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Article abstract

The Rattling Brook deposit is a low-grade, disseminated to stockwork-style gold deposit hosted by Precambrian granodiorite and adjacent Cambrian sedimentary rocks. Alteration and gold mineralization also occur in foliated and metamorphosed mafic dykes, likely of late Precambrian age. The auriferous granodiorite is in turn cut by relatively fresh, unaltered, and locally chilled diabase dykes, interpreted as Paleozoic post-mineralization intrusions. A fresh post-mineralization diabase gave a $^{40}\text{Ar}/^{39}\text{Ar}$ amphibole plateau age of 412.9 ± 4.3 Ma, which is interpreted as the time of its crystallization, and which provides a younger limit for the timing of gold mineralization. An altered, metamorphosed dyke of pre-mineralization timing gave an identical $^{40}\text{Ar}/^{39}\text{Ar}$ biotite plateau age of 412.6 ± 2.3 Ma, which is more difficult to interpret. It could represent post-metamorphic cooling, or alternatively, resetting of metamorphic biotite during alteration related to gold mineralization. In the first case, the age provides a reasonable upper limit for the timing of gold mineralization, provided that the ambient temperature during mineralization was not significantly above the closure temperature for Ar in biotite (~ 300 oC). On this basis, gold mineralization at Rattling Brook occurred during the latest Silurian or earliest Devonian, between 415 and 409 Ma. The possibility that mineralization occurred at temperatures above 300 oC, prior to 415 Ma, cannot be completely excluded, but it must be younger than ca. 430 Ma, the time of peak metamorphism in adjacent areas. In conjunction with sparse data on the ages of gold deposits elsewhere in Newfoundland, the results support two discrete episodes of mineralization corresponding to the Silurian-Devonian boundary (420–410 Ma) and middle to late Devonian (380–370 Ma). These age groupings resemble those defined by recent Re-Os isotopic studies of sulphides from vein-style gold deposits in the Meguma terrane of Nova Scotia and may in part correspond to the timing of intrusion-related gold in New Brunswick. Given the small amount of data from all of these areas, further interpretation is speculative. However, such hints of discrete orogen-scale episodes of gold mineralization, perhaps correlative with regional tectonothermal events, provide a powerful incentive for further geochronological studies of gold mineralization in the Appalachian orogen.

The timing of gold mineralization in White Bay, western Newfoundland: Evidence from $^{40}\text{Ar}/^{39}\text{Ar}$ studies of mafic dykes that predate and postdate mineralization

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ABSTRACT

The Rattling Brook deposit is a low-grade, disseminated to stockwork-style gold deposit hosted by Precambrian granodiorite and adjacent Cambrian sedimentary rocks. Alteration and gold mineralization also occur in foliated and metamorphosed mafic dykes, likely of late Precambrian age. The auriferous granodiorite is in turn cut by relatively fresh, unaltered, and locally chilled diabase dykes, interpreted as Paleozoic post-mineralization intrusions. A fresh post-mineralization diabase gave a $^{40}\text{Ar}/^{39}\text{Ar}$ amphibole plateau age of 412.9 ± 4.3 Ma, which is interpreted as the time of its crystallization, and which provides a younger limit for the timing of gold mineralization. An altered, metamorphosed dyke of pre-mineralization timing gave an identical $^{40}\text{Ar}/^{39}\text{Ar}$ biotite plateau age of 412.6 ± 2.3 Ma, which is more difficult to interpret. It could represent post-metamorphic cooling, or alternatively, resetting of metamorphic biotite during alteration related to gold mineralization. In the first case, the age provides a reasonable upper limit for the timing of gold mineralization, provided that the ambient temperature during mineralization was not significantly above the closure temperature for Ar in biotite (~ 300 °C). On this basis, gold mineralization at Rattling Brook occurred during the latest Silurian or earliest Devonian, between 415 and 409 Ma. The possibility that mineralization occurred at temperatures above 300 °C, prior to 415 Ma, cannot be completely excluded, but it must be younger than ca. 430 Ma, the time of peak metamorphism in adjacent areas. In conjunction with sparse data on the ages of gold deposits elsewhere in Newfoundland, the results support two discrete episodes of mineralization corresponding to the Silurian-Devonian boundary (420–410 Ma) and middle to late Devonian (380–370 Ma). These age groupings resemble those defined by recent Re-Os isotopic studies of sulphides from vein-style gold deposits in the Meguma terrane of Nova Scotia and may in part correspond to the timing of intrusion-related gold in New Brunswick. Given the small amount of data from all of these areas, further interpretation is speculative. However, such hints of discrete orogen-scale episodes of gold mineralization, perhaps correlative with regional tectonothermal events, provide a powerful incentive for further geochronological studies of gold mineralization in the Appalachian orogen.

RÉSUMÉ

Le gîte du ruisseau Rattling est un gîte aurifère à faible teneur qui, de disséminé, devient un stockwerk inclus dans de la granodiorite précambrienne et des roches sédimentaires cambriennes adjacentes. Une altération et une minéralisation aurifère se manifestent également dans des dykes mafiques métamorphisés et feuilletés, remontant probablement au Précambrien tardif. La granodiorite aurifère est à son tour recoupée par des dykes de diabase figés par endroits, non altérés et relativement sains, interprétés en tant qu'intrusions paléozoïques ultérieures à la minéralisation. Une diabase post-minéralisation inaltérée a accusé un âge plateau sur amphibole $^{40}\text{Ar}/^{39}\text{Ar}$ de $412,9 \pm 4,3$ Ma, ce qui est interprété comme le moment de sa cristallisation et rapproche la limite du moment de la minéralisation aurifère. Un dyke métamorphisé altéré, antérieur à la minéralisation, a livré un âge plateau sur biotite $^{40}\text{Ar}/^{39}\text{Ar}$ identique de $412,6 \pm 2,3$ Ma, qui est plus difficile à interpréter. Celui-ci pourrait représenter un refroidissement post-métamorphique ou, subsidiairement, une remise en place de la biotite métamorphique au cours de l'altération apparentée à la minéralisation aurifère. Dans le premier cas, la datation fournit une limite supérieure raisonnable quant au moment de la minéralisation aurifère, à condition que la température ambiante pendant la minéralisation n'ait pas été substantiellement supérieure à la température de convergence de l'AR dans la biotite (environ 300 °C). Le cas échéant, la minéralisation d'or du ruisseau Rattling est survenue au cours du Silurien tardif ou du Dévonien précoce, entre 415 et 409 Ma. La possibilité que la minéralisation se soit produite à des températures supérieures à 300 °C, avant 415 Ma, ne peut pas être entièrement exclue, mais la minéralisation doit être antérieure à 430 Ma environ, point culminant du métamor-

phisme dans les secteurs voisins. Outre les rares données sur les âges des gîtes d'or ailleurs à Terre-Neuve, les résultats appuient la manifestation de deux épisodes distincts de minéralisation correspondant à la limite siluro-dévonienne (420–410 Ma) et à la période du Dévonien moyen à tardif (380–370 Ma). Ces groupes d'âges ressemblent à ceux définis lors de récentes études isotopiques Re-Os de sulfures provenant de gîtes aurifères filoniens dans le terrane de Meguma en Nouvelle-Écosse et ils pourraient en partie correspondre au moment de l'apparition de l'or lié à une intrusion au Nouveau-Brunswick. Vu la somme restreinte de données tirées de l'ensemble de ces secteurs, effectuer une interprétation plus détaillée serait se livrer à des conjectures. Les indices comme les épisodes distincts de minéralisation aurifère à l'échelle orogénique, qui pourraient être corrélatifs avec des événements tectonothermiques régionaux, fournissent toutefois un puissant incitatif pour réaliser des études chronologiques plus poussées de la minéralisation aurifère dans l'orogène des Appalaches.

[Traduit par la rédaction]

INTRODUCTION

Epigenetic gold mineralization is inherently difficult to date using common geochronological techniques, as mineralized veins do not generally contain datable minerals. However, geological relationships combined with geochronological data locally permit the timing of mineralization to be bracketed precisely. This short paper reports new $^{40}\text{Ar}/^{39}\text{Ar}$ data from both mineralized and unmineralized mafic dykes at the Rattling Brook gold deposit in White Bay, western Newfoundland. These mafic dykes, respectively, predate and postdate gold mineralization, and the results indicate that mineralization is of latest Silurian or earliest Devonian age (415 to 409 Ma). These results add to the small number of precise ages for Paleozoic gold mineralization in the Maritime Appalachians, and substantiate interesting temporal patterns that merit wider discussion.

REGIONAL GEOLOGICAL FRAMEWORK

The study area lies in the Humber Zone of western Newfoundland, within the Appalachian orogen (Fig. 1, inset). The regional geology (Fig. 1) was described by Smyth and Schillereff (1982) and Kerr (2004, 2005). The oldest rocks are gneiss and granite of the Long Range Inlier, formed between ca. 1500 and ca. 980 Ma (Heaman *et al.* 2003). These rocks are cut by the late Precambrian Long Range mafic dykes (ca. 615 Ma; Stukas and Reynolds 1974; Kamo *et al.* 1989). The basement rocks and the dykes are unconformably overlain by Cambrian to Ordovician sedimentary rocks equivalent to the platformal sequence of the Gulf of St. Lawrence area (Kerr and Knight 2004). The basement rocks and their cover sequence are bounded to the west by an important lineament termed the Doucers Valley fault zone (Fig. 1), which remained active into the Carboniferous. The latest motion on this fault zone was dextral, but the earlier history is poorly known. To the east of the fault zone, Cambrian to Ordovician rocks are represented by deep-water sedimentary rocks, mafic volcanic rocks, and trondhjemitic plutonic rocks (Coney Head Complex), collectively termed the Southern White Bay Allochthon (Smyth and Schillereff 1982). Silurian volcanic and sedimentary rocks of the Sops Arm Group occur only to the east of the fault zone. The Sops Arm Group is mostly in fault contact with older rocks,

but an unconformable relationship to trondhjemitic of the Southern White Bay Allochthon is preserved in one location. Silurian and older rocks are intruded by granitoid rocks of the Gull Lake Intrusive Suite (398 ± 27/-7 Ma; Erdmer 1986) and the Devils Room granite (425 ± 10 Ma; Heaman *et al.* 2003). Carboniferous sedimentary rocks unconformably overlie all of the older rocks, including granite of the Gull Lake Intrusive Suite. The area was affected by Paleozoic orogenic episodes of Ordovician age (Taconic Orogeny) and Silurian-Devonian age (Salinic and/or Acadian Orogeny). Much of the present structural architecture reflects the later events, but it is not clear if they represent two discrete episodes in the White Bay area. However, regional folding affected rocks assigned to the latest Silurian (Pridoli Stage) on the basis of paleontology (Lock 1969).

Gold mineralization was first discovered in western White Bay in 1896, and auriferous quartz-carbonate veins of the Browning deposit (Fig. 1) produced briefly around 1904. Similar gold prospects occur elsewhere within sedimentary and volcanic rocks of the Silurian Sops Arm Group (Saunders 1991; Kerr 2006). Disseminated gold mineralization of the Rattling Brook deposit (Fig. 1) was discovered in Precambrian granitoid rocks in the 1980s, and was explored extensively prior to 1990 (Saunders and Tuach 1988, 1991). It is now the focus of a renewed exploration program aimed at both granitoid rocks and adjacent Cambrian to Ordovician sedimentary rocks, based on "Carlin-type" deposit models (Kerr 2004, 2005).

LOCAL GEOLOGY AND GOLD MINERALIZATION

The area around the Rattling Brook gold deposit is shown in Figure 2. Precambrian granitoid rocks outcrop in the west and Cambrian to Ordovician sedimentary rocks outcrop in the east, separated by a steeply east-dipping unconformity. The basal Cambrian strata belong to the Bradore and Forteau formations of the Labrador Group (Kerr and Knight, 2004). These sedimentary rocks are locally strongly deformed, and fault zones (Cobbler Head and Apsy Cove fault zones; Fig. 2), disrupt the sequence, placing Cambrian dolostone (Port au Port Group) or lower Ordovician limestone (St. George Group) against the older Cambrian rocks.

Gold mineralization at the Rattling Brook deposit is present

in four main areas termed the Incinerator Trail, Beaver Dam, Road, and Apsy zones (Fig. 2). The predominant host rock is Precambrian granodiorite, but mineralization also occurs in Cambrian Bradore Formation quartzite and in the basal limestone member of the Forteau Formation (Saunders and Tuach 1991; Kerr 2004, 2005). The granodiorite is cut by two generations of mafic dykes. The older dykes are metamorphosed, variably foliated, locally altered, and gold-bearing, whereas the younger dykes are fresh, unmetamorphosed, unaltered, and unmineralized. In this paper, the terms “metamorphosed dyke” and “fresh dyke” are used to denote the two types. The Cambrian sedimentary rocks do not contain metamorphosed dykes, implying that the latter are of Precambrian age, and likely part of the ca. 615 Ma Long Range dyke swarm. However, fresh diabase dykes cut Cambrian to Lower Ordovician sedimentary rocks in nearby outcrops and coastal exposures (Kerr and Knight 2004), and must be Middle Ordovician or younger. The mafic dykes in the study area have not been studied in detail, but Owen and Machin (1987) reported that metamorphosed Precambrian dykes located some 50 km to the northwest are subalkaline to locally alkaline, and record a polymetamorphic history. They were subjected to early low- and high-pressure events attributed to the Taconic Orogeny, followed by a greenschist-facies overprint.

Gold at Rattling Brook is associated with disseminated and veinlet-style pyrite and arsenopyrite in granodiorite, metamorphosed dykes, quartzite and limestone (Saunders and Tuach 1988, 1991; Kerr 2004, 2005). Alteration linked to mineralization consists of widespread early potassic alteration followed by local silicification and albitization associated with sulphide minerals and gold (Saunders and Tuach 1991; Kerr 2005). The mineralization is extensive, particularly in granodiorite, but the grades are relatively low (generally < 4 ppm Au). Minor Ag is present where Au values are high, but no associated base-metal enrichment occurs (Kerr 2005). The highest gold values are typically in metamorphosed dykes, mafic variants of the granodiorite, or impure carbonate rocks at the Bradore-Forteau formational boundary (Kerr 2004, 2005). The mineralization was initially classed as mesothermal (Saunders and Tuach 1991), but several features suggest similarities to sedimentary-rock hosted “micron” gold deposits (also known as “Carlin-type”) or non-carbonate-hosted disseminated gold mineralization (e.g., Poulsen *et al.* 2000). These features include high Au/Ag ratios, absence of base-metal enrichment, and the presence of associated elements such as As, Sb, Te, W, and Tl. Mineralized carbonate rocks show evidence of decarbonatization and silicification associated with Au enrichment, and mineralized zones contain gold in both arsenian pyrite and arsenopyrite (Kerr 2005; Wilton *et al.* 2007). The disseminated to stockwork-style mineralization at Rattling Brook differs from the vein-hosted gold mineralization in the nearby Sops Arm Group (Fig. 1), where gold is typically associated with Ag, Cu, Pb, and Zn sulphides in discrete quartz and quartz-carbonate veins (Saunders 1991, Kerr 2005). The gold mineralization and related alteration overprints all deformation in the various host

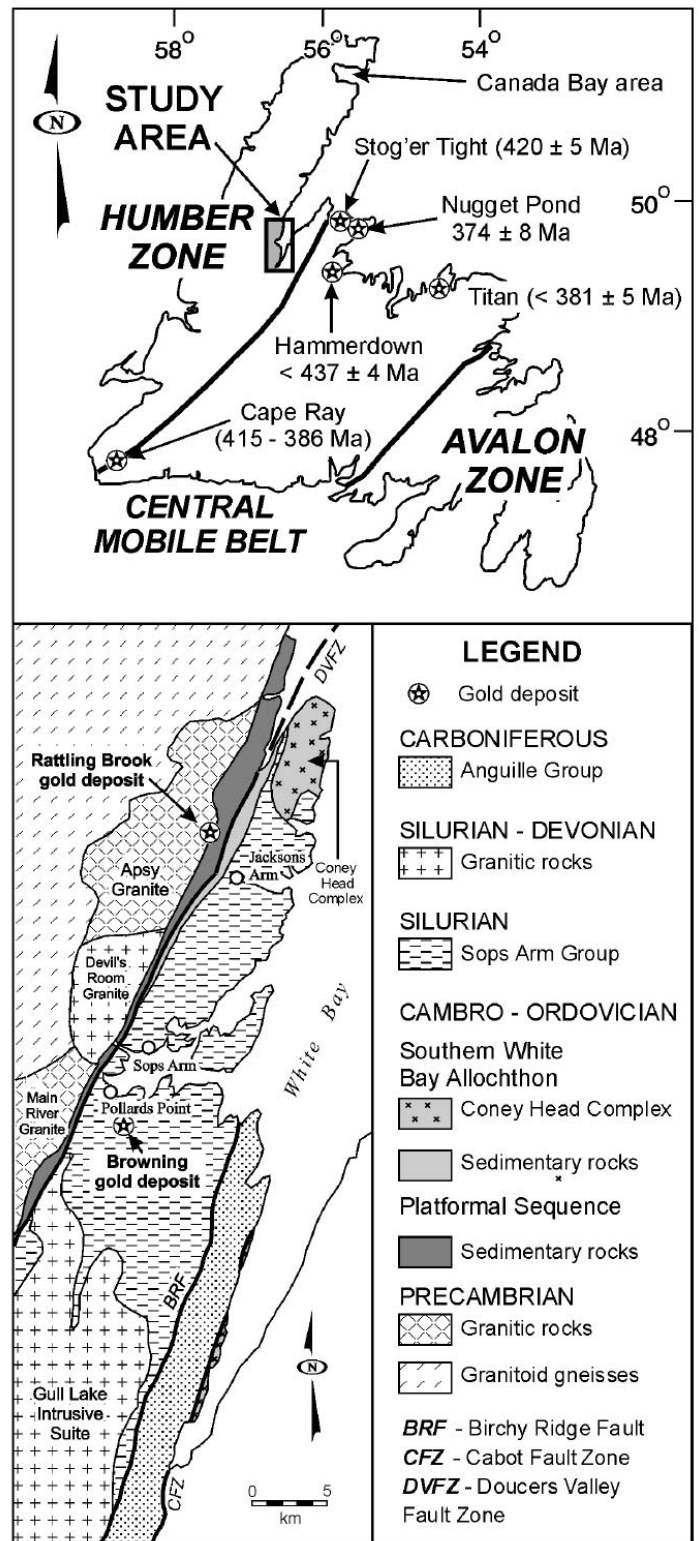


Fig. 1 Location of the study area in western Newfoundland and regional geology of the White Bay area, modified after Kerr (2006) and Smyth and Schillereff (1982). The inset map also shows the locations of other dated Paleozoic gold deposits in Newfoundland.

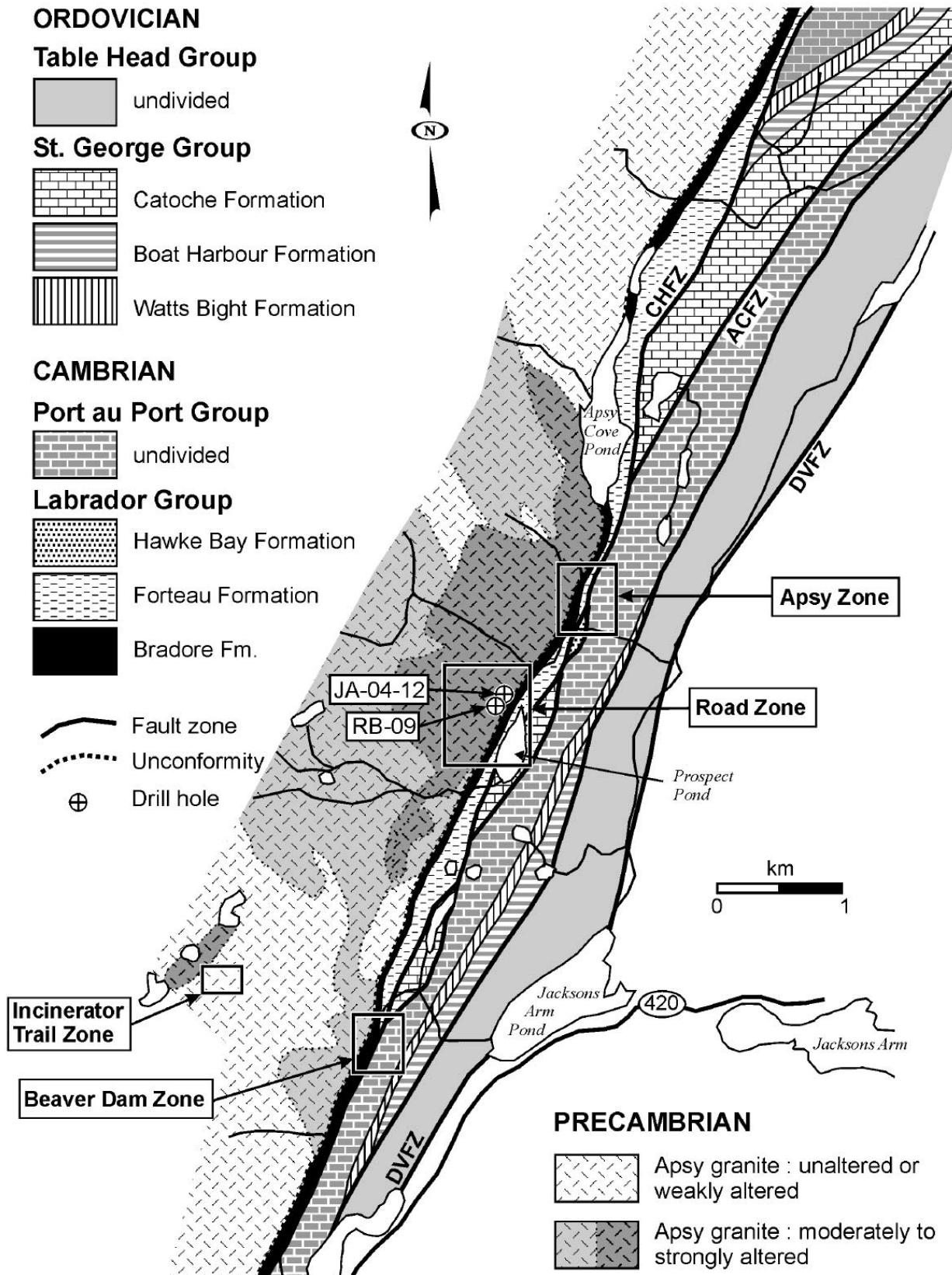


Fig. 2 Geology in the area of the Rattling Brook gold deposit, White Bay, showing the locations of drill holes sampled for this study. Diagram is modified after Kerr *et al.* (2006), with geology based on Kerr and Knight (2004) and work by BP Resources Canada. Abbreviations: DVFZ – Doucers Valley Fault Zone; CHFZ – Cobbler Head Fault Zone; ACFZ – Apsy Cove Fault Zone.

rocks, and the random geometric pattern of sulphide-rich veinlets, notable in granodioritic and quartzitic units, indicates that fluids were introduced in a régime characterized by brittle fracturing and brecciation.

The relationships between gold mineralization, alteration, mafic dyke emplacement, metamorphism, and deformation are very important in the context of this study, and were outlined by Kerr (2005). Mafic dykes that contain gold are foliated and recrystallized, suggesting that their emplacement predated metamorphism and deformation. Sericitic alteration developed along pre-existing foliation planes, but also overprinted the foliation in a pervasive manner, indicating that it postdated deformation. The sericitic zones are in turn overprinted by siliceous alteration zones associated with sulphides and gold enrichment. In contrast, unmineralized diabase dykes are fresh, retain igneous minerals and textures (including plagioclase phenocrysts), and have well-preserved chilled margins. These fresh dykes cut altered gold-bearing granodiorite, but are themselves unaltered and sulphide-free. The geological relationships indicate that the metamorphosed dykes were altered and mineralized following deformation and metamorphism, whereas the fresh dykes postdated the introduction of gold. The presence of high-grade gold mineralization in the metamorphosed dykes compared to the surrounding granodiorite is probably a reflection of their higher iron content; similarly, magnetite-bearing quartzite near the Bradore-Forteau formation boundary also hosts high-grade gold mineralization.

PREVIOUS GEOCHRONOLOGICAL CONSTRAINTS

Tuach and French (1986) recognized that gold mineralization occurred in granodiorite and metamorphosed dykes, but believed it to be absent from adjacent Cambrian-Ordovician sedimentary rocks. On this basis, they suggested that the mineralization was of late Precambrian age. Tuach (1987) suggested that local alteration and disseminated sulphides in the sedimentary rocks were due to local Paleozoic remobilization of Precambrian gold mineralization. The subsequent discovery of more extensive gold mineralization in the quartzite and limestone (McKenzie 1987) led Saunders and Tuach (1988, 1991) to suggest that mineralization was more likely of Silurian-Devonian age.

The porphyritic granodiorite host rock subsequently gave an imprecise U-Pb zircon age (lower intercept) of 1006 ± 82 Ma (Heaman *et al.* 2003). Kerr *et al.* (2006) attempted to date the gold mineralization directly using Re-Os isotopic analysis of pyrite and arsenopyrite. This effort was hampered by very low Re and Os contents in the sulphides and produced imprecise results. However, the Re-Os isochron age of 327 ± 58 Ma for sulphides in altered granodiorite confirms that the mineralization is Paleozoic, and argues against any remobilization of Precambrian gold into the younger sedimentary rocks (Kerr *et al.* 2006). Prior to the present investigation, there were no

attempts at dating the mafic dykes in the area, although Tuach and French (1986) and McKenzie (1987) both recognized that they might provide valuable timing constraints.

⁴⁰AR/³⁹AR GEOCHRONOLOGY

Sample Details

Samples analyzed in this study were collected from diamond drill core recovered from two closely adjacent drill holes at the Road zone (Fig. 2). The drill cores are archived at the Newfoundland Department of Natural Resources core storage facility in Pasadena, Newfoundland.

Sample KC-04-034 was collected from drillhole JA-04-12 (Fig. 2), completed by Kermod Resources Ltd., in 2004. This drillhole intersected a mixture of Precambrian granitoid rocks and fine-grained mafic intervals, both of which are variably altered and mineralized (Kerr 2005). The dated sample was collected at a depth of 34 m, from the centre of a fine-grained mafic interval interpreted as a metamorphosed dyke. Although chilled margins are not visible in the drill core, the same rock type forms dyke-like bodies in surface outcrops, and there is no indication that it was intruded by the surrounding granodiorite. The sample was taken from the least altered portion of the mafic interval, but contains some foliation-parallel sericitic alteration zones. The sampled interval contains only 0.09 ppm Au, but adjacent strongly altered and mineralized material contains up to 6.5 ppm Au.

Sample KC-04-036 was collected from drillhole RB-09 (Fig. 2), completed by BP Resources Canada Ltd., in 1985. This drillhole mostly intersected variably altered and mineralized Precambrian granitoid rocks, but it also encountered three intervals of fresh diabase, which exhibit clear chilled contacts against the granite, and show no signs of mineralization or alteration. The sample was collected at a depth of 62.3 m, and consists of fresh, dark grey-green diabase, with scattered plagioclase phenocrysts. The mineralized granodiorite above the diabase contains up to 2.3 ppm Au. Below the lower contact of the diabase dyke, a grey, siliceous rock type described in company logs as “diabase with green pyritic alteration” contains disseminated pyrite, and seems to grade downward into altered and mineralized granite. This altered material, sampled with the underlying granite, contains 1.4 ppm Au (McKenzie 1986). Petrographic studies show that the “diabase with green pyritic alteration” is actually a strongly recrystallized and altered granitoid rock, probably forming part of a “screen” preserved within the dyke (Kerr *et al.* 2006). The diabase dykes were not assayed by BP Resources as they contained no sulphides; however, subsequent analyses (A. Kerr, unpublished data) show only background levels of gold.

The metamorphosed dyke (KC-04-034) has granoblastic, foliated texture, and consists mostly of biotite, K-feldspar, and quartz, with lesser amounts of epidote and sericite. The biotite-rich (potassic) composition suggests either a parental lamprophyre dyke, or perhaps metasomatic effects that in-

roduced potassium prior to or during metamorphism. The fresh dyke (KC-04-036) retains good primary igneous textures, including plagioclase phenocrysts and oikocrystic mafic minerals. Primary orthopyroxene is accompanied by amphibole, most of which appears to be late magmatic. Fine-grained amphibole may be secondary or deuteric in origin, and the main alteration product of amphibole is chlorite. Both epidote and sericite occur locally in veinlets, but there is no sign of any wide-spread sericitic alteration. Analyzed material from KC-04-034 consisted of biotite-rich aggregates that also contained minor quartz; sericitic material was avoided. Analyzed material from KC-04-036 consisted of poor quality amphibole with opaque inclusions, probably iron oxides.

Results

Corrected argon isotopic data are listed in Table 1, and presented as gas-release spectra in Figures 3 and 4. Each gas-release spectrum plotted contains step-heating data from up to two aliquots, normalized to the total volume of ^{39}Ar released for each aliquot. Such plots provide a visual image of replicated heating profiles, evidence for Ar-loss in the low temperature steps, and the error and apparent age of each step.

The gas-release spectrum for sample KC-04-034 contains step-heating data from two aliquots (A and B). Both aliquots show well-defined multi-step plateaus representing 72% and 74%, respectively, of the total released ^{39}Ar . The combined

Table 1. Isotopic $^{40}\text{Ar}/^{39}\text{Ar}$ data.

Power ^a	Volume ^{39}Ar $\times 10^{-11}$ cc	$^{36}\text{Ar}/^{39}\text{Ar}$	$^{37}\text{Ar}/^{39}\text{Ar}$	$^{38}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}/^{39}\text{Ar}$	% ^{40}Ar ATM	* $^{40}\text{Ar}/^{39}\text{Ar}$	f_{39} ^b (%)	Apparent Age Ma ^c
KC-04-034 Biotite; J=0.00297700^d									
<i>Aliquot: A</i>									
2.8	0.3381	0.0189 ± 0.0102	0.018 ± 0.282	0.088 ± 0.012	49.17 ± 1.25	11.3	43.60 ± 3.25	0.7	220.2 ± 15.5
3.0	0.3933	0.0106 ± 0.0087	0.082 ± 0.243	0.085 ± 0.012	74.14 ± 1.14	4.2	71.00 ± 2.82	0.9	345.9 ± 12.5
3.5	0.8929	0.0039 ± 0.0038	0.261 ± 0.108	0.100 ± 0.011	84.08 ± 0.64	1.4	82.92 ± 1.30	2.0	398.0 ± 5.6
3.9	2.2086	0.0019 ± 0.0015	0.135 ± 0.044	0.111 ± 0.011	86.70 ± 0.77	0.7	86.13 ± 0.89	4.9	411.8 ± 3.8
4.2	2.7999	0.0017 ± 0.0012	0.001 ± 0.034	0.106 ± 0.011	86.52 ± 0.32	0.6	86.01 ± 0.48	6.2	411.3 ± 2.1
4.6	2.3027	0.0024 ± 0.0015	0.004 ± 0.041	0.096 ± 0.011	86.79 ± 0.39	0.8	86.07 ± 0.59	5.1	411.5 ± 2.5
5.5	2.3053	0.0024 ± 0.0014	0.028 ± 0.033	0.124 ± 0.011	87.07 ± 0.31	0.8	86.37 ± 0.52	5.1	412.8 ± 2.2
5.0	1.6226	0.0013 ± 0.0019	0.009 ± 0.054	0.101 ± 0.011	86.13 ± 0.38	0.4	85.74 ± 0.69	3.6	410.1 ± 3.0
6.0	3.3796	0.0011 ± 0.0009	0.004 ± 0.026	0.130 ± 0.011	86.78 ± 0.30	0.4	86.46 ± 0.41	7.4	413.2 ± 1.7
6.5	3.4826	0.0018 ± 0.0009	0.012 ± 0.025	0.130 ± 0.011	87.04 ± 0.31	0.6	86.50 ± 0.41	7.7	413.4 ± 1.8
7.0	3.6311	0.0019 ± 0.0010	0.010 ± 0.025	0.126 ± 0.011	87.44 ± 0.40	0.6	86.90 ± 0.51	8.0	415.1 ± 2.2
13.0	3.0546	0.0022 ± 0.0013	0.075 ± 0.030	0.122 ± 0.011	86.78 ± 1.46	0.8	86.12 ± 1.50	6.7	411.7 ± 6.4
<i>Aliquot: B</i>									
2.8	0.3389	0.0308 ± 0.0087	0.151 ± 0.280	0.113 ± 0.012	69.83 ± 1.15	13	60.730 ± 2.80	0.8	299.8 ± 12.7
3.0	0.7314	0.0048 ± 0.0039	0.068 ± 0.130	0.098 ± 0.011	75.44 ± 0.61	1.9	74.02 ± 1.30	1.6	359.3 ± 5.7
3.5	1.4586	0.0029 ± 0.0020	0.042 ± 0.065	0.101 ± 0.011	83.70 ± 0.38	1.0	82.84 ± 0.71	3.2	397.7 ± 3.0
3.9	1.0473	0.0074 ± 0.0028	0.132 ± 0.091	0.097 ± 0.011	86.78 ± 0.50	2.5	84.61 ± 0.95	2.3	405.3 ± 4.1
4.2	2.0093	0.0018 ± 0.0014	0.087 ± 0.047	0.098 ± 0.011	86.72 ± 0.81	0.6	86.19 ± 0.91	4.4	412.0 ± 3.9
4.6	1.6503	0.0018 ± 0.0017	0.082 ± 0.058	0.094 ± 0.011	86.59 ± 0.61	0.6	86.05 ± 0.79	3.6	411.5 ± 3.4
5.0	1.628	0.0018 ± 0.0018	0.102 ± 0.058	0.115 ± 0.011	86.66 ± 0.47	0.6	86.12 ± 0.70	3.6	411.8 ± 3.0
5.5	2.9118	0.0014 ± 0.0010	0.043 ± 0.033	0.122 ± 0.011	86.64 ± 0.28	0.5	86.24 ± 0.41	6.4	412.2 ± 1.8
6.0	1.9395	0.0006 ± 0.0015	0.050 ± 0.051	0.121 ± 0.011	86.65 ± 0.39	0.2	86.46 ± 0.59	4.3	413.2 ± 2.5
6.5	1.708	0.0014 ± 0.0017	0.084 ± 0.056	0.120 ± 0.011	86.74 ± 0.55	0.5	86.34 ± 0.74	3.8	412.7 ± 3.2
7.0	2.5298	0.0011 ± 0.0011	0.040 ± 0.038	0.111 ± 0.011	87.020 ± 1.268	0.4	86.699 ± 1.307	5.6	414.2 ± 5.6
13.0	1.1531	0.0026 ± 0.0025	0.461 ± 0.084	0.100 ± 0.011	86.51 ± 0.77	0.9	85.73 ± 1.06	2.5	410.1 ± 4.5
KC-04-036 Amphibole; J=0.00297340									
<i>Aliquot: A</i>									
3.0	0.0405	0.7047 ± 0.0495	67.225 ± 3.298	0.417 ± 0.044	286.01 ± 9.95	72.8	77.78 ± 16.56	2.3	375.3 ± 72.1
3.9	0.12	0.2397 ± 0.0152	53.223 ± 1.584	0.185 ± 0.018	156.95 ± 4.66	45.1	86.11 ± 6.03	6.8	411.3 ± 25.8
4.6	0.7023	0.0154 ± 0.0025	7.034 ± 0.194	0.033 ± 0.011	91.04 ± 0.87	5	86.49 ± 1.12	39.5	412.9 ± 4.8
5.0	0.4703	0.0175 ± 0.0037	11.750 ± 0.310	0.123 ± 0.012	91.67 ± 1.22	5.6	86.50 ± 1.62	26.5	412.9 ± 6.9
6.0	0.0908	0.0313 ± 0.0158	21.804 ± 1.278	0.226 ± 0.025	95.31 ± 4.43	9.7	86.05 ± 6.35	5.1	411.0 ± 27.1
6.5	0.0281	0.0557 ± 0.0946	37.844 ± 3.977	0.451 ± 0.050	109.34 ± 10.13	15.1	92.88 ± 29.64	1.6	439.9 ± 124.7
7.5	0.0537	0.0348 ± 0.0500	37.821 ± 2.372	0.474 ± 0.037	102.79 ± 5.62	10	92.50 ± 15.77	3	438.4 ± 66.4
13.0	0.2714	0.0137 ± 0.0107	15.424 ± 0.509	0.175 ± 0.013	90.76 ± 1.41	4.5	86.71 ± 3.44	15.3	413.8 ± 14.7

Notes: ^a As measured by laser in % of full nominal power (10W). ^b Fraction ^{39}Ar as percent of total run. ^c Errors are analytical only and do not reflect error in irradiation parameter J. ^d Nominal J, referenced to FCT-San = 28.03 Ma (Renne et al., 1994). All uncertainties quoted at 2sigma level.

weighted average $^{40}\text{Ar}/^{39}\text{Ar}$ age for 9 of 12 Ar release fractions from aliquot A and 8 of 12 from aliquot B is 412.6 ± 2.3 Ma, corresponding to a mean square of weighted deviates (MSWD) of 0.91. The Ar release fractions from the lowest power steps for each aliquot were excluded, as their younger apparent ages suggest Ar loss.

For amphibole from sample KC-04-036, an age plateau for 7 power steps represents 98% of the total argon released. The weighted average age for 5 of the 8 Ar release fractions (excluding the lowest power step, and fractions for power steps 6.5 and 7.5, which have large uncertainties) is 412.9 ± 4.3 Ma, corresponding to a MSWD of 0.013. An identical age is obtained

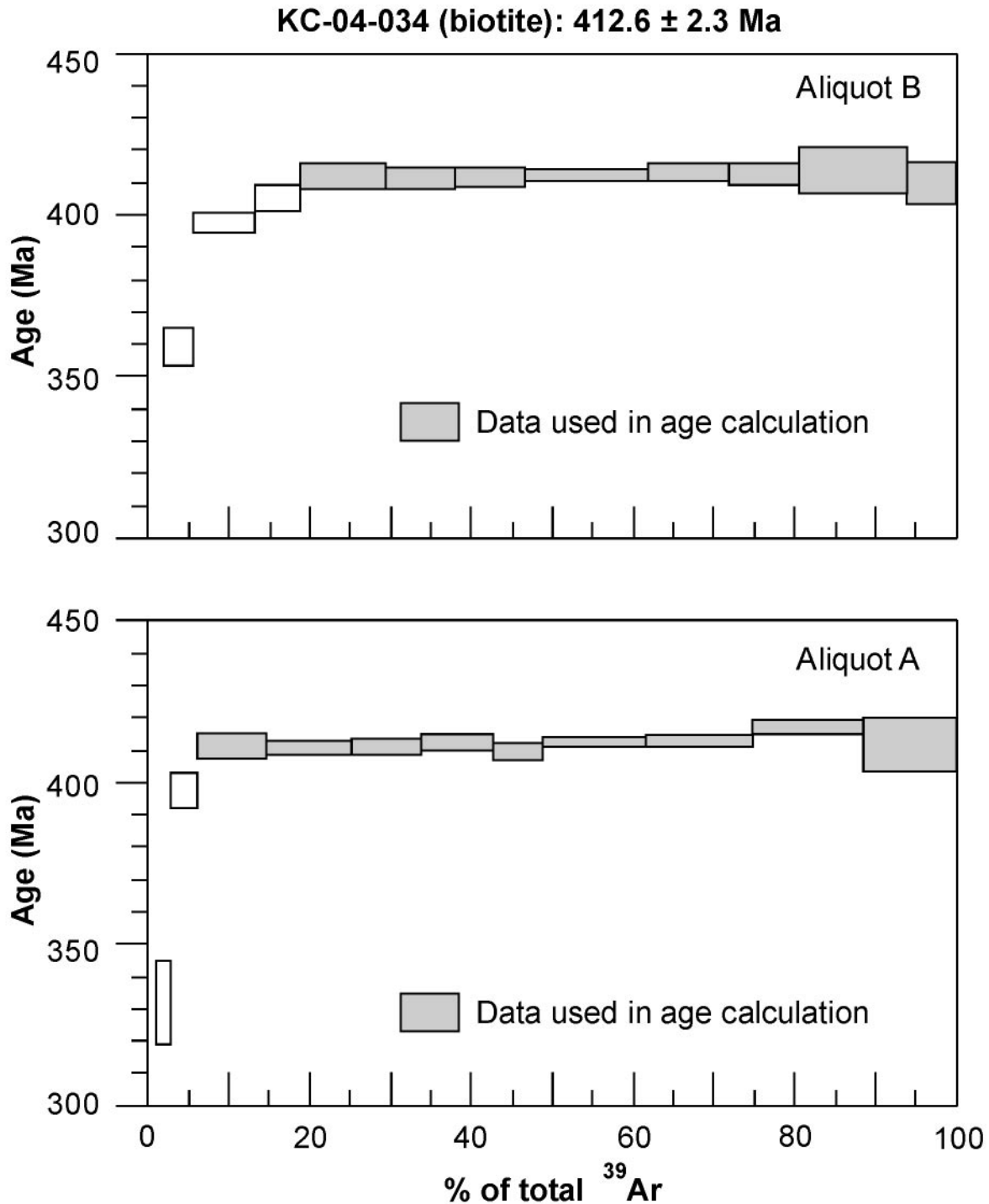


Fig. 3 Gas-release spectrum diagram showing $^{40}\text{Ar}/^{39}\text{Ar}$ ages plotted against percentage of total ^{39}Ar gas released during step heating of biotite separates from the pre-mineralization metamorphosed dyke (sample KC-04-034).

using a regression analysis for an isotopic correlation diagram (not shown). Excluding those steps having large uncertainties, the limited variation in Ca/K ratios (Fig. 4) suggests that the analyzed material is homogeneous.

The results demonstrate that the foliated, altered, metamorphosed, gold-bearing dyke (KC-04-034) and the fresh, unaltered, unmineralized diabase dyke (KC-04-036) have identical apparent ages within their respective errors. Accounting for errors, the apparent ages lie between 417 and 409 Ma, and correspond to latest Silurian or earliest Devonian time, using the current geological time scale (International Commission on Stratigraphy; Gradstein *et al.* 2004).

DISCUSSION

Interpretation of Ages

Geological relationships in surface outcrops and drill core indicate that the metamorphosed dyke was emplaced and metamorphosed prior to alteration and gold mineralization,

and that the fresh diabase was emplaced following alteration and mineralization. As the best gold grades at the Rattling Brook prospect typically occur in altered mafic rocks, the lack of alteration and mineralization in a dyke that has mineralized wall rocks is unlikely to be coincidental. However, as the closure temperatures for Ar in biotite and hornblende differ significantly, the identical apparent $^{40}\text{Ar}/^{39}\text{Ar}$ ages do not *necessarily* demonstrate simultaneous crystallization of the two mafic dykes, as discussed below.

The closure temperature for Ar in hornblende is around 550 °C (McDougall and Harrison 1999). Much of the hornblende in the post-mineralization diabase (KC-04-036) appears to be late-magmatic, and the fine grain size suggests rapid cooling and solidification. There is certainly no indication that this sample was subjected to high temperatures during some later event. The hornblende age for KC-04-036 is thus attributed to the time of crystallization, and provides a minimum age for hydrothermal alteration and gold mineralization in its granodioritic host rocks.

The closure temperature of Ar in biotite is typically around 300 °C, although it can be significantly higher (McDougall and

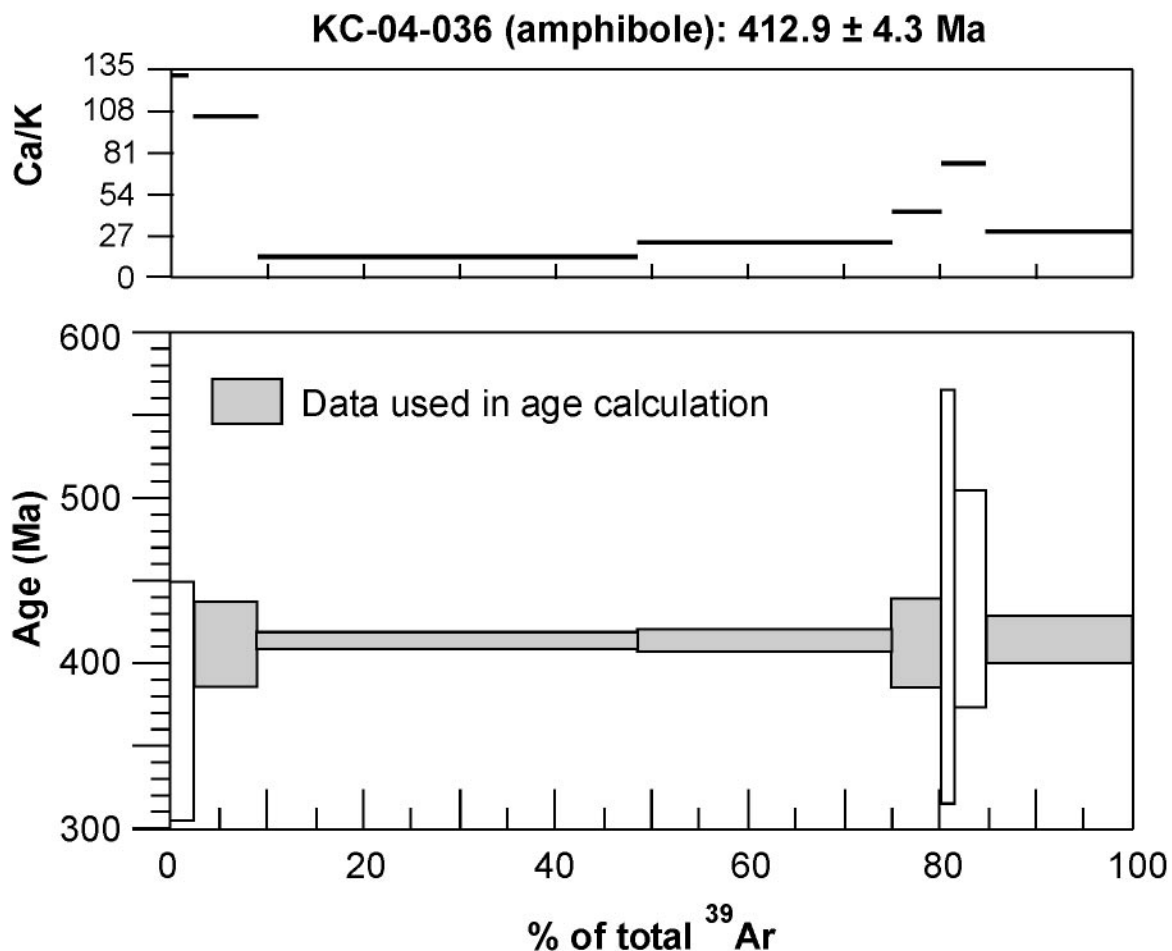


Fig. 4 Gas-release spectrum diagram showing $^{40}\text{Ar}/^{39}\text{Ar}$ ages plotted against percentage of total ^{39}Ar gas released during step heating of amphibole separates from the post-mineralization diabase (sample KC-04-036).

Harrison 1999). It is therefore unlikely that the $^{40}\text{Ar}/^{39}\text{Ar}$ age from the metamorphosed dyke (KC-04-034) actually records the time of its emplacement and crystallization, which is suspected to be late Precambrian. The result must instead be a younger cooling age linked to Paleozoic metamorphism. As the upper greenschist- to lowest amphibolite- facies metamorphic mineral assemblage reflects temperatures slightly above the closure temperature for biotite, the timing of peak metamorphism must be prior to 412.6 ± 2.3 Ma; data from other studies (see below) suggest that metamorphism occurred no earlier than 430 Ma.

The combined age data can be interpreted in two ways (Fig. 5). The first interpretation is that the age from the metamorphosed dyke records cooling through $\sim 300^\circ\text{C}$ soon after regional metamorphism. Cooling was quickly followed by alteration and mineralization, and then by emplacement of the fresh post-mineralization dyke. In this scenario, regional metamorphism occurred prior to 412.3 ± 2.3 Ma (Fig. 5a), but the *relative* timing of cooling through $\sim 300^\circ\text{C}$ and gold mineralization is less clear, as the latter could have occurred above the closure temperature for Ar isotopic systems in biotite (see discussion below). The second interpretation is that the age from the metamorphosed dyke actually records *resetting* of metamorphic biotite during alteration and gold mineralization, which was then followed quickly by emplacement of the fresh post-mineralization dyke. If the first interpretation is correct, the maximum age limit for mineralization could be greater than 412.6 ± 2.3 Ma, if the ambient temperature during the mineralization event was significantly above 300°C (Fig. 5a). If the second interpretation is correct, the average age of 412.6 ± 2.3 Ma from the metamorphosed dyke directly records the timing of mineralization, during which ambient temperatures must have exceeded 300°C (Fig. 5b). The temperature régime during gold mineralization is not directly constrained by fluid-inclusion studies, but Saunders and Tuach (1991) proposed 300 to 350°C , based on alteration types, and suggest 400°C as an upper limit, based on the presence of secondary microcline in altered granitoid rocks. This temperature estimate is in accordance with the inferred temperature range for most “mesothermal” gold mineralization, i.e., 250 to 400°C (e.g., Groves *et al.* 1998; Poulsen *et al.* 2000). Kerr (2005) presented evidence that mineralization at Rattling Brook has closer affinities to other gold deposit types that likely form at temperatures significantly below 300°C . From an observational perspective, the brittle fracturing of quartzitic rock types to accommodate mineralized veinlets corroborates this view. On the basis of these data, it seems unlikely that the ambient temperature during mineralization was higher than the closure temperature for Ar isotopic systems in biotite, and the first interpretation (Fig. 5a) is preferred. The identical ages from the metamorphosed dyke and the fresh, post-mineralization diabase are thus interpreted to closely reflect the timing of gold mineralization at the Rattling Brook deposit, and suggest that it formed in earliest Devonian times, between 415 Ma and 409 Ma. The age range is based on the slightly smaller errors for the pre-mineralization metamorphosed dyke, whereas use of

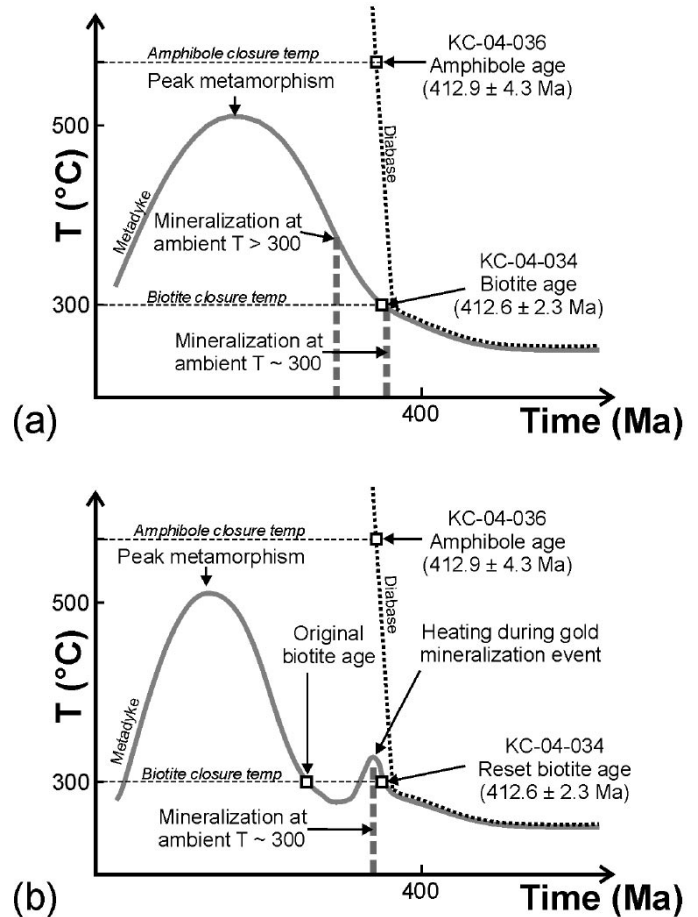


Fig. 5 Schematic illustrations of possible cooling paths for the pre-mineralization metamorphosed dyke and the post-mineralization diabase, with respect to the timing and temperature regime of gold mineralization. (a) Interpretation 1, in which the metamorphosed dyke cools following regional metamorphism. If the temperature regime during mineralization is below the blocking temperature for biotite, the age from the metamorphosed dyke provides an upper limit for mineralization. If the temperature regime during mineralization exceeds the blocking temperature for biotite, the metamorphosed dyke could actually be younger than the mineralization. (b) Interpretation 2, in which the metamorphosed dyke cools following metamorphism but is then reheated during gold mineralization, and its biotite age is reset in a thermal regime that exceeds the blocking temperature for biotite. See text for further discussion.

the less precise age from the post-mineralization dyke would suggest 417 Ma as the older limit.

However, the possibility that mineralization occurred prior to this time, at temperatures above 300°C , cannot be completely excluded without direct constraints on the thermal régime. In this context, it is important to note that U-Pb and ^{40}Ar - ^{39}Ar isotopic data from adjacent areas (see discussion below) suggest that regional metamorphism occurred ca. 430 Ma

ago, and this provides an *absolute* older limit for the timing of gold mineralization, even if our assumptions concerning the temperature of mineralization ultimately prove invalid.

Local Correlations

Several conclusions relevant to local geology may be drawn from the ⁴⁰Ar/³⁹Ar ages determined in this study (Fig. 6). The biotite cooling age for the metamorphosed dyke suggests that

peak metamorphism occurred during the Silurian. This timing is consistent with inferences from U-Pb and ⁴⁰Ar/³⁹Ar geochronological studies on the Baie Verte Peninsula and the Corner Brook area, which indicate peak metamorphism (amphibolite facies) at ca. 430 Ma, and cooling through 500 °C at ca. 430 to 420 Ma. (Cawood and Dunning 1993; Cawood *et al.* 1994). Slightly younger biotite cooling ages akin to those determined for the metamorphosed dyke would thus be expected, and Cawood *et al.* (1994) report a similar muscovite cooling age of

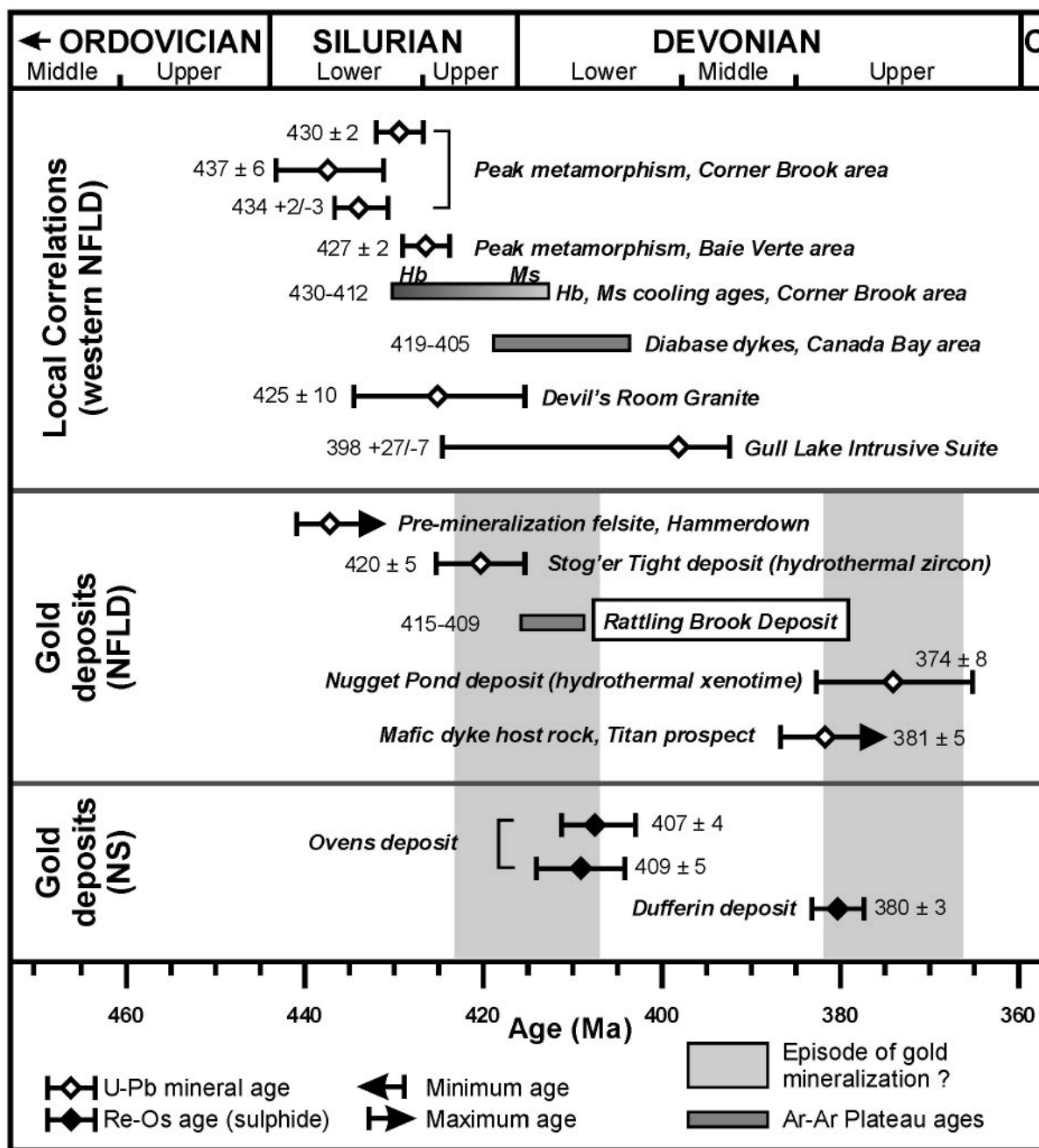


Fig. 6 Summary chart showing the results of ⁴⁰Ar/³⁹Ar dating at the Rattling Brook deposit and its relationship to geochronological constraints on the timing of metamorphism and plutonism in the study area, and ages of other Paleozoic gold deposits in Newfoundland and Nova Scotia.

413 ± 3 Ma. The result is consistent with the importance of the mid-Silurian Salinic Orogeny throughout the Newfoundland Appalachians (e.g., Dunning *et al.*, 1990).

The fresh, post-mineralization diabase dyke may be part of a more extensive earliest Devonian dyke swarm in western Newfoundland. Kerr and Knight (2004) reported that diabase dykes cut all of the Cambrian-Ordovician formations in the White Bay area, and they were emplaced after the rocks were folded, into zones of local dextral displacement. Similar mafic dykes occur in the Canada Bay area, north of the study area (Fig. 1, inset), where they cut time-equivalent Cambrian and lower Ordovician carbonate strata (Knight 1987). $^{40}\text{Ar}/^{39}\text{Ar}$ whole-rock plateau ages from several dykes in the Canada Bay area range from 419 ± 3 Ma to 405 ± 4 Ma (I. Knight and D. Lux, unpublished data), overlapping the age determined in this study.

Previous workers (e.g., Saunders and Tuach 1991) suggested a link between gold mineralization at Rattling Brook and local plutonic rocks. The age for gold mineralization suggested here is within the wide error envelopes of the Devils Room granite (425 ± 10 Ma; Heaman *et al.* 2003) and the Gull Lake Intrusive Suite (398 + 27 / -7 Ma; Erdmer 1986). A genetic link to these plutonic rocks thus remains possible, but it is far from proven. There is a striking absence of minor felsic intrusions in the area of the Rattling Brook gold deposit, aside from a narrow (< 3 m) felsic dyke in hole RB-12 that cuts mineralized granodiorite and appears to be of post-mineralization timing (McKenzie 1986).

Auriferous quartz-carbonate vein systems of more typical mesothermal aspect (e.g., the Browning deposit) are hosted by Silurian volcanic and sedimentary rocks of the Sops Arm Group (Saunders 1991; Kerr 2006; Fig. 1). Thin carbonate units in the host rocks contain mid-Silurian faunas, and the youngest sedimentary rocks in the Sops Arm Group contain uppermost Silurian (Pridoli Stage) faunas (Lock 1969). At face value, the latest Silurian – earliest Devonian gold mineralization event identified at Rattling Brook could also be responsible for the vein-style gold mineralization in the Silurian rocks. However, as discussed above, the characteristics and geochemical associations of the latter differ from those of Rattling Brook, and it remains possible that they are part of a discrete, younger gold mineralizing event (see discussion below).

Regional Correlations

Paleozoic epigenetic gold mineralization in Newfoundland occurs in rocks of Precambrian to Silurian age, but the most abundant host rocks are Cambrian and Ordovician. Most gold deposits in Newfoundland occur within the Dunnage Zone of the Central Mobile Belt (Fig. 1, inset). As discussed in the introduction, the difficulty of dating such deposits means that few reliable age constraints exist for comparative purposes (Fig. 6). At the Hammerdown gold deposit near Springdale (Fig. 1, inset) auriferous veins cut felsite dykes that share their structural trend. A U-Pb zircon age of 437 ± 4 Ma from the felsite provides an older limit for the timing of mineralization,

which is inferred to be Upper Silurian (Ritcey *et al.* 1995). At the Stog'er Tight gold deposit on the Baie Verte Peninsula (Fig. 1, inset), Ramezani *et al.* (2002) obtained a U-Pb zircon age of 420 ± 5 Ma from “hydrothermal” zircon in skarn-like, gold-bearing alteration. In contrast, a U-Pb xenotime age of 374 ± 8 Ma for pegmatitic quartz-feldspar-carbonate alteration associated with gold at the nearby Nugget Pond deposit (Fig. 1, inset) indicates late Devonian mineralization (Sangster and Pollard 2001; Sangster *et al.* 2007). More recently, McNicoll *et al.* (2006) obtained a U-Pb zircon (SHRIMP) age of 381 ± 5 Ma from a mafic dyke that hosts gold-bearing veins at the Titan Prospect, north of Gander (Fig. 1, inset). This age provides an older limit for the timing of mineralization, which must be post-Middle Devonian. Other constraints on the timing of gold mineralization are less definitive. For example, gold mineralization at the Cape Ray deposit in southwestern Newfoundland (Fig. 1, inset) is constrained only between 415 and 386 Ma (Dubé and Lauzière 1997).

Although the available dataset (Fig. 6) leaves much to be desired, it hints at two discrete periods of gold mineralization, one corresponding to the Silurian-Devonian boundary and the other to the middle to late Devonian. The data reported in this paper support this overall pattern, and suggest that the Rattling Brook deposit belongs to the earlier of these two mineralization events.

Outside Newfoundland, few data exist that reliably constrain the timing of gold mineralization in the northeastern Appalachian Orogen, but some information is available from New Brunswick and Nova Scotia.

New Brunswick is host to diverse styles of gold mineralization but information on the ages of the mineralized veins is mostly indirect. The best-known deposit is the Clarence Stream deposit, which is hosted by early Silurian sedimentary rocks of the Gander Zone. Mineralization here is classified as “intrusion-related”, and interpreted to be linked to the nearby Magaguadavic Granite, which gave a U-Pb zircon age of 396 ± 1 Ma (Bevier 1990). Muscovite from auriferous quartz veins yielded $^{40}\text{Ar}/^{39}\text{Ar}$ ages of ca. 389 Ma (Davis *et al.* 2004), and an upper limit is provided by associated and locally mineralized aplitic dykes that yielded a U-Pb monazite (microprobe) age of 390 ± 8 Ma (Thorne *et al.* 2002). The Lake George deposit, also within the Gander Zone of New Brunswick, is best known for its antimony mineralization, but also contains significant gold enrichment associated with scheelite and molybdenite (Lentz *et al.* 2002; Yang *et al.* 2003). Muscovite from auriferous veins gave $^{40}\text{Ar}/^{39}\text{Ar}$ ages of 411 ± 8 Ma and 413 ± 8 Ma, and Sb-bearing veins gave a $^{40}\text{Ar}/^{39}\text{Ar}$ muscovite age of 417 ± 8 Ma (Seal II *et al.* 1988). The Lake George Granite, believed to be genetically linked to the mineralization, gave a U-Pb age of 412 ± 2 Ma. The inferred age for this deposit is thus similar to that presented here from Rattling Brook, despite their markedly different metallogenic associations.

In Nova Scotia, a recent study by Morelli *et al.* (2005) in the Meguma terrane involved direct dating of sulphide minerals from two important gold deposits (Kontak *et al.* 1990). Two Re-Os arsenopyrite ages from the Ovens deposit gave identi-

cal results of 407 ± 4 Ma and 409 ± 5 Ma, whereas equivalent data from the Dufferin deposit indicate a significantly younger age of 380 ± 3 Ma (Morelli *et al.* 2005). Previous studies using $^{40}\text{Ar}/^{39}\text{Ar}$ methods on muscovite in the wall-rocks to veins had indicated a younger age of ca. 370 Ma, suggesting that there may be problems with the latter approach.

The discrete periods of gold mineralization indicated by the data from the Meguma terrane, and perhaps also New Brunswick, correspond well with those defined by this study and other information from Newfoundland summarized above (Fig. 6). Given the great distance between the areas in question, varied geological settings and the fact that they are separated by major faults representing terrane boundaries, the coincidence of this temporal pattern is intriguing. In terms of regional orogenic events recognized through the northern Appalachians, these periods of gold mineralization broadly correspond with definitions of the Acadian and so-called “Neoacadian” orogenies, rather than to the Silurian Salinic Orogeny (Robinson *et al.* 1998; van Staal 2007). Such correspondence raises the possibility that there may be orogen-scale mineralizing episodes linked to these late-stage thermotectonic events, as suggested by the distribution of so-called orogenic gold deposits through geological time (e.g., Goldfarb *et al.* 2001).

CONCLUSIONS

$^{40}\text{Ar}/^{39}\text{Ar}$ ages from altered, metamorphosed mafic dykes that predate mineralization and fresh diabase dykes that postdate mineralization provide a viable constraint on the timing of mineralization at the Rattling Brook gold deposit in western Newfoundland. These pre-mineralization and post-mineralization mafic dykes yield essentially identical $^{40}\text{Ar}/^{39}\text{Ar}$ ages of 412.6 ± 2.3 Ma (biotite) and 413 ± 4.3 Ma (hornblende), respectively. Interpretation is complicated by the differing closure temperatures of these minerals, but they are considered to closely bracket the timing of mineralization at 415 to 409 Ma, unless the ambient temperatures during mineralization were > 300 °C, which is considered unlikely. The possibility that mineralization is earlier than 415 Ma cannot be completely excluded, but it cannot be earlier than ca. 430 Ma, based on regional constraints.

In conjunction with U-Pb ages from other gold deposits in Newfoundland, these new data support the existence of two discrete mineralizing events, one at the Silurian-Devonian boundary and the other in the late Devonian. Interestingly, closely similar age groupings are indicated by recent Re-Os geochronological studies of gold deposits in the Meguma terrane of Nova Scotia, and may also be evident in $^{40}\text{Ar}/^{39}\text{Ar}$ and U-Pb data pertaining to mineralization in New Brunswick. It remains to be seen if this intriguing pattern will prove enduring, but it certainly provides an incentive for the acquisition of other data that might constrain temporal relationships between deformation, metamorphism, and gold mineralization elsewhere in Newfoundland and correlative regions of the Appalachian orogen.

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APPENDIX

Sample Processing and Analytical Techniques

Selected samples were crushed to granules in the range 0.25 to 0.50 mm. Several grains were loaded into aluminum foil packets along with a single grain of Fish Canyon Tuff Sanidine (FCT-SAN) to act as flux monitor (apparent age = 28.03 Ma; Renne *et al.* 1998). The sample packets were arranged radially inside an aluminum can. The samples were then irradiated for 12 hours at the research reactor of McMaster University in a fast neutron flux of approximately 3×10^{16} neutrons/cm². Laser $^{40}\text{Ar}/^{39}\text{Ar}$ step-heating analysis was carried out at the Geological Survey of Canada laboratories in Ottawa, Ontario. Upon return from the reactor, samples were split into several aliquots and loaded into individual 1.5 mm-diameter holes in a copper planchet. The planchet was then placed in the extraction line and the system evacuated. Heating of individual sample aliquots in steps of increasing temperature was achieved using a Merchantek MIR10 10W CO₂ laser equipped with a 2 mm x 2 mm flat-field lens. Heating was continued until the grains disintegrated or fused, releasing all gas. The released Ar gas was cleaned over getters for ten minutes, and then analyzed isotopically using the secondary electron multiplier system of a VG3600 gas source mass spectrometer. Details of data collection protocols can be found in Villeneuve and MacIntyre (1997) and Villeneuve *et al.* (2000). Error analysis on individual steps

follows numerical error analysis routines outlined in Scaillet (2000); error analysis on grouped data follows algebraic methods of Roddick (1988).

Neutron flux gradients throughout the sample canister were evaluated by analyzing the sanidine flux monitors included with each sample packet. A linear fit was interpolated against calculated J-factor and sample position. The error on individual J-factor values is conservatively estimated at $\pm 0.6\%$ (2 sigma).

Because the error associated with the J-factor is systematic and not related to individual analyses, correction for this uncertainty is not applied until calculation of dates from isotopic correlation diagrams (Roddick, 1988). The well-defined plateaus (Figs. 3 and 4) from different minerals indicate that excess ^{40}Ar was not present in the samples and all regressions are assumed to pass through the $^{40}\text{Ar}/^{36}\text{Ar}$ value for atmospheric air (295.5). All errors are quoted at the 2 sigma level of uncertainty.

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