After digestion, a predetermined volume of the rubidium spike solution was added volumetrically to one portion, and a predetermined volume of the strontium spike solution was added to a second portion. The respective mixtures were equilibrated by refluxing the resulting solutions with hydrochloric acid. No spike solution was added to the third portion, which was to be used for direct determination of the  $^{87}\text{Sr}/^{86}\text{Sr}$  ratio present in the sample.

No further extraction or purification, such as cation exchange chromatography, was made on the rubidium

portion. The resulting rubidium residue, in the chloride form, was analyzed mass spectrometrically.

After the spiking procedure, the two aliquots used for strontium analysis were treated similarly. The alkali perchlorates were leached by methanol and were removed from solution in a precipitate form by centrifuging the resulting slurry. Further purification of the strontium fraction was attained by cation exchange chromatography. The resulting strontium sample was analyzed in the chloride form as purified strontium chloride.

#### 5.4. Mass Spectrometry

A triple filament solid source assembly, single-focusing, sector-type mass spectrometer with a 10-inch radius of curvature and a ninety degree deflexion was used to determine all rubidium and strontium isotope analysis. The instrument was described by Penner (1970) and by Anderson (1974). A detailed discussion of the operating procedure is given in Appendix IV.

#### 5.5. Analytical Precision

Two interlaboratory standards were used to monitor the precision and accuracy of analyses made. Replicate analyses of the Eimer and Amend (E&A)  $\text{SrCO}_3$  standard (lot no. 492 327) were made to determine the precision in the measurement of  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios obtained from strontium analysis of samples. To determine the precision and accuracy

in the measurement of rubidium and strontium concentration obtained from spiked rubidium and spiked strontium analyses, replicate analyses of the NBS 70a K-feldspar standard (Compston et al., 1969) were made separately for rubidium and strontium analyses.

Periodic rubidium and strontium "blank" analyses were made to monitor contamination levels that may be present in rubidium and strontium analyses of samples.

## CHAPTER 6. ANALYTICAL RESULTS

### 6.1. Rubidium and Strontium Standards

Twenty-six replicate analyses of the Eimer and Amend SrCO<sub>3</sub> standard (lot No. 492 327) were made over a twenty-four month period to monitor the precision in the measurements of the <sup>87</sup>Sr/<sup>86</sup>Sr ratio in both spiked and unspiked strontium analyses. The average value of the <sup>87</sup>Sr/<sup>86</sup>Sr ratio was 0.7082 ± 0.0006 (1 sigma), and the standard deviation of a single measurement was 0.12% (1 sigma).

The precision and accuracy in the measurements of rubidium and strontium concentration determinations was obtained from replicate analyses of the NBS 70a K-feldspar standard. Data for the replicate analyses of this standard is listed in Table 3. The precision of the mean value (528.5 ppm) for eight rubidium analyses was 0.23% (2 sigma). The five total strontium concentration analyses yield a precision on the mean value (65.59 ppm) of 1.25% (2 sigma). The precision on the mean value (1.1980) for the determination of <sup>87</sup>Sr/<sup>86</sup>Sr ratio from spiked strontium analyses was 0.23% (2 sigma).

Student's "t" distribution statistics with (n-2) degrees of freedom, where n is the number of analyses, was

Table 3. Analytical Results for the NBS 70a II K-Feldspar Standard

Rubidium Data

| <u>Analyst</u> <sup>1</sup> |         | <u>Rb (ppm)</u> |
|-----------------------------|---------|-----------------|
| GRJ                         | 18/7/71 | 528.8           |
| "                           | 28/8/72 | 529.7           |
| "                           | 29/8/72 | 530.9           |
| "                           | 7/12/72 | 526.9           |
| RKA                         | 29/1/73 | 528.9           |
| PRB                         | 27/4/73 | 526.7           |
| "                           | 9/5/73  | 529.8           |
| "                           | 20/6/73 | <u>526.1</u>    |
| mean:                       |         | 528.5 ± 1.7 ppm |

Strontium Data

| <u>Analyst</u> <sup>1</sup> | <u>Date</u> | <u>Total Sr (ppm)</u> | <u><sup>87</sup>Sr*2 (ppm)</u> | <u><sup>86</sup>Sr (micromoles/g)</u> | <u>(<sup>87</sup>Sr/<sup>86</sup>Sr)N<sup>3</sup></u> |
|-----------------------------|-------------|-----------------------|--------------------------------|---------------------------------------|---|
| RKA                         | 31/1/73     | 66.11                 | 3.037                          | 0.07000                               | 1.1983  |
| "                           | 29/3/73     | 66.41                 | 3.049                          | 0.07134                               | 1.1980  |
| "                           | 21/5/73     | 65.14                 | 3.055                          | 0.06987                               | 1.2093  |
| "                           | 29/7/73     | <u>65.40</u>          | 3.015                          | 0.07024                               | <u>1.2001</u>   |
|                             |             | 65.77 ± 0.59 ppm      |                                |                                       | 1.2014 ± 0.0053                                       |

<sup>1</sup>Analyst: RKA (R. Anderson), PRB (P. Beaudoin), GRJ (G. Josse).

<sup>2</sup>Radiogenic Sr.

<sup>3</sup> <sup>87</sup>Sr/<sup>86</sup>Sr values normalized to <sup>86</sup>Sr/<sup>88</sup>Sr = 0.1194.

Table 4. Blank Analysis

Rb Data

| <u>Analyst</u> <sup>1</sup> | <u>Date</u> | <u>Rb</u><br><u>(micrograms)</u> | <u><sup>87</sup>Rb</u><br><u>(micrograms)</u> |
|-----------------------------|-------------|----------------------------------|---|
| GRJ                         | 24/7/72     | 0.038                            | 0.011   |
| "                           | 7/12/72     | 0.101                            | 0.028   |
| PRB                         | 8/5/73      | 0.014                            | 0.004   |
| GRJ                         | 6/6/73      | <u>0.028</u>                     | 0.008   |
| 0.045 ± 0.038 micrograms    |             |                                  |   |

Sr Data

| <u>Analyst</u> <sup>1</sup> | <u>Date</u> | <u>Sr</u><br><u>(micrograms)</u> |
|-----------------------------|-------------|----------------------------------|
| GRJ                         | 12/12/72    | 0.0024                           |
| "                           | "           | 0.0114                           |
| "                           | "           | <u>0.0173</u>                    |
| 0.0104 ± 0.0075 micrograms  |             |                                  |

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<sup>1</sup>Analysts: PRB (P. Beaudoin),  
GRJ (G. Josse).

used to convert the standard deviation to 2 sigma confidence limits (Spiegel, 1961, p.188).

### 6.2. Rubidium and Strontium Blank Analyses

To monitor the contamination levels present in rubidium and strontium analyses, rubidium and strontium blank analyses were made periodically. The determined contamination levels are listed in Table 4. The average contamination levels were:  $0.045 \pm 0.038$  micrograms Rb, and  $0.0104 \pm 0.0075$  micrograms Sr. The Rb "blank" correction was applied to the Elmes Island Formation because the average concentration level was low, (i.e., less than 10 ppm). The Rb "blank" correction was considered non-limiting, and hence negligible for the other units analyzed. Similarly, the Sr "blank" correction was found to be non-limiting and was considered negligible for the units analyzed.

### 6.3. Composition and Concentration of Spike Solutions

Isotope tracers enriched in the  $^{87}\text{Rb}$  isotope and isotope tracers enriched in the  $^{84}\text{Sr}$  isotope were supplied by Oak Ridge National Laboratories as salts of  $\text{RbCl}$  and  $\text{Sr}(\text{NO}_3)_2$ , respectively. The procedure for preparation of the respective spike solutions from these salts is given in Appendix II. The concentration of the strontium spike solution was calibrated with a gravimetrically prepared laboratory solution of  $\text{Sr}(\text{NO}_3)_2$  and the concentration of the rubidium spike solution was calibrated with a standard solution of

the NBS 984 standard RbCl. The isotope composition of each spike solution was measured directly on a mass spectrometer from dried aliquots of each spike solution. The concentrations and composition of each spike solution are listed in Table 5. During the course of this study, four solutions of the  $^{87}\text{Rb}$ -enriched spike solution and three solutions of the  $^{84}\text{Sr}$ -enriched spike solution were prepared.

#### 6.4. Duplicate Analyses

The standard deviation of replicate analyses was obtained by using the method of Youden<sup>1</sup>, a method that was illustrated by Reesman (1968, p.23). For example, replicate analyses for the Ham pluton yield a standard deviation on the mean of 0.83% and 0.28% for the analyses of rubidium and strontium concentration, respectively. From these coefficients of variation, a standard deviation on the mean of 0.88% for the determination of the  $^{87}\text{Rb}/^{86}\text{Sr}$  values was obtained. The standard deviation on the mean in the measurement of  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios from replicate analyses was 0.14%.

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<sup>1</sup>Youden (1951), p.16.

$$\text{standard deviation} = \pm \sqrt{\sum_{i=1}^n \frac{|1 - x_1^2|^2}{2n}}$$

where,

n = number of duplicate analyses,

$x_1^1, x_1^2$  = values for a duplicate analysis.



Table 5. Composition and Concentration of Spike Solutions

<sup>87</sup>Rb-Enriched Solutions

| <u>Spike</u> | <u>Date</u> | <u>Percent Composition</u> |                        | <u>Total Rb<br/>Concentration<br/>(micromoles/ml)</u> |
|--------------|-------------|----------------------------|------------------------|---|
|              |             | <u><sup>87</sup>Rb</u>     | <u><sup>85</sup>Rb</u> |   |
| IV           |             | 99.15                      | 0.85                   | 0.05898   |
| V            | Sept. 1971  | 99.15                      | 0.85                   | 0.05883   |
| VII          | July 1972   | 99.15                      | 0.85                   | 0.05902   |
| VII/2        | April 1973  | 99.14                      | 0.84                   | 0.05913   |

<sup>84</sup>Sr-Enriched Solutions

| <u>Spike</u> | <u>Date</u> |                        |                        |                        |                        | <u>Total Sr<br/>Concentration<br/>(micromoles/ml)</u> |
|--------------|-------------|------------------------|------------------------|------------------------|------------------------|---|
|              |             | <u><sup>84</sup>Sr</u> | <u><sup>86</sup>Sr</u> | <u><sup>87</sup>Sr</u> | <u><sup>88</sup>Sr</u> |   |
| IV/1         |             | 82.24                  | 3.71                   | 1.56                   | 12.49                  | 0.01110   |
| IV/2         | Aug. 1971   | 82.2                   | 3.67                   | 1.56                   | 12.5                   | 0.01110   |
| IV/3         | Dec. 1972   | 81.5                   | 3.66                   | 1.56                   | 12.1                   | 0.01126   |

## 6.5. Analytical Results from the File-Morton-Woosey Lakes Area

York's (1966) least squares cubic regression analysis program was used to calculate the respective isochrons. Constant percentage experimental errors of 0.15% and 1.5% were assigned to the  $^{87}\text{Sr}/^{86}\text{Sr}$  and  $^{87}\text{Rb}/^{86}\text{Sr}$  ratios, respectively, from which the weighting factors required for the program were obtained. These values were used in all isochron fits because the exact values used as weighting factors were found to have little effect on the determined age and intercept ( $^{87}\text{Sr}/^{86}\text{Sr}$ ) values. For each isochron, the error in age was quoted at 95% confidence limits (1.96 sigma), whereas the corresponding error quoted for the  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratio (i.e. the intercept value) was at 1.0 sigma limits. To convert the standard deviation of the intercept value to 95% confidence limits, Student's "t" distribution was employed using n-2 degrees of freedom, where n is the number of sample points regressed.

### 6.5.1. Elmes Island Formation

Seven samples were analyzed from the Elmes Island Formation of dacite flows (Table 6). A definitive isochron fitting of the sample points was not obtained because of the narrow spread in the range of the Rb/Sr values, the low mean value of the Rb/Sr ratio, and the excessive scatter of sample points about a best-fit least squares regression line. However, regression of four of the seven samples

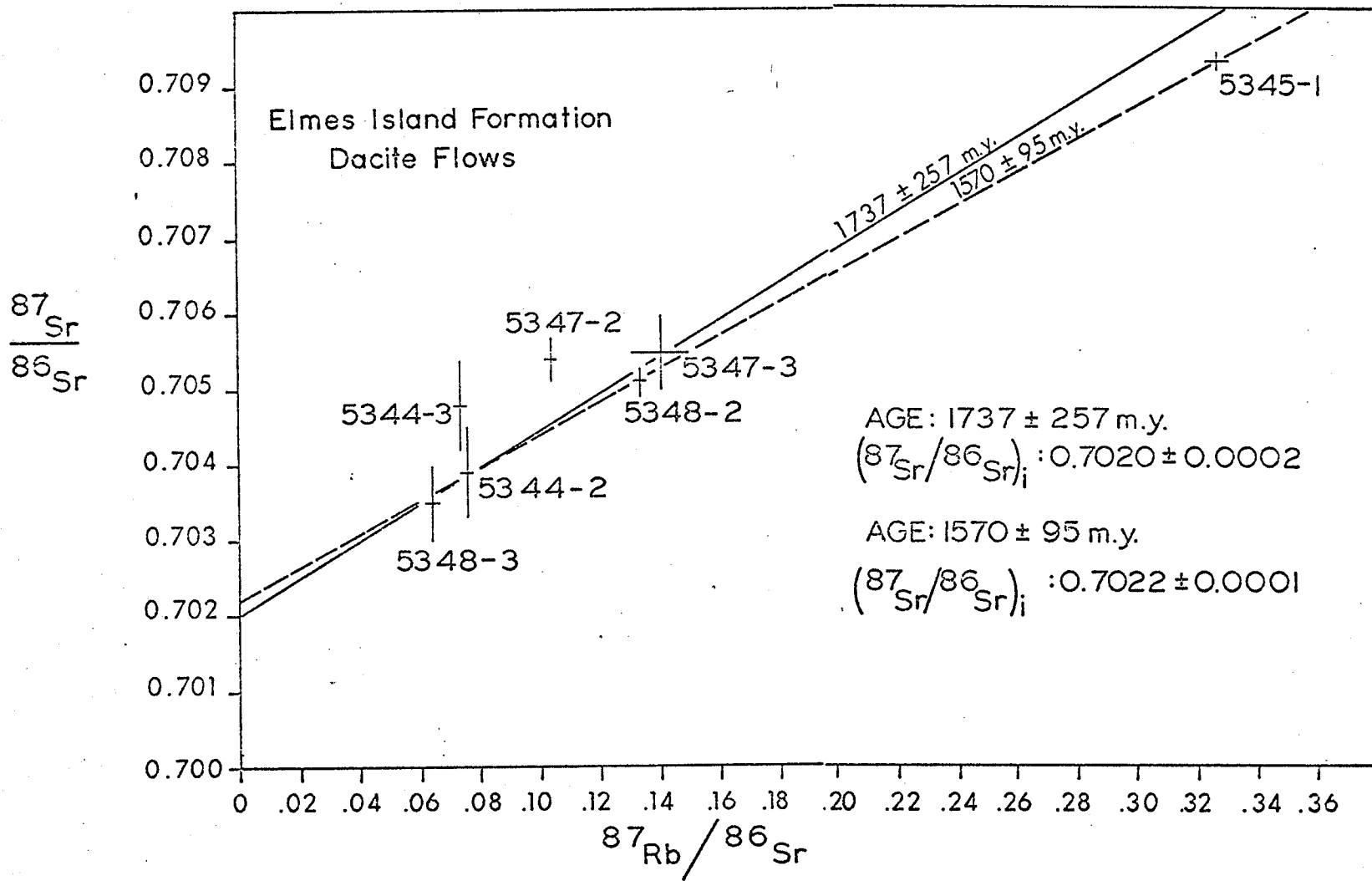
Table 6. Analytical Results from the Elmes Island Formation

| <u>Sample</u> | <u>Rb(ppm)</u>                      | <u>Sr(ppm)</u>                     | <u><math>^{87}\text{Rb}/^{86}\text{Sr}</math><br/>(atomic)</u> | <u><math>(^{87}\text{Sr}/^{86}\text{Sr})\text{N}^{\dagger}</math></u> | <u><math>(^{87}\text{Sr}/^{86}\text{Sr})\text{mean}</math></u> |
|---------------|-------------------------------------|------------------------------------|--|---|--|
| 5344-2        | 6.09<br><u>5.90</u><br>6.00±0.10    | 226.9                              | 0.0763   | 0.7039±0.0006   | 0.7039   |
| 5344-3        | 4.61                                | 179.9<br><u>182.0</u><br>181.0±1.0 | 0.0737   | 0.7055±0.0006<br>0.7052±0.0005<br>0.7040±0.0003*                      | 0.7048±0.0006  |
| 5345-1        | 18.60<br><u>18.34</u><br>18.47±0.13 | 163.2<br><u>165.5</u><br>164.4±1.1 | 0.3246   | 0.7094±0.0003<br>0.7092±0.0009*                                       | 0.7093±0.0001  |
| 5347-2        | 6.27<br><u>6.20</u><br>6.23±0.03    | 172.2                              | 0.1043   | 0.7057±0.0006<br>0.7051±0.0004*                                       | 0.7054±0.0003  |
| 5347-3        | 8.61<br><u>7.66</u><br>8.14±0.48    | 167.4                              | 0.1405   | 0.7050±0.0007<br>0.7063±0.0004*<br>0.7052±0.0002*                     | 0.7055±0.0005  |
| 5348-2        | 12.64<br><u>12.74</u><br>12.69±0.05 | 274.2<br><u>274.9</u><br>274.6±0.3 | 0.1332   | 0.7052±0.0004<br>0.7049±0.0005*                                       | 0.7051±0.0002  |
| 5348-3        | 6.91<br><u>6.78</u><br>6.84±0.06    | 306.5                              | 0.0646   | 0.7049±0.0006<br>0.7026±0.0007*<br>0.7027±0.0010*                     | 0.7035±0.0010  |

\*Unspiked Sr Analysis.

†All  $^{87}\text{Sr}/^{86}\text{Sr}$  values were normalized to the  $^{86}\text{Sr}/^{88}\text{Sr}$  value of 0.1194.

Figure 3. Partial regression of some of the analytical data from the Elmes Island Formation dacite flows. Regressing samples 5344-2, 5347-3, 5348-2, and 5348-3, an isochron age of  $1737 \pm 257$  m.y. and an initial ratio of  $0.7020 \pm 0.0002$  are defined (solid line). Regression of samples 5344-2, 5345-1, 5347-3, 5348-2, and 5348-3, gives an age of  $1570 \pm 95$  m.y. and an initial ratio of  $0.7022 \pm 0.0001$  (dashed line).



analyzed, (samples 5344-2, 5347-3, 5348-2, and 5348-3), yields an isochron age of  $1737 \pm 257$  m.y. and an  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratio of  $0.7020 \pm 0.0002$  (solid line, Figure 3). Regression of five of the seven samples analyzed, (samples 5344-2, 5345-1, 5347-3, 5348-2, and 5348-3), gives an age of  $1570 \pm 95$  m.y. and an  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratio of  $0.7022 \pm 0.0001$  (dashed line, Figure 3).

Samples 5344-3 and 5347-2 were omitted from either regression treatment because both points occur outside the assigned experimental error limits. This is illustrated by the error bars in Figure 3.

#### 6.5.2. Morton Porphyry

Eight samples were analyzed from the Morton porphyry (Table 7). An isochron age of  $1715 \pm 83$  m.y. and an  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratio of  $0.7027 \pm 0.0001$  are defined by the sample points (Figure 4).

#### 6.5.3. Ham Pluton

The seven samples analyzed from the Ham Pluton (Table 8) define an isochron age of  $1860 \pm 112$  m.y. and an  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratio of  $0.7019 \pm 0.0005$  (Figure 5). By deleting sample 1027-1 and regressing the remaining six samples, the resulting isochron defines an age of  $1880 \pm 85$  m.y. and an initial ratio of  $0.7019 \pm 0.0004$ . Although an improved definition of confidence limits is the result, sample 1027-1 should be retained in the

Table 7. Analytical Results from the Morton Porphyry

| <u>Sample</u> | <u>Rb</u><br><u>(ppm)</u>           | <u>Sr</u><br><u>(ppm)</u>          | <u><math>^{87}\text{Rb}/^{86}\text{Sr}</math></u><br><u>(atomic)</u> | <u><math>(^{87}\text{Sr}/^{86}\text{Sr})_{\text{N}}</math></u> <sup>†</sup> | <u><math>(^{87}\text{Sr}/^{86}\text{Sr})</math></u><br><u>Mean</u> |
|---------------|-------------------------------------|------------------------------------|--|---|--|
| 5180-1        | 12.06<br><u>12.54</u><br>12.30±0.24 | 540.7<br><u>537.7</u><br>539.2±1.5 | 0.0660   | 0.7052±0.0011*<br>0.7034±0.0003*  | 0.7043±0.0009  |
| 5180-3        | 8.23<br><u>7.84</u><br>8.03±0.21    | 550.4<br><u>547.0</u><br>548.7±1.7 | 0.0423   | 0.7034±0.0006<br>0.7043±0.0006<br>0.7038±0.0004*                            | 0.7038±0.0004  |
| 5180-5        | 16.16                               | 437.7                              | 0.1067   | 0.7052±0.0004*  | 0.7052   |
| 5181-1        | 9.84                                | 531.2                              | 0.0536   | 0.7035±0.0004<br>0.7042±0.0005*   | 0.7038±0.0003  |
| 5186-1        | 18.40                               | 439.1                              | 0.1211   | 0.7054±0.0008<br>0.7062±0.0004  | 0.7058±0.0004  |
| 5192-1        | 30.05                               | 222.4                              | 0.3905   | 0.7125±0.0007<br>0.7117±0.0004*   | 0.712±0.0004   |
| 5448-1        | 23.41<br><u>24.13</u><br>23.77±0.36 | 497.4<br><u>504.7</u><br>500.7±3.3 | 0.1372   | 0.7064±0.0007<br>0.7052±0.0004<br>0.7054±0.0004*                            | 0.7057±0.0005  |
| 5448-2        | 22.71                               | 507.9<br><u>519.1</u><br>513.5±5.6 | 0.1278   | 0.7068±0.0009<br>0.7054±0.0004*<br>0.7058±0.0007*                           | 0.7060±0.0005  |

\*Unspiked Sr analysis.

†All  $^{87}\text{Sr}/^{86}\text{Sr}$  values were normalized to  $^{86}\text{Sr}/^{88}\text{Sr} = 0.1194$ .

Figure 4: Rb-Sr whole rock isochron for the Morton  
Porphyry, a pre-to early kinematic intru-  
sion.



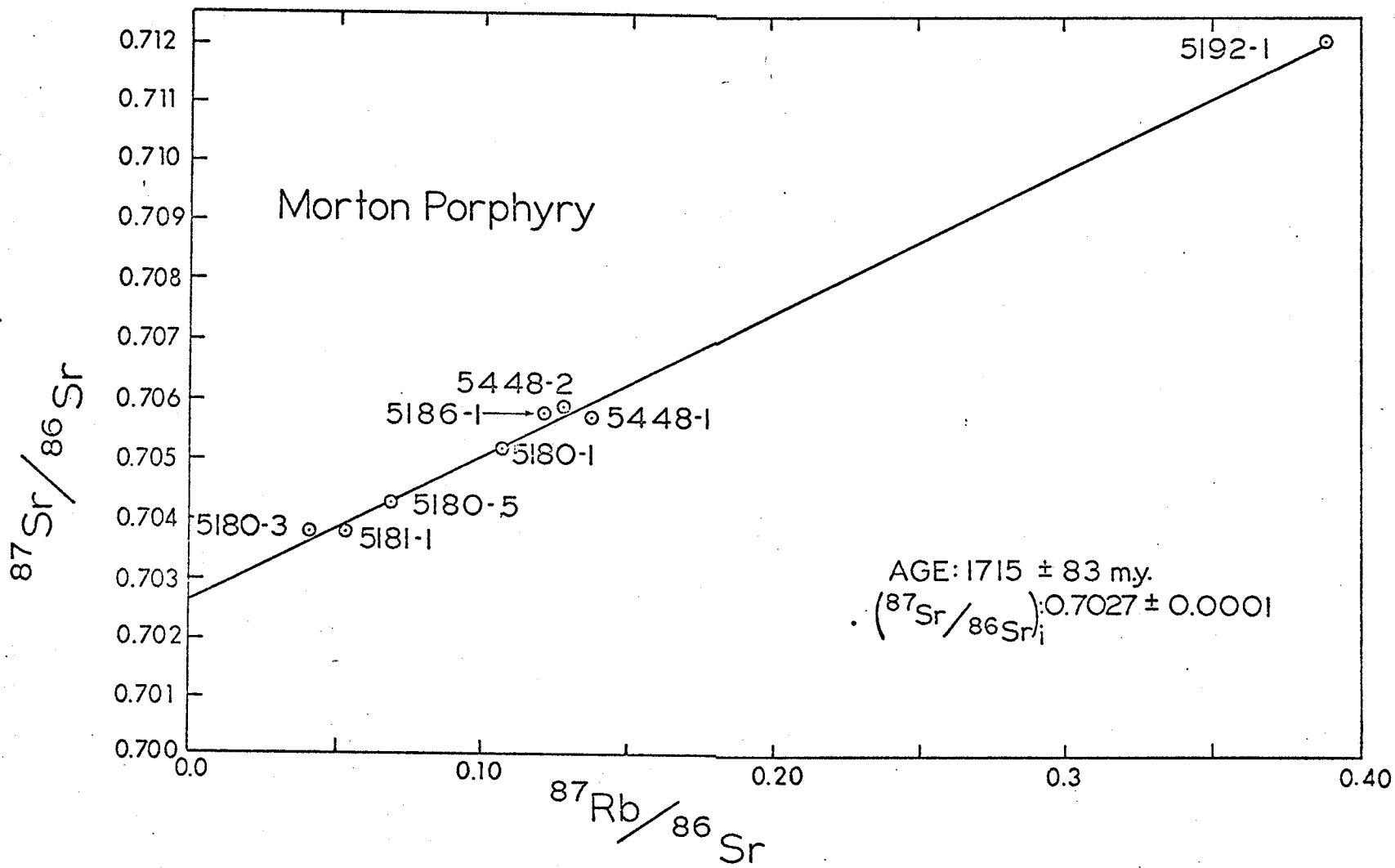


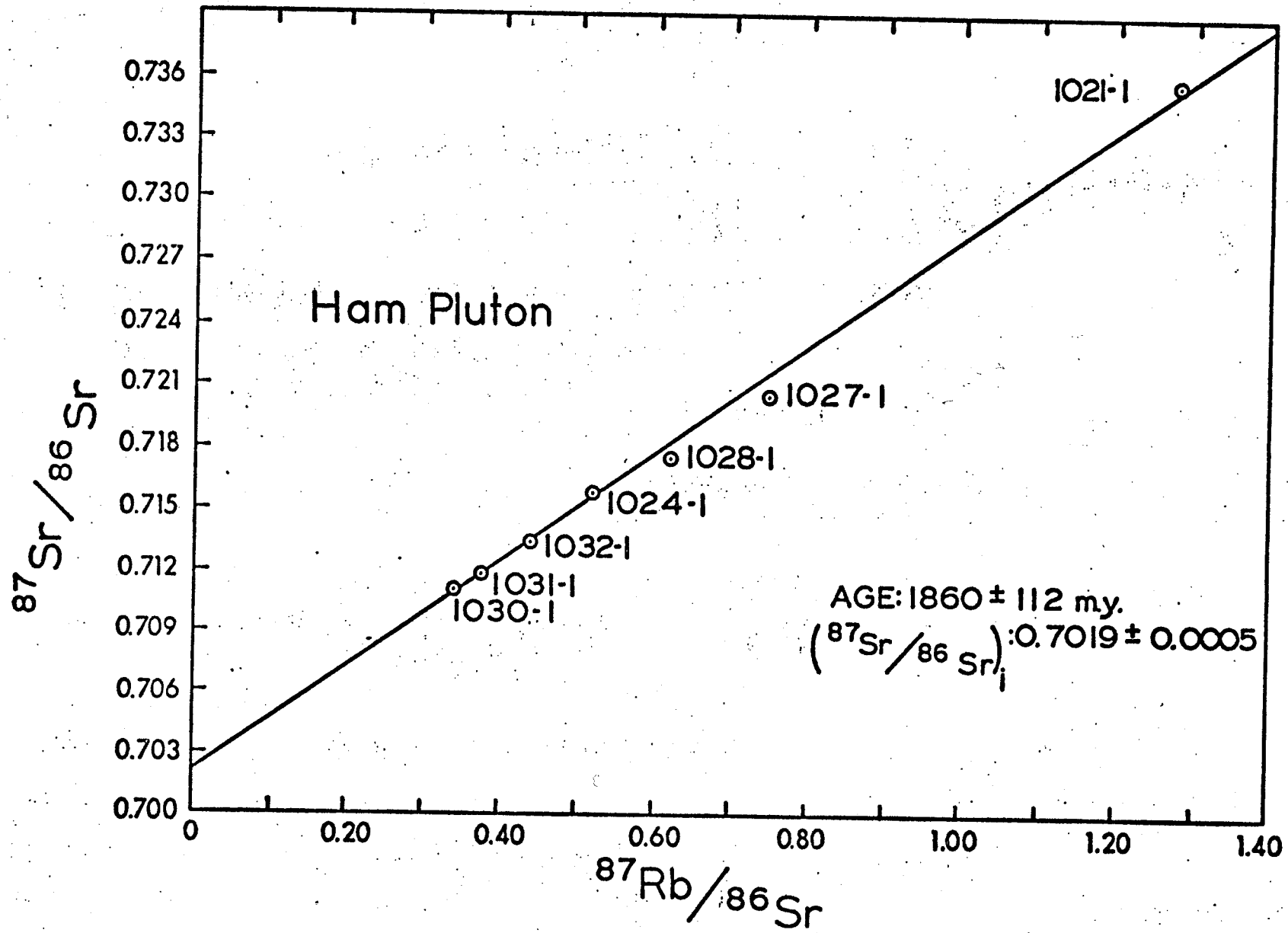
Table 8. Analytical Results from the Ham Pluton

| Sample | Rb<br>(ppm)                                    | Sr<br>(ppm)                        | $^{87}\text{Rb}/^{86}\text{Sr}$<br>(atomic) | $(^{87}\text{Sr}/^{86}\text{Sr})\text{N}^+$       | $(^{87}\text{Sr}/^{86}\text{Sr})$<br>Mean |
|--------|--|------------------------------------|---|---|---|
| 1021-1 | 122.5  | 276.6<br><u>276.8</u><br>276.7±0.1 | 1.2798<br><u>1.2790</u><br>1.2794           | 0.7361±0.0010<br>0.7359±0.0007<br>0.7370±0.0006*  | 0.7363±0.0004                             |
| 1024-1 | 48.1<br><u>48.4</u><br>48.3±0.2                | 270.2                              | 0.5150<br><u>0.5169</u><br>0.5159           | 0.7159±0.0010*                                    | 0.7159                                    |
| 1027-1 | 54.9<br><u>55.9</u><br>55.4±0.5                | 213.9                              | 0.7424<br><u>0.7549</u><br>0.7487           | 0.7204±0.0004<br>0.7209±0.0006*                   | 0.7206±0.0003                             |
| 1028-1 | 59.4   | 277.4                              | 0.6214                                      | 0.7188±0.0006<br>0.7165±0.0003*<br>0.7176±0.0004* | 0.7176±0.0007                             |
| 1030-1 | 42.8<br><u>43.2</u><br><u>42.4</u><br>42.8±0.3 | 374.1<br><u>373.0</u><br>373.5±0.5 | 0.3393                                      | 0.7105±0.0002<br>0.7117±0.0007                    | 0.7111±0.0006                             |
| 1031-1 | 47.9<br><u>48.3</u><br>48.1±0.2                | 374.8                              | 0.3714                                      | 0.7132±0.0006<br>0.7108±0.0009*                   | 0.7120±0.0012                             |
| 1032-1 | 49.2<br><u>49.9</u><br><u>48.6</u><br>49.2±0.5 | 324.0<br><u>322.9</u><br>323.0±1.0 | 0.4416                                      | 0.7139±0.0010<br>0.7127±0.0004*                   | 0.7133±0.0006                             |

\*Unspiked Sr run.

+All  $^{87}\text{Sr}/^{86}\text{Sr}$  values were normalized to  $^{86}\text{Sr}/^{88}\text{Sr} = 0.1194$ .

Figure 5. Rb-Sr whole rock isochron for the Ham pluton,  
a syn- to late kinematic intrusion.



regression because the sample lies within the experimental error limits.

#### 6.5.4. File Gneiss Dome

Ten samples were analyzed from the File gneiss dome (Table 9). An isochron age of  $1760 \pm 43$  m.y. and an  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratio of  $0.7038 \pm 0.0008$  are defined by the isochron (Figure 6).

#### 6.5.5. Late Pegmatite Dykes

The potassium feldspar mineral concentrates of two pegmatite samples from the pegmatite dykes were analyzed (Table 10). Assuming the  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratio to be 0.710, individual mineral ages of  $1741 \pm 55$  m.y. and  $1763 \pm 55$  m.y. were calculated for samples 966-1 and 1021-3, respectively.

Table 9. Analytical Results from the File Gneiss Dome

| Sample | Rb<br>(ppm)  | Sr<br>(ppm)  | $^{87}\text{Rb}/^{86}\text{Sr}$<br>(atomic) | $(^{87}\text{Sr}/^{86}\text{Sr})_{\text{N}^+}$ | $(^{87}\text{Sr}/^{86}\text{Sr})$<br>Mean |
|--------|--------------|--------------|---|--|---|
| 950-1  | 118.6        | 149.2        | 2.2959                                      | 0.7602±0.0014                                  | 0.7606±0.0004                             |
|        | <u>118.9</u> | <u>151.9</u> | <u>2.2620</u>                               | 0.7609±0.0010                                  |   |
|        | 118.8±0.2    | 150.6±1.4    | 2.2779                                      |  |   |
| 953-1  | 119.3        | 137.9        | 2.4963                                      | 0.7641±0.0012                                  | 0.7641                                    |
| 954-1  | 125.6        | 121.2        | 2.9778                                      | 0.7779±0.0006                                  | 0.7789±0.0010                             |
|        | <u>123.3</u> | <u>123.2</u> | <u>2.8924</u>                               | 0.7799±0.0003                                  |   |
|        | 124.5±1.2    | 122.0±1.0    | 2.9455                                      |  |   |
| 955-1  | 92.4         | 222.8        | 1.1892                                      | 0.7344±1.0010                                  | 0.7344                                    |
| 956-1  | 176.8        | 78.1         | 6.5521                                      | 0.8643±0.0011                                  | 0.8654±1.0011                             |
|        |              | <u>78.8</u>  | <u>6.4831</u>                               | 0.8665±0.0006                                  |   |
|        |              | 78.4±0.4     | 6.5176                                      |  |   |
| 957-1  | 112.7        | 184.1        | 1.7698                                      | 0.7473±0.0011                                  | 0.7473                                    |
|        | <u>113.3</u> |              | <u>1.7794</u>                               |  |   |
|        | 113.0±0.3    |              | 1.7746                                      |  |   |
| 958-1  | 130.0        | 119.9        | 3.1499                                      | 0.7842±0.0010                                  | 0.7838±0.0004                             |
|        | <u>130.6</u> | <u>119.7</u> | <u>3.1388</u>                               | 0.7834±0.0012*                                 |   |
|        | 130.3±0.3    | 119.8±0.1    | 3.1439                                      |  |   |
| 966-2  | 102.5        | 235.8        | 1.2567                                      | 0.7376±0.0012                                  | 0.7364±0.0012                             |
|        | <u>106.9</u> | <u>230.3</u> | <u>1.3404</u>                               | 0.7352±0.0007                                  |   |
|        | 104.7±2.2    | 233.1±2.7    | 1.2986                                      |  |   |
| 967-1  | 121.3        | 157.0        | 2.2151                                      | 0.7572±0.0010                                  | 0.7572                                    |
| 969-1  | 116.7        | 222.5        | 1.5164                                      | 0.7409±0.0009                                  | 0.7409                                    |
|        | <u>116.0</u> |              | <u>1.5062</u>                               |  |   |
|        | 116.3±0.3    |              | 1.5112                                      |  |   |

\*Unspiked Sr analysis.

+All  $^{87}\text{Sr}/^{86}\text{Sr}$  values were normalized to  $^{86}\text{Sr}/^{88}\text{Sr} = 0.1194$ .

Figure 6. Rb-Sr whole rock isochron of the File gneiss dome, a granitoid gneiss complex that was formed by the main regional metamorphic event.

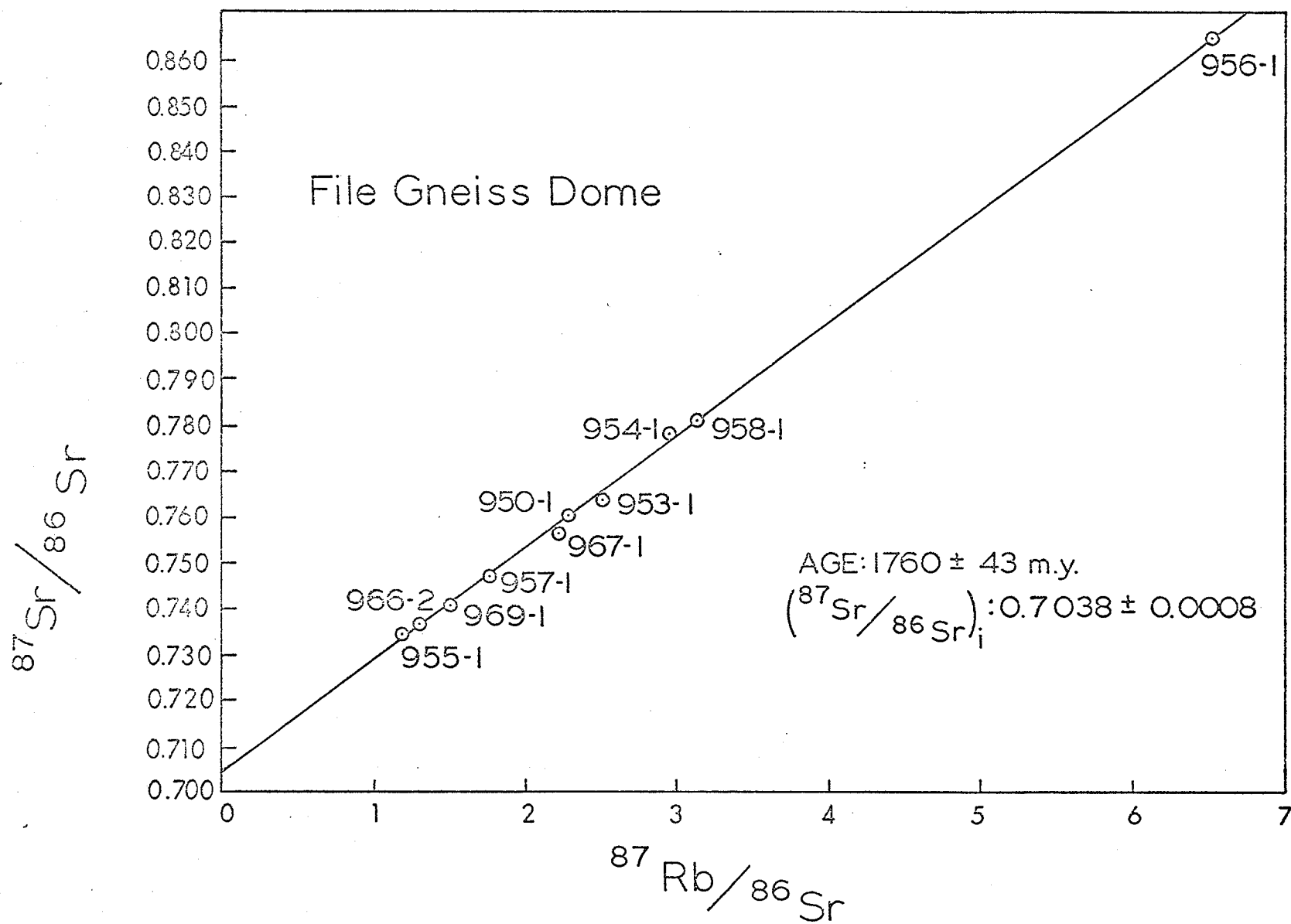




Table 10. Analytical Results from the Late Pegmatite Dykes

| <u>Sample</u> | <u>Rb</u><br><u>(ppm)</u> | <u>Total Sr</u><br><u>(ppm)</u> | <u><sup>87</sup>Sr*</u><br><u>(ppm)</u> | <u><sup>86</sup>Sr</u><br><u>(micromoles/g)</u> | <u>(<sup>87</sup>Rb/<sup>86</sup>Sr)</u><br><u>Atomic</u> | <u>(<sup>87</sup>Sr/<sup>86</sup>Sr)<sub>N</sub></u> | <u>Age<sup>†</sup>(m.y.)</u> |
|---------------|---------------------------|---------------------------------|---|---|---|--|------------------------------|
| 966-1         | 325.4                     | 72.1                            | 2.268                                   | 0.07862   | 13.461  | 1.0393±0.0010  | 1741 ± 55                    |
| 1021-3        | 363.1                     | 53.8                            | 2.568                                   | 0.05773   | 20.458  | 1.2185±0.0018  | 1763 ± 55                    |

\*Radiogenic Sr.

†Age calculations were obtained by assuming the <sup>87</sup>Sr/<sup>86</sup>Sr initial ratio to be 0.710.

## CHAPTER 7. INTERPRETATION OF ISOTOPIC DATA

Rubidium-strontium age determinations obtained from the File-Morton-Woosey Lakes area of the Flin Flon volcanic belt are listed in Table 11. The  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratios from these results are plotted in Figure 7, which is an isotope evolution diagram of strontium with time in the source regions of oceanic basalt and of continental crust. For comparative purposes, the initial ratios obtained by Mukherjee *et al.* (1971) at Flin Flon and by Coleman (1970) at Hanson Lake are included in Figure 7. This diagram can sometimes be used to distinguish between primary and recycled crustal material and to suggest a possible source region for the corresponding magma (Faure and Powell, 1972; Bruwash *et al.*, 1973).

### 7.1. Discussion of Isotopic Data and Age Relations

#### 7.1.1. Elmes Island Formation

Dacite flows of the Elmes Island Formation represent the felsic member of the 'second' volcanic cycle of Amisk volcanism recognized in the study area (Table 1). Because of the narrow range of Rb/Sr values and the low mean value of Rb/Sr ratios in this unit, least-squares regression of the sample points is characterized by a considerable scatter of sample points about the best-fitting mean

Table 11. Rb-Sr Age Determinations, File-Morton-Woosey  
Lakes Area

| <u>Unit Dated</u>   | <u>Age (95% Confidence)</u> | <u>(<sup>87</sup>Sr/<sup>86</sup>Sr) initial</u> |
|---|-----------------------------|--|
| Elmes Island Formation <sup>1</sup><br>(54°50'N by 100°27'W)  | 1737 ± 257 m.y.             | 0.7020 ± 0.0002                                  |
| Morton porphyry <sup>1</sup><br>(54°55'N by 100°26'W)         | 1715 ± 83 m.y.              | 0.7027 ± 0.0001                                  |
| Ham pluton <sup>1</sup><br>(54°55'N by 100°15'W)              | 1860 ± 112 m.y.             | 0.7019 ± 0.0005                                  |
| File gneiss dome <sup>2</sup><br>(54°55'N by 100°20'W)        | 1760 ± 43 m.y.              | 0.7038 ± 0.0008                                  |
| Late pegmatite dykes <sup>3</sup><br>calculated mineral ages: |                             |  |
| sample 966-1  | 1741 ± 55 m.y.              | 0.710, assumed                                   |
| sample 1021-3   | 1763 ± 55 m.y.              | 0.710, assumed                                   |

<sup>1</sup>Units representing the Flin Flon volcanic belt.

<sup>2</sup>Units representing the Kisseynew sedimentary gneiss belt.

<sup>3</sup>Late pegmatite dykes that intrude both the Ham pluton and the File gneiss dome.

isochron. Consequently, the data does not yield a definitive isochron (Figure 3), and the resulting age determination has a large uncertainty. For example, regression of four of seven samples analyzed, (samples 5344-2, 5347-3, 5348-2, and 5348-3), yields an isochron with an apparent age of  $1737 \pm 257$  m.y. and an  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratio of  $0.7020 \pm 0.0002$ . The three samples, (samples 5344-3, 5345-1, and 5347-2), that were excluded lie outside the experimental limits of error. If sample 5345-1 is included with the four previously regressed samples, the resulting isochron calculation yields an age of  $1570 \pm 95$  m.y. and an initial ratio of  $0.7022 \pm 0.0001$ . However, this apparent age of 1570 m.y. is considered to be too low for the Amisk Group and is discarded.

The radiometric age of  $1737 \pm 257$  m.y. for the Elmes Island Formation may represent the age of deposition of the dacite flows, or an updated age due to regional metamorphism during the Hudsonian orogeny. However, it is difficult to distinguish between the two possible interpretations because of the large uncertainty associated with the determined age. Since the age of  $1737 \pm 257$  m.y. is less than that of  $1860 \pm 112$  m.y. for the Ham pluton, a pluton that intrudes the volcanic strata but predates the regional metamorphism, and since the age of  $1737 \pm 257$  m.y. statistically overlaps the metamorphic age of  $1760 \pm 43$  m.y. for the File gneiss dome, the apparent age of  $1737 \pm 257$  m.y. can be interpreted to be a metamorphic age for the Elmes Island Formation that was updated during regional metamorphism.

The  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratio of  $0.7020 \pm 0.0002$  plots within the basalt field of Figure 7, suggesting that the parent material for the Elmes Island Formation dacite flows may have been derived from a similar source region. This does not preclude any crustal pre-history that may have occurred between the geological age of deposition for the dacite flows and its radiometric age of  $1737 \pm 257$  m.y.

#### 7.1.2. Morton Porphyry

The Morton porphyry is considered to have been intruded as pre- to early kinematic hypabyssal intrusions, that predate the main periods of deformation and metamorphism in the study area, but postdate the volcanic rocks in which they are set. However, according to Bailes (personal communication), its geological age relative to the gabbroic sills of the Josland Formation (Table 1) is not well established, and it could in fact be older. The porphyry bodies appear to be high level intrusions and may be subvolcanic, as indicated by their texture and mode of occurrence as numerous small dykes and plugs. The determined radiometric age of  $1715 \pm 83$  m.y. gives an age that is younger than the age of  $1860 \pm 112$  m.y. determined for the Ham pluton, a syn- to late kinematic intrusion that postdates the Morton porphyry. At 95% confidence limits, there is a statistical overlap of both ages, but there also is a statistical overlap with the age of  $1760 \pm 43$  m.y.

determined for the File gneiss dome. The radiometric age obtained for the Morton porphyry does not preclude a pre- to early kinematic time of intrusion. However, the radiometric age may also represent an updating of the rocks in response to the main regional metamorphic event that affected the area. The Morton porphyry has been foliated and regionally metamorphosed to upper greenschist facies conditions. The small dykes and plugs of tonalitic quartz feldspar porphyry composition that comprise the Morton porphyry could have responded as 'open' geochemical systems at this time. Therefore, the determined radiometric age of  $1715 \pm 83$  m.y. for the Morton porphyry should be interpreted as a possible minimum age of intrusion.

The  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratio of  $0.7027 \pm 0.0001$  (Figure 4) plots within the basalt field of Figure 7, suggesting that the parent material for the Morton porphyry may have been derived from a similar source region. The interpreted source region for oceanic basalts is the upper mantle (Faure and Powell, 1972, p.24). However, the initial ratio of 0.7027 does not preclude the possibility of a crustal pre-history for the Morton porphyry that may have occurred between its geological age of emplacement and its determined radiometric age. For example, if one assumes a possible parent material having a  $^{87}\text{Sr}/^{86}\text{Sr}$  ratio of 0.7020 and a maximum Rb/Sr ratio of 0.1, then the Morton porphyry could conceivably have had a crustal pre-history of approximately 250 m.y. prior to isotopic homogenization 1715 m.y.

ago as a result of metamorphism.

### 7.1.3. Ham Pluton

The Ham pluton is considered to be one of the older syn- to late kinematic 'granitic' plutons in the study area. The pluton is foliated and probably was metamorphosed to lower almandine-amphibolite facies, or even in part to upper almandine-amphibolite facies. Since intrusion of the pluton predates the main regional metamorphic event, but postdates Amisk volcanism and Missi sedimentation, its radiometric age of  $1860 \pm 112$  m.y. gives a minimum age for Amisk volcanism and Missi sedimentation, and all other rocks which predate the Ham pluton in the study area, such as the Morton porphyry. It also provides a minimum age for any early deformation that predates the Ham pluton.

The  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratio of  $0.7019 \pm 0.0005$  (Figure 5) for the Ham pluton plots within the basalt field of Figure 7. The low initial ratio suggests that the Ham pluton could not have been derived from much older continental crust material, but rather the pluton was derived from a source depleted in rubidium relative to strontium, such as the mantle. The value of the initial ratio is similar to the  $^{87}\text{Sr}/^{86}\text{Sr}$  ratio of 0.7018 which was calculated for the source regions of oceanic basalt having an average Rb/Sr ratio of 0.025 (Faure and Powell, 1972, p.25). However, the low initial ratio for the Ham pluton does not preclude the possibility that the pluton was affected by

the main regional metamorphic event or by a cooling period of some duration of time following intrusion. Either possibility is suggested by the scatter of sample points about the isochron that is indicated by the uncertainty of  $\pm 112$  m.y. in the determined radiometric age. As a result, the determined age of  $1860 \pm 112$  m.y. is interpreted as a minimum age of intrusion for the Ham pluton.

#### 7.1.4. File Gneiss Dome

The File gneiss dome is a granitoid complex derived from the Missi Group arkosic sedimentary rocks by recrystallization and potassium metasomatism. The radiometric age of  $1760 \pm 43$  m.y. is interpreted as the age of metamorphism for the File gneiss dome and the age of the main regional metamorphic event that affected the Kisseynew sedimentary gneiss belt. The radiometric age is comparable with the K-Ar biotite age of 1735 m.y. (GSC 60-73, Lowdon, 1961) obtained from a paragneiss at Kisseynew Lake, Bruce's (1918) type area for the Kisseynew gneisses. Both ages probably represent the age of the main regional metamorphic event of the Hudsonian Orogeny.

The  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratio of  $0.7038 \pm 0.0008$  (Figure 6) for strata of the File gneiss dome statistically overlaps the source regions for oceanic basalt and the source region for continental crust in Figure 7. The moderately high initial ratio appears to reflect this unit's complex crustal pre-history which includes metasomatic and



metamorphic generation from Missi Group arkosic sedimentary rocks.

#### 7.1.5. Late Pegmatite Dykes

The late pegmatite dykes represent the youngest intrusive event(s) of the study area. They postdate the main regional metamorphic event. The dykes intrude foliated bodies of rocks, such as the Ham pluton and the File gneiss dome, that were affected by the main regional metamorphism. By assuming the value of the  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratio to be 0.710, individual mineral ages of  $1741 \pm 55$  m.y. and  $1763 \pm 55$  m.y. were calculated for the two K-feldspar mineral concentrates analyzed. Thus, the individual mineral ages of 1741 m.y. and 1763 m.y. represent the best approximation of the age of the youngest intrusive event for the study area.

#### 7.2. $^{87}\text{Sr}/^{86}\text{Sr}$ Initial Ratios from the Flin Flon Volcanic Belt

In the application of strontium isotopes to the petrogenesis of igneous rocks,  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratios can be used to suggest the source regions for the material from which the igneous rocks crystallized. The value of the initial ratio depends upon the nature, the previous history, and in particular, the Rb/Sr ratio of the source regions. The nature of the source regions is represented by two generalized fields of initial ratios that are outlined in Figure 7, a development diagram of strontium isotope evolution with time in the source regions of oceanic basalt and the continental crust. The initial ratios of uncontaminated

Figure 7. Sr isotope evolution diagram for the Flin Flon volcanic belt, adapted from Faure and Powell (1972, p.26). The confidence limits for the  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratios are expressed at one sigma levels.

File-Morton-Woosey Lakes area (this work)

J-1 Elmes Island Formation dacite flows:  
1737  $\pm$  257 m.y., 0.7020  $\pm$  0.0002.

J-2 Morton porphyry: 1715  $\pm$  83 m.y., 0.7027  $\pm$  0.0001.

J-3 Ham pluton: 1860  $\pm$  112 m.y., 0.7019  $\pm$  0.0005.

J-4 File gneiss dome: 1760  $\pm$  43 m.y., 0.7038  $\pm$  0.0008.

Flin Flon area (Mukherjee et al., 1971)

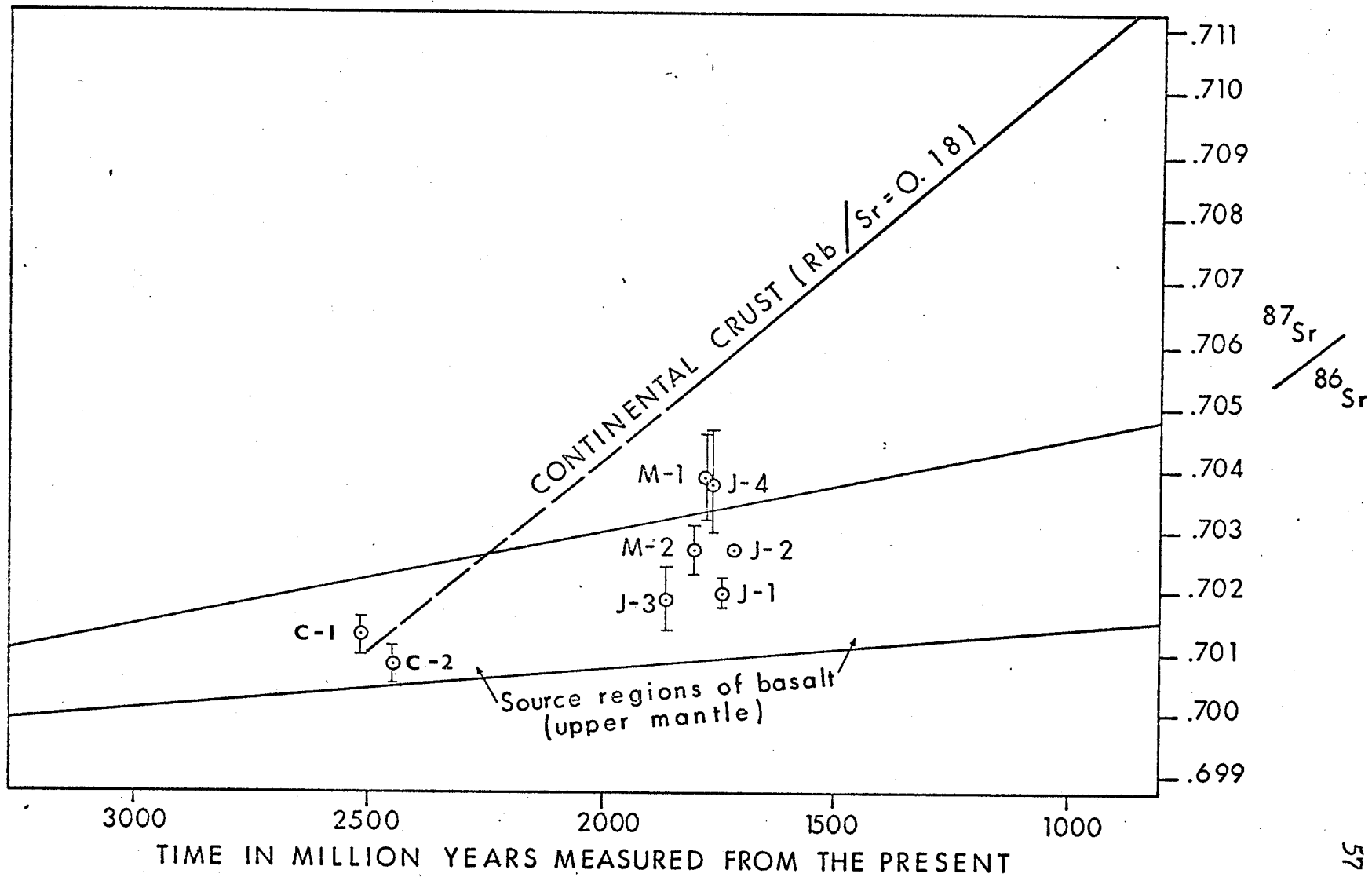
M-1 Amisk volcanics: 1775  $\pm$  89 m.y., 0.7039  $\pm$  0.0007.

M-2 Annabel Lake pluton: 1805  $\pm$  14 m.y.,  
0.7027  $\pm$  0.0004.

Hanson Lake area (Coleman, 1970)

C-1 Metavolcanics: 2521  $\pm$  60 m.y., 0.7013  $\pm$  0.0003.

C-2 Granitic pluton: 2446  $\pm$  16 m.y., 0.7008  $\pm$  0.0003.



oceanic basalt are considered to be the same as that of its source region in the upper mantle. The 'basalt field' in Figure 7 is defined by the initial ratios of oceanic basalt, that range in value from 0.702 to 0.706 and have a mean value of 0.7037. On the other hand, the "continental crust field" is more difficult to define because the  $^{87}\text{Sr}/^{86}\text{Sr}$  and Rb/Sr ratios are more difficult to determine. The  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios of the continental crust are quite variable since its range of values are determined by such variable factors as the Rb/Sr ratios present and the crustal history of the rocks. Faure and Powell (1972, p.26) obtained an estimate of 0.719 for the average  $^{87}\text{Sr}/^{86}\text{Sr}$  ratio in the continental crust having an average Rb/Sr ratio of 0.18. Initial ratios of igneous rocks that plot within the continental crust field of Figure 7 suggest that the parent material may have been derived from the reconstitution of older sialic material, from primary igneous material that was contaminated by a considerable amount of older crustal material, or, from the isotopic equilibration of strontium on a whole-rock scale by a metamorphic event.

For a more detailed discussion on the significance and use of  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratios as 'geological tracers' in the petrogenesis of igneous rocks, the reader is referred to the review by Faure and Powell (1972) and the application by Burwash *et al.* (1973).

In general, the low initial ratios obtained for the Elmes Island Formation (0.7020), the Morton porphyry (0.7027), and the Ham pluton (0.7019) plot within the basalt field of

Figure 7. The low initial ratios suggest that these rocks could not have been derived from much older continental crustal material, but rather from a source region depleted in rubidium relative to strontium, such as the upper mantle. However, as previously discussed, this interpretation does not preclude the possibility of any crustal pre-history that may have occurred between their geological ages of emplacement and the main regional metamorphic event that may have caused these units to undergo partial or complete isotopic homogenization.

The exception to this trend of low ratios is that obtained for the File gneiss dome (0.7038) which statistically overlaps the basalt and continental crust fields in Figure 7. The moderately high initial ratio indicates a crustal pre-history prior to isotopic homogenization of the Missi Group arkosic sedimentary rocks during regional metamorphism.

The  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratios obtained from volcanic and plutonic units by Mukherjee et al. (1971) at Flin Flon and by Coleman (1970) at Hanson Lake also plot within the basalt field in Figure 7. Coleman's Archean ages and Mukherjee's Aphebian ages are considered, by them, to represent igneous ages for the respective rock units. In discussing the significance of the low initial ratios obtained at Flin Flon, Mukherjee (1971, p.144) states that "it may be interpreted that previously existing continental crustal material did not contribute significantly to the Annabel Lake granodiorites or to Amisk volcanism."

## CHAPTER 8. SUMMARY AND CONCLUSIONS

Radiometric age determinations have been obtained by the rubidium-strontium whole rock isochron method from the File-Morton-Woosey Lakes area. The study area, which is located within the southeastern portion of the Churchill geological province of the Canadian Shield, straddles the boundary between two major easterly trending lithological belts: the Flin Flon volcanic belt (in the south), and the Kisseynew sedimentary gneiss belt (in the north).

The radiometric age of  $1860 \pm 112$  m.y. obtained for the Ham pluton, a syn- to late kinematic intrusion, is interpreted to be its minimum age of emplacement. The age is considered Aphebian, according to Stockwell's (1964, 1972) definition. It also is considered to provide a minimum age for the extrusion of the Amisk volcanic strata and deposition of the Missi sedimentary strata, rocks which it intrudes. In addition, it gives a minimum age for all other rocks in the study area that predate the Ham pluton, such as the Morton porphyry, and a minimum age for any early deformation that predates the pluton.

The age of regional metamorphism that affected the study area during the Hudsonian orogeny is given by the radiometric age of  $1760 \pm 43$  m.y. that was determined for the File gneiss dome, a granitoid gneiss complex.

The radiometric ages determined for the dacite flows of the Elmes Island Formation ( $1737 \pm 257$  m.y.), representing the Amisk volcanics, and for the Morton porphyry ( $1715 \pm 83$  m.y.), representing pre- to early kinematic intrusions, are interpreted to be probably metamorphic ages and not emplacement ages, and that these units were updated during regional metamorphism. The metamorphic age interpretation is preferred because: 1) the determined radiometric ages are younger than that obtained for the Ham pluton, an intrusion that postdates the Elmes Island Formation and the Morton porphyry, but predates the main regional metamorphic event; 2) both units were metamorphosed under greenschist facies conditions during regional metamorphism; and, 3) both ages statistically overlap the metamorphic age obtained for the File gneiss dome.

The late pegmatite dykes are post-kinematic intrusions that postdate all other rocks in the study area. The calculated mineral ages of  $1741 \pm 55$  m.y. and  $1763 \pm 55$  m.y. give the best approximation of the age of the youngest intrusive event in the study area.

The age determinations for the rock units analyzed can be subdivided according to their relation to the geological events recognized in the study area (Table 1). They are: 1) the Aphebian age of emplacement for the Ham pluton, which also provides a minimum age for Amisk volcanism, Missi sedimentation, and all other rocks and early deformation that predate the Ham pluton; 2) the age of regional metamorphism

during the Hudsonian orogeny is given by the metamorphic age of the File gneiss dome; 3) the probably metamorphic ages for the Elmes Island Formation dacite flows and the Morton porphyry; and, 4) the post-kinematic age determinations for the late pegmatite dykes.

Two trends are given by the  $^{87}\text{Sr}/^{86}\text{Sr}$  initial ratio values determined from the rock units analyzed. The rock units from the Flin Flon volcanic belt are characterized by low initial ratio values: Elmes Island Formation dacite flows ( $0.7020 \pm 0.0002$ ), Morton porphyry ( $0.7027 \pm 0.0001$ ), and the Ham pluton ( $0.7019 \pm 0.0005$ ). This characteristic trend suggests that these rocks could not have been derived from much older continental crustal material, but rather from a source region depleted in rubidium relative to strontium, such as the upper mantle. However, this does not exclude the possibility of any crustal pre-history that may have occurred between their geological ages of emplacement and the corresponding radiometric ages. For example, such a possibility is indicated for the Elmes Island Formation and the Morton porphyry, two rock units whose radiometric ages are considered metamorphic.

The exception to this trend of low initial ratios is given by the File gneiss dome ( $0.7038 \pm 0.0008$ ), a granitoid gneiss complex from the Kisseynew sedimentary gneiss belt. This moderately high initial ratio suggests a crustal pre-history for the paragneiss prior to isotopic homogenization of the Missi Group arkosic sedimentary rocks by recrystallization and metasomatism during regional metamorphism.



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**APPENDICES**



## APPENDIX I. SAMPLE DESCRIPTION

### I.1. Elmes Island Formations ( $54^{\circ}50'$ N by $100^{\circ}27'$ W)

- 5344-2 A light-grey, white weathering, fine grained to aphanitic dacite with a weak biotite foliation and disseminated pyrite. Iron oxide staining is present along fractures in the sample.
- 5344-3 A medium-grey, white weathering, fine-grained to aphanitic dacite with a weak biotite foliation and disseminated pyrite. Iron oxide staining is present along fractures in the sample.
- 5345-1 A medium-grey, white weathering, fine-grained to aphanitic dacite with a weak biotite foliation. It contains disseminated pyrite and chalcopyrite. Iron oxide staining is present along fractures in the sample.
- 5347-1 A light-grey, fine-grained to aphanitic dacite with small flattened phenocrysts of quartz. It contains disseminated pyrite. A small amount of iron oxide staining is present along the fractures in the sample.
- 5347-2 A light-grey, white weathering, fine-grained to aphanitic dacite with disseminated pyrite. No evidence of biotite foliation is present. Iron oxide staining occurs along fractures in the sample.
- 5347-3 A light-grey, white weathering, fine-grained to aphanitic dacite with disseminated pyrite. No evidence of biotite foliation is present. A small amount of iron oxide staining is present.
- 5348-2 A light-grey, fine-grained to aphanitic dacite with a strong biotite foliation and disseminated pyrite. A large amount of iron staining is present along fractures in the sample.

I.2. Morton porphyry (54°49'N by 100°26'W)

- 5180-1 A light grey, white weathering, felsic quartz feldspar, biotite-bearing porphyry with well developed phenocrysts of blue quartz and plagioclase (up to 10 m.m. in size), set in a fine-grained matrix. It contains disseminated pyrite. Iron oxide staining is present along cracks in the sample.
- 5180-3 A weakly foliated, light-grey, white weathering, felsic quartz feldspar, biotite-bearing porphyry with large subhedral to euhedral phenocrysts of plagioclase and blue quartz (up to 10 m.m.), set in a fine-grained matrix. It contains disseminated pyrite. A large amount of iron oxide staining is present throughout the sample.
- 5180-5 A weakly foliated, light-grey, white weathering, felsic quartz feldspar, biotite-bearing, porphyry with well developed phenocrysts of plagioclase and blue quartz (up to 10 m.m. in size), set in a fine-grained matrix. It contains accessory disseminated pyrite. A large amount of iron oxide staining is present.
- 5181-1 A light-grey, white weathering, felsic quartz feldspar, biotite-bearing porphyry with large well developed phenocrysts of plagioclase and quartz (up to 10 m.m.), set in a fine-grained matrix. It contains accessory disseminated pyrite. A large amount of iron oxide staining is present.
- 5186-1 A weakly foliated, medium-grey, felsic quartz feldspar, biotite bearing porphyry, with large subhedral to euhedral phenocrysts of plagioclase and blue quartz (up to 8 m.m. in size), set in a fine-grained to aphanitic matrix. It contains accessory disseminated pyrite. A small amount of iron oxide staining is present.
- 5192-1 A weakly foliated, light-grey, felsic quartz feldspar, biotite bearing porphyry, with large well developed phenocrysts of plagioclase and quartz (up to 10 m.m.), set in a fine-grained to aphanitic matrix. It contains disseminated pyrite. Iron oxide staining is present.

- 5448-1 A weakly foliated, light-grey felsic quartz feldspar, biotite bearing porphyry with well developed phenocrysts of plagioclase and quartz (up to 10 m.m.), set in a fine-grained matrix. It contains accessory disseminated pyrite. Iron oxide staining is prominent.
- 5448-2 A weakly foliated, light-grey felsic quartz feldspar, biotite-bearing porphyry with sub-hedral to euhedral phenocrysts of blue quartz and plagioclase (up to 10 m.m.), set in a fine-grained matrix. It contains accessory disseminated pyrite. Iron oxide staining is prominent.

I.3. Ham pluton ( $54^{\circ}55'N$  by  $100^{\circ}15'W$ )

- 1021-1 A white coloured, white weathering, massive medium-to coars-grained, biotite bearing, tonalite.
- 1024-1 A pink, massive, equigranular medium-grained, biotite-bearing, tonalite to granodiorite.
- 1027-1 A pink, massive porphyritic, medium-grained, biotite-bearing, tonalite to granodiorite, with small phenocrysts of potassium feldspar.
- 1028-1 A pink, massive, porphyritic, coarse-grained, biotite-bearing, tonalite to granodiorite, with phenocrysts of potassium feldspar. It also is weakly foliated.
- 1030-1 A pink, massive, weakly foliated, medium-to coarse-grained biotite-bearing, tonalite to granodiorite.
- 1031-1 A pink, massive porphyritic, coarse-grained, biotite-bearing, tonalite to granodiorite with phenocrysts of potassium feldspar.
- 1032-1 A pink, massive, equigranular, medium-grained, biotite-bearing, tonalite to granodiorite.

I.4. File gneiss dome ( $54^{\circ}55'N$  by  $100^{\circ}20'W$ )

- 950-1 A pink, gneissic, strongly foliated, fine-grained quartzofeldspathic biotite gneiss with small porphyroblasts of potassium feldspar.

- 953-1 A pink, gneissic, weakly foliated, fine-grained, quartzo-feldspathic biotite gneiss with small porphyroblasts of potassium feldspar.
- 954-1 A pink, white weathering, gneissic, weakly foliated, fine-grained, hornblende bearing, quartzo-feldspathic biotite gneiss with small porphyroblasts of potassium feldspar.
- 955-1 A greyish-pink, gneissic, moderately foliated, fine-grained, quartzo-feldspathic biotite gneiss with small porphyroblasts of potassium feldspar and accessory magnetite.
- 956-1 A pink, equigranular, massive, fine-to medium-grained, "granitic" gneiss with porphyroblasts of quartz and plagioclase. It is a quartzo-feldspathic biotite gneiss that does not possess a typical gneissic texture.
- 957-1 A greyish-pink gneissic, weakly foliated, fine-grained, equigranular, quartzo-feldspathic biotite gneiss with small porphyroblasts of potassium feldspar.
- 958-1 A pink, gneissic, weakly foliated, fine-grained, equigranular quartzo-feldspathic biotite gneiss with small porphyroblasts of potassium feldspar. It contains accessory disseminated pyrite.
- 960-2 A medium-grey gneissic, equigranular, moderately foliated, fine-grained quartzo-feldspathic biotite gneiss with porphyroblasts of potassium feldspar.
- 967-1 A greyish-pink, gneissic, strongly foliated, fine-grained, quartzo-feldspathic biotite gneiss with small porphyroblasts of potassium feldspar.
- 969-1 A pinkish-grey, gneissic, strongly foliated, fine-grained, quartzo-feldspathic biotite gneiss with small porphyroblasts of potassium feldspar.

#### I.5. Late pegmatite dykes

- 966-1 A pink, very coarse-grained pegmatite of granitic composition, containing large phenocrysts of perthitic microcline with graphic intergrowths of quartz and large phenocrysts of plagioclase with graphic intergrowths of quartz. It contains a small amount of biotite and accessory pyrite.

1021-3 A pink, coarse-grained, biotite-bearing, granitic pegmatite with large phenocrysts of microcline and quartz.

## APPENDIX II. DETAILED DISCUSSION OF CHEMICAL PROCEDURES

This treatment for preparation, selection, and chemical procedures is similar to the method outlined by Penner (1970).

### II.1. Sample Preparation and Selection

The weathered surfaces of the rock samples, weighing from 1 to 2 kilograms, were removed prior to sample processing. Each sample was ground to chip-size using a jaw crusher, and the sample chips were washed with distilled water. After drying, the chips were pulverized to a rock powder using a Bleuler grinder. Approximately 100 g. of the rock powder was selected from the bulk quantity by the process of "cone and quartering" to ensure homogeneity of the sample.

Preliminary rubidium and strontium concentrations were obtained from x-ray fluorescence analysis, (an ARL Quantometer x-ray fluorescence spectrometer was used). In general, these preliminary concentrations were from 25%-30% lower in value than the corresponding concentrations obtained from subsequent isotope dilution analysis. No correction factors for such effects as instrument drift, background, and absorption effects (such as electron shielding, secondary fluorescence, matrix effects), were

used for these preliminary concentrations.

These preliminary results were used for the selection of samples possessing a range of rubidium and strontium concentrations, expressed as a Rb/Sr ratio, suitable for whole-rock isochron studies. In addition to sample selection, the determination of sample weight required for precise mass spectrometric analysis and the corresponding volume of enriched spike selection needed to yield optimum isotopic ratios in the mixture of sample and spike were based on these preliminary results. The optimum spiking ratio used for the mixture of spike and sample was 0.5 for both the  $^{84}\text{Sr}/^{86}\text{Sr}$  ratio and  $^{85}\text{Rb}/^{87}\text{Rb}$  ratio measured for the respective mixtures.

## II.2. Chemical Procedure

### II.2.1. Dissolution and Digestion

Three separate aliquots, per sample, of a pre-determined weight (0.2 to 0.5 g. on the average), were precisely weighed on aluminum foil weighing boats and were carefully transferred to separate 100 ml. teflon beakers. These aliquots represent chemical preparations for a spiked Rb analysis, a spiked Sr analysis, and for an unspiked Sr analysis. The samples were dissolved in 20 ml. of 40% HF (manufactured by E. Merck; Suprapur) and were covered with teflon disks to allow the samples to reflux for 30 minutes at approximately 250°F on hot plates. 3 ml. of 70%  $\text{HClO}_4$  (G. Frederick Smith Chemical Co., double distilled) were

added to each aliquot to convert the sample to the perchlorate form, and the samples were refluxed for 8 hours at 250°F for sample digestion. In general, little or no undissolved matter remained at this point. The teflon covers were removed, and the samples were evaporated to a perchlorate mush. 20 ml. of double-distilled H<sub>2</sub>O was added to dissolve the mush, and the samples were evaporated to a perchlorate cake to remove any lingering HF acid from the sample. Care was taken to evaporate the samples to dryness without actually burning the sample brown.

#### II.2.2. Spiking Procedure

The appropriate volumes of <sup>87</sup>Rb-enriched spike solution and of <sup>84</sup>Sr-enriched spike solution were added volumetrically to the respective Rb and spiked Sr samples. In general, 2 ml. or 5 ml. were added to Sr samples and 5 ml. or 10 ml. to Rb samples. No spike solution was added to the unspiked Sr sample. 20 ml. of vycor distilled (constant boiling) 6.2N HCl were added to each spiked sample, the beakers were covered with the teflon disks, and the mixtures of sample and spike were equilibrated by refluxing on a hot plate for 30 minutes. The teflon covers were removed, and the spiked samples were evaporated to dryness.

#### II.2.3. Preparation of the Rb Sample

For the majority of rubidium samples prepared, the samples were not purified by the procedure of ion



exchange column chromatography. Originally, a number of samples from the File gneiss dome and the Ham pluton were concentrated and purified by eluting the Rb samples through calibrated ion exchange columns.

The Rb sample was taken into solution in 5 ml. of 1N HCl, and was transferred to a centrifuge tube. After centrifuging for 5 minutes, the liquid was decanted directly into a 10 ml. Hysil beaker and evaporated to dryness, as a chloride. The prepared sample contained iron, calcium, and other elements, but it was found that adequate mass spectrometric analysis could be made on this residue without necessitating the separation of the Rb fraction by ion exchange column chromatography.

In the original treatment, the supernatant liquid was decanted directly into a prepared ion exchange column. Once the sample was absorbed by the resin, one 5 ml. portion, two 10 ml. portions, and one 85 ml. portion of 1N HCl were added, allowing each portion to be completely absorbed, before adding the next. The desired Rb fraction was collected in the next 40 ml. portion of 1N HCl added. The collected 40 ml. fraction of eluted sample was evaporated to dryness as a sulfate by adding a drop of 10%  $H_2SO_4$  during evaporation.

#### II.2.4. Preparation of the Sr Samples

After the spiking procedure, the preparation of the spiked and unspiked Sr samples were treated similarly.

During the course of this work, the procedure for ion exchange column chromatography was altered. In the original method, an 8% cation exchange resin was used, whereas a 12% cation exchange resin was subsequently used (see following section on ion exchange columns).

The Sr samples were taken into solution in 5 ml. of methanol (Baker Instra-Analyzed, GC-Spectrophotometric Quality Solvent) to leach out the alkaline earth perchlorates, and were transferred to centrifuge tubes. After centrifuging, the supernatant liquids were decanted into 30 ml. Hysil beakers where 10 ml. of 6.2N HCl were added. The solutions were evaporated to dryness.

For elution of the samples using the 8% cation exchange resin, the dried samples were taken into solution in 5 ml. of 2.5N HCl, and were transferred directly to the prepared columns. After allowing the solution to be absorbed, one 5 ml. portion, two 10 ml. portions, and one 85 ml. portion of 2.5N HCl were added, allowing each portion to be completely absorbed before adding the next. The desired Sr fraction was collected in the next 40 ml. portion of 2.5N HCl added. This collected fraction was evaporated to dryness in the perchlorate form by adding a few drops of 10%  $\text{HClO}_4$  during evaporation.

For elution of samples using the 12% cation exchange resin, the dried Sr samples were taken into solution in 5 ml. of 6.2N HCl and were transferred to the prepared columns. Two 5 ml. portions and one 40 ml.

portion of 6.2N HCl were added, allowing each portion to be completely absorbed before adding the next. The Sr fraction was collected in the next 30 ml. portion of 6.2N HCl added. The collected Sr fraction was evaporated to dryness in the chloride form.

### II.3. Ion Exchange Columns

The columns used were made of 10 m.m. I.D. glass tubing fitted with a coarse fritted disc positioned about 3 cm. above the lower tapered end. The flow rate was fixed at approximately 5-10 ml. per hour by the porosity of the frit and the length of the resin used in the column. Two sets of columns, of a different design, were used for strontium extraction. On one set, the top of the column was terminated in a standard taper female ground glass joint to facilitate the positioning of a 200 ml. flask used to hold the acid; the flask was connected to a standard taper male ground glass joint by a 10 inch (25 cm.) length of narrow glass tubing. The purpose of this extension was to create a hydrostatic pressure to increase the flow rate of the acid through the resin to approximately 10 ml. per hour. The second set of columns were designed so that the top of the column was connected directly to the 200 ml. flask that was used to hold the acid. The set of columns used for rubidium separation were similar in design to this second set of strontium columns. Provision was made for all three sets of columns to have a resin length in the

column that varied from 9 to 10 inches (22 to 25 cm.).

Analytical reagent grade cation exchange resin AG-50W-X8-200-400 mesh, hydrogen form (Bio-Rad Laboratories), was used for the three sets of ion exchange columns in the original procedure. For the strontium columns, the resin was given a preliminary washing in 2.5N HCl, and then was added to the columns. Subsequent washing with 50 ml. of 6.2N HCl was made; this was followed by a washing with 150 ml. of 2.5N HCl. The columns were then ready for calibration and for use. In a similar manner, the resin for the rubidium columns was washed with 50 ml. of 1N HCl instead of the 2.5N HCl, and then was added to the columns to be washed with 50 ml. of 6.2N HCl and with 150 ml. of 1N HCl.

Analytical reagent grade cation exchange resin AG-50W-X12-200-400 mesh, hydrogen form (Bio-Rad Laboratories), was used for the modified procedure of ion exchange column chromatography for strontium separation. All washings of the 12% cation exchange resin were made with 6.2N HCl.

Calibration of the respective columns was made individually by eluting a suitably prepared sample through the columns. 10 ml. fractions of the elutant were collected, and each portion was analyzed on an atomic absorption spectrophotometer to determine the portions when the strontium fraction came out on the strontium columns, and when the rubidium fraction came out on the rubidium columns.

Following an extraction during the use of the columns, the strontium columns using the 8% cation exchange resin were cleaned by passing consecutive volumes of 50 ml. of 6.2N HCl and 100 ml. of 2.5N HCl; the strontium columns using the 12% cation exchange resin were cleaned with 100 ml. of 6.2N HCl; and, the rubidium columns were cleaned with 50 ml. of 6.2N HCl and 100 ml. of 1N HCl. The resin in the columns, when not in daily use, was kept moist by adding several ml. of 1N HCl, 2.5N HCl, and 6.2N HCl to the rubidium columns, to the strontium columns using the 8% cation exchange resin, and to the strontium columns using the 12% cation exchange resin, respectively.

#### II.4. Preparation of Potassium Feldspar Mineral Concentrates from Pegmatite Samples

The procedure for the separation of the potassium feldspar mineral concentrates, from other constituent minerals present in the pegmatite samples, was based on Nilssen's (1967) method and that used previously in this laboratory.

The two pegmatite samples analyzed (samples 966-1 and 1021-3) were crushed and pulverized to a rock powder in the manner previously outlined for the preparation of the whole rock samples. The rock powder was passed through a sieve and the -60+100 mesh fraction was collected. The very fine particle or dust fraction was carefully removed by washing with distilled water and by decanting several times. After drying, the -60+100 mesh fraction was

suspended in bromoform (specific gravity = 2.89), contained in a separatory funnel, and the mafic mineral fraction was removed as the "sink" fraction. The specific gravity of the heavy liquid was lowered by dropwise addition of acetone to the bromoform. The "sink" fraction was removed. Repetition of this procedure gave a series of "sink" fractions of steadily decreasing specific gravity. These respective "sink" fractions were washed with acetone, to remove any lingering traces of bromoform and to dry the fractions. The last and lightest "sink" fraction was considered to comprise pure potassium feldspar. The purity of the feldspar concentrate was checked by refractive index measurements using the method of Kerr (1959, pp.57-63).

#### II.5. Preparation of the Spike Solutions

Isotope tracers enriched in the  $^{87}\text{Rb}$  isotope and isotope tracers enriched in the  $^{84}\text{Sr}$  isotope were supplied by Oak Ridge National Laboratories as salts of  $\text{RbCl}$  and of  $\text{Sr}(\text{NO}_3)_2$ , respectively. The respective spike solutions were prepared by G. S. Clark from these isotopically enriched tracer salts. The salts were dried overnight at  $120^\circ\text{F}$ ., and were allowed to cool in a dessicator. The spike salts were carefully weighed and carefully transferred to 200 ml. volumetric flasks where they were diluted to volume with 0.5N  $\text{HCl}$ . A 10 ml. aliquot of the  $^{84}\text{Sr}$ -enriched spike solution was transferred volumetrically to a 1000 ml. volumetric flask, where the stock solution was

diluted to volume using 60 ml. of 6.2N HCl and double-distilled water. A 15 ml. aliquot of the  $^{87}\text{Rb}$ -enriched spike solution was transferred volumetrically to a separate 1000 ml. volumetric flask, and the stock solution was diluted to volume with 60 ml. of 6.2N HCl and double-distilled water.

Composition analysis of the respective spike solutions were measured mass spectrometrically on dried 10 ml. aliquots of each spike solution separately.

The concentration of the strontium spike solution was calibrated with a gravimetrically prepared laboratory standard solution of  $\text{Sr}(\text{NO}_3)_2$  (Baker analyzed, reagent grade), and the concentration of the rubidium spike solution was calibrated with a solution of RbCl (NBS 984 RbCl, assay-isotopic standard). The isotopic ratios necessary to determine the concentration and composition of these spike solutions were measured mass spectrometrically.

### APPENDIX III. ISOTOPE DILUTION ANALYSES

An isotope dilution analysis is an addition analysis in which a precisely measured volume of a tracer (spike) solution (whose concentration and isotopic composition are precisely known) is added to an accurately weighed sample to be analyzed. In applying this method to age determinations, the isotopic abundances of rubidium or strontium present in a rock sample are determined, in addition to the total concentration of rubidium or strontium. The corresponding isotopic ratios are calculated from the isotopic abundances.

#### III.1. Isotope Dilution Calculation for Rubidium

The method used for isotope dilution calculations of rubidium is described by Penner (1970). The notations used for this method are given below:

$m$  = concentration of each isotope or total rubidium in the mixture of spike and sample;

$t$  = concentration of each isotope or total rubidium in the spike;

$s$  = concentration of each rubidium isotope in the sample; and

$A$  = total sample concentration of rubidium, in units or micromoles.



The isotope dilution equation used for rubidium calculations was:

$$(1) \left( \frac{{}^{85}\text{Rb}}{{}^{87}\text{Rb}} \right)_m = \frac{({}^{85}\text{Rb})_t + ({}^{85}\text{Rb})_s}{({}^{87}\text{Rb})_t + ({}^{87}\text{Rb})_s}$$

From the natural composition of rubidium:

$$(2) ({}^{85}\text{Rb})_s = 0.722A$$

$$(3) ({}^{87}\text{Rb})_s = 0.278A$$

Using the isotopic composition, concentration, and volume of the rubidium spike solution used, both  $({}^{85}\text{Rb})_t$  and  $({}^{87}\text{Rb})_t$  are determined. Therefore, equation (1) is reduced to a single unknown, the total concentration (A) of rubidium in the sample. From equation (1), the concentration (A) of rubidium is calculated uniquely in terms of the measured ratio  $({}^{85}\text{Rb}/{}^{87}\text{Rb})_m$ . Given the concentration of rubidium (A), the concentration of  ${}^{87}\text{Rb}$ , in micromoles/g., was calculated from equation (3) using the natural composition of rubidium and the sample weight used.

All rubidium isotope dilution calculations were made using a data reduction program written by A. Penner for use on an Olivetti-Underwood Programma 101 desk calculator.

### III.2. Isotope Dilution Calculation for Strontium Analysis

The following discussion of isotope dilution calculation for strontium is similar to that outlined by Penner (1970) and Anderson (1974). The notations described by Anderson (1974, p.47) are listed below:

m = pertaining to the mixture of spike and sample,

such as the concentration of each isotope or total strontium, the isotopic composition of each isotope, or the isotopic ratio of an isotope pair;

t = concentration of each isotope or total strontium in the spike;

s = concentration of each strontium isotope in the sample;

A = total concentration of strontium in the sample, in units of micromoles.

The calculations required in the calculation of the atomic abundance of each of the four strontium isotopes are from Anderson (1974, p.47-48). However, Anderson's equations (7) through (10) contain an erratum. The denominator should read  $(\text{Sr}/^{86}\text{Sr})_m$  and not  $A/({}^{86}\text{Sr})_m$ . The equations are:

$$(4) \quad ({}^{84}\text{Sr}/{}^{86}\text{Sr})_m = \frac{({}^{84}\text{Sr})_t + ({}^{84}\text{Sr})_s}{({}^{86}\text{Sr})_t + ({}^{86}\text{Sr})_s}$$

$$(5) \quad ({}^{84}\text{Sr})_s = 0.0056A$$

$$(6) \quad ({}^{86}\text{Sr})_s = 0.0986A$$

$$(7) \quad A = \frac{({}^{84}\text{Sr})_t - ({}^{84}\text{Sr}/{}^{86}\text{Sr})_m ({}^{86}\text{Sr})_t}{0.0986 ({}^{84}\text{Sr}/{}^{86}\text{Sr})_m - 0.0056}$$

$$(8) \quad (\text{Sr})_m = A + (\text{Sr})_t$$

$$(9) \quad (\text{Sr}/{}^{86}\text{Sr})_m = ({}^{84}\text{Sr}/{}^{86}\text{Sr})_m + ({}^{87}\text{Sr}/{}^{86}\text{Sr})_m + ({}^{88}\text{Sr}/{}^{86}\text{Sr})_m + 1$$

$$(10) \quad ({}^{88}\text{Sr}/\text{Sr})_m = \frac{({}^{88}\text{Sr}/{}^{86}\text{Sr})_m}{(\text{Sr}/{}^{86}\text{Sr})_m}$$

$$(11) \quad ({}^{87}\text{Sr}/\text{Sr})_m = \frac{({}^{87}\text{Sr}/{}^{86}\text{Sr})_m}{(\text{Sr}/{}^{86}\text{Sr})_m}$$

$$(12) \quad ({}^{86}\text{Sr}/\text{Sr})_m = \frac{1}{(\text{Sr}/{}^{86}\text{Sr})_m}$$

$$(13) \quad ({}^{84}\text{Sr}/\text{Sr})_m = \frac{({}^{84}\text{Sr}/{}^{86}\text{Sr})_m}{(\text{Sr}/{}^{86}\text{Sr})_m}$$

$$(14) \quad ({}^{88}\text{Sr})_s = (\text{Sr})_m ({}^{88}\text{Sr}/\text{Sr})_m - ({}^{88}\text{Sr})_t$$

$$(15) \quad ({}^{87}\text{Sr})_s = (\text{Sr})_m ({}^{87}\text{Sr}/\text{Sr})_m - ({}^{87}\text{Sr})_t$$

$$(16) \quad ({}^{86}\text{Sr})_s = (\text{Sr})_m ({}^{86}\text{Sr}/\text{Sr})_m - ({}^{86}\text{Sr})_t$$

$$(17) \quad ({}^{84}\text{Sr})_s = (\text{Sr})_m ({}^{84}\text{Sr}/\text{Sr})_m - ({}^{84}\text{Sr})_t$$

In the above calculations, equations (4) through (7) lead to the determination of the total concentration of strontium in the sample (A). In equations (5) and (6), the values 0.0056 and 0.0986 refer to the fractional isotopic composition of  ${}^{84}\text{Sr}$  and  ${}^{86}\text{Sr}$  in natural strontium, respectively. The total concentration of strontium in the mixture of spike and sample,  $(\text{Sr})_m$ , is determined from equation (8). The sum of the four isotope ratios present in the mixture,  $(\text{Sr}/{}^{86}\text{Sr})_m$ , is given by equation (9), where  $({}^{86}\text{Sr}/{}^{86}\text{Sr})_m$  is assumed equal to 1.0. Equations (10) through (13) lead to the calculation of the fractional composition of each strontium isotope in the mixture of spike and sample. The isotope concentration of  $({}^{88}\text{Sr})_s$ ,  $({}^{87}\text{Sr})_s$ ,  $({}^{86}\text{Sr})_s$ , and  $({}^{84}\text{Sr})_s$  are determined by equations (14), (15), (16), and (17). The isotope ratios  $({}^{88}\text{Sr}/{}^{86}\text{Sr})_s$ ,  $({}^{87}\text{Sr}/{}^{86}\text{Sr})_s$ , and  $({}^{84}\text{Sr}/{}^{86}\text{Sr})_s$  are obtained from the following calculations:

$$(18) \quad ({}^{88}\text{Sr}/{}^{86}\text{Sr})_s = ({}^{88}\text{Sr})_s / ({}^{86}\text{Sr})_s$$

$$(19) \quad ({}^{87}\text{Sr}/{}^{86}\text{Sr})_s = ({}^{87}\text{Sr})_s / ({}^{86}\text{Sr})_s$$

$$(20) \quad ({}^{84}\text{Sr}/{}^{86}\text{Sr})_s = ({}^{84}\text{Sr})_s / ({}^{86}\text{Sr})_s$$

At this stage of data reduction, the determined isotope ratios of ( $^{88}\text{Sr}/^{86}\text{Sr}$ )<sub>s</sub>, ( $^{87}\text{Sr}/^{86}\text{Sr}$ )<sub>s</sub>, and ( $^{84}\text{Sr}/^{86}\text{Sr}$ )<sub>s</sub> are approximate in value because neither the normalizing factor, to correct for isotopic fractionation, nor the "alpha" correction factor, to adjust the relative isotopic composition of the four strontium isotopes, has been applied. If the "alpha" correction factor is to be applied, it was applied at this stage of data reduction. If no "alpha" correction is necessary, the following procedure of applying the "alpha" correction factor was omitted, and the procedure of applying the normalizing factor was made directly.

The "alpha" correction is an adjustment of the isotopic composition of the relative isotope abundances of strontium in the sample, and this correction is applied to a sample that has a much larger than natural value of ( $^{87}\text{Sr}/^{86}\text{Sr}$ )<sub>s</sub>. Such a condition is present in rubidium-rich phases such as pegmatites, where the presence of radiogenic ( $^{87}\text{Sr}$ )<sub>s</sub> is reflected in the larger than natural value of ( $^{87}\text{Sr}/^{86}\text{Sr}$ )<sub>s</sub>. The relative atomic abundance of ( $^{86}\text{Sr}$ )<sub>s</sub> in the sample is a function of the ( $^{87}\text{Sr}/^{86}\text{Sr}$ )<sub>s</sub> composition in the sample, since the other isotope ratios of ( $^{88}\text{Sr}/^{86}\text{Sr}$ )<sub>s</sub> and ( $^{84}\text{Sr}/^{86}\text{Sr}$ )<sub>s</sub> are invariant in nature. It was shown by Penner (1970, p.16) that the use of the "alpha" correction factor will increase the calculated total strontium concentration in the sample, and will significantly improve the calculated ( $^{84}\text{Sr}/^{86}\text{Sr}$ )<sub>s</sub> value that is very sensitive to this correction. On the other hand, it will not affect the

calculated concentration of  $(^{86}\text{Sr})_s$ , and will only affect the  $(^{87}\text{Sr}/^{86}\text{Sr})_s$  value very slightly.

The calculations required to apply the "alpha" correction factor are listed below.

$$(21) \quad (^{84}\text{Sr})_s = 0.0056 (\text{alpha}) A$$

$$(22) \quad (^{86}\text{Sr})_s = 0.0986 (\text{alpha}) A$$

$$(23) \quad \text{alpha} = \frac{(^{84}\text{Sr}/^{86}\text{Sr})_n + (^{87}\text{Sr}/^{86}\text{Sr})_o + (^{88}\text{Sr}/^{86}\text{Sr})_n + 1.0}{(^{84}\text{Sr}/^{86}\text{Sr})_n + (^{87}\text{Sr}/^{86}\text{Sr})_s + (^{88}\text{Sr}/^{86}\text{Sr})_n + 1.0}$$

where,

$$(^{84}\text{Sr}/^{86}\text{Sr})_n = 0.0568,$$

$$(^{88}\text{Sr}/^{86}\text{Sr})_n = 8.375, \text{ and}$$

$$(^{86}\text{Sr}/^{86}\text{Sr})_n = 1.0,$$

are the natural isotopic ratios in the sample;

$(^{87}\text{Sr}/^{86}\text{Sr})_s$  = the determined preliminary value from equation (19), and

$(^{87}\text{Sr}/^{86}\text{Sr})_o = 0.706$ , an arbitrary value.

The value of "alpha" obtained from equation (23) is substituted into equations (21) and (22). Equations (21) and (22) then are substituted into equation (7), and the calculations associated with equations (7) through (20) are repeated. From this iteration, a new set of adjusted values for the total concentration of strontium in the sample (A), and the sample isotope ratios  $(^{88}\text{Sr}/^{86}\text{Sr})_s$ ,  $(^{87}\text{Sr}/^{86}\text{Sr})_s$ , and  $(^{84}\text{Sr}/^{86}\text{Sr})_s$  are determined. More than one iteration of equations (7) through (20) may be required until two successive estimates of  $(^{87}\text{Sr}/^{86}\text{Sr})_s$  are of equivalent value.

To correct for the variable degree of isotopic fractionation that occurs during mass spectrometric analysis, the application of the normalizing factor was used. The measured isotope ratios,  $(^{88}\text{Sr}/^{86}\text{Sr})_m$ ,  $(^{87}\text{Sr}/^{86}\text{Sr})_m$ , and  $(^{84}\text{Sr}/^{86}\text{Sr})_s$  are proportionally adjusted (i.e. normalized) to  $(^{88}\text{Sr}/^{86}\text{Sr})_s = 8.375$ . The calculations required for the normalization procedure are listed below.

$$(24) \quad (88/86)_N = 8.375 / (^{88}\text{Sr}/^{86}\text{Sr})_s$$

$$(25) \quad (^{88}\text{Sr}/^{86}\text{Sr})_{m,N} = (88/86)_N (^{88}\text{Sr}/^{86}\text{Sr})_m$$

$$(26) \quad (^{87}\text{Sr}/^{86}\text{Sr})_{m,N} = (1/2) [1.0 + (88/86)_N] (^{87}\text{Sr}/^{86}\text{Sr})_m$$

$$(27) \quad (^{84}\text{Sr}/^{86}\text{Sr})_{m,N} = [1.0 / (88/86)_N] (^{84}\text{Sr}/^{86}\text{Sr})_m$$

In the above calculations,  $(88/86)_N$  is the normalizing factor that is determined uniquely for that particular strontium mass spectrometric analysis. In equations (25), (26), and (27), the three measured isotope ratios of the mixture of spike and sample are proportionally adjusted (normalized).

Equations (25), (26), and (27) are substituted into equations (7) through (13), and the overall calculation procedure from equations (7) through (20) was repeated. If the "alpha" correction factor was used, equations (21) and (22) are used instead of equations (5) and (6). The "alpha" factor determined above was used. If no "alpha" correction was used, equations (21) through (23), and the procedure associated with them, was omitted. If the "alpha" correction was used, the total concentration of strontium in the sample (A) was obtained from equations (7), (21), (22), and

(27). In addition,  $(^{86}\text{Sr})_s$  was determined from equation (22). The respective concentration values for A and  $(^{86}\text{Sr})_s$  were divided by the sample weight used in the analysis. The purpose was to standardize the concentration unit to micromoles/g. If no "alpha" correction was used, (A) and  $(^{86}\text{Sr})_s$  were determined directly from equations (6), (7), and (27), and from the sample weight used.

### III.3. Calculation of the Radiogenic $^{87}\text{Sr}$ Content

Radiogenic  $^{87}\text{Sr}$  content was calculated from rubidium-rich samples, such as K-feldspar mineral concentrates from pegmatite samples, or from the NBS 70a K-feldspar standard. The calculation used, was after York and Farquhar (1972, p.31, 57), and is listed below.

$$(28) \quad (^{87}\text{Sr})_s^* = (^{86}\text{Sr})_s \left[ (^{87}\text{Sr}/^{86}\text{Sr})_s - (^{87}\text{Sr}/^{86}\text{Sr})_i \right]$$

where  $(^{87}\text{Sr})_s^*$  = concentration of radiogenic strontium in the sample,  $(^{86}\text{Sr})_s$  = concentration of  $^{86}\text{Sr}$  in the sample;  $(^{87}\text{Sr}/^{86}\text{Sr})_s$  = determined sample ratio, and  $(^{87}\text{Sr}/^{86}\text{Sr})_i$  = initial ratio at the time of crystallization, assumed to be 0.710.

The concentration of  $(^{86}\text{Sr})_s$  must be determined before equation (28) can be used. Since the relative isotopic composition of  $^{88}\text{Sr}$ ;  $^{86}\text{Sr}$ ;  $^{84}\text{Sr}$  is constant in nature, the relative isotopic abundance of  $^{86}\text{Sr}$  in the sample is dependent upon the abundance of  $^{87}\text{Sr}$ ; the abundance of  $^{87}\text{Sr}$  is reflected by the value of  $(^{87}\text{Sr}/^{86}\text{Sr})_s$ . Two methods of similar result can be used to calculate the

relative isotopic abundance of  $^{86}\text{Sr}$ , that in turn determines the concentration value for ( $^{86}\text{Sr}$ )s. To calculate the relative isotopic abundance (and concentration) of ( $^{86}\text{Sr}$ )s, equations (22) and (23) can be used. Alternatively, the relative isotopic abundance of  $^{86}\text{Sr}$  can be determined directly from the isotopic composition of strontium in the sample. To illustrate both procedures, the pegmatite sample 966-1, a K-feldspar mineral concentrate, is used. For sample 966-1, the total strontium concentration is 72.1 ppm, and the ( $^{87}\text{Sr}/^{86}\text{Sr}$ )s ratio is 1.0393. Since the nonradio-genic isotopes of strontium are considered invariant in nature, then ( $^{88}\text{Sr}/^{86}\text{Sr}$ )n = 1.0. These three isotope ratios, plus that for ( $^{87}\text{Sr}/^{86}\text{Sr}$ )s are summed:

$$\Sigma = (8.375 + 1.0393 + 1.0 + 0.0568) = 10.4711.$$

The following tabular calculation was made to calculate the relative isotopic abundance of strontium and to calculate the atomic weight of strontium. The relative isotopic abundance of  $^{86}\text{Sr}$  was found to be 0.0955 and the corresponding atomic weight of strontium was found to be 87.58.

| <u>Isotope</u>   | <u>Relative Isotopic Abundance in Sample</u> |   | <u>Isotopic<sup>1</sup> Atomic Mass(amu)</u> |   | <u>Atomic Weight</u> |
|------------------|--|---|--|---|----------------------|
| $^{88}\text{Sr}$ | $8.375/\Sigma = 0.7998$                      | x | 87.9056                                      | = | 70.3087              |
| $^{87}\text{Sr}$ | $1.0393/\Sigma = 0.0993$                     | x | 86.9089                                      | = | 8.6257               |
| $^{86}\text{Sr}$ | $1.0/\Sigma = 0.0955$                        | x | 83.9094                                      | = | 8.2043               |
| $^{84}\text{Sr}$ | $0.0568/\Sigma = \underline{0.0054}$         | x | 83.9134                                      | = | <u>0.4397</u>        |
|                  | 1.0000                                       |   |  |   | 87.5784              |

<sup>1</sup>Handbook of Chemistry and Physics, 49th edition, The Chemical Rubber Co., Cleveland, Ohio, (1968), p. B24-B26.



The concentrations of ( $^{86}\text{Sr}$ )<sub>s</sub> was obtained from the following calculation:

$$\begin{aligned} (29) \quad (^{86}\text{Sr})_s &= (\text{abundance of } ^{86}\text{Sr}) (\text{Total common Sr}) \\ &= (0.0955) (72.1 \text{ ppm}) = 6.886 \text{ ppm.} \end{aligned}$$

Alternatively, from equation (22), the "alpha" value for the sample was 0.9685. Substituting this value into equation (22), the concentration of ( $^{86}\text{Sr}$ )<sub>s</sub> was also obtained.

$$\begin{aligned} (22) \quad (^{86}\text{Sr})_s &= 0.0986 (\text{alpha}) A \\ &= (0.0986) (0.9685) (72.1 \text{ ppm}) \\ &= 6.886 \text{ ppm.} \end{aligned}$$

This value is identical to that determined above using equation (29). Substituting the values of ( $^{86}\text{Sr}$ )<sub>s</sub> = 6.886 ppm, ( $^{87}\text{Sr}/^{86}\text{Sr}$ )<sub>s</sub> = 1.0393, and ( $^{87}\text{Sr}/^{86}\text{Sr}$ )<sub>i</sub> = 0.710 into equation (28), the radiogenic concentration of  $^{87}\text{Sr}$  present in the sample then was calculated.

$$\begin{aligned} (^{87}\text{Sr})_s^* &= 6.886 \text{ ppm} (1.0393 - 0.710) \\ &= 2.268 \text{ ppm.} \end{aligned}$$

## APPENDIX IV. MASS SPECTROMETRY

### IV.1. Description of the Mass Spectrometer

The following description of the mass spectrometer is similar to that described by Penner (1970) and Anderson (1974).

All isotopic measurements were made on a triple filament thermal ionization solid source assembly, single (direction) focusing mass spectrometer with a ten inch radius of curvature and a ninety degree deflexion.

The ionization source assembly used was similar to that described by Craig (1959). Picker nuclear beads (style #297157) were used as filament beads. Two tantalum sample filaments and one rhenium center filament were welded to the posts of the Picker nuclear filament bead. The dimensions of the filament material used was 0.030" wide and 0.001" thick (H. Cross and Company). The prepared filament beads were mounted in the source assembly with non-magnetic stainless steel blocks. Separate ionization sources were used for rubidium and strontium analysis.

The vacuum system consists of an oil diffusion pump backed by an oil-sealed mechanical fore pump at the source end, and a Varian Vac Ion Pump (50 liters/sec.) at the collector end. On-line gates valves were used to isolate the collector end and oil diffusion pump from the

source end of the vacuum system for venting and changing samples.

Signal detection and amplification of the ion current was attained with a Cary Model 401 vibrating reed electrometer (VRE). The signal was recorded on a Leeds and Northrup Speedomax Model W/L ten inch strip chart recorder equipped with an expanded scale measurement recording circuit to integrate the VRE output with the chart recorder. The principle of operation of the expanded-scale has been described by Damon (1966, p.13) and Shields (1966, pp.11-13).

A Fluke high voltage power supply (Model 410B) was used to provide the voltage (D.C.) required for the "accelerating" voltage of the produced ion current in the ionization source assembly. The filament currents and vibrating reed electrometer were stabilized using a Sola transformer and a Sorensen voltage regulator.

#### IV.2. Operating Procedure

The filament bead to be used for either rubidium or strontium analysis was outgassed in the source region of the mass spectrometer for a period of 30 minutes. The side and centre filaments were outgassed by passing a current of 2.5 amp. and 5.0 amp., respectively.

The prepared sample to be analyzed, stored in a beaker, was taken into solution by adding a few drops of double-distilled water. A clean glass micropipette, that

was discarded after use, was used to transfer the sample slurry and solution to the surface of the outgassed tantalum side filaments. The sample was evaporated to dryness by passing an electric current of 1.8 amps.

The procedure of mounting the source was aided by the use of on-line gate valves to isolate the source region from the oil diffusion pump, the ion beam tube, and the collector end, where a constant vacuum was maintained. A mechanical roughing pump was used for rapid pumpdown of the source region. Once a pressure of 50 microns was attained, the on-line gate valve to the oil diffusion pump was opened, and the mechanical roughing pump was isolated. For removal of the source, the source region was isolated from the oil diffusion pump, the ion beam tube, and the collector end where a constant vacuum will be maintained. The source region was then vented with dry nitrogen to facilitate removal of the source from the source end.

After a pressure of  $2.0 \times 10^{-7}$  millimeters of mercury was attained in the source end by the oil diffusion pump, the sample was outgassed by passing a current through the side and centre filaments. A current of 0.1 amp. on the side filaments and 1.8 amp. on the centre filament was used to outgas rubidium samples, whereas 4.0 amp. on the centre filament was used to outgas strontium samples. Outgassing of the samples for 30 minutes was usually sufficient to ensure that the pressure in the source region was below  $2.0 \times 10^{-7}$  millimeters of mercury necessary for analysis.

During mass spectrometric analysis, magnetic peak switching was used to measure a set of isotope pairs.

During strontium analysis, the operating mode was to use: 1) an accelerating voltage of 4000 volts (D.C.); 2) filament currents between 0.1 and 0.5 amp. for the side sample filaments, and 3) a filament current between 4.5 and 5.5 amp. for the centre ionizing filament. For spiked strontium measurements, three separate sets between 10 and 12 scans of the isotope ratios,  $^{88}\text{Sr}/^{86}\text{Sr}$ ,  $^{87}\text{Sr}/^{86}\text{Sr}$ , and  $^{84}\text{Sr}/^{86}\text{Sr}$ , were measured at expanded scale. The base line was scanned between each set of isotope ratios measured to monitor base line resolution of the isotope peaks, and to monitor the presence of any rubidium contamination that may be present. In addition, the base line was scanned before and after the measurement of the three sets of isotope ratios. The procedure for unspiked strontium measurements was similar to that outlined for spiked strontium measurements, with the exception that no measurement of the  $^{84}\text{Sr}/^{86}\text{Sr}$  ratio was made.

For rubidium analysis, an accelerating voltage of 3500 volts (D.C.) was used. Filament currents between 0.3 to 0.7 amp. on the side sample filaments and between 1.8 to 2.2 amp. on the centre ionizing filaments were maintained. During a rubidium measurement, one set of 10 or 12 scans of the isotope pair  $^{85}\text{Rb}/^{87}\text{Rb}$  was taken.

#### IV.3. Reproducible Analysis

The quality of analysis made by mass spectrometric

analysis often is influenced by a number of variable factors. Controlling the variability of these factors where possible, generally results in consistent and improved reproducible data. The stability and sensitivity of ionic emission during an analysis were dependent on such factors as: 1) the quality of the prepared sample, 2) the consistency in the manner of sample mounting onto the side filaments; 3) the position of the side filaments relative to the centre filament, 4) the quality of vacuum maintained during analysis; 5) the degree of matrix effects; and, 6) the alignment of the centre filament with the principal slit of the source assembly.

By outgassing the sample before an analysis, the risk of pressure surges during an analysis was minimized and the pressure of the vacuum system was maintained constant. Proper resolution between the isotope mass peaks was maintained when the pressure was kept constant without the system being subject to pressure surges.

Matrix effects were evident in rubidium analysis because the sample was prepared as a residue. Care was exercised while mounting the sample residue onto the side filaments. If too much residue was mounted, the resulting sample sensitivity generally was decreased. This would necessitate a larger than average sample filament current. In addition, the sample would remain gassy during analysis, and the vacuum system often would be subject to pressure surges.

Proper centre filament alignment with the principal slit of the source assembly was necessary in attaining a workable resolution between the isotope mass peaks. Good resolution was necessary for the resolution of the mass 87 peak from the mass 88 peak in strontium analysis.

#### IV.4. Rubidium Contamination

In strontium isotope analysis containing rubidium contamination (as recognized by the presence of the mass 85 peak), the standard correction procedure of assuming the isotope ratio of the contaminating rubidium to be of normal composition was used (i.e.  $^{87}\text{Rb}/^{85}\text{Rb} = 0.385$ ). However, Pringle (1970) suggests that such a procedure is invalid because the isotope ratio of the contaminating rubidium is not of normal composition. This would be particularly evident if such contamination was caused by a "memory effect" of a previous spiked rubidium analysis. As a result, strontium isotope analyses were discarded if the contaminating rubidium contained 1) the presence of a spiked rubidium composition (i.e. a "memory effect"); 2) the presence of the mass 85 peak in a "growing" mode; and 3) the presence of  $^{85}\text{Rb}$  greater than or equal to 1% of the measured mass 87 peak.