Glacial Geology and Geochronology of Peggy's Cove Region

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Abstract

Peggy's Cove region is ideal for estimating paleo-ice margin retreat due to the high density of large (>2m) quartz bearing boulders in the area, for estimating glacial erosion rates, and is strategically situated near the coast so glacial chronology will help provide a link between marine and terrestrial chronologies. Seven boulders and four bedrock surfaces were sampled for exposure dating, and a surficial geology map with ice flow direction was determined for the area. The hypothesis (H) and question (Q) asked were; (H) The boulders on bedrock ridges near Peggy's Cove represent an ice marginal retreat between 17 to 15.9±0.3 kyr; and (Q) What is the effectiveness of glacial erosion on the Halifax Pluton during the Wisconsinan glaciation? Exposure dating using ^{10}Be, a cosmogenic nuclide was used to estimate the exposure age of the boulders, and a comparison of the concentrations of ^{10}Be in the boulders and bedrock surfaces lead to a simple glacial erosion model. The results of exposure dating showed a remarkable agreement between the terrestrial cosmogenic nuclide (TCN) exposure ages of the boulders, and calibrated marine and terrestrial ^{14}C dates. The weighted mean age of 6 boulders (excluding one outlier) indicates an ice marginal retreat at 16.4 ± 2.8 ka. Snow cover and erosion have probably had less than a 1% effect, and we assume continuous exposure since initial deglaciation. The ages imply that the ice was gone before the Younger Dryas, and ice flow readings indicate an ice flow direction of southwestern and southeastern, which is consistent with recent literature for the area. The glacial erosion on the streamlined ridges ranges from 0 to 2.6 m or 0 to 4.5 cm/kyr.

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Chapter 1: Introduction

In the 1980s and 1990s, two developments made major contributions to paleo-ice sheet dynamics, relative sea level studies, and paleoclimatology in Atlantic Canada. The first was a significant effort of mapping and radiocarbon dating of offshore glacial features by the Department of Natural Resources and the Geological Survey of Canada. The second was the rejuvenation by Stea et al. (1998) and others of the dynamic multi-ice cap model for the deglaciation proposed by Grant (1977). This conceptual model contrasts with the large-scale Laurentide Ice Sheet cover models that persist in the recent literature (e.g. Peltier, 1994). A new generation of complex thermomechanical ice sheet models (Fastook, 2002) have been kindled by the advances in ice sheet modeling. The new models are highly sensitive to the details of local ice marginal retreat history, ice flow direction, and ice geometry interpreted from field data. However, our picture of the Wisconsinan glaciation ice dynamics in Atlantic Canada remains limited by two important aspects. (1) There are over 500 radiocarbon dates that help constrain glacial sediment age, but the age control is insufficient to precisely determine the timing of ice marginal positions on land or offshore. (2) The paleo-ice flow data determined from striated or streamlined bedrock surfaces cannot be precisely dated. In other words, it is unclear if the observed striations represent flow of the last ice phase, or if the striations were formed from an earlier ice phase or advance.

To improve on these limitations, a study was conducted near Peggy's Cove, Nova Scotia, Canada to (1) tie the offshore record of deglaciation with the terrestrial record, (2) evaluate the existing calibrated radiocarbon-chronology-based conceptual models (e.g.

Piper and Fehr, 1991; Scott and Medioli, 1980; Stea et al., 1998), and (3) to use cosmogenic isotopes to help recognize landforms that persisted throughout the entire Wisconsinan glaciation (e.g the landforms only experienced a small amount of glacial erosion).

The Peggy's Cove region is ideal for determining the timing of ice marginal retreat and the amount of glacial erosion. There is a high density of large (>2m) quartz bearing boulders in the area, and a lack of surficial sediments so glaciated bedrock ridges are well exposed. The question of local ice margin retreat has been extensively evaluated by the Geological Survey of Canada (e.g. Piper and Fehr, 1991), and the Nova Scotia Department of Natural Resources (e.g. Stea et al., 1998) by radiocarbon dating of organic material (e.g. shells and wood) found in and around glacial sediments. These radiocarbon dates provide the age of sediment deposition but rarely provide a precise estimate of ice margin retreat. Individual ^{14}C dates in Nova Scotia have not conformed to a simple retreat model, and therefore have yielded different interpretations (Dyke, 2002). Furthermore, there have not been any detailed radiocarbon studies within 20 km of Peggy's Cove region.

Cosmogenic nuclide exposure dating provides a means to precisely date the timing of ice marginal positions. By utilizing the boulders and exposed bedrock ridges near Peggy's Cove, the timing of ice margin retreat and the amount of glacial erosion can be determined. The hypothesis (H) is the boulders on bedrock ridges near Peggy's Cove represent an ice margin retreat between 17 to 15.9 ka. The estimated timing of ice marginal retreat for the area is 15.9 to 17 ka based on the Chignecto Phase ice advance from Stea et al (1998), and a glacial readvance date of 15.9 ± 0.3 ka cal. from Josenhans and Lehman (1999). Additionally, the effectiveness of glacial erosion on the Halifax Pluton will be estimated, and this may help in selecting ice flow indicators for early vs late paleo ice flow history.

The concentration of ^{10}Be, a terrestrial in situ cosmogenic nuclide (TCN) will be used to determine the exposure ages of multiple suitable boulders. The concentration of ^{10}Be in the boulders will be compared to the bedrock to estimate the amount of glacial erosion on ridges in the Peggy's Cove region. The boulders and bedrock surfaces picked for sampling all had to meet stringent requirements outlined by Gosse and Phillips (2001) for sampling in the field (summarized in 3.2.1). Samples collected from seven boulders and four bedrock surfaces were physically and chemically processed to extract ^{10}Be in order to prepare the samples for accelerator mass spectrometry (AMS). The samples were sent to Lawrence Livermore Laboratory in California for AMS analysis. The AMS, chemical, and sample attribute data used to calculate the duration of exposure for each sampled boulder. A comparison of ^{10}Be concentration in the boulders and the bedrock surfaces allowed for the determination of glacial erosion depths.

A detailed surficial geology map on the scale of 1:10000 was constructed from field observations and aerial photographs of the Peggy's Cove region. Important glacial features (e.g. stoss-lee ridges and streamlined ridges), ice flow direction, and surficial deposits (e.g. exposed bedrock surfaces and lakes and lacustrine deposits) were observed in order to complete the map. A map of the sampled boulders and bedrock surfaces was completed using the aerial photograph and GPS locations of the samples.

The exposure ages of the seven boulders ranged from 15.7 to 35.8 kyr, and interpreted to indicate an ice marginal retreat at 16.4 ± 1.7 ka. The timing of ice marginal retreat determined by exposure dating is slightly earlier than previously thought by Stea et al. (1998) and Stea and Mott (1998), but within the window of time determined from the offshore record (16.8 ± 0.26 cal ka) and the land record of deglaciation in Nova Scotia (Stea et al., 1998). The ridges sampled experienced a wide range of glacial erosion from 0 to 2.5 m or 0 to 4.5cm/kyr, during the entire Wisconsinan glaciation. Probable reasons and implications of this erosion are proposed.

Chapter 2: Background Information

2.1 Glacial History of Nova Scotia

Nova Scotia has been covered and uncovered by glacier ice margin advances and retreats throughout the Pleistocene, with the last glacial maximum approximately at 23.7 ± 0.4 cal ka (Stea et al., 1998). To understand the context of exposure ages on boulders in the Peggy's Cove region, it is useful to review the relevant literature and evidence for the glacial stratigraphy and history for the Wisconsinan glaciation in southern Nova Scotia. The evidence of the glacial record for southern Nova Scotia comes from marine and terrestrial observations, and Peggy's Cove is in a strategic position to assist in the dating and correlation of the marine and terrestrial records. Radiocarbon ages that had to be calibrated, are noted by cal ka or cal kyr, and were calibrated using Calib 4.3 (refer to Appendix 1 for original dates).

Until recently, two opposing and long-standing conceptual models for the extent of the ice margin during the glaciation of Nova Scotia and Atlantic Canada have been discussed. The "maximum model" refers to extensive Late Wisconsinan northern ice sheets that covered Atlantic Canada (Denton and Hughes, 1981). The ice sheets extended across most of the continental shelf (Gipp and Piper, 1989)(Figure 1a). The ice sheets spread from local land-based centres located in Atlantic Canada, and would have been fringed by ice shelves at the ice margin (Grant, 1987). During glaciations, cold-based ice would exist on land (Grant, 1987). The "minimum model" refers to a restricted ice sheet cover over Atlantic Canada during the late Wisconsinan that would not have major offshore components (Denton and Hughes, 1981)(Figure 1b), and would extend just beyond the present coastline (Gipp and Piper, 1989). The regional ice sheet would leave exposed areas of land or nunataks, and would originate from the Appalachian ice sheet (Grant, 1987). Based on present deglacial chronology and correlation with the terrestrial and offshore record, attributes of both models appear to fit the deglacial history of Atlantic Canada (Stea et al., 1998), but not one single ice sheet. Instead multiple domes with the ice extent of the maximum model.

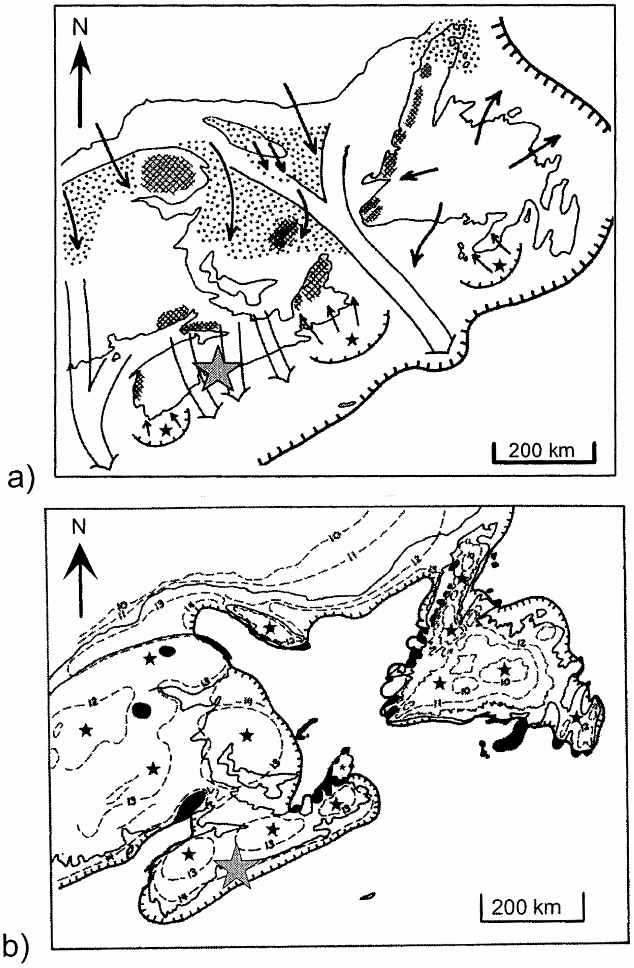


Figure 1 Maximum (a) and Minimum (b) models after Grant (1987). The ice margin is represented by the bold line, and the grey large star represents the Peggy's Cove region in (a) and (b). (a) Ice rises or offshore ice domes are represented by the small black stars, dotted areas represent areas of shield erratics, the cross-hatching shows cold-based ice zones, ice streams are represented by the large arrow, and ice flow direction is represented by the small arrows. (b) The ice centers are represented by the small black star, and nunataks are the black areas (Grant, 1987).

Of the first to designate different Atlantic Canada ice flows into ice flow phases were Rampton et al. (1984). Rampton et al. (1984) used glacial evidence in New Brunswick to distinguish 6 ice flow phases of Wisconsinan glaciation in New Brunswick. In Nova Scotia, King and Fader (1986) divided the Wisconsinan glaciation into 3 main stages: the Early Wisconsinan (approximately 120 to 60 Ka), Mid-Wisconsinan (approximately 65 to 23 Ka), and Late Wisconsinan (approximately 23 to cal. 11.5 ± 0.3 ka). Later, Stea et al. (1998) attempted a more formal nomenclature after Rampton et al. (1984), and divided the Late Wisconsinan glaciations of Nova Scotia into 5 ice-flow phases. The five ice-flow phases are: the Caledonia Phase (ca. 75 to 40 ka)(Figure 2); Escuminac Phase (25 to 21.4 cal ka)(Figure 5); Scotian Phase (21.4 ±0.35 to 17.9 ±0.3 cal ka)(Figure 6); Chignecto Phase (15.6 ± 0.35 to 14.8 ± 0.55 cal ka)(Figure7); the Colins Pond Phase (12.7 ± 0.17 cal ka) (Figure 16). The glacial history of Nova Scotia will be discussed according to the ice flow phases 1 to 4 that Stea et al. (1998) has designated, and Phase 5 will be discussed in section 2.3. The relevance of each phase to the Peggy's Cove area will be discussed at the end of each section. The extent of ice margin advance offshore of Nova Scotia will be discussed here, and the glacial geology offshore of Nova Scotia will be described in section 2.2.

2.1.1 Caledonia Phase (Early to Mid-Wisconsinan ice-flow phase 1)

The earliest and most extensive ice-flow in Nova Scotia for which there is a record occurred during the Caledonia phase in an eastward flow direction that was later cross cut by a southeastward flow direction (Stea et al., 1992). Stea et al. (1992)

suggested that the eastward flow direction may represent an older, separate phase of glaciation that may go back to the Illinoian (140 to 180 ka), but Stea et al. (1998) decided it was Early Wisconsinan. The direction of ice-flow was determined from striation patterns, striated boulder pavements, till fabric, and erratics (Stea et al., 1992). The source of ice-flow may have been from an ice center in New Brunswick called the Gaspereau ice center (Rampton et al., 1984), but there is still uncertainty regarding the source (Stea et al., 1998). During this phase, glacier ice extended to the edge of the Scotian Shelf (Stea et al., 1998).

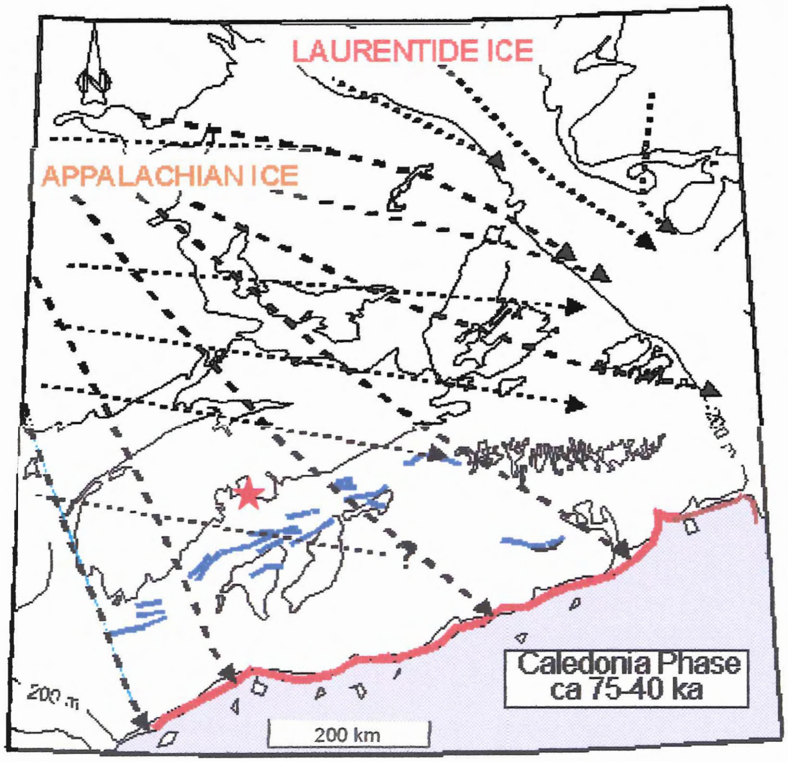


Figure 2 Caledonia Phase: The dashed arrows represent ice flow direction, the red line represents the extent of the ice margin, the blue lines represent the Scotian Shelf end moraine complex. The red star represents Peggy's Cove region (From Stea et al., 1998).

In southwestern Nova Scotia, three tills overlie a wave cut rock bench, 4-6 m above sea-level: (1) Little Brook Till, (2) Red Head Till, and (3) the Unnamed Till (Stea et al., 1992)(Figure 3). The Little Brook Till overlies the wave-cut rock bench and is a stony, massive diamicton that contains clasts derived from local sources (Stea et al., 1992).The till may have been deposited by a Nova Scotian based ice cap (Grant, 1980). The Red Head Till named by Grant, (1980) is a matrix supported diamicton with a greyish-red colour, and contains erratics from New Brunswick (Stea et al., 1992). The till is considered to be a lodgement till (Stea et al., 1992).Shell fragments from the Bay of Fundy are located in the Red Head Till and are dated at >38 uncal ka (Stea et al., 1992).The Little Brook Till, and the Red Head Till may have been formed during the Illinoian glaciation (Stea et al., 1992).The Salmon River Sand overlies the Red Head Till in southern Nova Scotia, and may be of Sangamonian age based on the warm-water fauna, and shells found in the sand (Clarke and Grant, 1972). Nielsen (1974) interpreted the sands as middle Wisconsinan glaciomarine deposit. Two possibilities for the origin of the sand were proposed by Grant and King (1984): (1) the sand is part of a thrust sheet, and (2) the sands are a glaciomarine deposit, deposited during the middle Wisconsinan (Nielson, 1974). Overlying the Salmon River Sands is a till called the Unnamed Till (Stea et al., 1998).

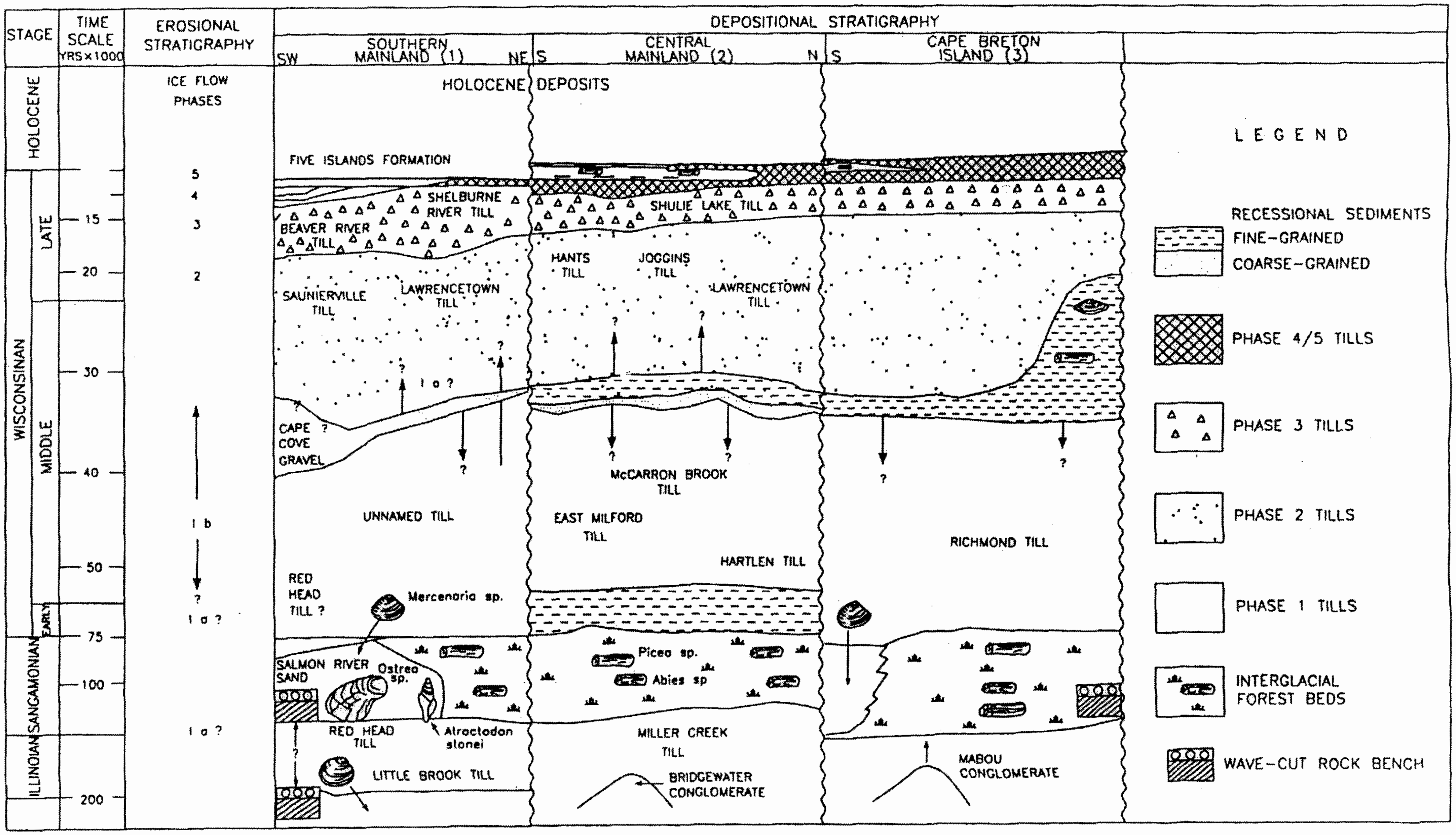


Figure 3: Depositional Stratigraphy of Nova Scotia (From Stea et al., 1998)

At the end of the Caledonia phase, the glacier margin reached to the Scotian Slope, terminating in 300-600 m deep water (Stea et al., 1998) (Figure 4). During the mid-Wisconsinan (ca. 40-22 ka), the ice sheet retreated leaving the outer regions of the Gulf of Maine, the St. Lawrence River and parts of the Scotian Shelf ice-free (Stea et al., 1998).

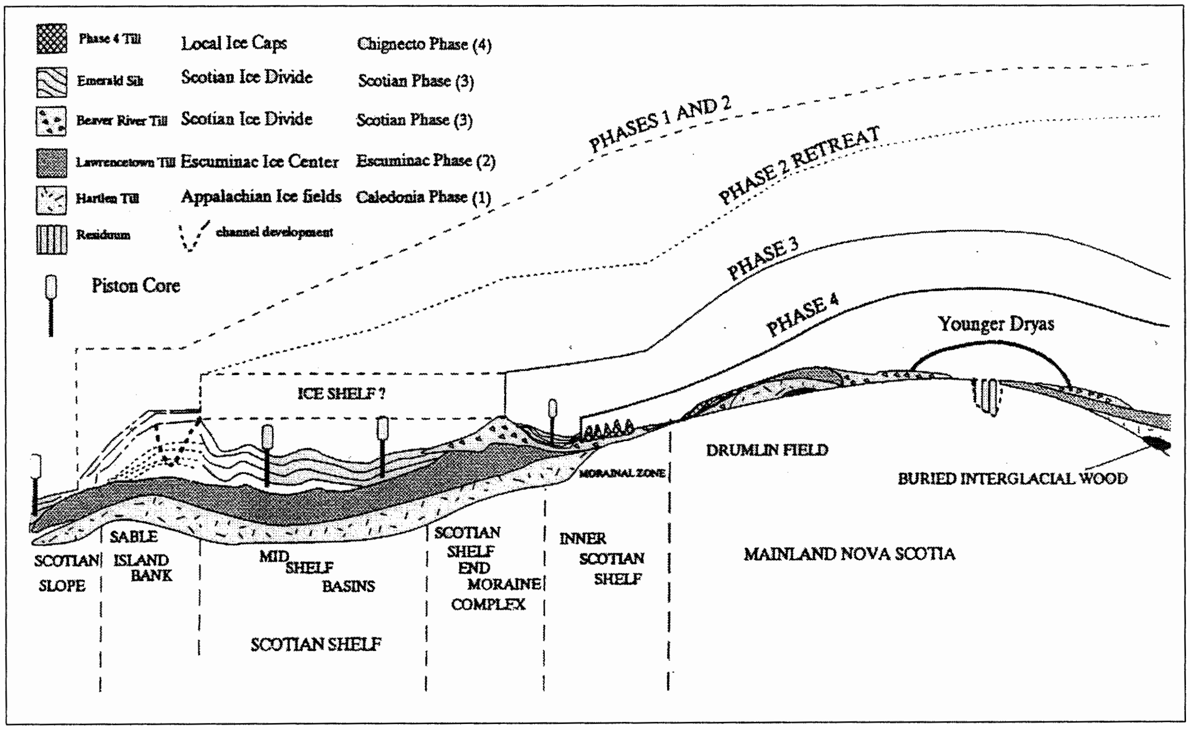


Figure 4 Conceptual Cross section of ice sheet extent from Mainland Nova Scotia to the Scotian Slope (After Stea et al., 1998).

During the Caledonia Phase, Peggy's Cove region was covered by ice that extended out onto the Scotian Shelf (Stea et al., 1998)(Figure 2). The ice margin retreated at the end of the Caledonia phase, but Peggy's Cove region likely remained covered by ice (Stea et al., 1998).

2.1.2 Escuminac Phase (late Wisconsinan ice-flow phase 2)

The Escuminac Phase corresponds to the last glacial maximum during the Wisconsinan period. During the Escuminac phase, the ice-flow direction moved from the eastward and southeastward direction, to a southward and southwestward flow direction recorded by striated outcrops in Nova Scotia and Prince Edward Island (Stea et al., 1998) (Figure 5). The striated outcrops are a result of the Escuminac ice centre (Rampton et al., 1984) positioned north of Prince Edward Island on the Magdalen Shelf, (Stea et al., 1992) and the abutting Chignecto glacier, which was active for a short time prior to the Scotian Phase (Stea et al., 1998). The Escuminac ice centre is thought to have flowed into Chignecto Bay, and the Bay of Fundy by Rampton et al. (1984), Stea (1983), and others. Also, Stea et al. (1992) believed that ice flow from the local ice centre was radial. The "maximum model" of Grant (1987) best describes the extent of ice during the Escuminac phase, which reached to the edge of the Scotian Shelf (Stea et al., 1998).

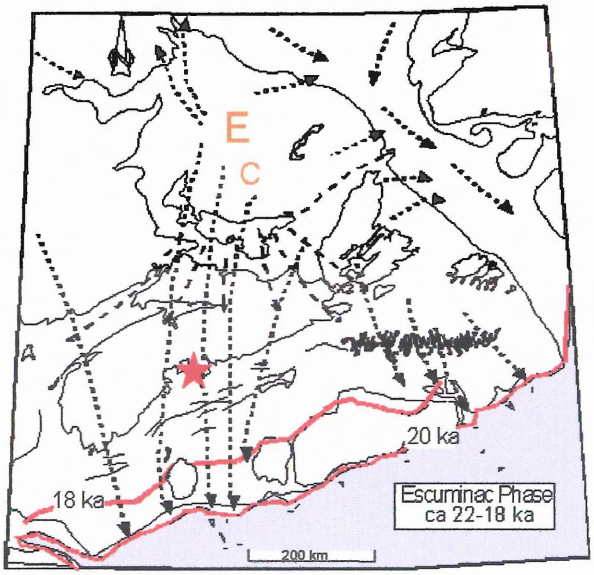


Figure 5 Escuminac Phase: The dashed black arrows represent ice flow direction, the red line represents the extent of the ice margin, E is the Escumianc ice center, and C is the Chignecto glacier. Peggy's Cove region is represented by the red star (From Stea et al. 1998).

In southern Nova Scotia two tills are present, the Saunier Till, and the Lawrencetown Till that overly gravel deposits named the Cape Cove Gravel (Stea et al., 1998) (Figure 2.3). The Lawrencetown Till was deposited during southward transport of sediment from the Magdalen Shelf onto the Meguma zone terrain (Stea et al., 1994). The distinctive red colour of the Lawrencetown Till is from red, hematitic sediments that were transported by ice flow from the Magdalen Shelf (Stea et al., 1994). A large percentage of foreign pebbles, from the Cobequid Highlands of the Avalon zone, characterize this till (Stea et al., 1998).

The ice margin during the Escuminac Phase reached to the edge of the Scotian Shelf (Figure 4), and ice streams flowed from the glacier across the Scotian Shelf to the outer banks, and stopped in 300 m of water (Stea et al., 1998). From offshore seismic records and core samples, ice-marginal tills form wedges that intrude into red-brown glaciomarine sediments (King and Fader, 1986). The ice-marginal tills are thought to represent an ice-flow event that occurred between 18 to 21 uncal. Ka (add 2.5 kyr for approximated calibration) (Stea et al., 1998).

The retreat of the ice margin during the Escuminac Phase was initiated by sea-level rise and calving (Stea et al., 1998). As the Escuminac Phase glacier began to retreat near the shelf edge, it may have contributed to ice rafted debris that appears as a Heinrich layer (H2) in cores from the deep ocean (Stea et al., 1998). The Heinrich layer (H2) has been dated between 18.9 to 21.4 ka (Heinrich, 1988). Ice streams formed underneath the Escuminac ice sheet, and flowed around Sable Island forming large channels (Stea et al., 1998). The Emerald Basin began to become free of ice between 20.8 ± 0.35 to 21.4 ± 0.35 cal ka (Gipp and Piper, 1989) based on linear extrapolation between two radiocarbon dates (King and Fader, 1986). The LaHave Basin deglaciation started around 23.7 ± 0.4 cal ka, and ice withdrew from the basin around 22 ± 0.36 cal ka (Piper and Fehr, 1991). The Peggy's Cove region remained ice covered during this phase, and was influenced by the mainly Southern ice flow (Stea et al., 1998) (Figure 5).

2.1.3 Scotian Phase (late Wisconsinan ice-flow phase 3)

Following the glacial maximum Escuminac Phase, glacier ice began to thin and undergo major ice dynamic changes as topography became an increasingly more important influence (Stea et al., 1998). The Escuminac ice centre began to disintegrate due to ice-stream drawdown into marine channels, and the Scotian ice divide was formed over Nova Scotia as a result of the disintegrating ice centre (Stea et al., 1998) (Figure 6). Ice flow from the divide, was funnelled northwestward into the Bay of Fundy, and southeastward over the eastern shore of Nova Scotia (Stea et al., 1998). During this Scotian phase, the ice margin reached to the edge of the Scotian Shelf End-Moraine Complex (Stea et al., 1998) (Figure 6).

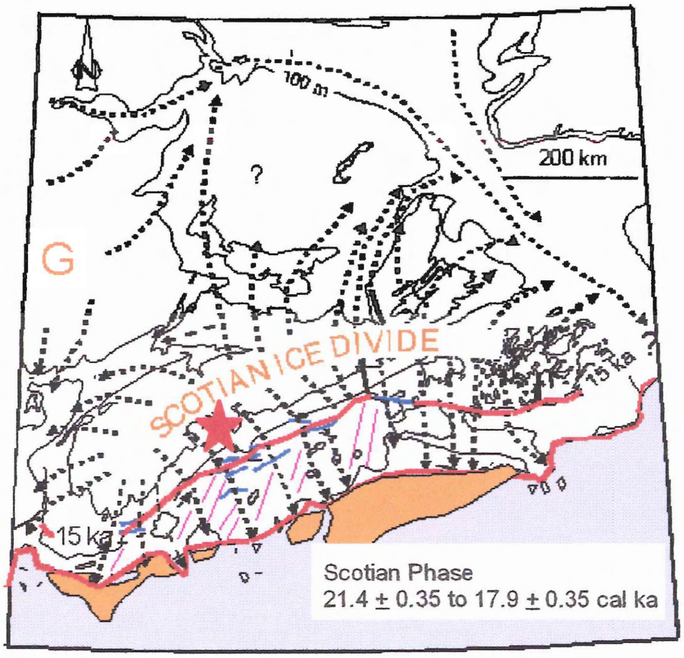


Figure 6 Scotian Phase: The dashed lines with arrows represent ice flow direction, orange area is emerging marine landscapes, the red line is ice margin extent, G represents the Gaspereau ice center in New Brunswick, and the Scotian Ice divide is a local ice center over Nova Scotia. The red star represents Peggy's Cove region (From Stea et al., 1998).

In southern Nova Scotia two tills are observed, the Beaver River Till and the Shelburne River Till (Stea et al., 1998) (Figure 3). The type section for the Beaver Till facies for the Scotian phase is located near Yarmouth, approximately 400 km from Peggy's Cove on the southern tip of Nova Scotia (Stea et al., 1998). The Beaver Till is a sandy, stony till derived from local bedrock sources, and characterised by angular to subangular cobbles, and boulders (Stea et al., 1998). The till has been interpreted by Stea et al. (1989) to be an ice-divide till. The till forms a till plain over the Atlantic coast and in drumlin sections, and overlies the Lawrencetown and Hartlen Tills (Stea et al., 1998) (Figure 3). There was no information found on the Shelburne till.

Climatic warming and sea-level rise following the outer shelf lowstand around 20.2 ± 0.35 to 17.9 ± 0.3 cal ka (King, 1994) initiated calving and rapid retreat of the Scotian Shelf ice margins (Stea et al., 1998). The Scotian Shelf end moraine complex formed during this phase between 18.5 ± 0.3 to 20.2 ± 0.35 cal ka (Stea et al., 1998). Heinrich event (H1) sediments offshore have been dated at 13.4 to 15.0 ka (Grousset et al., 1993), and may be due to ice rafted debris from the retreating Scotian ice margin (Stea et al., 1998). The Emerald Silt facies A and B were deposited as ice receded out of marine areas on the inner Scotian Shelf (Stea and Mott, 1998). The ice margin had retreated close to the inner continental shelf of Nova Scotia by 16.8 ± 0.26 cal ka, and most of the ice was removed from the Gulf of Maine, Bay of Fundy, and the Laurentian Channel between 15.6 ± 0.35 to 17.9 ± 0.3 cal ka (Stea et al., 1998). The DeGeer Sea moved into sunken coastal regions, and beaches were formed as the ice melted (Stea et al., 1998). These beaches are now located 30 m above present sea-levels (Grant, 1989; Stea et al., 1987).

During the Scotian Phase the Scotian ice flowed southeastward over the Eastern shore and the margin retreated to the Inner Scotian Shelf (Stea et al., 1998). Peggy's Cove remained under ice as sea-level rose (Stea et al., 1998).

2.1.4 Chignecto Phase (Latest Wisconsinan ice-flow phase 4)

Ice advance during the Chignecto phase was in a westward direction across Nova Scotia from a number of small local ice caps (Stea et al., 1998) (Figure 7). The local ice caps were the South Mountain ice cap located over the southern tip of Nova Scotia, the

Chignecto glacier located in the Northumberland Srait area, and the Antigonish-Chedabucto Bay Glacier located in the Antigonish Highlands (Stea et al., 1998). During this phase, the ice extended to just offshore of the present-day Nova Scotian coastline (Stea et al., 1998).

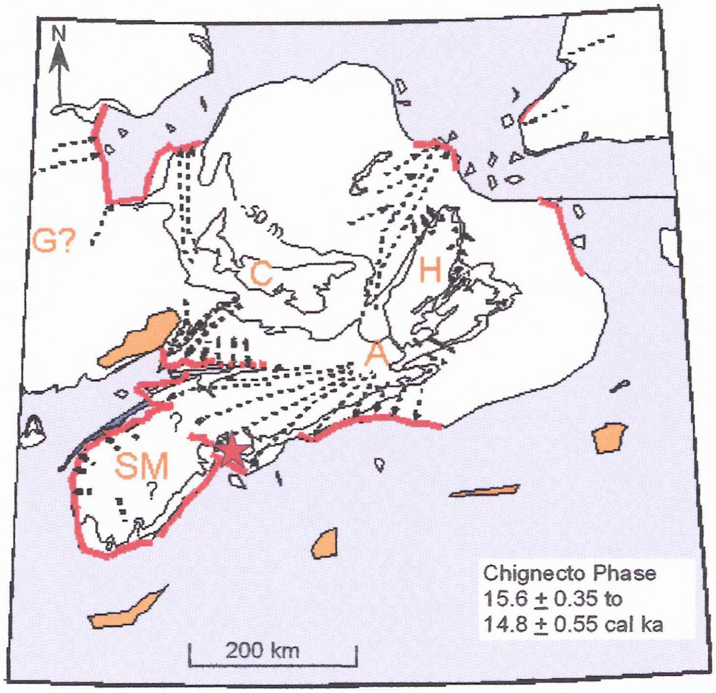


Figure 7 Chignecto Phase: The orange areas are emerging marine landscapes, the red lines are the ice margin extent, the dashed lines with arrows represents ice flow direction, and the letters represent local ice centers (G = Gaspereau ice center; C = Chignecto glacier; H =Cape Breton Highlands; A = Antigonish Highlands and Chedabucto Bay; SM = South Mountain). The red star represents Peggy's Cove region (Adapted from Stea et al., 1998).

A cross-valley moraine called the Gilbert Valley moraine is thought to have formed between 13.3 ± 0.15 to 15.9 ± 0.3 cal ka based on basal lake dates of wood (AMS) (Stea and Mott, 1989). The wood pieces were located in lakes that were situated in front and behind the ice margin during glaciation (Stea and Mott, 1989). The ice margin is marked by outwash deltas that grade to 40+ m above sea level on the Bay of Fundy coast (Stea et al., 1987).

Hummocky ground moraines were deposited throughout the marginal regions of the ice sheet as the glaciers ablated by downwasting (Grant, 1977). The Atlantic Coast was ice free at 14 ± 0.17 cal ka, and the deglaciation of southern Nova Scotia began slightly earlier (Stea and Mott, 1998). Relative sea-level of –65 m was recorded around 13.6 ± 0.18 cal ka on the Inner Scotian Shelf (Stea et al., 1998). Relative sea-level rose quickly after 13.6 ± 0.18 cal ka, causing erosion on the Inner Scotian Shelf that lead to a surface with few depositional features (Stea et al., 1998). Between cal. 10.1 ± 0.16 to 13.3 ± 0.1 relative sea-level rising slowed, which may mark the passage of the peripheral bulge as the glacier retreated (Quinlan and Beaumont, 1982; Stea et al., 1998).

During the Chignecto Phase, ice advances over Nova Scotia terminating just offshore of the present-day coastline (Stea et al., 1998). Peggy's Cove remained under ice, and sea-level rose, submerging more of the central Scotian Shelf (Stea et al., 1998) (Figure 7).

2.2 Glacial History of Offshore Nova Scotia

The glacial history of the Scotian Shelf is important for correlation of glacial activities on land and offshore. The first work on the glacial history of the Scotian Shelf, divided the shelf into three main physiographic zones called the inner zone (Inner Scotian Shelf), middle zone (Mid Scotian Shelf), and the outer zone (Drapeau and King, 1972; King, 1970). The following summary provides a brief overview of the glacial record on the Inner Scotian Shelf near Peggy's Cove region.

2.2.1 Inner Scotian Shelf

Stea et al. (1994) divided the inner Scotian Shelf into 5 subzones that are parallel to the coast of Nova Scotia. In a landward direction, the 5 zones are: (1) the Scotian Shelf End-Moraine Complex (King et al., 1972), (2) the Basin Zone, (3) the Outcrop Zone (Forbes et al., 1991), (4) the Morainal Zone (Stea et al., 1993), and (5) the Truncation Zone (Stea et al., 1994).

The Scotian Shelf End-Moraine complex occurs 30 to 40 km offshore (King and Fader, 1986) (Figure 8), as a series of large ridges parallel to the present coast, and represents the extent that the ice margin reached during Phase 2 or the Escuminac Phase (Stea et al., 1998). Three incoherent seismic units in the Scotian Shelf end moraine complex correlate with the Hartlen Till from central Nova Scotia (King et al., 1972).

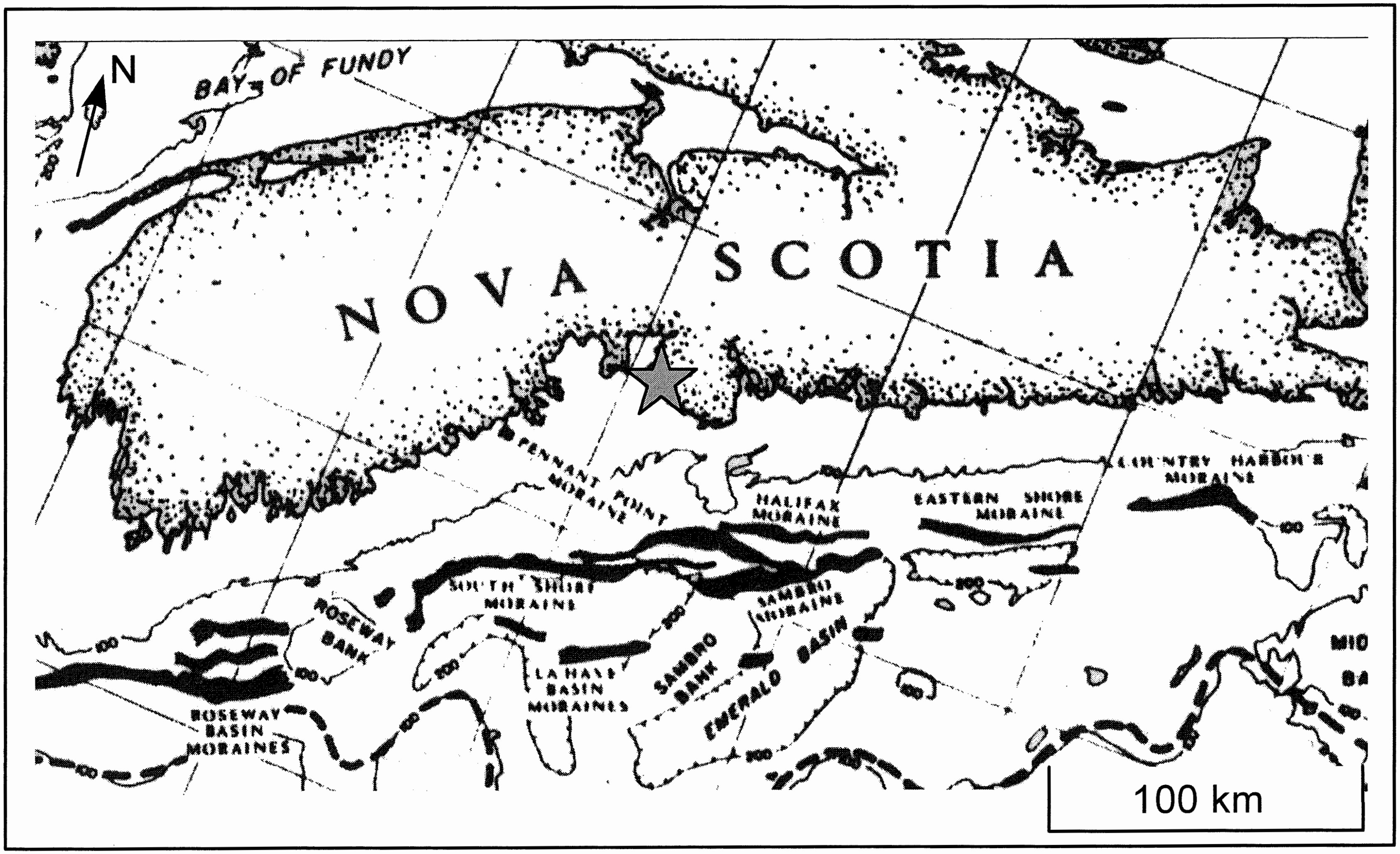


Figure 8 Location of the Scotian Shelf end moraine complex on the Inner Zone of the Scotian Shelf. The black areas represent the Scotian Shelf end moraine complex, and the grey star represents Peggy's Cove region (After King and Fader, 1986).

The Basin Zone of the Inner Scotian Shelf is divided in three zones; (1) Scotian Shelf Drift, (2) Emerald Silt, and (3) the LaHave Clay (King and Fader, 1986). The Scotian Shelf Drift is a dark greyish brown till consisting of poorly sorted sandy clay, silt, and gravel that overlies the bedrock surface as a continuous uniform layer (King and Fader, 1986). Till tongues are wedgeshaped bodies of till associated with the stratigraphic transition zones between the Emerald Silt and the Scotian Shelf Drift (King and Fader, 1986). The Emerald Silt facies has a conformable style of deposition, has been interpreted as a glaciomarine deposit (King and Fader, 1986), and may have been deposited during a retreat of a partially floating ice margin towards land, during ice-flow Phase 3 or 17.9 ± 0.35 to 21.4 ± 0.3 cal ka. (Stea et al., 1998) The LaHave Clay is a greyish brown clay grading to silt, confined mostly to depressions and basins on the shelf (King and Fader, 1986), and is thought to be a Holocene marine deposit (Stea et al., 1994).

The outcrop zone is a large area of acoustic basement devoid of surface sediments located in 80 to 120 m water depth (Forbes et al., 1991). The lack of sediments may be due to three erosional mechanisms: (1) coastal transgression and wave-erosion, (2) local glacial erosion, and (3) subglacial meltwater erosion (Stea et al., 1994).

The type section for the Morainal zone is located off of Sheet Harbour (Stea et al., 1994) (Figure 9). The most prominent feature along the Morainal Zone is a series of symmetrical ridges arranged in a staggered system (Stea et al., 1994). The ridges are composed of a matrix-supported diamicton with cobbles, and erratics randomly scattered along the crests (Stea et al., 1994). Goldthwait (1989) interpreted these ridges as marginal or subglacial moraines, and Stea et al. (1998) believed that the ridges formed during ice-flow Phase 4 or the Chignecto Phase.

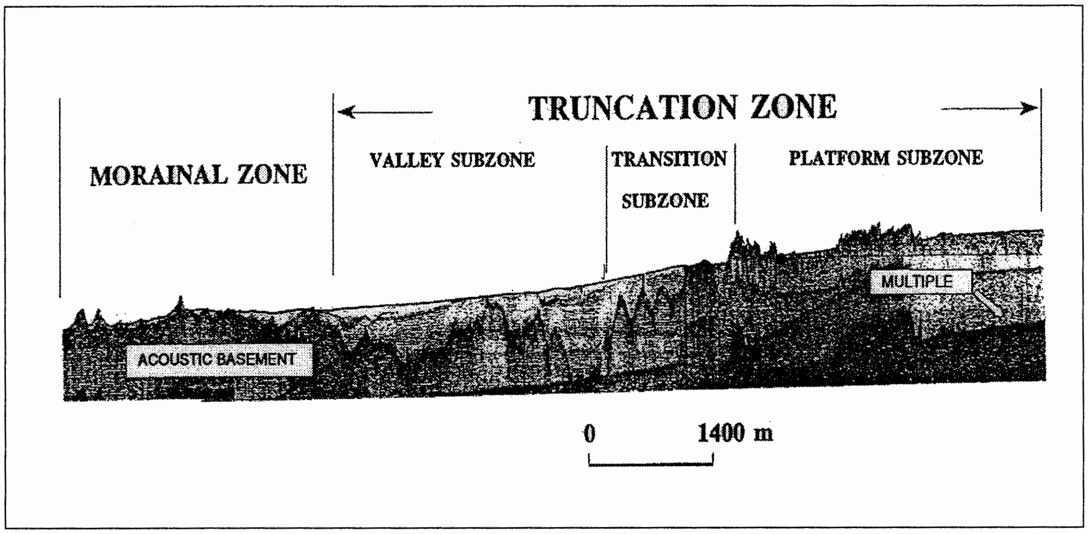


Figure 9 Transect off of Sheet Harbour showing the Morainal Zone, and the Valley subzone, Transition subzone and the Platform subzone of the Truncation zone (From Stea et al., 1994).

In the Morainal zone, DeGeer Moraines are orientated perpendicular to the southwestward ice flow direction during Chignecto Phase (Stea et al., 1998). DeGeer moraines or cross-valley moraines (Gipp, 2001) are narrow ridges with steep-sides, and consist of till (King and Fader, 1986). The orientation of the moraines may represent the extent that the Southern margin of the Antigonish Highlands reached offshore of Nova Scotia (Stea et al., 1998).

The truncation zone is located from water depths of 90 m to the present-day shoreline of Nova Scotia (Stea et al., 1994). Stea et al. (1994) subdivided the Truncation Zone into 4 subzones: (1) the Valley Subzone, (2) the Transition Subzone, (3) the Platform Subzone, and (4) the Esturaine Subzone (Figure 2.10). The Valley Subzone is located in 90 m of water, and consists of a sediment-infilled valley 40-70 m deep (Stea et al., 1994). The transition zone is a steep ramp, or terrace leading to a planar surface or the Platform Subzone (Stea et al., 1994) (Figure 9). Located in the transition Zone is the Sambro Delta that is a progradational feature (Stea et al., 1994). The top of the ramp surface, and the topset/foreset contact of the Sambro Delta has been interpreted to be the former sea-level at –65 m, and has been dated at 13.6 ± 0.18 cal ka (Stea et al., 1994). The Platform Subzone starts in water depths of 65 m, and is defined as a low relief surface cut into the bedrock, that is thought to represent a paleo-shoreline (Stea et al., 1994) (Figure 9). The Estuarine Subzone is confined to water depths of less than 50 m, and is located in the marine valleys (Stea et al., 1994).

A study by Piper et al., (1986) looked at the offshore record of the Inner Scotian Shelf located near the South Shore from Peggy's Cove to Lockeport (Figure 10).

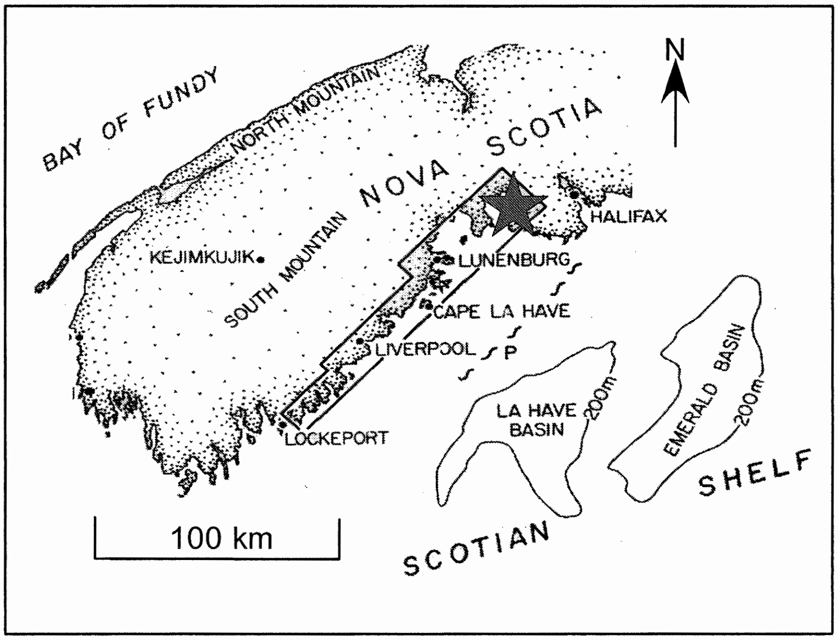


Figure 10 Location of Inner Scotian Shelf study of the South shore by Piper et al., 1986. Peggy's Cove region is represented by the grey star (After Piper et al., 1986).

St. Margaret's Bay is a large depression orientated in a south-southeast direction near Peggy's Cove (Piper et al., 1986). In the shallow water of St. Margaret's Bay, Sable Island sand and gravel covers eroded till platforms (Piper et al., 1986). In St. Margaret's Bay, flat-topped drumlins are well preserved, and are 10's of metres high allowing some drumlins to emerge as islands (Piper et al., 1986). An extensive drumlin field called the Lunenburg Drumlin Field extends between St. Margaret's Bay and the LaHave Estuary (Flint, 1971) (Figure 11). The drumlins consist of red till that is similar to the terrestrial Lawrencetown Till found in southern and central Nova Scotia (Piper et al., 1986). Beneath some of the drumlins in the Lunenburg area is a grey till (Nielson, 1976) that is similar to the Hartlen Till in Central Nova Scotia (Piper et al., 1986). During the lowstand of –120 m, offshore till was modified by wave erosion and the subsequent transgression (Piper et al., 1986).

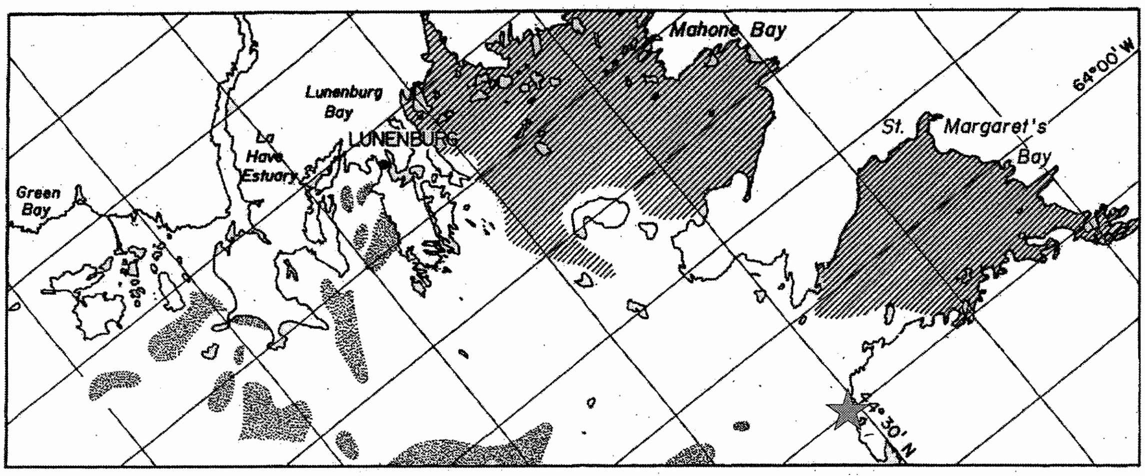


Figure 11 Distribution of Till on the South Shore Inner Scotian shelf. The parallel lines represent areas of till in bays, the dots represent eroded areas of till on the continental shelf, and the grey star represents Peggy's Cove region (From Piper et al., 1986).

Two cores (74-01 and 77-12) were obtained from the central basin of St, Margaret's Bay, and one core (73-4) was obtained from the mouth of St. Margaret's Bay (Piper et al., 1986). Core 74-01 is from acoustic unit d of King (1970) that is a transparent unit of mud that may be equivalent to the LaHave Clay. Facies 2a and 2b of core 77-12 is similar to the Emerald Silt, and facies 1a and 1b are similar to the LaHave clay and the Sable Island sand and gravel (Piper et al., 1986).

2.2.2 Relative Sea-Level

The concept of a migrating peripheral bulge in relation to ice sheets is important for understanding relative sea-level. The peripheral bulge is created by mantle material that is pushed to the side as the weight of the ice sheet pushes down on the surface (Quinlan and Beaumont, 1981). The mantle material that is pushed to the side causes uplift of a flexed lithosphere and forms the peripheral bulge near the ice margin (Scott et al., 1987). When the ice sheets begin to melt, the change in the distribution of overlying weight causes the mantle material that has been pushed aside to flow back under the rebounding lithosphere, which allows the bulge to migrate towards the centre of the receding ice sheet (Scott et al., 1987). The migration of the bulge is not able to keep up with the more rapidly receding ice margin, and will continue to migrate long after the ice sheets have receded (Quinlan and Beaumont, 1981). Different relative sea-level histories of different locations are produced as the bulge migrates through the area causing the locations to experience uplift, and then subsidence after the bulge has continued past the locations (Quinlan and Beaumont, 1981).

Quinlan & Beaumont (1981) used a simplified lithospheric flexure model to divide coastal regions into four relative sea-level zones. Zone A sites may have been subaerially exposed or exist in shallow water, and occur onshore or offshore (Quinlan and Beaumont, 1981). Zone B sites may contain an erosional unconformity that marks the transition of falling relative sea-level (Quinlan and Beaumont, 1981). Zone C sites are characterized by the lack of raised markers, and Zone D is located beyond the area that the glacial bulge reached (Quinlan and Beaumont, 1981). The model produced by Quinlan and Beaumont (1981), can be related to the “maximum” and “minimum” ice models. The "maximum model” has the four zones of relative sea-level in Atlantic Canada with Nova Scotia in zone B and the Scotian Shelf in zone C (Figure 12a). The “minimum model” shows Atlantic Canada in zones B-D of relative sea-level, with Nova Scotia and parts of the Scotian Shelf in zone C, and the outer Scotian Shelf in zone D (Quinlan and Beaumont, 1981) (Figure 12b). Peggy’s Cove falls in zone B for the “maximum model”, and zone C for the “minimum model”.

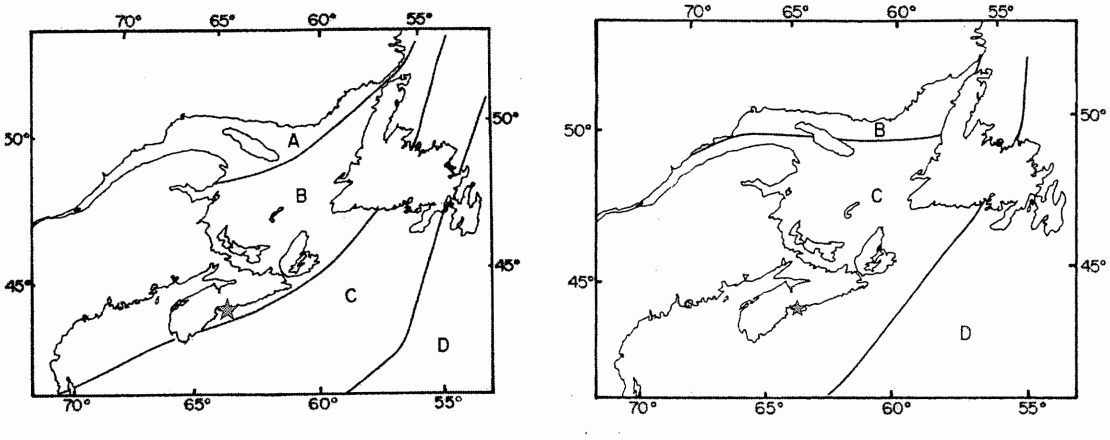


Figure 12 The relative sea-level zones in relation to the “maximum” ice model (a), and the “minimum” ice model (b). The small grey star represents Peggy’s Cove region .(Quinlan and Beaumont, 1981).

The sea-level curve for the Atlantic coast of Nova Scotia located near Chezzetcook Inlet, correlates with zone C of the relative sea-level models from Quinlan and Beaumont (1981) (Figure 13). In zone C, relative sea-level fell as the peripheral bulge approached, then sea-level rose until present after the peripheral bulge had past (Scott et al., 1987).

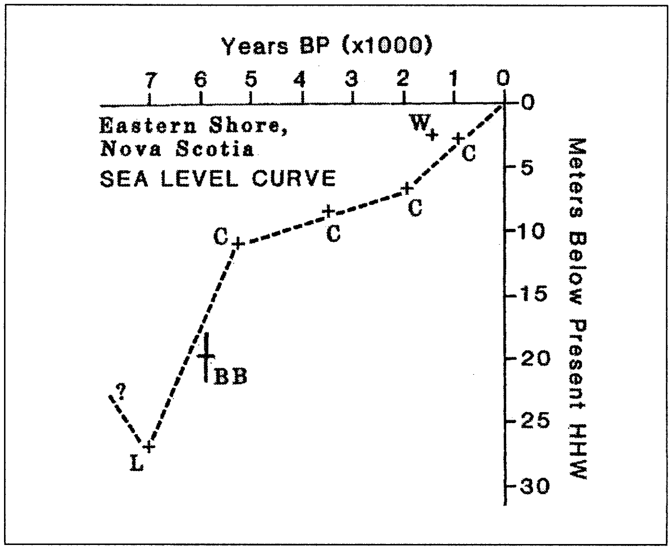


Figure 13 Sea level curve for the Eastern Shore, Nova Scotia over the last 7 uncal ka (From Scott et al., 1987).

A six stage evolutionary model summarises the sedimentation pattern on the coast of Nova Scotia (Scott et al., 1987)(Figure 14). During stage 1, NW to SE elongate scours where produced by Pleistocene glaciations, and also deposited drumlin till, and lodgement or ablation till (Scott et al., 1987). Stage 2 transgressions where produced after the forebulge passed, and relative sea-level began to rise (Scott et al., 1987). Stages 3 to 6 represent a cyclic barrier evolution of generation, retreat, destruction, and landward migration before re-establishment (Scott et al., 1987).

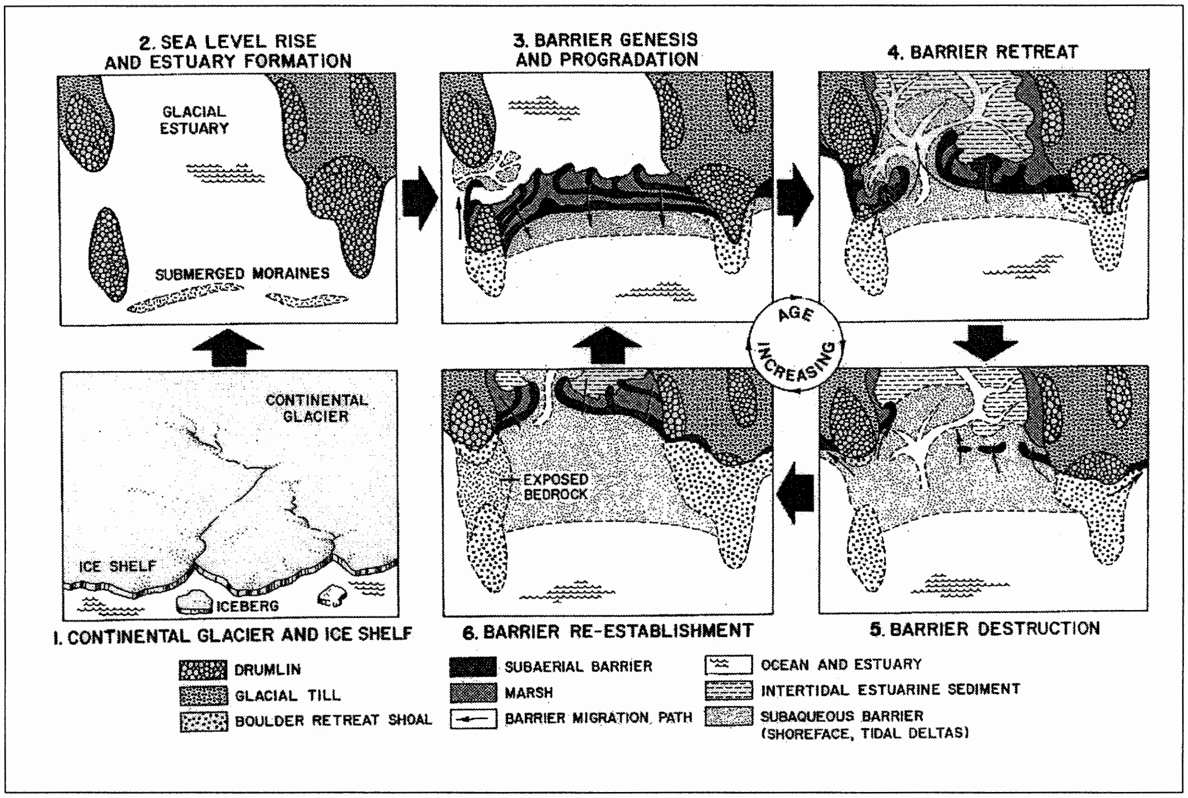


Figure 14 An evolutionary model showing the six stages of sedimentation patterns in zone C of the Atlantic Coast, Nova Scotia (From Scott et al., 1987).

New data from Halifax harbour by Edgecombe et al. (1999) has been added to the relative sea-level curve to produce a more complete sea-level curve for the inner Scotian Shelf (Figure 15). The sea-level curve shows that from 8.4 to 7.0 ka, Halifax Harbour experienced an emergence, and from 7.0 ka to present, Halifax harbour has been submerging (Edgecombe et al., 1999). Zone C from Scott et al. (1987), and Quinlan and Beaumont (1981) corresponds to the new sea-level curve (Edgecombe et al., 1999). The data for Halifax Harbour is the closest available data to Peggy’s Cove region for the relative sea-level curve, and the new relative sea-level curve for the Inner Scotian shelf may be applied to the region.

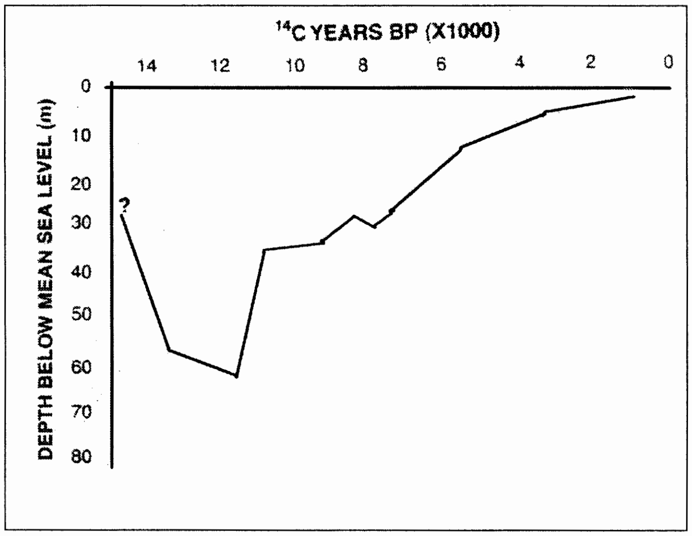


Figure 15 The new relative sea-level curve for the Scotian Inner Shelf produced by Edgecombe et al., (1999). The curve was redrawn from Stea et al. (1994).

2.3 Paleoclimatology

Any paleoclimate information on land we have, is mostly from paleovegetation records, fossil pollen, and preserved beetle assemblages (Clarke and Grant, 1972; Miller, 1995). Marine organisms from offshore have also been used (Mott and Grant, 1985). The $\delta^{18}O$ is dominated by ice-volume changes due to glacial and interglacial periods (Chappell and Shakleton, 1986). Using the marine oxygen isotope record, it is convenient to subdivide the last 150 kyr into four paleoclimate stages; Sangamon (oxygen isotope stage 5: substage 5a to 5e), Wisconsinan (oxygen isotope stage 4-2), Younger Dryas (after oxygen isotope stage 2), Holocene (oxygen isotope stage 1) (Ruddiman and McIntyre, 1981).

The Sangamonian interglacial period (oxygen isotope stage 5) lasted from 127 to 73 kyr (Ruddiman and McIntyre, 1981). There is not much evidence of the Sangamonian in Nova Scotia because glaciers during the Wisconsinan have eroded or buried most of the stratigraphic evidence. There has been some evidence found by de Vernal et al., (1983) in Cape Breton, and de Vernal et al., (1984) along East Bay; Mott and Grant (1985) provided an overview of the paleoenvironments based on previous work. In Nova Scotia, Mott and Grant (1985) divided the organic deposits into three Sangamonian stratigraphic units each containing a distinct pollen spectrum. The first interval (substage 5e) was the warmest interval during the Sangamonian and was much warmer than the present day climate (Mott and Grant, 1985). The second interval (substage 5b) was very similar to the present climate, and the third interval (substage 5a or maybe Stage 4) fluctuated between warm and cool periods, with each cool period being colder than its predecessor (Mott and Grant, 1985).

The Wisconsinan glaciation lasted from ca. 75 kyr to its glacial maximum at ca. 20 kyr (Stea et al., 1998). After ca. 20 ka, deglaciation occurred as the glacier retreated to remnant ice caps over Nova Scotia as the climate warmed (Stea et al., 1998). There is not much evidence of paleoclimate due to continuous ice cover throughout the Wisconsinan.

The Younger Dryas Chronozone (Younger Dryas) is a climatic cooling that occurred after a period of climate warming during the last interglacial period (Stea et al., 1998). The Younger Dryas lasted for 1.3 kyr years from 12.8 to 11.5 cal ka (Taylor et al., 1993). Some possible mechanisms for the origin of the Younger Dryas are ice shelf breakup (Mercer, 1969), melt-water diversion from the Laurentide ice sheet (Broecker et al., 1985), or a change in deepwater formation in the North Atlantic Ocean (Broecker et al., 1985). The Younger Dryas event is recorded in Atlantic Canada by buried organic deposits, and lake sediments (Mott et al., 1986). After deglaciation, vegetation began to invade the region and the buried organic deposits began to accumulate as shallow pond sediments, mire sediments, and organic soils (Mott et al., 1986). Mineral sediments where deposited on top of the organic deposits due to a climatic shift attributed to the Younger Dryas that affected depositional environments (Mott et al., 1986). Deposits of fluvial gravel and sand, or lacustrine sand, silt, and clay, or diamicton may truncate the peat deposits (Stea and Mott, 1989). Miller (1995) looked at beetle assemblages at Hirtles in southwestern Nova Scotia to determine paleoclimate. The mean July temperatures at 12.3 ± 0.17 cal ka were 15°C (Miller, 1995). Spooner (1998) found that the Younger Dryas mineral oscillation in western Nova Scotia is sporadic. This leads to the possibility that western Nova Scotia was free of ice after 13.5 ± 0.3 cal ka, and there was not a Younger Dryas readvance (Spooner, 1998). Stea et al., (1998) and Miller and Elias (2000) propose that during the Younger Dryas, Peggy's Cove region remained free of ice advances (Figure 16).

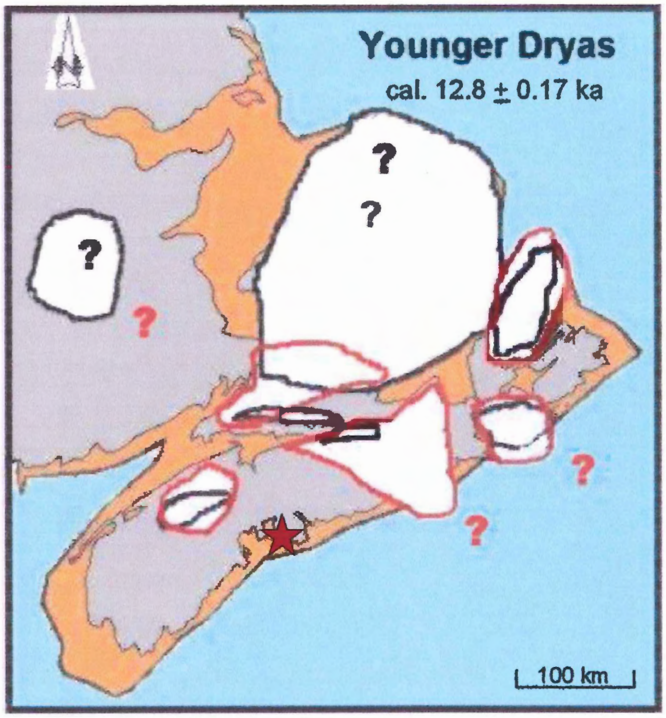


Figure 16 Ice extent during the Younger Dryas or Colins Pond Phase. The black lines represent ice extent from Stea et al., (1998), and the red lines represent ice extent from Miller and Elias (2000). The red star represents Peggy's Cove region (Compiled from Stea et al., 1998; Miller and Elias, 2000)

2.4 Physiography

The study area is located in the Meguma terrane of southeastern Nova Scotia (Tate and Clarke, 1997). The surficial geology of the field area consists primarily of exposed granite bedrock of the South Mountain Batholith (SMB), with some surficial deposits of till and Holocene stream and colluvial sediments. A study by Finck and Graves (1987) produced a surficial geology map showing the Quaternary deposits and glacial geology of the Halifax Peninsula. In the Peggy's Cove region, Finck and Graves show bedrock with B soil horizon developed on the bedrock, and possible thin till occurrences in bedrock hollows.

The bedrock of the region consists primarily of the Halifax Pluton of the SMB (Kontak et al., 2002) (Figure 17). The SMB is a large, peraluminous intrusive body that consists of 13 different plutons (MacDonald et al., 1992) composed of granodiorite-monzogranite-leucogranite units (MacDonald and Horne, 1988). The plutons range from 20 to 25 km thick, and cover 10,000 km^2 of southern Nova Scotia (Garland, 1953). The plutons can be grouped into two stages: Stage 1 plutons are intruded by Stage 2 plutons, and are dominated by biotite granodiorite, and biotite monzogranite. Stage 2 plutons are dominated by leucomonzogranite, and leucogranite (Horne et al., 1992). The Halifax Pluton is a zoned pluton divided into Stage 1 pluton of biotite granodiorite to biotite monzogranite, and a Stage 2 pluton of muscovite-biotite leucomonzogranite (Kontak et al., 2002).

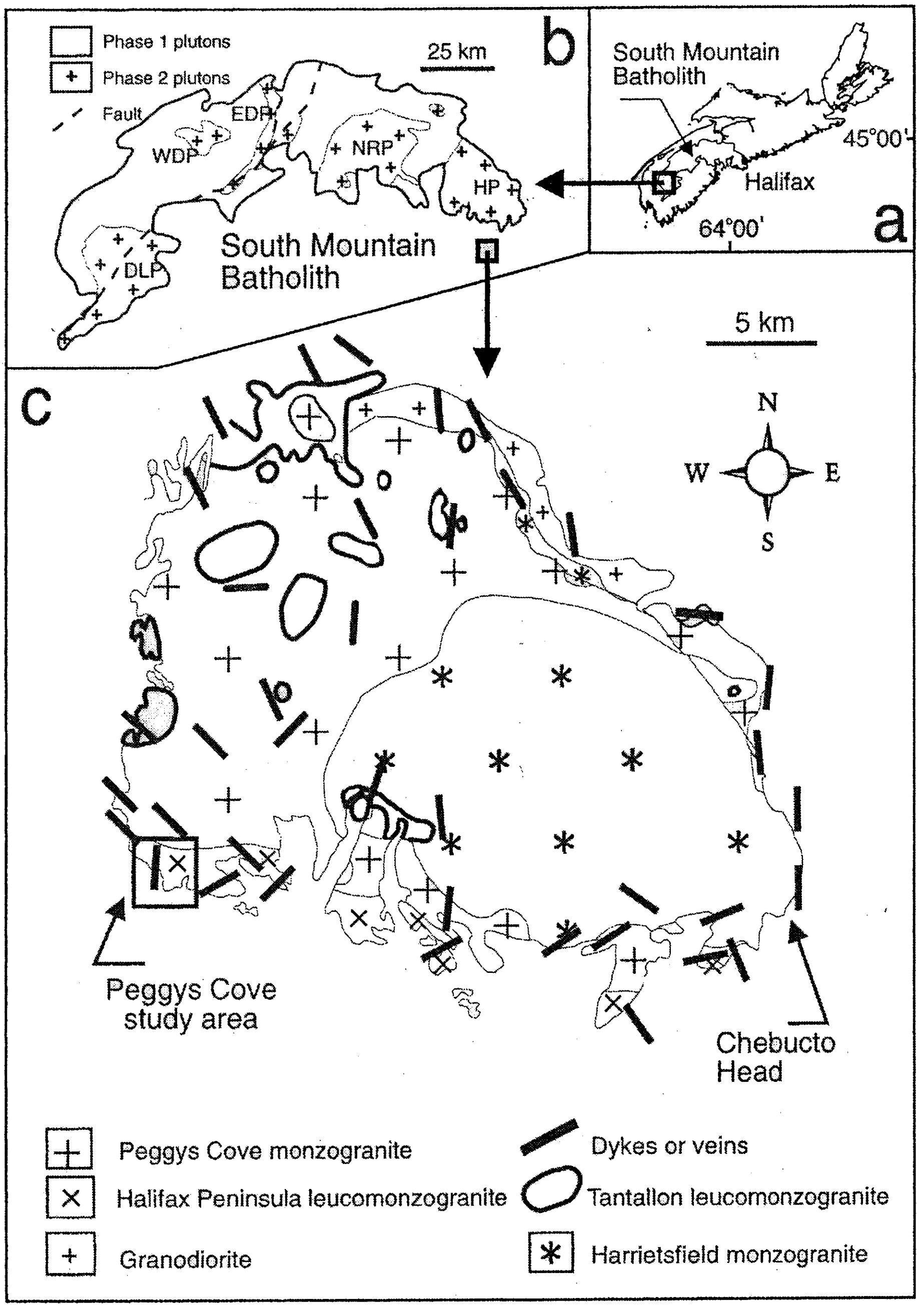


Figure 17 Location of study by Kontak et al. (2000) at Peggy's Cove. The map shows the location of different granite types, and dykes and veins in the Halifax Pluton (From Kontak et al., 2000).

The primary flow structures in the Halifax Pluton are the alignment of megacrysts, and xenoliths (Horne et al., 1992). The megacrysts are aligned parallel with the long axis of large, tabular feldspar crystals (Horne et al., 1992). The patterns of the megacrysts as seen in the outcrops, are planar or irregular swirling patterns, and no lineations have been defined yet (Horne et al., 1992). The xenoliths are aligned by the long axis of elongate xenoliths, and are parallel to megacryst alignment (Horne et al., 1992).

The Halifax Pluton is characterized by a dominant northwest trending joint system (315°), and a poorly developed northeast trending joint system (063°) (Horne et al., 1992). The northwest trending joints have surfaces that are straight, continuous, planar, with regular spacing in the outcrops (Horne et al., 1992). These joints form long, narrow ridges that are continuous, and may host quartz ± greisen veins, and granitic dykes (Horne et al., 1992). Care needs to taken to prevent misinterpreting the joint-bounded linear northwest-southwest ridges as glacigenic. The northeast trending joint is irregularly spaced, discontinuous, with a curviplanar surface (Horne et al., 1992). It is very rare to find dykes and veins with the same orientation (Horne et al., 1992). The fracture density in the immediate field area ranges from low (fracture spacing > 3m) to moderate (spacing 20-50 cm). This may have a bearing on the effectiveness of the ability of the glacier to erode or pick up parts of the bedrock and carry it away.

The subtle difference in the granites makes it difficult to conclude from field observations that the boulders on the bedrock ridges are true erratics, i.e. that have been transported glacially from a source that has a different lithology from the ridge on which they sit. The bedrock lithology in the immediate field area is dominantly Peggy's Cove 370 Ma Halifax Peninsula leucomonzogranite (Stage 2) that is a peraluminous granite (Kontak et al., 2002). Peraluminous granites have a molecular proportion of alumina that exceeds the combined proportion of sodium and potassium oxides (Bates and Jackson, 1984). Four types of xenoliths are seen in the Peggy's Cove monzogranite: (1) Meguma-like xenoliths with primary sedimentary features; (2) fine-grained equigranular xenoliths; (3) fine- to medium-grained xenoliths with abundant megacrysts of quartz, alkali feldspar, and plagiocalse, and (4) medium- to coarse-grained xenoliths with a similar appearance to the surrounding bedrock (MacDonald and Horne, 1988). The leucomonzogranite is medium- to coarse-grained, and devoid of any obvious structural fabric (Kontak et al., 2002). The finer grained aplite dykes are excellent at preserving glacial polish, and the striations are preserved in essentially all rock types.

Chapter 3: Methods

3.1 Mapping of Glacial Data

The glacial geology of the field area was mapped at a scale of 1:10000 using air photos, and topographic maps. A GPS was used to mark the position of the boulders, bedrock sample locations, and striation measurements. The air photos were used to look for three main glacially related features; (1) anything flat (i.e. deltatops, outwash plain, floodplain, or paleo-lake bed); (2) anything linear or curved, but continuous (i.e. paleo-lake shorelines, marine shorelines or raised beaches, or weathering zones); and (3) special features (i.e. eskers, erratics, stoss-lee features, roches moutonees, meltwater channels or paleostream deposits).

The mapping of glacier ice flow was performed by selecting suitable striations, which are usually located on aplite veins in the country rock with good glacial polish. The striations are often very fine, and best observed on sunny days. A GPS reading is taken for the location of the striations, and a compass (suunto MC-2) reading for the striation trend is recorded. The ice-flow directions were averaged if within about a 10^{o} range, and compiled on the glacial map.

Other important features looked for in the field were, rouche mountonees, fracture density of the bedrock, and stoss-lee ridges, and were recorded on the glacial geology map.

3.2 Field Sampling Techniques

To test the hypothesis, that the boulders represent a retreat of the ice margin between 17 to 13 kyr; seven boulders were sampled. In order to get an estimation of glacial erosion rates, the measurement of ^{10}Be concentration in the bedrock ridges was compared to the boulders. The field sampling techniques are very important, because the numerical ages are only as good as the samples that are collected (Gosse and Phillips, 2001). Peggy's Cove region is ideal for determining the timing of ice margin retreat. A boulder used for exposure dating must: (1) contain quartz (2) have a > 2 m diameter, (3) have a horizontal (2π) top surface, (4) have experienced no significant vegetation cover, (5) have little probability of snow cover, (6) have not rolled since deposition, and (7) exhibit weak indications of weathering and erosion (Gosse and Phillips, 2001). The top surface of the boulder must be horizontal in order to reduce foreshortening of cosmic rays. To obtain the desired degree of precision, multiple boulders must be sampled, each must meet the required standards for sampling and have a clear stratigraphic position. In the Peggy's Cove region, the only reasonable genesis of the large boulders often perched on ridges, is that they were deposited by glacial ice (i.e. they are erratics). A description of each boulder is recorded in the field book. The description includes a location of the boulder using a GPS including elevation, thickness of sample collected, a drawing of the boulder with the location of the sample, height and volume of the boulder, rock type, topographical shielding of cosmic radiation (large landforms in the area), possibility of till and snow cover, percentage of lichen covering the boulder, indications of grusification, and any gnammas or rills that are present, and the height and type of vegetation surrounding the boulder (Figure 18).

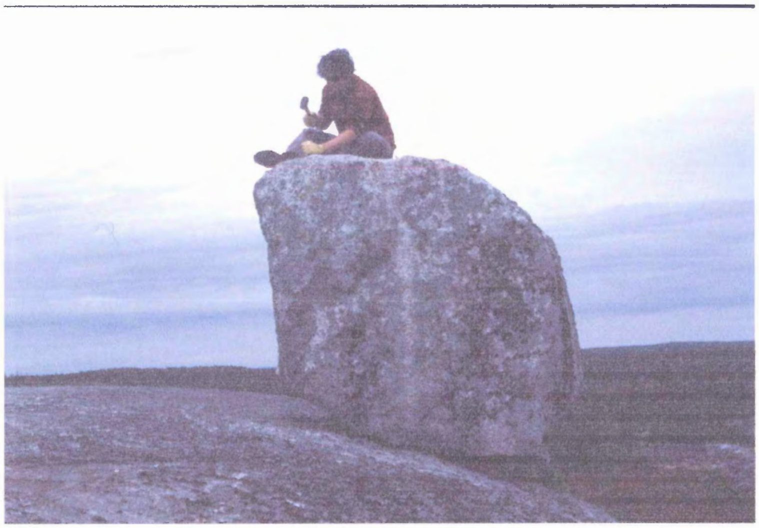


Figure 18 Example of typical sampled boulder, NS-PEGGY-02-003

In addition to boulders sampled, four bedrock samples located on high ridge crests were obtained in the same locality as the boulders, using similar sampling criteria, and making sure there was no possibility of topographically shielding from an adjacent boulder (Figure 19).

Figure 19 Example of a typical wind swepted ridge, NS-PEGGY-02-009, also showing the use of a cutoff saw to help collect samples.

The erratics and bedrock samples were sampled with a chisel and 2.5 lb sledge hammer, but it was found that the erratics were too hard, and sampling was very slow. A 10 inch cut off saw was rented from McFarlands in Halifax, and the proposed boulder and bedrock sampling areas were criss-crossed with the saw, cutting a diamond pattern into the surface (Figure 19). A chisel and sledgehammer were then used to chip out the diamond shapes. When sampling, a sample area was 30 cm from any edge, and samples were no deeper than 3 cm. A 1 kg size sample was collected at each site to ensure that enough quartz had been collected for use of TCN (Gosse and Phillips, 2001). Two large ziplock freezer bags were used for each sample with the sample ID number written in 3 places. Important information about each sample is shown in Table 1.

Table 1 Sample numbers, type of sample, location, elevation and sample thickness.

Field Sample Number	**Type of Sample**	**Latitude** (degrees/ feet)	**Longitude** (degrees/ feet)	**Elevation** (km)	**Thickness** (cm)
NS-PEGGY-02-001	Boulder	N 44 30.139	W 063 53.829	0.037	3
NS-PEGGY-02-002	Boulder	N 44 30.179	W 063 53.853	0.035	3
NS-PEGGY-02-003	Boulder	N 44 30.140	W 063 53.646	0.038	1.5
NS-PEGGY-02-004	Boulder	N 44 30.163	W 063 53.845	0.036	3
NS-PEGGY-02-005	Boulder	N 44 29.961	W 063 53.972	0.037	2
NS-PEGGY-02-006	Boulder	N 44 29.769	W 063 53.480	0.044	2.3
NS-PEGGY-02-007	Boulder	N 44 29.777	W 063 53.330	0.054	3
NS-PEGGY-02-008	Bedrock	N 44 30.130	W 063 53.635	0.038	1.7
NS-PEGGY-02-009	Bedrock	N 44 29.969	W 063 53.976	0.037	2.4
NS-PEGGY-02-010	Bedrock	N 44 29.765	W 063 53.470	0.037	2.1
NS-PEGGY-02-011	Bedrock	N 44 29.783	W 063 53.339	0.054	2.5

3.3 Terrestrial in situ Cosmogenic Nuclides (TCN)

3.3.1 Background Information on TCN

The Peggy's Cove region has no media for radiocarbon dating, and in places where there was, it would not give the right bounds on the timing of deglaciation. A new method used by Nishiizumi et al., (1986) determined the production rate of ^{10}Be and ^{26}Al to look at the implications for erosion rates, Lal (1991) determined production and erosion rates of in situ nuclides, and Gosse et al., (1995a) used in situ ^{10}Be to date boulders sitting on the crests of moraines in Wyoming.

Terrestrial in situ cosmogenic nuclides (TCN) are nuclides that are produced by the interaction of secondary cosmic rays with atoms in Earth's surface material. (Gosse and Phillips, 2001) TCN refers to nuclides (^{26}Al, ^{10}Be, ^{3}He, ^{14}C, ^{21}Ne, and ^{36}Cl) that are produced in situ, in exposed rocks on the Earth (Gosse and Phillips, 2001). The TCN used in this study is radioactive ^{10}Be, which is produced in quartz at a rate of approximately 5 atoms per gram of quartz per year (Stone, 1999).

The galactic cosmic radiation (GCR) that is responsible for the production of TCN, originates partly from the Milky Way galaxy, and partly from outside our galaxy (Ligenfelter and Flamm, 1964). The GCR consists of energetic particles that are composed of 83% protons, 13% α-particles, 3% electrons, and 1% heavier nuclei (Smart and Shea, 1985).

The GCR makes its way through the interplanetary magnetic fields until it reaches the terrestrial magnetic field (Masarik et al., 2001). The GCR that reaches the terrestrial geomagnetic field must have a certain momentum and rigidity to enter the geomagnetic field (Masarik et al., 2001). The low-energy GCR will be reflected back into space and only high energy particles can penetrate at low latitudes, so relatively more GCR penetrates the atmosphere near the north and south poles, than at the equator (Gosse and Phillips, 2001). Due to the variation in GCR reaching different latitudes, different production rates of TCN must be determined for different latitudes (Gosse and Phillips, 2001).

The GCR that reaches the top of the atmosphere, is considered to be primary radiation, and interacts with the nuclei of atoms located in the atmosphere, forming a cascade of particles (Allkofer and Grieder, 1984). The cascade of particles formed, called secondary radiation, consists of secondary nucleons (e.g. protons and neutrons) and mesons (e.g. kaons and muons), and other particles that have the same properties as the primary radiation (Allkofer and Grieder, 1984). The interaction of secondary neutrons with atoms in the atmosphere propagates down towards Earth's surface, and penetrates the upper few meters of the Earth's crust (Gosse and Phillips, 2001). Secondary muons are lighter particles and do not interact with matter as much, so they penetrate deeper into the rock (Gosse and Phillips, 2001). The secondary radiation enters the rocks at the surface, and interacts with atoms in the minerals of the rocks (e.g. Si and O in quartz) producing cosmogenic nuclides (e.g. ^{10}Be) (Gosse and Phillips, 2001). ^{10}Be is made from both neutron and muon interactions, but near the surface neutron spallation accounts for 97.8% of the ^{10}Be produced in quartz (Gosse and Phillips, 2001).

The production rate of a TCN for a given geomagnetic latitude and elevation, is the rate at which the cosmic rays produce ^{10}Be in the target mineral (quartz) (Gosse and Phillips, 2001). The production rate of ^{10}Be at Peggy's Cove ranges from 4.89 to 4.99 atoms/g of quartz/year. The production rate is affected by latitude, elevation, and sample thickness The half-life of ^{10}Be is 1.5 Myr, and the concentration of ^{10}Be is affected by the decay of the nuclide, but this does not affect the samples in this study because the boulders are less than 20 kyr (Figure 20). The production rate of ^{26}Al is approximately 6 times faster than ^{10}Be, but ^{26}Al also decays faster (half life = 700 kyr) (Figure 20).

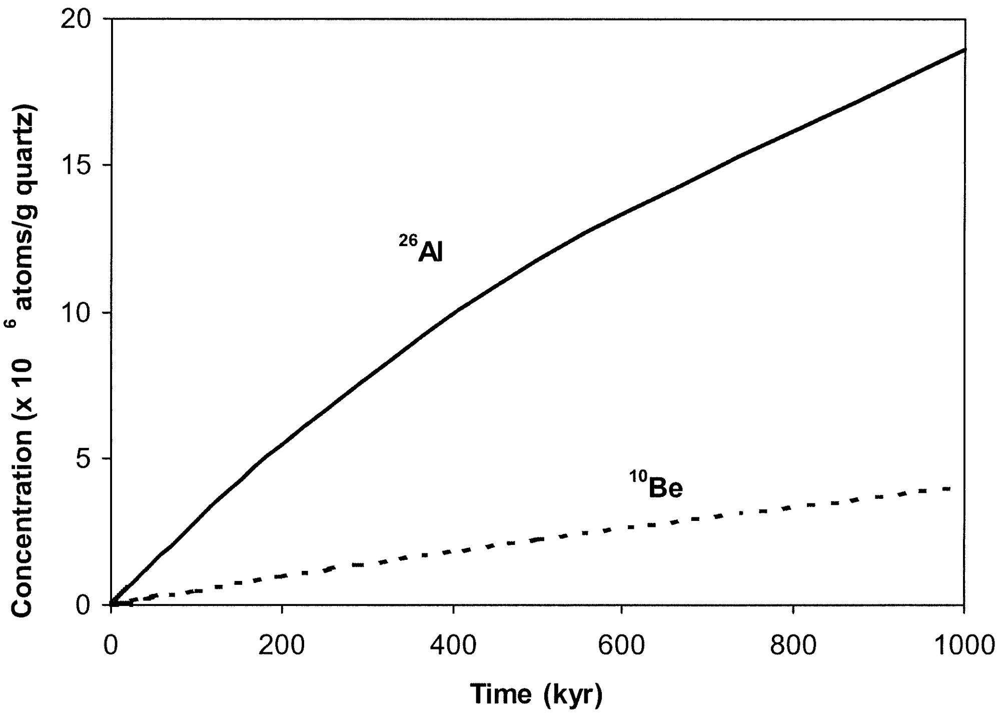


Figure 20 Accumulation rate of ^{10}Be and ^{26}Al in quartz, over 1 Myr

The production of TCN decreases exponentially with depth below the surface of the exposed landform and is calculated by the following:

$$P = P_o e^{-d\rho/\Lambda} \qquad \text{Equation 1}$$

(Gosse and Phillips, 2001), where P is the production rate at a certain depth (atom g^{-1} yr^{-1}), Po is the production rate at the surface (atom g^{-1} yr^{-1}), d is the depth of the sample (cm), ρ is density of the sample (g cm^{3}), and Λ is the apparent vertical attenuation length (g cm^{-2}).

The secondary radiation (e.g. slow muons and fast neutrons) penetrates to different depths depending on their attenuation length (1500 g cm^{-2} and 150 g cm^{-2}, respectively) (Figure 21). This is why it is important to collect the top 3 cm of a surface for sampling in order to obtain the most ^{10}Be possible.

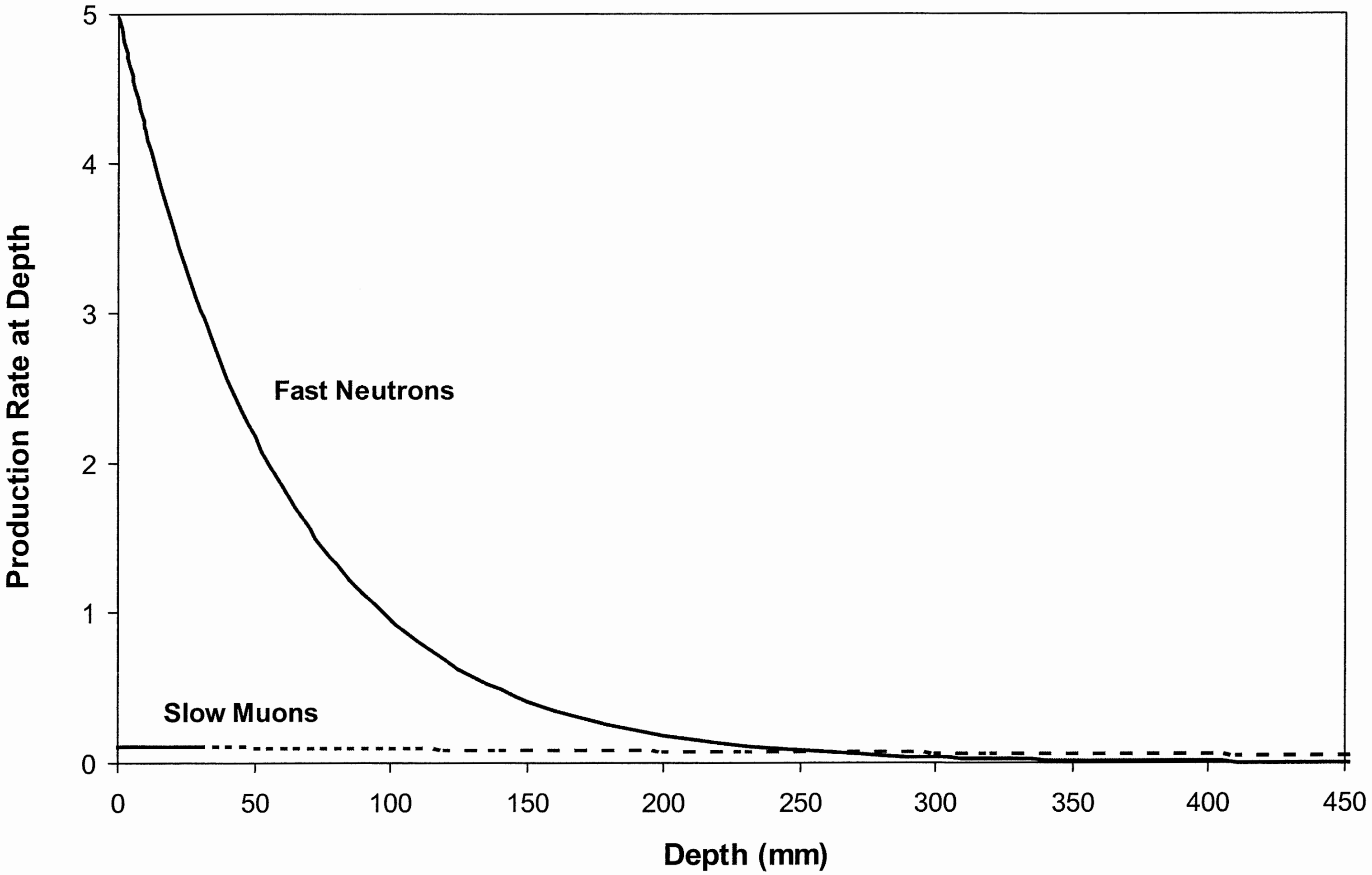


Figure 21 Production of nuclides at depth due to fast neutrons and slow muons.

3.3.2 Physical and Chemical Pretreatment

The procedure for physical and chemical pre-treatment has been adapted from the procedure outlined in the 2002 Dal-CNEF laboratory procedure, and can be found in Appendix 2. Crushing and grinding were performed in the Crystal Isolation Facility in the basement of the Dunn Building, Dalhousie University. A physical and chemical worksheet was filled out for each sample, and a hand specimen was saved in case thin section or additional inspection was needed. The remaining sample was crushed in the jaw crusher, then ground with a BICO disc pulverizer. The ground sample was sieved using <150μm, 150-250μm, 250-355μm, 355-500μm, >500μm sieves to obtain the optimum grain size 355 – 500μm needed for the chemical pre-treatment. Each grain size fraction was weighed, sealed into a medium ziplock freezor bag, labelled in 3 places with the grain size and field sample number, and archived in case the sample required reanalysis.

The next steps in physical and chemical pre-treatment were performed in the wet lab of the Cosmogenic Nuclide Extraction Facility (CNEF), located at Dalhousie University, room 4617. Type 1 pure water is used throughout the pre-treatment to minimize the introduction of Be and B. Aqua regia was used to disegregate polymorphic grains, weaken micas, and help begin the purification to quartz. Aqua regia is a 3:1 ratio of $HCl:HNO_3$. The amount of aqua regia needed, depends on the amount of sample that is available (typically 400 ml). Approximately, 600 g of the 355-500μm grain size were used, unless there was not enough sample, then the 250-355μm grain size was also used.

HF etching was performed to partially leach the silicate grains, and begin to remove the outer layer of the quartz grains to remove any meteoric ^{10}Be attached on the grains. An ultrasonic treatment was used to separate the quartz from the non-quartz phases, by weakening any feldspars in the sample and removing them (Kohl and Nishiimuzi, 1992). The ultrasonic treatment involves HF to partially leach the mineral grains. After two cycles in the ultrasonic tank, the samples were removed, rinsed, and dried in preparation for abrasion. Abrasion is used to help remove the softer non-quartz phases from the sample. For abrasion, 20 g of a sample was put into each of 3 abradors, and compressed air was forced into each abrador chamber causing the sample to disintegrate in the chamber. Since quartz is usually harder than other minerals in the sample, the quartz breaks down the softer minerals. After abrasion, the sample was rinsed to remove any powder produced during abrasion, and more Ultrasonic cycles were run until the sample appeared to be mostly quartz.

Aluminum testing for non quartz silicates was performed using a qualitative test, EM Quant Test Strips, which has been shown to be sufficiently comparable to ICP.AES (CNEF laboratory procedure). If the quartz concentrate had 125 ppm of Al or less, the samples are sufficiently quartz-pure to proceed with the Nuclide Extraction Procedure (Table 2). If the samples contain > 125 ppm Al, the isotope chemistry is more difficult, so the sample is returned for more leaching and abrasion.

Table 2 Weight and ppm for Al test

Sample #	CNEF #	Digestion Vessel ID	Weight (g)	ppm
NS-PEGGY-02-001	1095	D1	1.0005	50-125
NS-PEGGY-02-002	1096	D11	1.0034	50
NS-PEGGY-02-003	1097	D9	1.0003	125
NS-PEGGY-02-004	1098	D2	1.0007	125
NS-PEGGY-02-005	1099	D10	1.0005	125
NS-PEGGY-02-006	1100	D3	1.0009	125
NS-PEGGY-02-007	1101	D4	1.0015	125
NS-PEGGY-02-008	1102	D5	1.0011	125
NS-PEGGY-02-009	1103	D6	1.0005	125
NS-PEGGY-02-010	1104	D7	1.0002	50-125
NS-PEGGY-02-011	1105	D8	1.0009	50-125

3.3.3 Nuclide Extraction Procedure

The procedure for Nuclide Extraction Procedure has been adapted from the procedure outlined in the 2002 Dal-CNEF laboratory procedure, and can be found in Appendix 2.The chemistry data obtained during nuclide extraction can be found in Appendix 3. The amount of quartz concentrate required for the Quartz Dissolution and Be Spiking must be determined before proceeding further. It is important to determine the amount of ^{10}Be atoms required to obtain an optimum ratio of ^{10}Be/^{9}Be ($>1\text{x}10^{-14}$) that the accelerator mass spectrometer can measure. Samples collected from low elevations have low production rates, and samples exposed for short time periods will have low ^{10}Be concentrations. Therefore, more quartz is required to make precise AMS measurements on the Peggy's Cove samples. The optimum mass required can be predicted with the following equation:

$$m_T = \frac{R\left[{}^{m_c}\!/_{C}\right]N_A \Big/ W_{^{9}Be}}{EP_{^{10}Be}} \qquad \text{Equation 2}$$

m_T = total mass of sample required
E = exposure period (14 kyr)
$P_{^{10}Be}$ = production rate of 10Be (5 atoms/g of quartz/ year)
R = ratio of ^{10}Be/^{9}Be ($1\text{x}10^{-14}$)
m_c = mass of carrier (0.5 g)
C = concentration of Be in carrier (1000 μg/g)
N_A = Avagadros number ($6.02\text{x}10^{23}$)
$W_{^{9}Be}$ = molecular weight of ^{9}Be (9.027 g)

40 g of sample was dissolved in the quartz dissolution and Be spiking. No Aluminum carrier was added to the samples because the Al concentration from the Quant-Em sticks indicated that there was 50 – 125 ppm of native Al already in the quartz. Furthermore, it is unlikely that ^{26}Al will be measured in these samples.

An anion column chemistry procedure was necessary to further purify the samples of elements other than Be. The unwanted elements are removed using anion resin that is composed of a lattice of strongly basic anion exchangers and quaternary ammonium functional groups attached to styrene divinylbenzene copolymer. The styrene has a very high ion exchange capacity that allows for the substitution of, and holding of different anions on the surface of the styrene spheres. Many anions (e.g. S^-, Br^-) are removed and some cations are converted to negative molecular ions prior to eluting ($FeCl_2^-$) and removed as well.

Further purifying of Be sample is continued in the controlled precipitation. Controlled precipitation allows for the separation of the sample from elements that precipitate as hydroxides. At a pH of 6.5-9.5, the Be, Al, Fe, and Ti are insoluble hydroxides, but Ca and other soluble alkali hydroxides are discarded.

Final ion chromatography of Be is completed with a cation resin which is composed of sulfonic acid functional groups attached to styrene divinylbenzene copolymer lattice. The Cation Column Chemistry is performed to separate Be from all remaining elements. The Be is converted to an oxide powder (typically 1 mg) that is a target for AMS.

3.3.4 Final Product and the Accelerator mass Spectrometer (AMS)

The Be oxide powders targets were mixed with pure niobium metal and loaded into small metal Al target holders designed for accelerator mass spectrometry at Lawrence Livermore National Laboratory.

The Accelerator Mass Spectrometry measures the isotopic ratio (R) of the stable and cosmogenic isotope for a given element (Gosse and Phillips, 2001) (Finkel and Suter, 1993). For ^{10}Be, the isotope ratio is ^{10}Be/^{9}Be, is typically analyzed in each sample for 5 minutes or longer until 5% precision is attained.

3.3.5 Data reduction & Error Analysis

In order to calculate the ^{10}Be concentration in a quartz sample, the AMS and chemical data (quartz mass, carrier mass, and blank subtractions) are used:

$$N_{meas} = R_{10/9} m_c \frac{\left[\frac{N_A}{A_{Be}}\right]}{m_{qtz}} \qquad \text{Equation 3}$$

(Gosse and Phillips, 2001), where N_{meas} is the measured concentration of ^{10}Be (atoms g^{-1}), $R_{10/9}$ is the ratio of the concentration of ^{10}Be and ^{9}Be (unitless), m_c is the mass of the carrier added to the sample (g), N_A is Avogadro's number, A_{Be} is the atomic weight of Be, and m_{qtz} is the mass of quartz sample (g). Next, the component of the nuclide concentration produced during the geologic event (t) being dated, needs to be calculated. Typically it is initially assumed that the surface had never been exposed prior to being dated, and that erosion or burial during the event is negligible. Finally, to calculate the exposure duration recorded by the sample, production rates for each sample must be calculated (latitude, longitude, thickness, topographic shielding) and used with the decay constant according to:

$$T = -\frac{1}{\lambda_{10}} \ln\left[1 - \frac{N_{10}\lambda_{10}S\rho}{P_{10}\Lambda(1 - e^{-S\rho/\Lambda})}\right]$$ Equation 4

(Gosse et al., 1995a), where T is exposure time (yr), λ_{10} is the decay constant (yr^{-1}), N_{10} is the concentration of ^{10}Be in the sample (atoms g^{-1}), P_{10} is the local production rate of ^{10}Be in quartz (g atoms^{-1}yr^{-1}), ρ is the average density of the sample (g cm^{-3}), Λ is the thickness of the rock that reduces cosmic-ray flux (g cm^{-2}), and S is the thickness (cm) of a sample taken from the surface of a boulder.

There are two types of errors in calculating exposure durations: (1) random errors, and (2) systematic error (Gosse and Phillips, 2001). Random errors include sample characteristics (e.g. thickness or burial), sample preparation and analysis (e.g. gravimetric addition carrier or contamination from physical processing), and mass spectrometric measurements (e.g. poisson statistics) (Gosse and Phillips, 2001). Systematic errors include radionuclide halflife, production rate (e.g. basic calibration or attenuation lengths), temporal variations (e.g. cosmic ray flux or geomagnetic paleointensity), stable element measurements, carrier and standard uncertainties, fractionation, spectrometry, other assigned constants, and calculation errors (Gosse and Phillips, 2001). Random errors affect precision, and random and systematic errors affect the accuracy of calculations (e.g. ages or erosion rates) (Gosse and Phillips, 2001). The total uncertainty in a calculated age can be represented by:

$$\sqrt{\sum random^2 + \sum systematic^2}$$ Equation 5

An important uncertainty that must be taken into account when calculating the age is the rate of erosion. If the rate of erosion is high, the boulders or bedrock surfaces will have lower ^{10}Be than if the surface was not eroded, due to the exponential decrease in production with depth (Figure 21). Aplite dykes observed in the field are more resistant to weathering than the granite bedrock, and are useful in determining post-glaciation erosion. The height that the dyke protrudes form the granite is measured, it is assumed that the height difference between the aplite dyke and the bedrock is the amount of post-glacial erosion over the last 16.5 kyr, and erosions was constant since deglaciation. The constant rate of erosion after glaciation observed in the field was 1.0 cm over 16.5 kyr, which gives a rate of erosion of 0.06 mm/kyr. Erosion is considered negligible in Peggy's Cove region because the effect is only 0.5%.

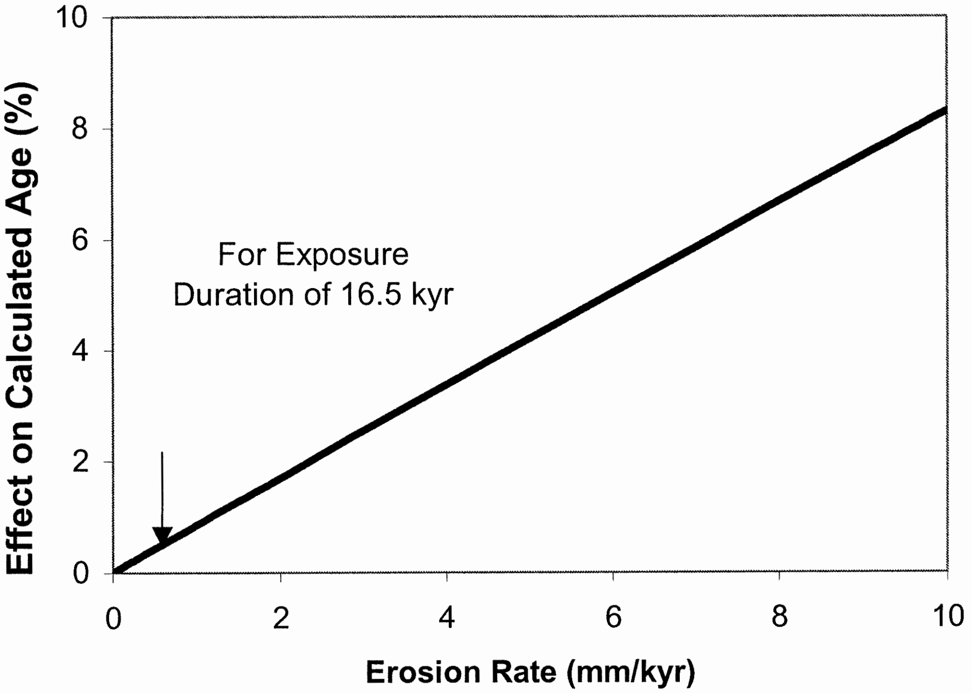


Figure 22 The effect of the rate of erosion on calculated exposure age, the arrow represents observed erosion in Peggy's Cove region.

Chapter 4: Results

4.1 Field Results

Seven boulders were exposure dated (Figure 23), and four bedrock surfaces (Figure 24) were measured for glacial erosion estimation (refer to Appendix 4 for field data of sample boulders and bedrock surfaces). The dominant lithology of the boulders is a coarse grained granite that is very similar to the Halifax Pluton leucomonzogranite that makes up the underlying bedrock (Kontak et al., 2002). The boulder heights range from 1.6 to 3.1 m, and 3 out of the 7 boulders are perched on smaller boulders. All sample sites are located on wind swept ridges, with little or no surrounding vegetation. Bedrock samples NS-PEGGY-02-009, and NS-PEGGY-02-010 are located on bedrock ridges with moderate fracture spacing (approximately < 50 cm), bedrock samples NS-PEGGY-02-008, and NS-PEGGY-02-011 are located on bedrock ridges with low fracture spacing (approximately > 3 m).

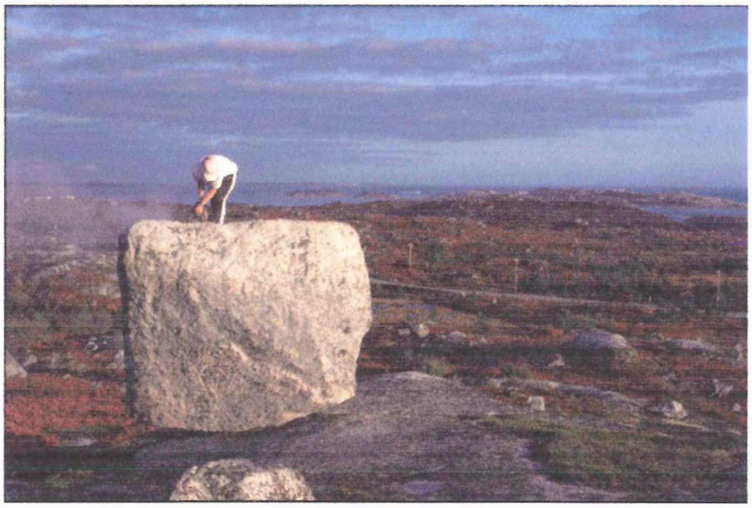


Figure 23 Example of sampled boulder NS-PEGGY-02-007. Note the flat nature of the top surface, and location of sample site away from the boulder edges.

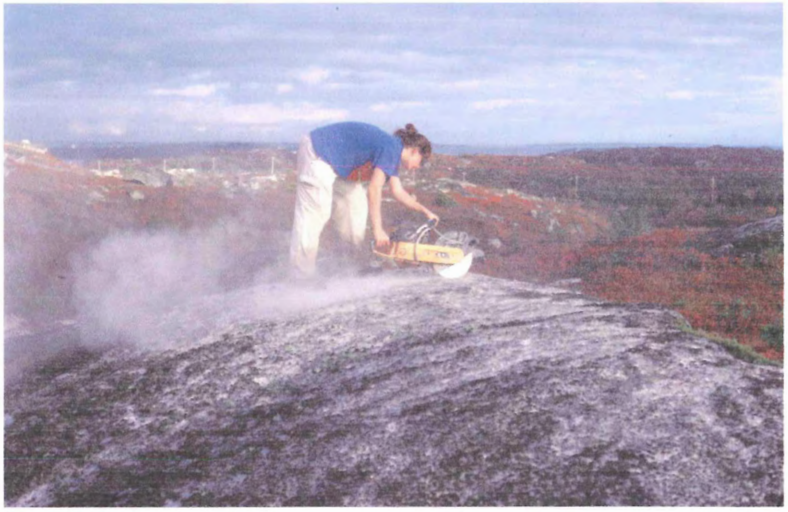


Figure 24 Example of sampled bedrock location NS-PEGGY-02-010. Note the streamlined nature of the ridge (ice flowed from left to right).

A map of the glacial geology (Figure 25) and the surficial geology (Figure 26) of the area was determined from field data and arial photographs. Striation readings from glacial polish reveal two general cross cutting directions, 160° and 224°. Determination of the relative timing of the two directions was not possible. The only large glacial features observed in the field area were stoss-lee ridges. The surficial geology map shows the location of Halifax Pluton granite, streamlined ridges, till veneer, lakes, marshes and lacustrine deposits, and anthropogenic disturbances. The till veneer is only observed between the ridges.

Figure 25 Sample location in the Peggy's Cove region.

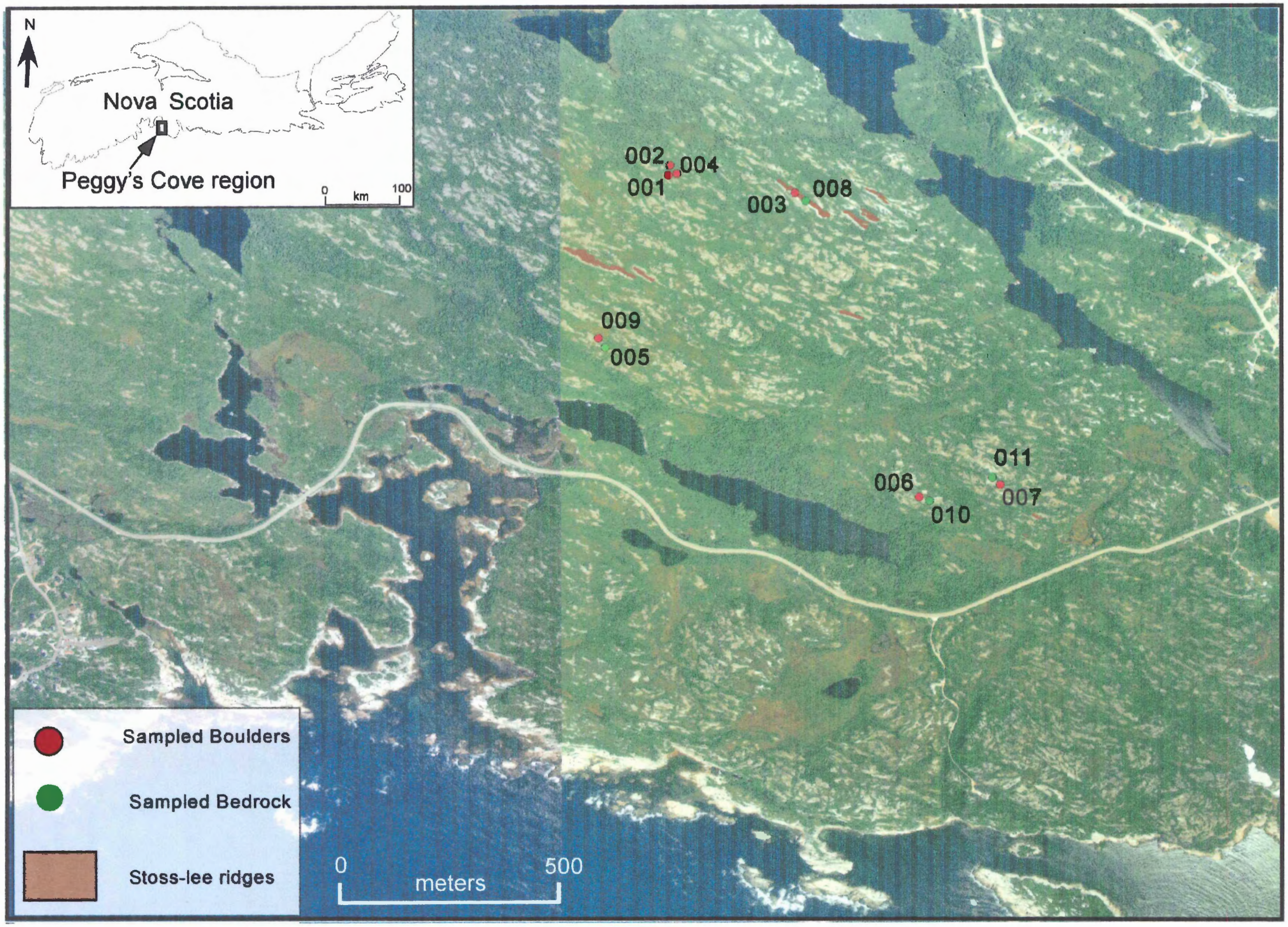
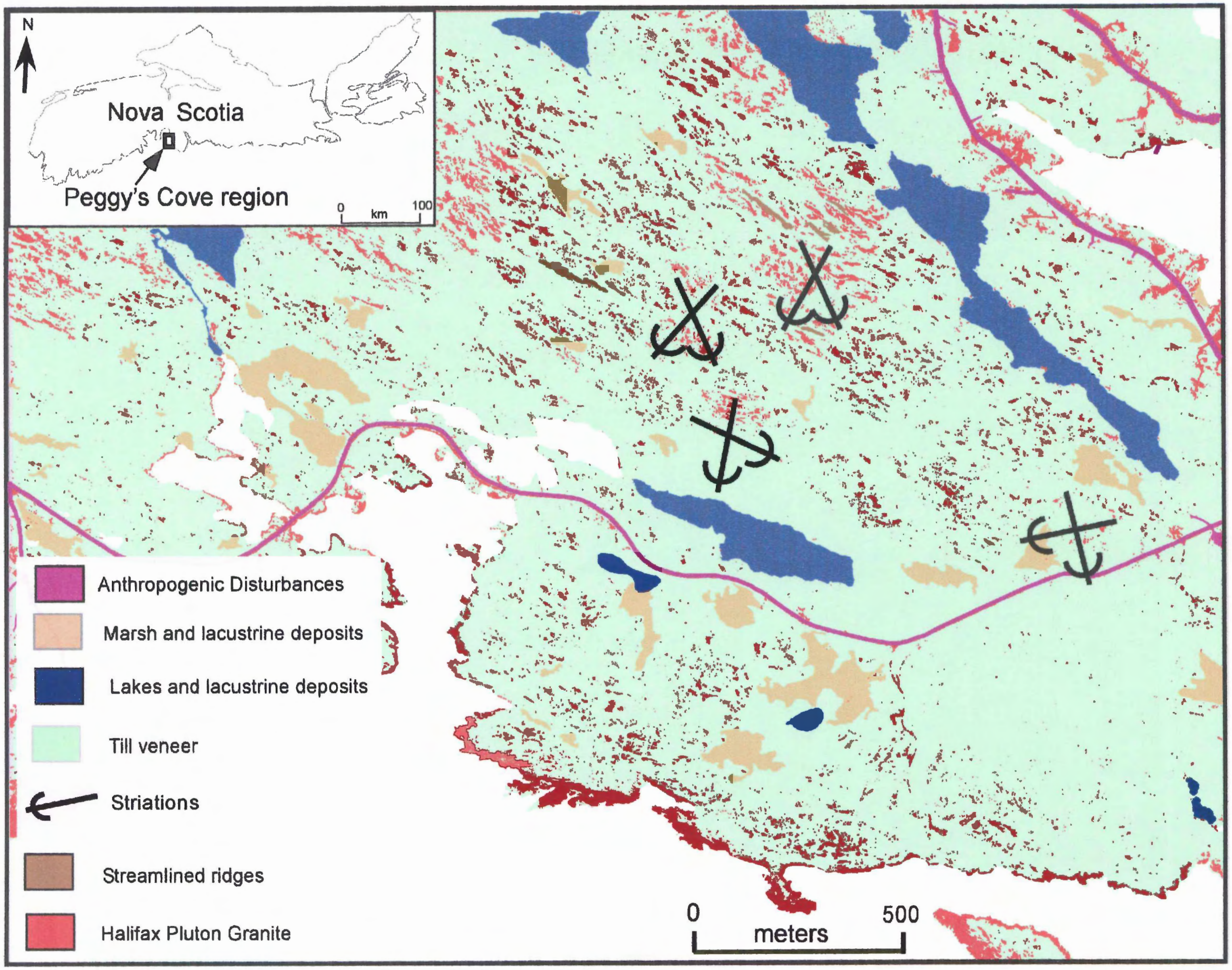

N
Nova Scotia
Peggy's Cove region
0
km
100
002
004
001
003
008
009
005
011
006
007
010
Sampled Boulders
Sampled Bedrock
Stoss-lee ridges
0
meters
500

Figure 26 Surficial Geology Map of Peggy’s Cove Region


N
Nova Scotia
Peggy's Cove region
0 km 100
Anthropogenic Disturbances
Marsh and lacustrine deposits
Lakes and lacustrine deposits
Till veneer
Striations
Streamlined ridges
Halifax Pluton Granite
0 meters 500

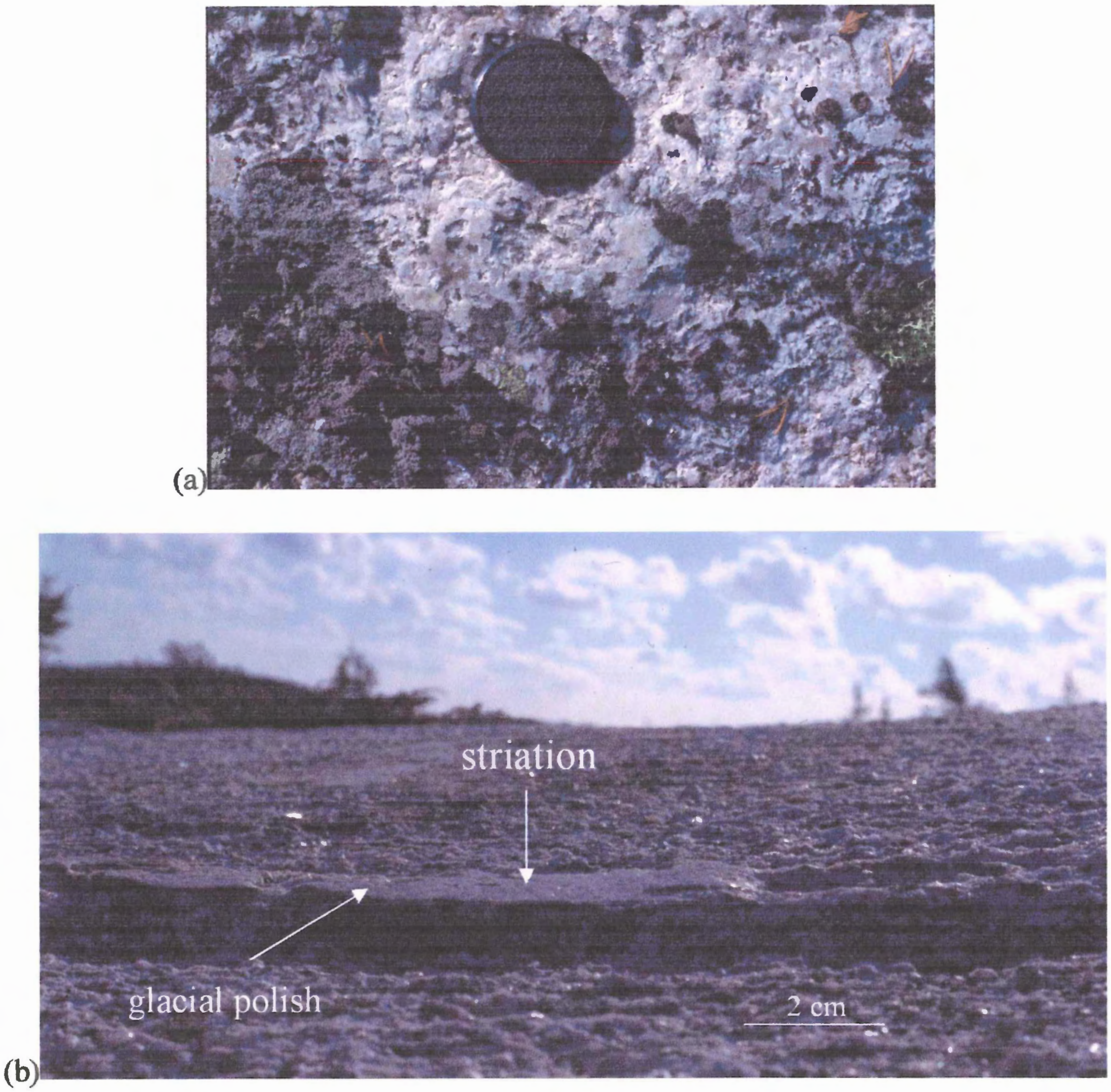


Figure 27 (a) top view of glacial polish on a vein; (b) an example of glacial polish with striations on a protruding aplite dike. Note the protrusion is approximately 1.5 cm.

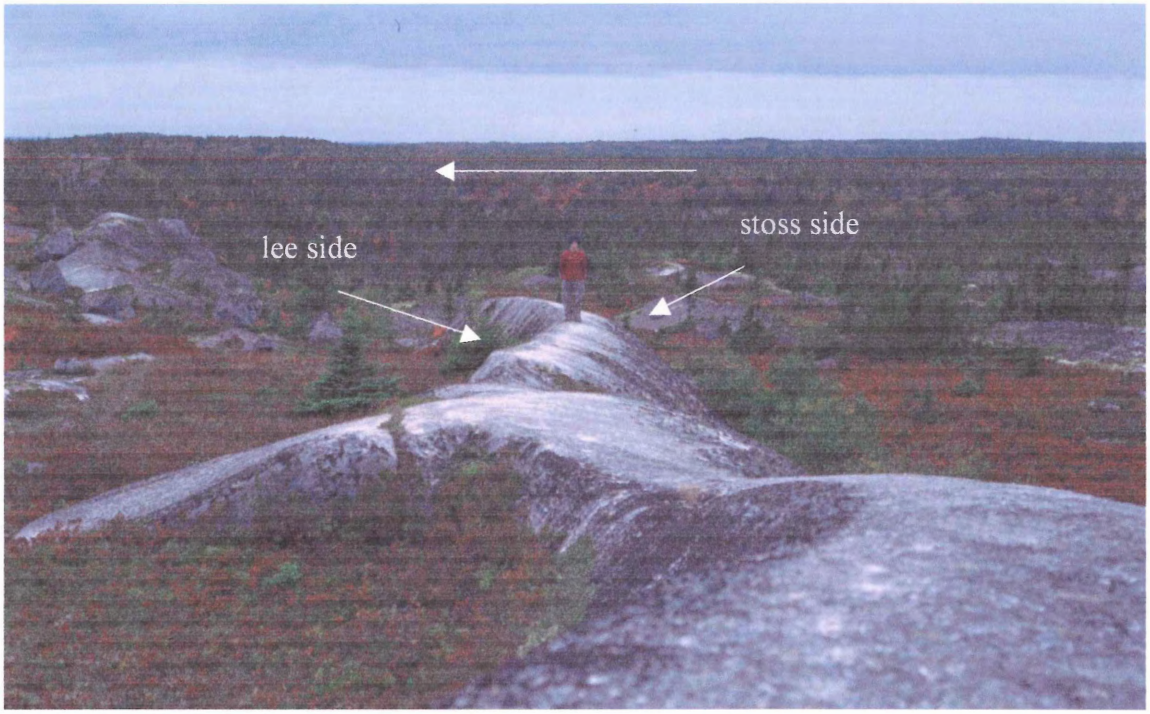


Figure 28 Example of a stoss-lee ridge, showing the stoss (up ice), and the lee (down ice) side. The large arrow indicates ice flow direction.

4.2 ^{10}Be boulder exposure ages

Two geochemical blanks were prepared and analysed with the boulder and bedrock samples. One blank had too little ^{10}BeO to be analysed with sufficient counts on the accelerator. The other blank has a ^{10}Be/^{9}Be ratio of $9.1\ 10^{-15} \pm 1.1 \times 10^{-15}$ atom/atom. This is consistent (within 1σ) of the geochemical blanks for the past two years (N=12) at the University of Kansas. The geochemical blank is used to indicate geochemical and AMS background. ^{10}Be background subtractions were no more than 9% for any given sample. This is higher than desired, but predictable because the samples were exposed for a short duration and at sea level so the concentrations of ^{10}Be in the samples were low (^{10}Be/^{9}Be range from 6.0×10^{-13} to 9.3×10^{-14} atom/atom).

The duration of exposure of the seven boulders ranges from 15.7 to 35.8 kyr, and the mean age of all the boulders is 19.9 ± 1.8 kyr. When the exposure age of the boulders vs. relative boulder position is graphed (Figure 29), the boulders show a low relative scatter, i.e. the exposure age's fall within a small range except one. The position of the boulders from the shore does not show any correlation with the exposure ages. The uncertainty ranges shown on the graph for each sample, are only the uncertainty from the AMS measurements. It is important to note that 4 out of 6 of the boulders fall within, or very close to the bracket age range of 15.9 to 17 kyr. The estimated timing of ice marginal retreat for the area is based on the Chignecto Phase ice advance from Stea et al (1998), and a glacial readvance date of 15.9 ± 0.3 ka cal. from Josenhans and Lehman (1999).

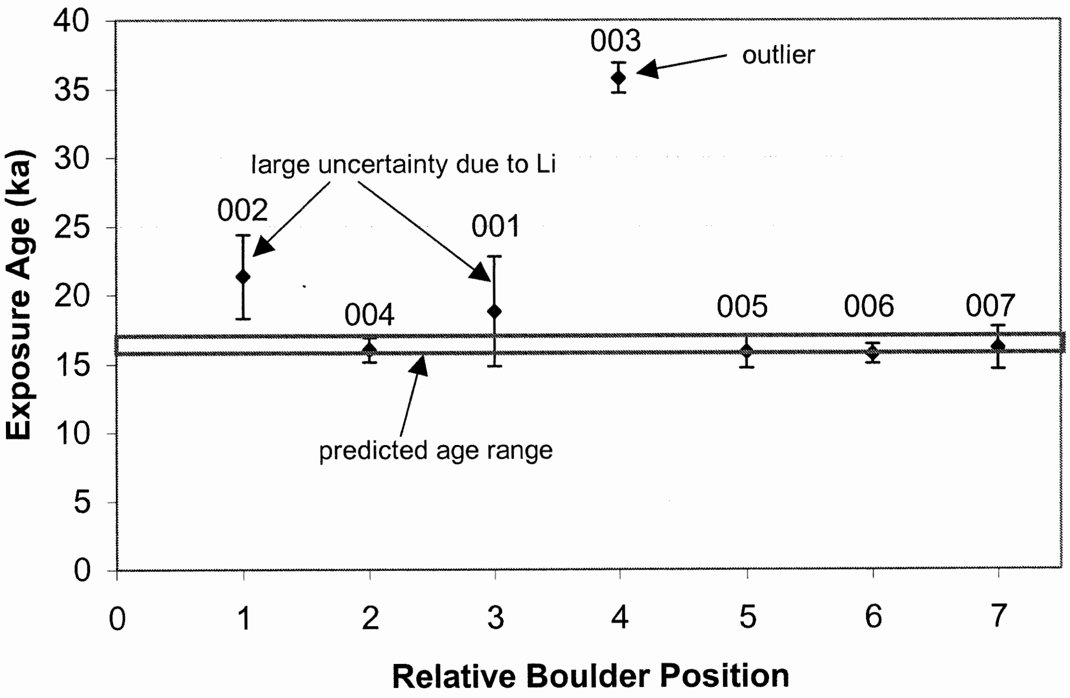


Figure 29 Relative boulder position is the position in an ice flow direction, with sample 7 being furthest down ice.

The concentrations of the bedrock (8.84×10^4 to 4.95×10^4 atoms g^{-1}) are higher than the mean concentrations of the boulders (8.48×10^4 atoms g^{-1}), and give a larger age range than the boulders (Figure 30).

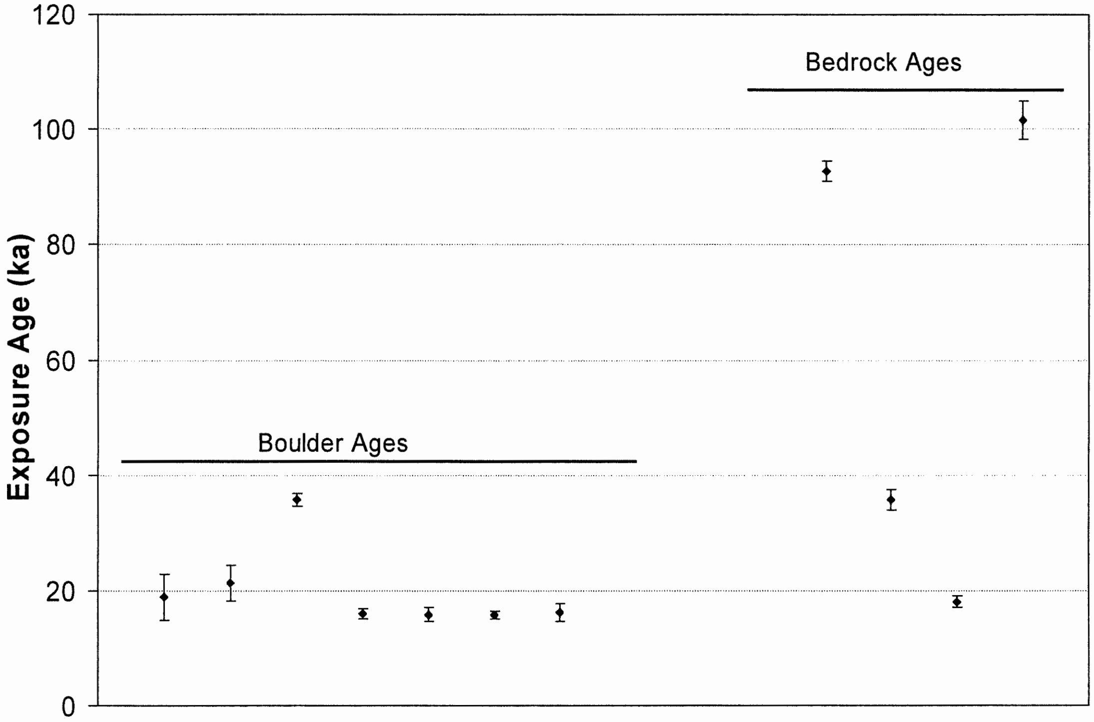


Figure 30 Graph of calculated boulder and bedrock exposure ages. Notice that the bedrock ages have a much larger range than the boulder ages.

Chapter 5: Interpretations

5.1 Ice flow direction

Ice flow direction was determined from striations found on the glacial polish of veins in the bedrock. Two crosscutting striation directions were observed on a number of veins; (1) a southwestward direction (180 ° to 260 °), and (2) a southeastward (100° to 160 °). It was not possible to determine which set of striations was younger in the field. By comparing the striation directions to the ice flow phases of Stea et al., (1998), southeastward direction corresponds to the Scotian ice flow phase (21.4 ± 0.4 to 17.9 ± 0.3 cal ka), and the southwestward ice flow direction corresponds to the Chignecto ice flow phase (15.6 ± 0.4 to 14.8 ± 0.6 cal ka).

5.2 Boulder exposure ages

The ratio of ^{10}Be/^{9}Be of the blank is used to correct the samples for any contamination that may have occurred during sample processing (refer to Appendix 5 for the AMS data). The background corrected ratio of ^{10}Be/^{9}Be of the samples is then used in equation 3 to determine the concentration of ^{10}Be (N_{meas}) in each sample. Next, the production rate for each sample is calculated, taking into account muonic contribution, latitude, altitude, site production rate, and thickness of the sample that reduces cosmic ray flux, horizon, snow cover, and the slope of the top surface of the boulder (refer to Appendix 6). Finally, the duration of exposure for each boulder is determined according to equation 4. Table 3 shows important sample data, and a complete spreadsheet including all the data used in the calculation of exposure duration can be found in Appendix 6.

Table 3 Sample Data (The error recorded for the Be Age only includes the uncertainty of the AMS precision).

CNEF ID	Sample ID	Elevation (km)	Height of Boulder (m)	Thickness (cm)	Quartz mass (g)	Mass of be Carrier Solution (g)	Concentration (mg / ml)	Age	
								Be Age (kyr)	Error (kyr)
1095	NS-PEGGY-02-001	0.037	1.6	3	42.4251	0.502	1015	18.8	4.0
1096	NS-PEGGY-02-002	0.035	3.1	3	41.0031	0.5103	1015	21.3	3.0
1097	NS-PEGGY-02-003	0.038	2.1	1.5	40.016	0.5057	1015	35.8	1.1
1098	NS-PEGGY-02-004	0.036	2.5	3	40.1252	0.5059	1015	16.0	0.9
1099	NS-PEGGY-02-005	0.037	3.0	2	40.0191	0.5095	1015	15.8	1.2
1100	NS-PEGGY-02-006	0.044	2.0	2.3	40.2198	0.5005	1015	15.7	0.7
1101	NS-PEGGY-02-007	0.054	3.0	3	40.0065	0.5006	1015	16.1	1.5
1144	Blank				0	0.529	1015		
1102	NS-PEGGY-02-008	0.038		1.7	40.0282	0.5034	1015	92.7	1.8
1103	NS-PEGGY-02-009	0.037		2.4	40.0298	0.5098	1015	35.7	1.7
1104	NS-PEGGY-02-010	0.037		2.1	40.0049	0.511	1015	18.0	1.0
1105	NS-PEGGY-02-011	0.054		2.5	40.0012	0.5011	1015	101.6	3.4
1145	Blank				0	0.52	1015		

Random and systematic errors affect precision, and the accuracy of calculated ages and erosion rates (Gosse and Phillips, 2001). The random errors are AMS precision, thickness, erosion rate, and snow cover. Erosion rate is considered negligible in the Peggy's Cove field area because the observed erosion over 16.4 kyr is 1 cm, and the effect on the age is approximately 0.5% (Figure 22). Snow cover is also considered negligible due to the location of samples on wind swept ridges. For individual data pertaining to concentrations, the random errors are used. For individual data pertaining to ages, the following equation adapted from equation 5 is used:

$$\sqrt{A^2 + R^2} \qquad \text{Equation 6}$$

Where A is the 1σ AMS error , and R represents the other random errors (approx. 0.06) (Gosse and Phillips, 2001). The error is then multiplied by the age to get the error in kyr. A is 10.3% and R is 0.06%, giving a total uncertainty for the ages of 10.3% or 1.7 kyr.

When the sample exposure ages are graphed (Figure 30), the exposure duration of the bedrock surfaces shows a large range (18 to 101.6 kyr) compared to the duration of exposure for the seven boulders. The large scatter of the bedrock exposure ages is most likely due to the spatial variation in glacial erosion during glaciation(Gosse et al., 1995b). It is important to graph the bedrock exposure ages in order to note whether there has been an effect due to glacial erosion, and erosion during exposure on the ages. The effect of glacial erosion on the sampled bedrock will be discussed in section 5.3, and the discussion from this point on will only include the sampled boulders. When the boulder exposure ages are graphed against relative boulder position inland from the coast (Figure 29), there is no apparent trend. It is important to note that four out of the six sampled boulders fall within, or close to the predicted exposure age range of 15.9 to 17 kyr.

A statistical analysis was performed on the seven boulder exposure ages. Sample NS-PEGGY-02-003 is an outlier, and was tested by subtracting the mean age of the boulders without the outlier, from the outliers age and dividing by the standard deviation. It was found that NS-PEGGY-02-003 was 7.1 standard deviations outside the mean. The weighted mean of the six boulders is 16.4 ± 1.7kyr (total uncertainty). The mean age of the boulders excluding the statistical outlier sample NS-PEGGY-02-003 is 17.3 kyr, and the standard deviation (1σ) is 2.8 kyr. The coefficient of variation is 17%, and was determined by dividing the standard deviation by the weighted mean, and multiplying by a 100. The AMS precision ranges from 2% to 21%, but the precisions above 7% are likely due to an artifact of the effects of high Li concentrations in the quartz (Gosse, personal-communication.).

The Peggy's Cove region was free of ice, based on the weighted mean, at 16.4 ± 1.7 ka. It is not possible to determine the duration of retreat of the ice margin over the 2.0 km field area, because the duration of retreat is less than the 1.5 kyr precision of the dating technique. It has been interpreted by Stea et al. (1998) that the last ice flow phases, the Chignecto Phase, lasted from 15.6 ± 0.4 to 14.8 ± 0.6 cal ka. On the continental shelf, the LaHave basin was ice free at 22 ± 0.4 cal ka (Piper and Fehr, 1991), and the Emerald basin deglaciation began between 20.8 ± 0.4 to 21.4 ± 0.4 cal ka (Gipp and Piper, 1989). During the Scotian Phase, the Scotian Shelf end moraine complex (SSEMC) was deposited between 18.5 ± 0.4 to 20.2 ± 0.4 cal kyr, and the ice margin had retreated to the edge of the SSEMC at 17.9 cal ka (Stea et al., 1998). Most of the ice was removed from the Gulf of Maine, Bay of Fundy, and the Laurentian Channel between 15.6 ± 0.4 to 17.9 ± 0.3 cal ka before the last ice flow phase (Chignecto Phase) (Stea et al., 1998). The exposure age of 16.4 ± 1.7 ka is consistent with previous radiocarbon ages of the Atlantic Coast.

5.3 Glacial Erosion Rate

The amount of glacial erosion of the bedrock ridges can be approximated by a simple model that uses the ^{10}Be concentrations in the boulders and bedrock surfaces. It is important to note that glacial erosion is the amount of bedrock removed during the last glaciation, which is different from erosion during post-glacial exposures. The model determines the depth of the bedrock that was eroded during glaciation by taking into account exposures before and after glaciation. The assumptions for the model are: (1) the last glaciation began 75 kyr (Stea et al., 1998), (2) glaciation ended 16.4 ka, (3) the timing since glaciation is 16.4 kyr, (4) the bedrock ridges were exposed for 88 kyr prior to the start of the last glaciation, and (5) the mean boulder concentration has negligible inheritance. Assumption 4 was determined by taking the maximum exposure period of the bedrock ridges prior to glaciation if no glacial erosion occurred. The equation used for the model is:

$$N^{*} = N_{88kyr}\left[e^{\left(\frac{-\rho z}{\Lambda}\right)}\right] \qquad \text{Equation 7}$$

where N^* is the ^{10}Be concentration in the bedrock before glaciation (atom g^{-1}), but after the effects of glacial erosion (i.e. if glacial erosion happened on the first day of the Wisconsinan), N_{88kyr} is the concentration of ^{10}Be in the bedrock (atom g^{-1}) before glaciation and before the bedrock was eroded by the glacier, ρ is the density of the bedrock (g cm^{-3}), Λ is the attenuation length of cosmic ray flux (g cm^{-2}), and z is the thickness of bedrock (cm) that must have been removed to get N^*. N* is determined from the equation below:

$$N^* = \left[N_{meas} + N^{decay} - N^{postglacial}\right]$$

Equation 8

where N_{meas} is the concentration of ^{10}Be measured in the samples by AMS (atom g^{-1}), N^{decay} is the amount of ^{10}Be concentration decayed over 75 kyr during burial (atom g^{-1}), and $N^{postglacial}$ is the mean concentration of ^{10}Be in the boulders (atom g^{-1}). Also, a correction for the decay during exposure of ^{10}Be of each sample was performed.

The amount of bedrock removed during glaciation (z), and the averaged rate of glacial erosion is summarized in Table 4.

Table 4 Modeled glacial erosion rates of bedrock ridges

Field ID	Z (cm)	Glacial erosion (cm/kyr)
NS-PEGGY-02-008	7	0.1
NS-PEGGY-02-009	85	1.5
NS-PEGGY-02-010	263	4.5
NS-PEGGY-02-011	0	0

Sampled sites NS-PEGGY-02-008 and NS-PEGGY-02-010 were checked, and a low fracture density of the bedrock at NS-PEGGY-02-008 supported the low glacial erosion. A high fracture density, making it easier for the glacier to pluck blocks of the bedrock as it moves over the area, supports the high glacial erosion at NS-PEGGY-02-010.

Chapter 6: Conclusion

The hypothesis: the boulders represent a retreat of the ice margin between 17 to 15.9 kyr was proven true by the calculated exposure duration age of six of the seven sampled boulders. Only six of the sampled boulders ages were used because sample NS-PEGGY-02-003 was 7 standard deviations outside the mean and considered an outlier. Sample NS-PEGGY-02-003 is an outlier probably due to inheritance or previous exposure giving a older age than the other six boulders. The weighted mean age of 16.4 ± 1.7 ka of six of the boulders falls within the predicted age range of 15.9 to 17 kyr. The exposure age for deglaciation for the Peggy's Cove region shows remarkable consistency with previous radiocarbon dates, glacial readvance date of 15.9 ± 0.3 ka cal. from Josenhans and Lehman (1999), and the Chignecto Phase from ice flow models by Stea et al (1998). The radiocarbon dates have a more precise uncertainty than the ^{10}Be exposure ages because there was a problem with the Li content of the quartz. This caused a problem with the accelerator when the samples were run, which lead to high uncertainty values for some of the samples. The ice flow directions recorded in the field correspond to the Scotian and Chignecto ice flow phases of Stea et al., (1998). The duration of retreat of the ice margin over the 2.0 km field area is less than the precision of the dating technique, and therefore cannot be determined.

The question of effectiveness of glacial erosion on the bedrock ridges was answered by simple modeling of glacial erosion rates based on the comparison of ^{10}Be concentrations in the boulders and bedrock surfaces. The rate of glacial erosion appears to be dependent on the fracture density of the bedrock, and ranged from 0 to 2.6 m. Bedrock surfaces that had little or no erosion probably persisted throughout the entire Wisconsinan glaciation.

Future work should include further dating of boulders located in boulder fields around Nova Scotia. By dating boulders from similar boulder fields, it would be possible to date the rate of ice margin retreat, and provide more data in order to correlate the terrestrial and offshore record. A more extensive study of glacial erosion would provide more accurate information to determine if bedrock has, or has not been eroded during the last glaciation, and therefore which glaciation or ice flow phase the glacial features correspond to.

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Appendices

Appendix 1 Calibrated Radiocarbon Ages

Uncalibrated Ages	Uncertainty	Type	Publication	Calibrated Ages	Uncertainty
10 ka			(King and Fader, 1986)	11.5	0.25
ca. 18 -15 ka			(Stea et al., 1998)	21.4-17.9	0.35-0.3
ca. 13-12.5 ka			(Stea et al., 1998)	15.6-14.8	0.35-0.55
ca. 10.8 ka			(Stea et al., 1998)	12.8	0.17
17.5-18 ka		mollusc shells	(Gipp and Piper, 1989)	20.8-21.4	0.35-0.35
20 ka			(Piper and Fehr, 1991)	23.7	0.4
18.5 ka			(Piper and Fehr, 1991)	22	0.36
17-15 ka			(King, 1994)	20.2-17.9	0.35-0.3
15.5-17 ka			(Stea et al., 1998)	18.5-20.2	0.3-0.35
14 ka			(Stea et al., 1998)	16.8	0.26
13-15 ka			(Stea et al., 1998)	15.6-17.9	0.35-0.3
11.4 - 13.2 ka		basal lake (wood)	(Stea and Mott, 1989)	13.3-15.9	0.15-0.3
ca. 12 ka			(Stea and Mott, 1989)	14	0.17
9-11 ka			(Quinlan and Beaumont, 1982; Stea et al., 1998)	10.1-13.3	0.16-0.1
11650 yr BP	110	mussel valve fragment	(Stea et al., 1994)	13.6	0.18
10800-10400 ka			(Miller, 1995)	12.8-12.3	0.17-0.35
11500 BP			(Spooner, 1998)	13.5	0.3

Appendix 2 Lab Procedure

The procedure for Physical and Chemical pre-treatment, and nuclide extraction have been slightly adapted from the Specific Safety and Laboratory Standard Operating Procedures for the Preparation of Cosmogenic Nuclide ^{10}Be and ^{26}Al Oxides from Rock by John Gosse (2002).

Physical and Chemical Pretreatment Procedure

Sample Crushing and Grinding

1. Turn dust vacuum system on, put on safety goggles, mask, ear plugs, and secure long hair before starting sample crushing and grinding.
2. Record sample information required on the physical preparation worksheet. Remember to save a small sample piece for hand specimen and put in a medium, heavy-duty ZipLock freezer bag, labelling in three places with the sample number.
3. Remove soil and lichens form the outer surface of the sample pieces by scrubbing with water.
4. Always assume the jaw crusher, disc pulverizer, and adjacent surfaces are dirty and clean them with the brushes, pressurized air, and vacuum.
5. Make sure all the sieves that are going to be used have been cleaned and are free of dust particles. Sieve sizes used are <150μm, 150-250μm, 250-355μm, 355-500μm, >500μm.
6. If the sample pieces are too large for the jaw crusher, reduce the size with the sledgehammer. (Any sample pieces that touch the floor are not to used)
7. Crush the sample in the jaw crusher, putting larger pieces through twice if needed.
8. Sieve the crushed material, using the >500μm grain size to be grinded in the disc pulverizer.
9. The disc pulverizer discs should be about 3mm apart for the first round of grinding. Slowly add sample to the disc pulverizer to reduce the amount of powder. When the collection tray is full, sieve the sample, and continue putting the sample through the disc pulverizer, until all the sample has been

pulverized. When the sample has been sieved, take the >500μm, and pulverize again until there is about 10 g left of the >500μm. Adjust the plates so they are closer together if needed to reduce the >500μm.

10. When the pulverizing is finished, label six medium heavy-duty ZipLock freezer bags with the sample number and grain size in three places and put the medium bags into a large heavy-duty ZipLock freezer labelled in three places.
11. Take sample and more thoroughly sieve with the sieve shaker to increase ease for the chemical pretreatment.
12. Before starting another sample, all devices used must be thoroughly cleaned with the brushes, pressurized air, vacuum, and acetone if devices are particularly dirty.
13. When finished crushing and grinding for the day. Take samples up to lab and record the weight of each grain size on the physical worksheet and on the computer master spreadsheet.

Aqua Regia

Aqua regia is performed on the sample to dissolve iron and other metals from the sample to make sample purification to quartz easier.

1. In the perchloric fumehood, preheat the two hotplates to 200°C.
2. Label one 1 L Teflon beaker, and one lid per sample with a Fisher Scientific black pen in two places with CNEF #, Field #, and the date. Only 4 Teflon beakers fit on one hotplate. Use one stir stick per sample, always keeping stir stick in with the sample it is being used for at the time.
3. Prepare the aqua regia by mixing HCL and HNO_3 at a volume ratio of 3:1 in an 800 ml Nalgene bottle. Use a plastic cylinder to measure HCL, and a glass cylinder to measure HNO_3.
4. Weigh 600 g of the 355-500μm grain size into one Teflon beaker labelled with the same CNEF and Field numbers. If there is not enough 355-500μm grain size, the 250-355μm grain size may also be used. If the sample size is

still small, reduce the amount of aqua regia used per sample. Record the grain size used and the weight of each sample in the lab book. Use 400 ml of aqua regia for samples weighing 400 – 600 g, use 300 ml for samples weighing 300-400 g, and use 200 ml for samples weighing 200-300 g.

5. Slowly add the determined amount of aqua regia to each sample, put lid on, and put on hotplate for 2 hours. Stir each sample every 15-20 minutes, by removing sample from hotplate, stirring, and returning to the hotplate.
6. Clean cylinders by rinsing with water in the fumehood and dumping into the acid neutraliser bucket. Add soda ash to neutralise the acid to a pH of ≥4. Clean each cylinder with soap, rinse with tap water and then rinse with deionized water and put to dry.
7. When the samples have finished boiling for 2 hours, remove the samples from the hotplates and allow to cool. Use the acid neutraliser bucket, adding soda ash as needed. Pour the liquid from one Teflon beaker into the acid neutraliser bucket, be careful not to lose any sample. Rinse the sample with water until there is no yellow colour left on the sample from the aqua regia. Decant any micas in the sample if possible. When the acid neutraliser bucket is full and the acid is neutralised, pour down the sink. Repeat rinsing for each sample.

HF Etching

1. In the perchloric fumehood, preheat hotplate to 200oC.
2. Pour 80 ml of deionised water into a disposable beaker.
3. Add HF up to the 260 ml mark on the disposable beaker.
4. Pour HF and deionized water into sample.
5. Measure 20 ml of HNO3 using a plastic cylinder and add to the sample.
6. Repeat steps 2 – 5 for each sample.
7. Put each sample on the hotplate for 20 –30 minutes, stirring frequently.
8. Remove from hotplate and cool.

9. Decant liquid into the acid neutraliser bucket, adding soda ash and emptying bucket as needed. Rinse sample with tap water 5 – 10 times until the pH of the water is > 4.
10. Rinse sample thoroughly with deionized water.
11. Put sample on hotplate at 150°C to dry keeping the lid propped up by the stirring road to allow for water vapour to escape.
12. Repeat steps 9-11 for each sample.
13. Once samples have dried, label a 250 ml Nalgene bottle with the CNEF #, Field #, date, and After HF etching on both sides of the bottle, and label the lid with the CNEF # and the Field #. If the sample is larger than one bottle, fill two bottles and put an elastic around both bottles.
14. Clean the Teflon beakers by boiling with a small amount of aqua regia for 5-10 minutes. Take off hotplate, pour liquid into acid neutraliser bucket, and wash beaker with soap. Remove the writing on the beaker with acetone. Rinse the cleaned beaker with deionized water ands et to dry.

Ultrasonic Quartz Separation

The object of the ultrasonic quartz separation is to separate the quartz from the non-quartz phases in the sample, and to weaken any feldspars in the sample in preparation for abrasion. The following procedure has been adapted from Kohl and Nishiizumi (1992).

1. Weigh approximately 60±1 g of sample into one 4L Nalgene bottle until the all the sample has been weighed out. Record in the lab book, what bottles each sample has gone into. Only nine 4L bottles can be run in the Ultrasonic tank at one time. If there is a time constraint, 120±1 g may be weighed into each bottle in order to run more samples at once.
2. Fill each 4L bottle with deionized water to the top of the handle mark. Only remove the cap from one bottle at any one time.
3. Measure a ratio of HF: HNO_3: deionized water at 162:114:4000 ml. Add the appropriate amount of acid to each 4L bottle. When measuring HF, only a

plastic cylinder may be used. The ratio of acid will change according to the amount of sample and how may runs the sample has had in the ultrasonic tank.

4. Put each bottle in the ultrasonic tank, leaving the lid loose to prevent pressurization.
5. If necessary, fill the tank with tap water to 2 cm above the handle mark on the 4L bottles. Close the tank lid. There is no measurable evaporation of water from the tank at 90°C over a 12 hour period when the lid is closed.
6. Set the ultrasonic tank temperature to the black mark on the dial, and turn on the ultrasonic power generator for the corresponding tank. The tank may be run for 8 – 16 hours. It is convenient to run overnight.
7. When the run time is up, turn off all the dials and open the tank lid. Take the bottles out and allow to cool.
8. Decant the liquid into the acid neutralizer bucket, making sure not to lose any sample. If the samples are being run again, rinse 3 times with deionized water, if the samples are going to be dried, rinse 5 times with deionized water. Add soda ash to the bucket, and neutralize to a pH of ≥ 5 before pouring down the sink.
9. Repeat steps 2-8 if sample is going to be run again. If sample is going to be dried, go to step 10. Samples must have two runs in the ultrasonic tank, dried for abrasion, abraded, and then run in the ultrasonic tank till the ppm of Al reading is <25 for the Al test. About 5-7 more runs, depending on the sample.
10. Label a Teflon beaker and lid with CNEF #, Field #, date, and how many runs in the ultra sonic tank have be done. Put sample from bottles into a Teflon beaker by rinsing the sample with deionized water into the beaker. Make sure to combine samples that had to be separated for the ultrasonic tank. Put sample to dry on hotplate at 150°C.
11. Clean bottles with soapy water, and rinse with tap water. When the bottles are cleaned, rinse with deionized water, put the lid on, dry off the outside of the bottle, and put on the counter.

Aluminium Test

This test will provide an approximate concentration of Al in the sample after Ultrasonic cycles are complete. It is necessary to have a low Al concentration in the sample to make the nuclide extraction procedure easier.

1. Weigh 1 g (±0.001 g precision) of sample into a 50 ml digestion vessel. Record the weight, and digestion vessel number that each sample is put into.
2. Add enough HF to cover the sample, approximately 10 ml, in each digestion vessel. Put the lid back on the vessel, leaving slightly loose to allow for the build of pressure.
3. Put each digestion vessel on a hotplate at 60°C for 8 hours, or until the sample is dissolved.
4. When the sample is dissolved, turn the hotplate up to 150°C to evaporate the acid, Leave the lid slightly adjare to allow for an escape route for the vapour. This step can be left overnight.
5. When the sample is evaporated, use the Merkoquant Aluminium test.
6. Label a plastic test tube, and lid for each sample with the digestion vessel number and the CNEF number.
7. Take one digestion vessel and rinse the vessel with HCl to dissolve the white powder that is left from step 4. Pour the liquid into the labelled test tube.
8. Add HCl to bring the liquid level up to the 5ml mark.
9. Repeat steps 7 – 8 for each sample.
10. Take one sample, and bring the pH to 13 by adding reagent 1. Approximately 3 – 8 drops depending on sample. Shake sample to mix solution. Check pH by using a disposable pipette, and put one drop on pH paper.
11. When pH reaches 13, use disposable pipette to put a drop on the reaction zone of the test strip for 1 second. Shake off the excess liquid.
12. Apply one drop of reagent 2 to the reaction zone, and allow to react for 1 min.

13. Shake the excess reagent off of the strip and wait 1 minute.
14. Compare the colour of the reaction zone to the colour scale.
15. If the reading is 25 or less, the sample is ready for the nuclide extraction procedure. If the reading is greater than 25, more ultrasonic cycles are needed.

Nuclide Extraction Procedure

The following methods have been adapted Kohl and Nishiizumi (1992).

Quartz Dissolution and Be Spiking

1. Use a 250ml digestion vessel. The digestion vessel must be acid-cleaned by boiling in nitric acid for at least 5 hours. *Do not allow glass to come in contact with the sample because glass contains* ^{10}*B, an important isobar of 10Be that can reduce the precision of* ^{10}*Be/*^{9}*B measurements with AMS.*
2. Record vessel ID, and other important information. For every seven samples, add one chemical blank.
3. Take one sample, weigh the digestion vessel, and record the weight.
4. Tare the scale, weigh 40 g ($\underline{+}$0.0001g precision) of the quartz concentrate into the digestion vessel, and record weight.
5. Tare the scale again, and measure 0.5 g of a Be carrier, by determining how much Be carrier is needed for all the samples. Rinse out a 15 ml disposable beaker with a small amount of Be carrier, and then measure out the predetermined amount into the digestion vessel, using a disposable 5 ml pipette. <u>Never put anything into the Be carrier bottle.</u> Record the weight. Put the lid on the digestion vessel. *The carrier is a substance made up of mostly* ^{9}*Be with a known amount of* ^{10}*Be which is added to (1) increase the oxide mass so that there is sufficient target for the AMS, and (2) preclude us from having to carry all of the* ^{10}*Be through the entire chemistry.*
6. Repeat steps 3 – 5 for all the samples being processed. When completed, proceed to step 7.

7. Add the same amount of Be carrier to the chemical blank. Everything that is done to the samples, must now be done to the chemical blank for every step after 7.
8. Record carrier ID, concentration, date, density, and total mass transferred.
9. Keep all containers closed, immediately seal the beryllium carrier with parafilm, and refrigerate.
10. Add the following acids per 5 g of quartz: 20 ml of HF, 1 ml of $HClO_4$, and 5ml of aqua regia. It is a good idea to mix the acids together first, and then add the proportional volumes to each sample. Heat the samples at 125°C until all the quartz is dissolved. Leave the lids loose to allow for pressurization. *The HF, and* $HClO_4$ *will help dissolve the rock, and the nitric acid in the aqua regia helps to prevent the formation of* CaF_2.
11. Add additional HF and nitric acid (4:1 ratio) if necessary to complete the dissolution. Dissolution may take 1 to 4 days.
12. Tilt the caps on the digestion vessel, and raise the hotplate temperature to 200°C and evaporate to complete dryness (takes 1 – 2 days). When the evaporation is about 50% complete, add 15 ml of HNO_3 to help prevent Be from precipitating with F^-. Check the hotplate temperature using surface thermometers.
13. Record the colour and size of each sample. Make sure that the crusts are completely dry.
14. Let cool. Add 15 ml (or more if necessary) of $HClO_4$ acid. Completely dissolve the solid. Rinse the inside of the digestion vessel and the cap with deionized water, keeping the deionized water in the vessel. Then evaporate to dryness at 200°C. Do not add HF. *All of the F should now be gone, and much of the Be will be present as* $BeClO_4$.
15. Cool to <100°C. Add 15 ml of concentrated HNO_3, dissolve the solid, and evaporate to dryness at 100°C. Use more HNO_3 acid if the sample mass was >20 g of quartz. If the sample does not dissolve, add more HNO_3 acid.

16. Completely dissolve the dried sample in 15 ml of 2% HCl acid. Rinse the sides with an additional 5ml of 2% HCl acid. If the sample does not dissolve, add more 2% HCl.
17. Transfer each sample solution into two disposable 15 ml centrifuge tube or one 30ml tube. Rinse the digestion vessels with more 2% HCl, and decant into the centrifuge tubes. Centrifuge at 1500 rpm for 15 minutes. Rinse the digestion vessel with deionized water. Note the colour of the solutions, and the prescience of insolubles. Remove the insolubles, and store them until the Be and Al oxides have been completed.

ICP Subsampling & Al Spiking

The sample plus the Be-carrier is subsampled to determine the concentration of native Al in the rock with an ICP.AES. For these samples, no Aluminum carrier is added.

1. Label one 10 ml volumetric flasks with sample CNEF number. Label two 20 ml ICP scintillation vials with the sample CNEF number and A or B.
2. Using the analytical balance, weigh the 100 ml glass volumetric flask, and record the weight. *Do not include the caps when weighing.* The flask should be acid cleaned, and dry. Do not store the sample in volumetric flask for an extended time period. When finished with the flask, thoroughly rinse with aqua regia, then rinse 3 times with Type1 deionized water, dry in a clean oven at 110°C, cool, and cap.
3. Using the large transfer pipette, transfer the centrifuged supernates dissolved in ca. 20 ml 2% HCl into the weighed 100 ml volumetric. There should be no solid particles transferred. Rinse the Teflon digestion vessel with additional 2% HCl, and add to the volumetric. Bring the volume up to 100 ml with 2% HCl. Weigh the flask with the 100 ml solution, and record the weight. Rinse and wipe out the Teflon digestion vessels and covers with kimwipes, rinse again, and cover.
4. Shake the volumetric flask to mix the sample, and acid.

5. Tare the balance with volumetric A. Pipette 5 ml of the sample solution from the 100 ml volumetric into Volumetric A. Record the mass transferred. Bring the volume up to 10 ml with 2% HCl, and pour into the ICP scintillation bottle A.
6. Tare the balance with Volumetric B. Pipette 10 ml of the sample from the 100 ml volumetric into volumetric B. Record the mass transferred.
7. Weigh the 100 ml volumetric, and sample solution again. The mass removed, around 15 ml, should equal the sum of the sample masses in the ICP aliquots A, and B.
8. To the blank, transfer 1 g of Al carrier, unless it was predetermined that no ^{26}Al/^{27}Al measurement will be made on the samples. Record the mass, carrier ID, date, and density of Al-carrier added.
9. Pour the 85 ml of spiked sample solution back into the same Teflon digestion vessel that it was dissolved in.
10. Send the samples to the Dalhousie Metallurgical lab for analysis in order to determine how to proceed with the anion chemistry.

Anion Column Chemistry

If the columns are already conditioned, got to step 1, if the columns are not conditioned, go to step 12.

1. Evaporate the 80 ml of sample to dryness at 100 – 120°C (Takes several hours).
2. Dissolve a few drops of 9N HCL, and let stand for at least 4 hours. *This will convert many of the cations to chlorides.*
3. Transfer to centrifuge tubes, rinse digestion vessel with more 9N HCL, and bring up to 10 ml. The solutions will be clear and may be colourless or coloured.
4. Centrifuge for at least 10 minutes at a minimum of 3500 rpm.

5. Drain any 9N HCL in the columns. Discard. Record the anion column ID for each sample on WK6_Anion Column.
6. With the stopcock closed, gently transfer the sample (avoid any residue on the bottom) onto the conditioned anion column. Try not to stir up the resin or make depressions in the top of the resin. Cover columns with a plastic 15 ml beaker with a hole in the bottom to reduce contamination from dust.
7. Open the column and collect sample in the same digestion vessel that was used for each sample (wipe it clean with a clean kimwipe before reusing). Lower the column if there is any splash.
8. Elute the Be, Al, and other cations further with 30 ml 9N HCL, and collect that in same digestion vessel. The eluant should be colourless.
9. Add 5 ml of 4.5 N HCL from a transfer pipette. Collect this as Anion Supernate in a labeled 100 ml bottle.
10. Add 100 ml of 1N HCL, and collect this with the Anion Supernate.This liquid may appear yellow due to the $FeCl_4$.
11. Rinse resin with 50ml of deionized water and discard.
12. Recondition the anion columns by gently lowering the pH again. The bottles have been coded according to the order in which they should be added to the columns. (1) 50 ml of 1 N HCL (bottle A1) (2) 50 ml 4.5 N HCL (bottle A2), and (3) 100 ml 9 N HCL (bottle A3). Leave enough 9N HL in the column to prevent drying (approximately 2 mm high). Write any comments on WS6 worksheet.

Controlled Precipitation

This step will separate the elements that precipitate as hydroxides from other elements in the sample. Be, Al, Fe, and Ti are all insoluble as hydroxides at 6.5<pH<9.5.

1. Evaporate anion elute to dryness at 125°C. (Takes several hours)

2. Dissolve in 10 drops of 1N HCL from a large transfer pipette. Meanwhile, mix a 2% NH_4Cl buffer by first diluting 20% optimal NH_4OH to 2% by measuring 10 ml of NH4OH, then filling to 100 ml with deionized water. Add 100 ml of 0.5N HCl to 200ml of NH_4Cl. Now mix a 1:1 solution with 200 ml of the NH_4Cl buffer, and 200 ml of 0.5N HCl. Pour 8 ml of this 1:1 solution into each Teflon vessel. You should get precipitate here.
3. Transfer to a 15 ml centrifuge tube. Rinse Teflon vessels with 2+ml of 1:1 solution. The bottom of the vessels will appear greasy due to the NH_4OH. Centrifuge for 10 minutes at 3500 rpm. You should get a lot of precipitate here.
4. Decant by pipetting into a clean centrifuge tube (to remove any insoluble particles). Heat in a water bath at 50 to 60°C until thermal equilibrium is reached.
5. Add drops of 1:1 optimal grade HN_4OH: H_2O to bring sample to a pH = 8. Add four or five transfer pipette drops first, check pH, then add single drops. Do not raise the pH>10 if possible. If this happens, drop the pH to <5 by adding concentrated HCl. The precipitate will form a cloud in the sample. You should get about 1 ml of precipitate.
6. Centrifuge the samples for 15 minutes at 3500 rpm.
7. Check the pH of the solution. *There should be a little drop in pH as the precipitate has been removed. If the pH is below 6, redo step 4 & 5.*
8. Decant, save the liquid with the Anion Supernate.
9. Wash with approximately 8 ml of deionized water. Vortex. Centrifuge again.
10. Repeat step 8.
11. If necessary, repeat step 8, and record on the worksheet the final approximate precipitate volume.
12. Decant, and discard the solution.

Cation Column Chemistry

The object is to remove unwanted elements from the sample, and to separate the desired elements so concentrates of Be and Al can be collected.

1. Dissolve the precipitate in 5ml concentrated HCl. Evaporate to dryness at 125°C.
2. Redissolve in 2.5 ml 1N HCl, and 2.5 ml 0.5N HCl.
3. Transfer to centrifuge tube. Rinse the digestion vessel with about 1 or 2 ml of 0.5N HCl. Try to keep centrifuge column to about 7-8 ml. Centrifuge for 10 minutes at 1500 rpm to separate any undissolved particles.
4. Record the column ID for each sample on WK8_CationColumn. We can sue the cation resin for about 5 or 6 runs before it needs to be replaced. If the columns are already conditioned, go to step 5. If the columns need to be conditioned, do to step 12.
5. Columns should contain a small amount of 0.5N HCl. Allow the columns to drain through, and discard the solution. Add 4 ml of deionized water carefully with a disposable pipette. Discard in the Erlenmeyer flask.
6. Pipette all the sample (about 11 ml) into the appropriate column. Discard eluant.
7. Using the top loading balance, add 220 ml of 0.5 N HCL (bottle C6). Pipette the first 15 ml in order to prevent disturbing the resin, then pour the remaining in. Collect as Cation Supernate.
8. Add 200 ml of 0.5 N HCL (bottle C7). Collect this eluant as Be-Sample.
9. Add 30 ml of 1N HCL (bottle C8). Save as the Be-sample
10. Add 100 ml of 4.5N HCl to release the Al (bottle C9). Approximately 88% or more of the total Al in the original sample should be recovered. Save as the Al-sample.
11. Condition the cation columns. The bottles have been coded according to the order in which they should be added to the columns. If the columns contain 4.5N HCL, then add the bottles in order: C1-C2-C3-C4-C5. If you have just added new resin, then condition in this order: C3-C2-C1-C2-C3-C4-C5.

100 ml 9N HCl (bottle C1)
50 ml 4.5N HCl (bottle C2)
50 ml 1N HCl (bottle C3)
50 ml type 1 water (bottle C4)
100 ml 0.5N HCl (bottle C5)

Leave enough in the column to over the top of the resin to prevent drying.

Be Sample

This step is to prepare the Be Sample solution into an oxide which the AMS can use.

1. Prepare the quartz vials. Rinse them with Type 1 water. Do not use soap. Then boil them at 150°C in a Teflon beaker with water:HF:HNO_3 mixture of 80:10:10. Rinse the vials many times in Type 1 water. Place in oven, and dry at 110°C. Cool in covered container.
2. Evaporate the Be-Sample from the cation column in the same digestion vessel for about 8 hours at 125°C.
3. Add 2-5ml of 20% perchloric acid. Evaporate over several hours at 200°C. *To mix 20% perchloric acid, add 285ml ultrapure grade 69-72%* $HClO_4$ *acid to 715ml Type 1 water.*
4. Add another 2-5ml of 20% perchloric acid, and evaporate it at 200°C.
5. Dissolve the sample in 8 ml of 0.5N HCL. Let the sample dissolve for at least 30 minutes.
6. Transfer to 15 ml centrifuge tube. Rinse vessel with a little water.
7. Centrifuge at 3500 rpm for 15 minutes, and decant by pipette into clean centrifuge tubes. Any white residue may indicate that there is a significant TiO_2 phase in the sample, note this.
8. Heat the centrifuge tubes in a 50-60°C deionized water bath until thermal equilibrium.
9. Precipitate the $Be(OH)_2$ by bringing the solution to a pH=9.2. To do this, we use the highest ammonia gas available.

- (i) Attach a new micropipette tip to the hose from the NH3 tank. Wipe each tip with a kimwipe.
- *(ii)* Clamp the hose to a ring stand so the pipette tip dangles at a height, so that they protrude halfway into the centrifuge tubes in a test tube rack.
- *(iii)* Remove a centrifuge tube from the water bath, dry with a kimwipe. With the gas on, place the tip approximately halfway into the centrifuge tube, and bubble the gas through the solution, slowly for about 8 seconds. Check for a cloudy precipitate.
- *(iv)* Check the pH by dropping a microbiology loop into the sample, and dabbing a piece of pH paper with 0.5 pH unit resolution. The desired pH is 9.2 to 9.3. Add 1N HCL to lower pH if necessary. If the pH exceeds 12 at any time, acidify with optima grade HCl, and begin again.

10. Centrifuge the sample for 15 minutes at 3500 rpm. Test the pH of each sample after centrifugation. If the pH is less than 8, redo step 8. If the pH>8, record the volume of precipitate, decant Supernate.
11. Wash the decanted precipitate with about 9 ml of Type 1 water, vortex, and centrifuge.
12. If the volume of precipitate is greater than 1ml, repeat step 11 once more.
13. Record on WK9_BeSample the mass of the quartz vials assigned to each sample.
14. Make a map of the sample locations in the furnace holder. The marker ink will not survive the furnace.
15. In the dry lab, add a minimum amount of Type 1 water, and slurry the precipitate. Samples that are too watery have a greater chance of boiling, and exploding, which of course we want to avoid.
16. Transfer the sample with a fine transfer pipette into the designated quartz vial. Try to get every drop. Place the vial into the furnace sample holder, and cover the vial immediately with an alumina crucible.
17. Heat the ocean at 120°C for at least 2-3 hours.
18. Let samples cool. Scrape the sample oxide down from the walls of the vial.

19. Place the sample holder in a room temperature muffle furnace. Covert to BeO by heating in 850°C furnace for a minimum of 1 hour.
20. Cool while covered, record the mass of the sample+vial. Label the quartz vial with the CNEF #.
21. In the target loading station, add niobium power to the vial at a ratio of 5:1 Niobium to oxide. Cover the vial with weighing paper, and parafilm. Store the sample or proceed to target loading.

Final Product and Accelerator Mass Spectrometry (AMS)

The procedure for target loading was communicated by Lab Technician Guang Yang (December 2002).

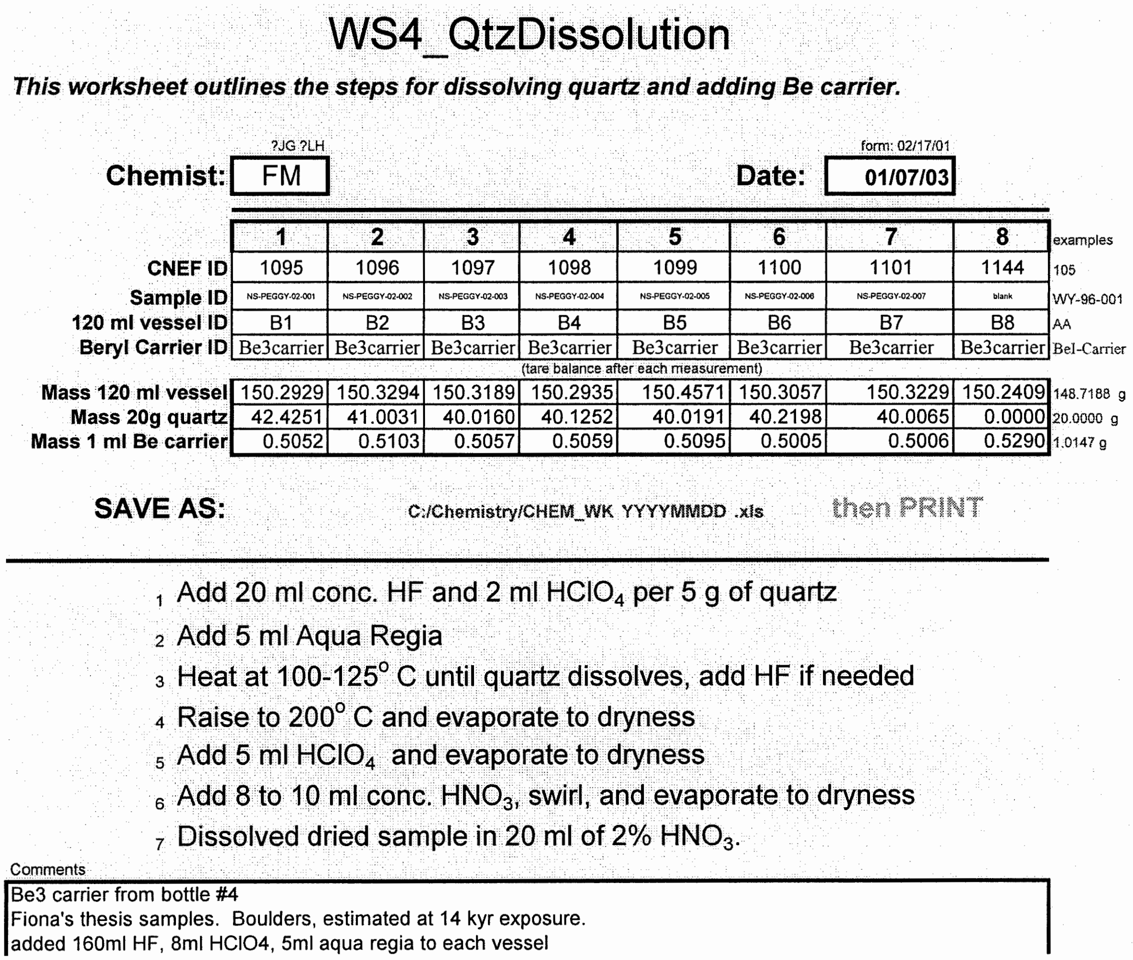
Target Loading Procedure

The Be oxide powders are loaded into metal targets, which are then sent to the Lawrence Livermore Accelerator in California.

1. Take the dust cover off of the target loading station in the dry lab.
2. Take one sample, and use a small metal spatula to remove the sample from the walls of the quartz vial.
3. Estimate how much sample is inside the vial to determine how much Niobium, or Silver powder is to be added. *The Niobium powder is added to the Be oxide powder in a 1:1 ratio, and the Silver powder is added to the Al oxide powder in a 1:1 ratio. At least 1 mg of Niobium or Silver powder must be added per sample.*
4. Take a piece of weighing paper, fold it in half, and then spread open again. Pour the sample from the quartz vial onto the weighing paper. If the sample is coarse grained, fold the paper around the sample, and hit the paper with the hammer to reduce the grain size. If the powder is already fine grained, procede to step 5.
5. Put three pieces of weighing paper on the target holder in case any of the sample is spilled. Put the target on the target holder, and pour the sample into the hole, in the center of the top of the target. *Use an Al target for the Be oxide, and a Stanley steel target for the Al oxide.*

6. Clean a No. 55 drill blank with sand paper to remove any dirt on the surface. Take a kimwipe, and use isopropyl alcohol to finish cleaning the drill blank.
7. Put the drill blank in the hole of the target, and hit the drill blank with the hammer to make sure that all the sample is at the bottom of the hole in the target. Label the target with the sample number.
8. Put the target into a 1.2 ml cryogenic vial, and screw on the lid. Wrap the lid with parafilm, and label the vial with the sample number.
9. When all the samples have been loaded, send to the Lawrence Livermore Accelerator.

Appendix 3: Chemistry Data



WS4_QtzDissolution

This worksheet outlines the steps for dissolving quartz and adding Be carrier.

?JG ?LH

Chemist: FM

form: 02/17/01

Date: **01/07/03**

	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	examples
CNEF ID	1095	1096	1097	1098	1099	1100	1101	1144	105
Sample ID	NS-PEGGY-02-001	NS-PEGGY-02-002	NS-PEGGY-02-003	NS-PEGGY-02-004	NS-PEGGY-02-005	NS-PEGGY-02-006	NS-PEGGY-02-007	blank	WY-96-001
120 ml vessel ID	B1	B2	B3	B4	B5	B6	B7	B8	AA
Beryl Carrier ID	Be3carrier	Be3carrier	Be3carrier	Be3carrier	Be3carrier	Be3carrier	Be3carrier	Be3carrier	BeI-Carrier

(tare balance after each measurement)

Mass 120 ml vessel	150.2929	150.3294	150.3189	150.2935	150.4571	150.3057	150.3229	150.2409	148.7188 g
Mass 20g quartz	42.4251	41.0031	40.0160	40.1252	40.0191	40.2198	40.0065	0.0000	20.0000 g
Mass 1 ml Be carrier	0.5052	0.5103	0.5057	0.5059	0.5095	0.5005	0.5006	0.5290	1.0147 g

SAVE AS: **C:/Chemistry/CHEM_WK YYYYMMDD .xls** then PRINT

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1. Add 20 ml conc. HF and 2 ml $HClO_4$ per 5 g of quartz
2. Add 5 ml Aqua Regia
3. Heat at 100-125° C until quartz dissolves, add HF if needed
4. Raise to 200° C and evaporate to dryness
5. Add 5 ml $HClO_4$ and evaporate to dryness
6. Add 8 to 10 ml conc. HNO_3, swirl, and evaporate to dryness
7. Dissolved dried sample in 20 ml of 2% HNO_3.

Comments

Be3 carrier from bottle #4
Fiona's thesis samples. Boulders, estimated at 14 kyr exposure.
added 160ml HF, 8ml HClO4, 5ml aqua regia to each vessel

WS5_ICP Aliquot and Al spiking

This worksheet outlines the steps for collecting ICP aliquots and adding Al carrier.

FM ?

Chemist: FM

form: 02/17/01

Date: 01/16/03

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1 **Label two 10 ml volumetric flasks, A & B per sample (16)**

2 **Label three ICP bottles with NewfID and A B & C per sample (24)**

	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	examples
CNEF ID	**1095**	**1096**	**1097**	**1098**	**1099**	**1100**	**1101**	**1144**	105
Sample ID	NS-PEGGY-02-001	NS-PEGGY-02-002	NS-PEGGY-02-003	NS-PEGGY-02-004	NS-PEGGY-02-005	NS-PEGGY-02-006	NS-PEGGY-02-007	blank	WY-96-001
Al carrier ID	Al-carrier4	Al-carrier4	Al-carrier4	Al-carrier4	Al-carrier4	Al-carrier4	Al-carrier4	Al-carrier4	ALI-carrier
Quant-EM est. Al in qtz	50	50	125	125	125	125	125	0	ppm
Volume carrier to add to smpl	-1.12	-1.05	-4.00	-4.02	-4.00	-4.03	-4.00	1.00	ml

Tare between mass measurements

Mass100 ml volumetric	56.8550	57.1251	56.9031	57.2359	57.2388	56.6726	57.3973	57.2515	66.9239 g
100ml volumetric+sample+HCl	157.6321	158.1559	157.7274	158.0457	158.0970	157.4552	158.1164	157.9383	166.9875 g
Mass 5 ml smpl pipetted to vol A	4.9985	5.1352	5.0044	5.0086	5.0149	5.0307	5.0040	5.0122	5.0000 g
Final Mass of 100 ml vol and smpl	142.5836	142.9302	142.6380	142.9976	142.9641	143.2357	142.9811	142.7484	1.0100 g
Mass Al carrier to remaining (row18)									1 0100 g

% of original sample before carrier	85.0%	84.8%	84.9%	85.0%	84.9%	85.8%	84.9%	84.8%	85%

PRINT this form

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3 Wipe digestion vessel and cover clean

4 Transfer the 80 ml sample back into vessel

5 Bring contents of volumetrics A and B to 10 ml

6 Transfer contents volumetrics A&B to ICP bottles A&B

WS6_Anion Column Chemistry

This worksheet outlines the steps for the Anion Column Chemistry

FM ?

form: 02/17/01

Chemist: FM **Date:** 01/17/03

Print this page

☐1 Evaporate 80 ml to dryness at 100-120°C (will take at least 3 hrs)

☐2 Dissolve in 10 ml 9N HCl (let stand for several hours)

☐3 Transfer to 15 ml centrifuge tubes, rinse digestion vessels with 9N HCl to bring volume in tube to 10 ml

☐4 Centrifuge at 1500 rpm or higher for minimum of 10 minutes

☐5 Allow any 9 N HCl in columns to drain out; discard

Column ID	A	B	C	D	E	F	G	H	AnionColumnID
NEWF ID	1095	1096	1097	1098	1099	1100	1101	1144	105
Sample ID	NS-PEGGY-02-001	NS-PEGGY-02-002	NS-PEGGY-02-003	NS-PEGGY-02-004	NS-PEGGY-02-005	NS-PEGGY-02-006	NS-PEGGY-02-007	blank	WY-96-001

☐6 With stopcock closed, pipet sample (avoid residue)onto columns.

☐7 Collect sample in same (wiped)120 ml teflon vessel

☐8 Elute with 30 ml 9 N HCl, and collect that, close stopcock

☐9 5 ml 4.5 N HCl, collect Anion Supernate in labeled 100 ml bottle

☐10 100 ml 1 N HCl, collect Anion Supernate

☐11 50 ml deionized water. Discard.

☐12 **CONDITION ANION COLUMN**

(bottle A1) 50 ml 1N HCl, discard
(bottle A2) 50 ml 4.5 N HCl, discard
(bottle A3) 100 ml 9 N HCl, discard, but retain acid approx. 2 mm above resin

Comments

evaporated sample to dryness
added 10ml 9 N Hcl
transferred to centrifuge
completed anion chemistry for samples in B1-B4 digestion vessels
completed anion chemistry for samples in B5-B8 digestion vessels

WS7_Controlled Precipitate

This worksheet outlines the steps for the controlled precipitation chemistry

FM ?

Chemist: FM

form: 02/17/01

Date: 01/24/03

Print this page

1 Evaporate "anion" elute to dryness at 125°C

2 Dissolve in 10 ml of a 1:1 solution of 0.5N HCl and 2% NH_4Cl

3 Transfer to 15 ml centrifuge, centrifuge for 10 minutes

4 Decant into clean test tube, heat in water bath at 60°C

5 Add drops of 1:1 $NH_4OH:H_2O$ to pH=7 (5 drops first then single)

6 Centrifuge for 15 minutes

7 Check pH of liquid, if less than pH=7, redo step ☐5

8 Decant, save with Anion Supernate

9 Wash with deionized water, vortex, centrifuge, decant

10 Wash with deionized water, vortex, centrifuge, decant

11 Wash with deionized water, vortex, centrifuge, decant

NEWF ID	1095	1096	1097	1098	1099	1100	1101	1144	#
Sample ID	NS-PEGGY-02-001	NS-PEGGY-02-002	NS-PEGGY-02-003	NS-PEGGY-02-004	NS-PEGGY-02-005	NS-PEGGY-02-006	NS-PEGGY-02-007	blank	
Approx. vol. Ptte	0.8	0.7	0.9	0.8	0.6	0.9	0.7	0.1	

Comments

Added the wrong solution to B2-B4
Had to add HCL to remove ppt
completed

c

WS8_Cation Column Chemistry

This worksheet outlines the steps for the Cation Column Chemistry

FM ?

Chemist: FM

form: 02/17/01

Date: 01/31/03

Print this page

☐1 Dissolve in 5 ml conc. HCl and evaporate to dryness at 125°C

☐2 Redissolve in 2.5 ml 1N HCl and 2.5 ml 0.5 N HCl

☐3 Transfer to centrifuge tube, rinse with 1 ml 0.5N, and centrifuge

Column ID	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**1**	examples
CNEF ID	1095	1096	1097	1098	1099	1100	1101	1144	105
Sample ID	NS-PEGGY-02-001	NS-PEGGY-02-002	NS-PEGGY-02-003	NS-PEGGY-02-004	NS-PEGGY-02-005	NS-PEGGY-02-006	NS-PEGGY-02-007	blank	WY-96-001

☐4 Pipette 8-10 ml sample into designated conditioned cation column

☐5 add 220 ml 0.5 N HCl (bottle C6) to column

☐6 collect as Cation Supernate

☐7 Add 200 ml 0.5N HCl (bottle C7)

☐8 Eluant into 250 ml bottle, save as Be-sample

☐9 Add 30 ml 1.0 N HCl (C8)

☐10 Eluant into 250 ml bottle, save as Al-sample

☐11 Add 100 ml 4.5 N HCl (C9)

☐12 Eluant into 250 ml bottle labeled Al-sample

☐13 **CONDITION CATION COLUMN**

(bottle C1) 60 ml 9N HCl
(bottle C2) 30 ml 4.5 N HCl
(bottle C3) 30 ml 1 N HCl
(bottle C4) 30ml water
(bottle C5) 60 ml 0.5 N HCl

WS9_Be Sample Chemistry

This worksheet outlines the steps to prepare the BeO sample

FM ?

Chemist: FM

form: 02/17/01

Date: 02/02/03

Print this page

☐$_1$ Evaporate Be Sample from column in digestion vessels at 125°C

☐$_2$ Add 2-5 ml 20% perchloric and evaporate at 200oC

☐$_3$ Again, add 2-5 ml 20% perchloric and evaporate at 200°C

☐$_4$ Dissolve sample in 8 ml of 0.5 N HCl (optima grade)

☐$_5$ Transfer to 15 ml centrifuge tube

☐$_6$ Centrifuge and decant into clean centrifuge tube

☐$_7$ Heat centrifuge tubes in water bath at 60°C

☐$_8$ Precipitate $Be(OH)_2$ using Matheson ultimate grade ammonia gas

Gently bubble NH_3 with clean pipet tip on hose

for ca.15 bubbles, or ca. 8-12 sec until ptte forms

Optimum pH=9.2; 1N HCl may be added

☐$_9$ Centrifuge 15 min., decant (save and redo ☐$_8$ if pH of liquid is < 8)

☐$_{10}$ Wash with water, vortex, centrifuge for 10 min, and decant

☐$_{11}$ Record mass quartz vials, label, and place them in furnace holder

NEWF ID	1095	1096	1097	1098	1099	1100	1101	1144	105
Sample ID	NS-PEGGY-02-001	NS-PEGGY-02-002	NS-PEGGY-02-003	NS-PEGGY-02-004	NS-PEGGY-02-005	NS-PEGGY-02-006	NS-PEGGY-02-007	blank	WY-96-001
Mass Qtz Vial	2.1816	2.2459	2.211	2.2748	2.2757	2.178	2.2319	2.2719	2.1400 g
Mass Vial+Spl	2.1828	2.247	2.2117	2.2757	2.2763	2.1789	2.2328	2.2719	2.1410 g
Mass Spl	0.0012	0.0011	0.0007	0.0009	0.0006	0.0009	0.0009	0	1 mg

☐$_{12}$ Add 1 small drop of water with micropipet, slurry precipitate

☐$_{13}$ Transfer sample into quartz vial, cover with alumina vial

☐$_{14}$ Heat in oven at 120°C for 2-3 hours

☐$_{15}$ Let cool and scrape sample down from walls of quartz tube

☐$_{18}$ Place in furnace. Convert to BeO in furnace at 850°C for minimum 1 hr

☐$_{19}$ Determine mass of vial + sample

WS4_QtzDissolution

This worksheet outlines the steps for dissolving quartz and adding Be carrier.

?JG ?LH

Chemist: FM **Date:** 02/20/01

	1	2	3	4	5	examples
CNEF ID	1102	1103	1104	1105	1145	105
Sample ID	NS-PEGGY-COVE-008	NS-PEGGY-COVE-009	NS-PEGGY-COVE-010	NS-PEGGY-COVE-011	BLANK	WY-96-001
120 ml vessel ID	B9	B10	B11	B12	B13	AA
Beryl Carrier ID	Be3carrier	Be3carrier	Be3carrier	Be3carrier	Be3carrier	BeI-Carrier
			(tare balance after each measurement)			
Mass 120 ml vessel	150.2753	150.1606	150.2877	150.1790	150.2666	148.7188 g
Mass 20g quartz	40.0282	40.0298	40.0049	40.0012	0.0000	20.0000 g
Mass 1 ml Be carrier	0.5034	0.5098	0.5110	0.5011	0.5020	1.0147 g

SAVE AS: C:/Chemistry/CHEM_WK YYYYMMDD

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1. Add 20 ml conc. HF and 2 ml $HClO_4$ per 5 g of quartz
2. Add 5 ml Aqua Regia
3. Heat at 100-125^{o} C until quartz dissolves, add HF if needed
4. Raise to 200^{o} C and evaporate to dryness
5. Add 5 ml $HClO_4$ and evaporate to dryness
6. Add 8 to 10 ml conc. HNO_3, swirl, and evaporate to dryness
7. Dissolved dried sample in 20 ml of 2% HNO_3.

Comments

Added 40 g of quartz to digestion vessels
Be carrier from bottle 4
added 160 ml (HF), 5 ml (Aqua regia), 8 ml (HClO4)
added 6ml of HClO4
added HNO3
added HCl instead of 2% HCl to B10, B11, B12
completed B9, B13 with 2% HCl
added 20ml HCl to blank, put B10, B11, B12, B13 to dry

WS5_ICP Aliquot and Al spiking

vorksheet outlines the steps for collecting ICP aliquots and adding Al c

FM ?

Chemist: FM **Date:** 12/04/01

☐ 1 **Label two 10 ml volumetric flasks, A & B per sample (16)**

☐ 2 **Label three ICP bottles with NewfID and A B & C per sample (24)**

	1	2	3	4	5	examples
CNEF ID	1102	1103	1104	1105	1145	105
Sample ID	NS-PEGGY-COVE-008	NS-PEGGY-COVE-009	NS-PEGGY-COVE-010	NS-PEGGY-COVE-011	BLANK	WY-96-001
Al carrier ID	Al-carrier4	Al-carrier4	Al-carrier4	Al-carrier4	Al-carrier4	ALI-carrier
Quant-EM est. Al in qtz	125	125	50	125	0	ppm
Volume carrier to add to smpl	-14.63	-14.63	-1.50	-14.63	1.00	ml
	Tare between mass measurements					
Mass100 ml volumetric	57.2351	56.9029	57.2513	57.1659	56.6728	66.9239 g
100ml volumetric+sample+HCl	157.9621	157.6482	157.9938	158.0224	157.3739	166.9875 g
Mass 5 ml smpl pipetted to vol A	5.0009	5.0090	5.0086	5.0033	4.9999	5.0000 g
Mass 10 ml smpl pipetted to vol B	10.0024	10.0005	10.0141	10.0053	10.0051	5.0000 g
Final Mass of 100 ml vol and smpl	142.9120	142.6038	142.9264	142.9633	142.3179	1.0100 g
Mass Al carrier to remaining (row18)					1.0476	1.0100 g
% of original sample before carrier	0.8495	0.8496	0.8493	0.8494	0.8494	0.0100 g

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☐ 3 Wipe digestion vessel and cover clean

☐ 4 Transfer the 80 ml sample back into vessel

☐ 5 Bring contents of volumetrics A and B to 10 ml

☐ 6 Transfer contents volumetrics A&B to ICP bottles A&B

WS6_Anion Column Chemistry

This worksheet outlines the steps for the Anion Column Chemistry

FM ?

Chemist: FM **Date:** 01/27/03

Print this page

☐ 1 Evaporate 80 ml to dryness at 100-120°C (will take at least 3 hrs)

☐ 2 Dissolve in 10 ml 9N HCl (let stand for several hours)

☐ 3 Transfer to 15 ml centrifuge tubes, rinse digestion vessels with 9N HCl to bring volume in tube to 10 ml

☐ 4 Centrifuge at 1500 rpm or higher for minimum of 10 minutes

☐ 5 Allow any 9 N HCl in columns to drain out; discard

Column ID	**A**	**B**	**C**	**D**	**E**	AnionColumnID
NEWF ID	1102	1103	1104	1105	1145	105
Sample ID	NS-PEGGY-COVE-008	NS-PEGGY-COVE-009	NS-PEGGY-COVE-010	NS-PEGGY-COVE-011	BLANK	WY-96-001

☐ 6 With stopcock closed, pipet sample (avoid residue)onto columns.

☐ 7 Collect sample in same (wiped)120 ml teflon vessel

☐ 8 Elute with 30 ml 9 N HCl, and collect that, close stopcock

☐ 9 5 ml 4.5 N HCl, collect Anion Supernate in labeled 100 ml bottle

☐ 10 100 ml 1 N HCl, collect Anion Supernate

☐ 11 50 ml deionized water. Discard.

☐ 12 **CONDITION ANION COLUMN**

(bottle A1) 50 ml 1N HCl, discard
(bottle A2) 50 ml 4.5 N HCl, discard
(bottle A3) 100 ml 9 N HCl, discard, but retain acid approx. 2 mm above resin

Comments

cv

WS7_Controlled Precipitate

orksheet outlines the steps for the controlled precipitation ch

FM ?

Chemist: FM **Date:** 01/29/03

Print this page

☐ 1 Evaporate "anion" elute to dryness at 125°C

☐ 2 Dissolve in 10 ml of a 1:1 solution of 0.5N HCl and 2% NH_4Cl

☐ 3 Transfer to 15 ml centrifuge, centrifuge for 10 minutes

☐ 4 Decant into clean test tube, heat in water bath at 60°C

☐ 5 Add drops of 1:1 $NH_4OH:H_2O$ to pH=7 (5 drops first then single)

☐ 6 Centrifuge for 15 minutes

☐ 7 Check pH of liquid, if less than pH=7, redo step ☐ 5

☐ 8 Decant, save with Anion Supernate

☐ 9 Wash with deionized water, vortex, centrifuge, decant

☐ 10 Wash with deionized water, vortex, centrifuge, decant

☐ 11 Wash with deionized water, vortex, centrifuge, decant

NEWF ID	1102	1103	1104	1105	1145	#
Sample ID	NS-PEGGY-COVE-008	NS-PEGGY-COVE-009	NS-PEGGY-COVE-010	NS-PEGGY-COVE-011	BLANK	
Approx. vol. Ptte	2.5	2.7	0.9	2.1	0.5	

Comments

WS8_Cation Column Chemistry

his worksheet outlines the steps for the Cation Column Chemistr

FM ?

Chemist: FM **Date:** 01/30/03

Print this page

☐ 1 Dissolve in 5 ml conc. HCl and evaporate to dryness at 125°C

☐ 2 Redissolve in 2.5 ml 1N HCl and 2.5 ml 0.5 N HCl

☐ 3 Transfer to centrifuge tube, rinse with 1 ml 0.5N, and centrifuge

Column ID	**1**	**2**	**3**	**4**	**5**	**6**	examples
CNEF ID	0	1102	1103	1104	1105	1145	105
Sample ID	0	NS-PEGGY-COVE-008	NS-PEGGY-COVE-009	NS-PEGGY-COVE-010	NS-PEGGY-COVE-011	BLANK	WY-96-001

☐ 4 Pipette 8-10 ml sample into designated conditioned cation column

☐ 5 add 220 ml 0.5 N HCl (bottle C6) to column

☐ 6 collect as Cation Supernate

☐ 7 Add 200 ml 0.5N HCl (bottle C7)

☐ 8 Eluant into 250 ml bottle, save as Be-sample

☐ 9 Add 30 ml 1.0 N HCl (C8)

☐ 10 Eluant into 250 ml bottle, save as Al-sample

☐ 11 Add 100 ml 4.5 N HCl (C9)

☐ 12 Eluant into 250 ml bottle labeled Al-sample

☐ 13 **CONDITION CATION COLUMN**

(bottle C1) 60 ml 9N HCl
(bottle C2) 30 ml 4.5 N HCl
(bottle C3) 30ml 1 N HCl
(bottle C4) 30ml water
(bottle C5) 60 ml 0.5 N HCl

WS9_Be Sample Chemistry

This worksheet outlines the steps to prepare the BeO sample

FM ?

Chemist: FM **Date:** 02/02/03

Print this page

☐1 Evaporate Be Sample from column in digestion vessels at 125°C

☐2 Add 2-5 ml 20% perchloric and evaporate at 200oC

☐3 Again, add 2-5 ml 20% perchloric and evaporate at 200°C

☐4 Dissolve sample in 8 ml of 0.5 N HCl (optima grade)

☐5 Transfer to 15 ml centrifuge tube

☐6 Centrifuge and decant into clean centrifuge tube

☐7 Heat centrifuge tubes in water bath at 60°C

☐8 Precipitate $Be(OH)_2$ using Matheson ultimate grade ammonia gas

Gently bubble NH_3 with clean pipet tip on hose
for ca.15 bubbles, or ca. 8-12 sec until ptte forms
Optimum pH=9.2; 1N HCl may be added

☐9 Centrifuge 15 min., decant (save and redo ☐8 if pH of liquid is < 8)

☐10 Wash with water, vortex, centrifuge for 10 min, and decant

☐11 Record mass quartz vials, label, and place them in furnace holder

NEWF ID	1102	1103	1104	1105	1145	105
Sample ID	NS-PEGGY-COVE-008	NS-PEGGY-COVE-009	NS-PEGGY-COVE-010	NS-PEGGY-COVE-011	BLANK	WY-96-001
Mass Qtz Vial	2.2548	2.181	2.2229	2.1239	2.2071	2.1400 g
Mass Vial+Spl	2.2559	2.19	2.224	2.1251	2.2078	2.1410 g
Mass Spl	0.0011	0.009	0.0011	0.0012	0.0007	1 mg

☐12 Add 1 small drop of water with micropipet, slurry precipitate

☐13 Transfer sample into quartz vial, cover with alumina vial

☐14 Heat in oven at 120°C for 2-3 hours

☐15 Let cool and scrape sample down from walls of quartz tube

☐18 Place in furnace. Convert to BeO in furnace at 850°C for minimum 1 hr

☐19 Determine mass of vial + sample

Appendix 4: Field data of sampled boulders and bedrock surfaces

Sample Number	**NS-PEGGY-02-001**	**NS-PEGGY-02-002**	**NS-PEGGY-02-003**	**NS-PEGGY-02-004**
Type of Sample	Boulder	Boulder	Boulder	Boulder
Longitude **Latitude**	44° 30.139N 063° 53.829W ±7 m	44o 30.179N 063o 53.853W ±4 m	44o 30.140N 063o 53.646 ± 7 m	44o 30.163N 063o 53.845W ± 5 m
Elevation	37 m	35 m	38 m	36 m
Placement	The boulder is perched on a west hill slope of bedrock ridge.	The boulder is on a west hill slope of a bedrock ridge.	The boulder is perched on the stoss side of a stoss-lee ridge	The boulder is perched on the west side of a gently sloping ridge
Lithology of sample	Coarse grained granitic gneiss	Coarse grained granodiorite.	Coarse grained granodiorite.	Coarse grained granodiorite.
Size/ geometry	Height = 1.6 m Volume = 8 m^3 horizontal surface	Height = 3.1 m Volume = 50 m^3 Surface slopes <10° with horizontal areas	Height = 2.1 m Volume = 4 m^3 Flat top surface is horizontal.	Height = 2.5 m Volume = 12 m^3 Flat top surface is horizontal.
Vegetation cover on sample	100% lichen covered	95% lichen covered	80% lichen covered	95% lichen covered.
Topographical shielding	none	none	None	None
Snow/till cover	No possibility	No possibility	No possibility	No possibility.
Grassification, gnammas, or rills	1 rill	none	None	None
Vegetation surrounding erractic	Bedrock is mostly bare	Bedrock is covered by small shrubs with bare areas.	Bedrock is bare.	Bedrock is bare with a few small shrubs.

Sample Number	**NS-PEGGY-02-005**	**NS-PEGGY-02-006**	**NS-PEGGY-02-007**
Type of Sample	Boulder	Boulder	Boulder
Longitude **Latitude**	44o 29.961 N 063 53.972 W ± 6 m	44o 29.769 N 063 53.480 W ± 6 m	44o 29.777 N 063o 53.330 W ± 4 m
Elevation	37 m	44 m	54 m
Placement	The boulder is sitting on horizontal bedrock, against a ridge	The boulder is situated on the west side of a ridge.	The boulder is perched on the west side of a ridge.
Lithology of sample	Coarse grained granodiorite.	Coarse grained granodiorite.	Coarse grained granodiorite.
Size/ geometry	Height = 3 m Volume = 18 m^3 Horizontal ridge along the top surface.	Height = 2 m Volume = 12 m^3 Surface is dipping with horizontal areas.	Height = 3 m Volume 15 m^3 Surface is dipping 9o.
Vegetation cover on sample	100% lichen covered	40% lichen covered.	30% lichen covered.
Topographical shielding	None	None	None
Snow/till cover	None	None	None
Grassification, gnammas, or rills	Some grassification	None	Some grassification.
Vegetation surrounding erractic	Exposed bedrock ridges are lichen covered, with small shrubs.	Bare bedrock with very small shrubs.	Bedrock is bare.
Sampling location, thickness	Sampled on boulder top staying 30 cm from any edge	Sampled on boulder top staying 30 cm from any edge	Sampled on boulder top staying 30 cm from any edge

Sample Number	**NS-PEGGY-02-008**	**NS-PEGGY-02-009**	**NS-PEGGY-02-010**	**NS-PEGGY-02-011**
Type of Sample	Bedrock surface	Bedrock surface	Bedrock surface	Bedrock surface
Longitude **Latitude**	44o 20.130 N 063o 53.635 ± 4 m	44o 29.969 N 063o 53.976 W ± 4 m	44o 29.765 N 063 53.470 W ± 5 m	44o 29.783 N 063 53.339 W ± 4 m
Elevation	38 m	37 m	37 m	54 m
Sample Location	Top of a 6 m high stoss-lee ridge near NS-PEGGY-02-003.	Top of a 4 m stoss-lee ridge near NS-PEGGY-02-005	Top of a 2 m high stoss-lee ridge near NS-PEGGY-02-006	Top of a 8 m high stoss-lee ridge near NS-PEGGY-02-011
Lithology of sample	Coarse grained granite.	Coarse grained granite.	Coarse grained granite	Coarse grained granite
Vegetation cover on sample	20% lichen covered.	80% lichen covered.	60% lichen covered.	40% lichen covered
Topographical shielding	None	none	None	None
Snow/till cover	Possibility	Possibility	Possibility	Possibility
Grassification, gnammas, or rills	None	None	None	None
Sample Thickness	No deeper than 3 cm., staying 30 cm form any edge.	No deeper than 3 cm., staying 30 cm form any edge.	No deeper than 3 cm., staying 30 cm form any edge.	No deeper than 3 cm., staying 30 cm form any edge.

Appendix 5: AMS Data

CENTER FOR ACCELERATOR MASS SPECTROMETRY

Lawrence Livermore National Lab

NAME	CAMS #	B F.O.M.	^{10}Be/Be ratio		Background		Bkgd-corrected		
			ratio	±	ratio	±	ratio	±	percision
JG 1095	BE16541	0.99	1.272E-13	2.500E-14	9.11E-15	1.63E-15	1.188E-13	2.505E-14	21%
JG 1096	BE16542	0.98	1.362E-13	1.815E-14	9.11E-15	1.63E-15	1.279E-13	1.823E-14	14%
JG 1097	BE16543	1.00	2.215E-13	6.284E-15	9.11E-15	1.63E-15	2.138E-13	6.491E-15	3%
JG 1098	BE16544	0.99	1.033E-13	5.046E-15	9.11E-15	1.63E-15	9.480E-14	5.302E-15	6%
JG 1099	BE16545	0.99	1.024E-13	6.725E-15	9.11E-15	1.63E-15	9.390E-14	6.919E-15	7%
JG 1100	BE16546	0.99	1.042E-13	3.953E-15	9.11E-15	1.63E-15	9.570E-14	4.275E-15	4%
JG 1101	BE16547	0.99	1.063E-13	9.185E-15	9.11E-15	1.63E-15	9.779E-14	9.327E-15	10%
JG 1102	BE16536	1.00	5.538E-13	1.061E-14	9.11E-15	1.63E-15	5.484E-13	1.073E-14	2%
JG 1103	BE16537	1.00	2.176E-13	9.934E-15	9.11E-15	1.63E-15	2.099E-13	1.007E-14	5%
JG 1104	BE16538	0.99	1.146E-13	5.805E-15	9.11E-15	1.63E-15	1.062E-13	6.029E-15	6%
JG 1105	BE16539	1.00	6.114E-13	2.010E-14	9.11E-15	1.63E-15	6.063E-13	2.016E-14	3%
JG 1144	BE16540						No Current		
JG 1145	BE16535	0.96	9.054E-15	1.626E-15			9.115E-15	1.626E-15	18%

1) 10Be/9Be ratios were normalized to a standard (LLNL) with a 10Be/9Be ratio of 3.00E-12 .

2) Unless otherwise indicated, ratios were corrected for spurious counts resulting from boron contamination in the sample.
A Boron Figure of Merit of 1 indicates that there were no spurious counts, an F.O.M. of .95 corresponds to a 5% boron correction, etc.

3) Background-corrected ratios were calculated using the background indicated, based on BE16535.

Appendix 6: Calculated age worksheet

CNEF ID	Sample ID	Sample Density (g/cm3)	Thickness (cm)	Initial Production Rate (atoms/g/yr)	Muonic Contribution (fraction)	Latitude (deg.deg)	Altitude (km)	Site Production Rate (atoms/g/yr)
1095	NS-PEGGY-02-001	2.6	3	5.1	0.02	44.50	0.037	5.023268371
1096	NS-PEGGY-02-002	2.6	3	5.1	0.02	44.50	0.035	5.014584681
1097	NS-PEGGY-02-003	2.6	1.5	5.1	0.02	44.50	0.038	5.027624328
1098	NS-PEGGY-02-004	2.6	3	5.1	0.02	44.50	0.036	5.018926575
1099	NS-PEGGY-02-005	2.6	2	5.1	0.02	44.50	0.037	5.023191503
1100	NS-PEGGY-02-006	2.6	2.3	5.1	0.02	44.50	0.044	5.053666389
1101	NS-PEGGY-02-007	2.6	3	5.1	0.02	44.50	0.054	5.097613135
1144	Blank							
1102	NS-PEGGY-02-008	2.6	1.7	5.1	0.02	44.50	0.038	5.027620002
1103	NS-PEGGY-02-009	2.6	2.4	5.1	0.02	44.50	0.037	5.023194958
1104	NS-PEGGY-02-010	2.6	2.1	5.1	0.02	44.50	0.037	5.023106832
1105	NS-PEGGY-02-011	2.6	2.5	5.1	0.02	44.50	0.054	5.097615808
1145	Blank							

Imperically defined perameters		
attenLn	150	(g/cm2)
attenLmuonair	227	(g/cm2)
Be_decay	4.56018E-07	yr-1

		Adjustments (1 = no effect)						
CNEF ID	Sample ID	Thickness	Horizon	Geom Sloped	Snow	Final Production Rate (atoms/g/yr)	Qtz mass (g)	Carrier-ID
1095	NS-PEGGY-02-001	0.974444868	1	1	1	4.894898087	42.4251	Be3carrier
1096	NS-PEGGY-02-002	0.974444868	1	1	1	4.88643631	41.0031	Be3carrier
1097	NS-PEGGY-02-003	0.987111938	1	1	1	4.962827994	40.016	Be3carrier
1098	NS-PEGGY-02-004	0.974444868	1	1	1	4.890667246	40.1252	Be3carrier
1099	NS-PEGGY-02-005	0.982865239	1	1	1	4.937120317	40.0191	Be3carrier
1100	NS-PEGGY-02-006	0.980328939	1	1	1	4.954255411	40.2198	Be3carrier
1101	NS-PEGGY-02-007	0.974444868	1	1	1	4.967342961	40.0065	Be3carrier
1144	Blank						0	Be3carrier
1102	NS-PEGGY-02-008	0.985410321	1	1	1	4.95426864	40.0282	Be3carrier
1103	NS-PEGGY-02-009	0.979485452	1	1	1	4.920146383	40.0298	Be3carrier
1104	NS-PEGGY-02-010	0.982018832	1	1	1	4.932785503	40.0049	Be3carrier
1105	NS-PEGGY-02-011	0.978642935	1	1	1	4.988745696	40.0012	Be3carrier
1145	Blank						0	Be3carrier

CNEF ID	Sample ID	Concentration-Be (mg/ml)	Density (g/ml)	mass carrier soln (g)	^{9}Be Atoms Added (atoms)	^{10}Be/^{9}Be Corr. for Bckgrnd & Boron	^{10}Be/^{9}Be Error
1095	NS-PEGGY-02-001	1015	1.013	0.502	3.27E+19	1.188E-13	2.505E-14
1096	NS-PEGGY-02-002	1015	1.013	0.5103	3.33E+19	1.279E-13	1.823E-14
1097	NS-PEGGY-02-003	1015	1.013	0.5057	3.30E+19	2.138E-13	6.491E-15
1098	NS-PEGGY-02-004	1015	1.013	0.5059	3.30E+19	9.480E-14	5.302E-15
1099	NS-PEGGY-02-005	1015	1.013	0.5095	3.32E+19	9.390E-14	6.919E-15
1100	NS-PEGGY-02-006	1015	1.013	0.5005	3.26E+19	9.570E-14	4.275E-15
1101	NS-PEGGY-02-007	1015	1.013	0.5006	3.26E+19	9.779E-14	9.327E-15
1144	Blank	1015	1.013	0.529	3.45E+19		
1102	NS-PEGGY-02-008	1015	1.013	0.5034	3.28E+19	5.484E-13	1.073E-14
1103	NS-PEGGY-02-009	1015	1.013	0.5098	3.32E+19	2.099E-13	1.007E-14
1104	NS-PEGGY-02-010	1015	1.013	0.511	3.33E+19	1.062E-13	6.029E-15
1105	NS-PEGGY-02-011	1015	1.013	0.5011	3.27E+19	6.063E-13	2.016E-14
1145	Blank	1015	1.013	0.52	3.39E+19	9.115E-15	1.626E-15

CNEF ID	Sample ID	10Be atoms counted	10Be conc. (atom/g qtz)	Erosion Rate (cm/yr)	Density of Rock (g/cm^3)	Calc Erosion Rate (g/cm^2·yr)	Calc Be Age (yr)	Be Age (kyr)	Error (yr)
1095	NS-PEGGY-02-001	3889493	91679	0	2.6	0	18810	18.8	4.0
1096	NS-PEGGY-02-002	4254445	103759	0	2.6	0	21338	21.3	3.0
1097	NS-PEGGY-02-003	7047673	176121	0	2.6	0	35778	35.8	1.1
1098	NS-PEGGY-02-004	3126644	77922	0	2.6	0	15991	16.0	0.9
1099	NS-PEGGY-02-005	3118870	77935	0	2.6	0	15843	15.8	1.2
1100	NS-PEGGY-02-006	3122605	77639	0	2.6	0	15727	15.7	0.7
1101	NS-PEGGY-02-007	3191491	79774	0	2.6	0	16119	16.1	1.5
1144	Blank								
1102	NS-PEGGY-02-008	17995895	449580	0	2.6	0	92677	92.7	1.8
1103	NS-PEGGY-02-009	6976057	174272	0	2.6	0	35709	35.7	1.7
1104	NS-PEGGY-02-010	3537310	88422	0	2.6	0	17999	18.0	1.0
1105	NS-PEGGY-02-011	19806044	495136	0	2.6	0	101567	101.6	3.4
1145	Blank								