Zircons from a Pegmatite Cutting Eclogite (Gridino, Belomorian Mobile Belt): U-Pb-O and Trace Element Constraints on Eclogite Metamorphism and Fluid Activity

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Received: 15 April 2020; Accepted: 19 May 2020; Published: 21 May 2020

Abstract: This report presents new data on U-Pb geochronology, oxygen isotopes, and trace element composition of zircon from a pegmatite vein crosscutting an eclogite boudin on Stolbikha Island, Gridino area, Belomorian mobile belt (BMB). The zircon grains occur as two distinct populations. The predominant population is pegmatitic and shows dark cathodoluminescence (CL); about a third of this population contains inherited cores. The second zircon population is typical of granulite and exhibits a well-defined sectorial (mosaic) zoning in CL. Both the inherited cores and sectorial in CL zircons appear to have been captured from metabasites as xenocrysts during the pegmatite vein formation. A U-Pb age of 1890 ± 2 Ma for the main zircon population is interpreted as the age of the pegmatite injection. This value is close to the age threshold for the BMB eclogites (~1.9 Ga) and unambiguously defines the upper age limit for the eclogite metamorphism. The pegmatite formation is thus related to partial melting events that occurred during the retrograde amphibolite-facies metamorphism shortly after the eclogitization. A U-Pb date of 2743 ± 10 Ma obtained for the sectorial in CL zircons is considered as the age of the granulite-facies metamorphism established previously within the BMB. The values of δ18O in the zircon populations overlap in a broad range, i.e., δ18O in the pegmatitic zircons varies from 6.1‰ to 8.3‰, inherited cores show a generally higher δ18O of 6.7–8.8‰, and in the captured granulitic zircons δ18O is 6.2–7.9‰. As a result of fluid attack during the final stage of the pegmatite vein formation, the composition of the pegmatitic zircons in terms of non-formula elements (REE, Y, Ca, Sr, Ti) has become anomalous, with the content of these elements having been increased by more than tenfold in the alteration zones. Our data provide new constraints on the timing of eclogite metamorphism within the BMB and show that the late-stage pegmatite-related fluids exerted a very pronounced influence on trace element abundances in zircon, yet had no significant impact on the isotopic composition of oxygen.

Keywords: zircon; trace elements; rare earth elements; U-Pb zircon geochronology; eclogites; pegmatites; Gridino; Belomorian mobile belt
1. Introduction

The age of eclogite metamorphism of the Belomorian mobile belt (BMB) has recently turned out to be an urgent issue not only for this particular region, but for Precambrian geology in general. The U-Pb geochronology of zircon from BMB eclogitized basites provided Archean age estimates of 2.7–2.8 Ga, which were interpreted as the age of eclogite-facies metamorphism (e.g., [1–6]). However, it is generally accepted that no crustal eclogites of Archean age occur anywhere on Earth because of the high geothermal gradients and relatively low thickness of the crust in the Archean (e.g., [7]). Hence, the eclogites are often considered to represent evidence for plate tectonics of the modern type (e.g., [8] and references therein). Provided that 2.7–2.8 Ga ages are geochronological evidence, these could make the BMB the unique example of the Archean eclogite metamorphism and thus reinforce the model of plate tectonics at an early stage of the formation of the Earth’s crust. On the other hand, the conclusions on the Archean age of the BMB eclogites have not been adequately supported by a study of trace element distribution in zircon, whereas the results of U-Pb dating of zircons are often quite arbitrarily interpreted (e.g., [6]).

There are two key sites of eclogite occurrences within the BMB, the Salma and Gridino regions. Previously, several mutually exclusive models on the origin of Gridino eclogites have been put forward. These models provided contrasting age estimates for the eclogites, i.e., two stages of eclogite metamorphism (2.72 and 2.42 Ga, [9]), only a single Archean stage of metamorphism (e.g., [6,10]), or only a Proterozoic (Svecofennian, ~1.9 Ga) age of the eclogites [8,11–17]. For eclogites of similar mineral composition in the northwestern part of the BMB (Salma eclogites), the age of eclogitization has not been unequivocally constrained as being Archean as well, with our results showing rather the Svecofennian (~1.9 Ga) age of the process [18–21]. Further detailed geochemical studies of eclogites from the Gridino area are required to resolve the contradictions and contribute to the unraveling of the complex history of metamorphic events within the BMB.

During a preliminary investigation of zircon grains from a pegmatite vein that cuts a reference eclogite boudin on Stolbikha Island, Gridino region, a single zircon showing an anomalous rim composition was recognized (the total content of Y and REE in this grain reaches 17 wt%; sample 101 in [22]). Here, we conducted a comprehensive isotope-geochemical study of zircons recovered from a more representative specimen (101d) from this pegmatite vein. These data allow us to provide some additional constraints on the age of eclogitization within the Gridino area based on a study of zircons from the younger pegmatitic assemblage that directly cut the eclogite. We also investigate oxygen isotopes and the behavior of Y, REE and other non-formula elements in the zircons to offer insights into the effect of late-stage fluids on the zircon during the pegmatite vein formation.

2. Geological Setting

Gridino village is located on the Karelian coast of the White Sea in the central part of the BMB (Figure 1). Owing to the good outcrop conditions, variety of rocks, and the preservation of cross-cutting relationships, the Gridino region has become the object of detailed investigations by many researchers worldwide ([23] and references therein). The Gridino area is composed predominantly of gneisses of various compositions. The most ancient formations of the region, garnet amphibolites and metamorphosed ultramafic rocks, occur as bodies ranging in size from the first cm to 4–5 m, often clustered within the gneisses. Volodichev et al. [9] interpreted the gneisses and bodies of rocks of various composition (including eclogites) as the enclosing rocks and podiform fragments of the Archean melange, respectively. The gneisses are cut by dikes of metabasites of several age generations. The widespread dikes of high-Mg and high-Cr metagabbros of the ~2.44–2.36 Ga lherzolite-gabbro-norite complex mark the onset of the Proterozoic and thus are of particular importance for the geology of the region [23].

The Gridino area is characterized by structural heterogeneity due to the uneven structural-metamorphic reworking of the Archean crust during the Paleoproterozoic era [24]. The most complex structures are observed in areas where the gneisses and metabasite fragments are intensively
folded and disturbed by brittle faults. These structures are cut by undeformed Paleoproterozoic metabasite dikes (including metagabbronorites), which indicates their Archean age. There is no evidence of eclogites being cut by the undeformed dikes of metabasites in the region.

The early Paleoproterozoic structure of areas where the dikes of metagabbronorites and other metabasites are deformed to various degrees is much simpler. This is primarily because Paleoproterozoic deformations completely or partially obliterated pre-existing structural fragments and contributed to the formation of gneisses with a steep dip of banding. The deformations are associated with Paleoproterozoic steeply dipping ductile shear zones of different ages and thickness. The eclogitization of basites is associated with Svecofennian (~1.9 Ga) ductile shear zones [13,15,25].

Areas with a low-angle dip of thin gneiss banding are also characterized by a simple structure. These areas were formed under the amphibolite-facies conditions in gently dipping ductile shear zones, which are younger compared to the steeply dipping ones [24]. Despite the abundance of metabasite dikes in the Gridino region, the gneisses with a low-angle dip of banding are not crosscut by the metabasite dikes, but instead contain eclogite bodies and boudins of gabbronorite dikes and also appear to enclose boudins of younger metabasite dikes. This evidence indicates the formation of the low-angle ductile shear zones at the final (Late Svecofennian) stage of the structural-metamorphic evolution of the BMB after the eclogitization of mafic rocks.

The study area is located ~10 km southeast of the village of Gridino, on Stolbikha Island. This area represents an inner part of the youngest gently dipping ductile shear zone. Here among the metabasite bodies eclogitized mineral assemblages occur. The bodies of metabasites (amphibolites and retro-eclogites) are distributed chaotically among the gneisses with a low angle of thin banding. As a rule, these bodies (boudins) show their own fabrics and exhibit lineation or foliation, which is occasionally folded. The metabasites’ bodies are enveloped by thin gneiss banding, with the latter being folded in the proximity to the metabasite bodies, especially in areas near their clusters. There are no penetrative textures in the finely-banded gneisses and metabasite boudins. The size of the metabasite boudins is commonly less than 10 m. They comprise ~5–10% of the outcrop area.

A previous study of the Gridino eclogites [9] has established the P-T parameters of the prograde metamorphism at 14.0–17.5 kbar and 740–865 °C. Yu et al. [8] determined the peak P-T conditions
of the eclogitization in the Gridino area at 725–750 °C and more than 18 kbar. The retrograde amphibolite-facies metamorphism has been constrained at 8.2–6.5 kbar and 630–715 °C [9].

The studied eclogite boudin that is cut by the pegmatite vein is located on the southeastern coast of the island (Figure 2). It was passively involved in a gently dipping ductile shear zone formed under the amphibolite-facies conditions. Zircon grains recovered from this boudin have been previously studied in [9–12,17], whereas rock-forming minerals of the boudin are dealt with in [8,21]. The boudin is composed of eclogite and banded symplectitic apoecloligite with a gradual transition between the two. The banding of symplectitic apoecloligite is due to the uneven distribution of clinopyroxene-plagioclase symplectite (after omphacite), plagioclase, and amphibole. Eclogites showing weak mineral lineation compose the northeastern part of the boudin. The enclosing rocks are finely-banded amphibole and biotite gneisses. The contact zone, not exceeding 30–40 cm in thickness, experienced intensive amphibolite metamorphism.

The pegmatite vein injection is one of the events that occurred at the final stage of the structural-metamorphic development of the region. The pegmatite vein, from which sample 101d was taken, strikes approximately east-west and dips subvertically (Figure 2). The contacts between the vein and the eclogite are distinctively crosscutting, with the vein transecting the mineral lineation of the eclogite. The vein is characterized by a constant thickness of 20–30 cm and exhibits simple zoning, with a selvage composed of feldspar and a core of quartz. The core accounts for about a third of the vein thickness.

![Figure 2](image-url)

**Figure 2.** (a) West-south-westward view of a boudin composed of banded symplectitic apoecloligite (outlined by outer white dotted line) and eclogite (outlined by inner white dotted line). The boudin is crosscut by a subvertical pegmatite vein (outlined by red dotted line). The enclosing rocks are finely-banded amphibole gneisses showing platy parting. Stolbikha Island, Gridino area. (b) Pegmatite vein (thickness 20–30 cm); small red star indicates the site from which sample 101d was collected.
3. Methods

Zircon grains were recovered from the pegmatite (sample 101d) by conventional magnetic and heavy liquid techniques. Once separated, the zircon grains were mounted in epoxy together with TEMORA-2 [26] and 91500 [27] zircon standards. Spots for in situ analyses were selected by using both transmitted and reflected light images in order to avoid cracks and inclusions. Cathodoluminescence (CL) images were obtained at the Centre for Isotopic Research of the All-Russian Geological Research Institute using a MX2500S (CamScan Electron Optics Ltd., Cambridge, UK) scanning electron microscope (SEM) coupled with a QLI/QUA2 CL and operated at 12 kV, 5–7 nA and a working distance of 31–33 mm. Back-scattered electron (BSE) imagery of zircon was carried out at the Institute of Precambrian Geology and Geochronology of the Russian Academy of Sciences using a JSM-6510 LA SEM coupled with an energy-dispersive system JED-2200 (JEOL Ltd., Akishima, Japan).

Measurements of U, Th, and Pb isotopes in zircon were performed using an IMS-1280HR ion microprobe ( Cameca, Gennevilliers, France) at the Institute of Geology and Geophysics, Chinese Academy of Sciences. The instrument description and analytical procedure can be found in [28]; and only a brief summary is given here. The primary O\(^2–\) ion beam spot was about 20 × 30 µm in size. Positive secondary ions were extracted with a 10 kV potential. In the secondary ion beam optics, a 60 eV energy window was used, together with a mass resolution of ~5400 (at 10% peak height), to separate Pb\(^+\) peaks from isobaric interferences. A single electron multiplier was employed in ion-counting mode to measure secondary ion beam intensities by peak jumping mode. Analyses of the reference zircon TEMORA-2 (417 Ma, [26]) were interspersed with unknown grains. U and Th concentrations were calibrated against zircon standard 91500 (Th = 29 ppm and U = 81 ppm, [27,29]). A long-term uncertainty of 1.5% (1σ RSD) for \(^{206}\)Pb/\(^{238}\)U measurements of the standard zircons was propagated to the unknowns [30]. Data reduction was carried out using Isoplot/Ex version 2.49 [31].

Oxygen isotope compositions in zircon were determined at the Institute of Geology and Geophysics of the Chinese Academy of Sciences on an IMS-1280 ( Cameca, Gennevilliers, France) ion microprobe. Analytical procedures are similar to those described in [32,33]. The primary Cs\(^+\) beam spot was up to ~15 µm in size. The measured \(^{18}\)O/\(^{16}\)O ratios were normalized to standard VSMOW (\(^{18}\)O/\(^{16}\)O = 0.0020052). We performed oxygen isotope measurements at spots located directly adjacent to pits after U-Pb isotope analyses. Before measuring, the sample was re-polished to remove the pits from previous analyses. The instrumental mass fractionation (IMF) was corrected using TEMORA-2 zircon standard. The second reference zircon 91500 was analyzed as an unknown to ascertain the veracity of the IMF. Eight measurements of 91500 zircon during the course of the present study yielded an average δ\(^{18}\)O value of 10.1 ± 0.4‰ (2SD). It is in agreement with the value of 9.9‰ for the 91500 zircon standard [29].

Measurements of zircon trace element composition were performed using an IMS-4f ( Cameca, Gennevilliers, France) ion microprobe at the Yaroslavl’ branch of the Institute of Physics and Technology of the Russian Academy of Sciences. We mainly followed the analytical procedure described in [4,34]. The primary O\(^2–\) ion beam spot size was ~20 µm. Each analysis was averaged from 3 measurement cycles. Accuracy of trace element measurements was up to 10% and 20% for concentrations of more than 1 ppm and between 0.1 and 1 ppm, respectively. To construct REE distribution spectra, the composition of zircon was normalized to that of chondrite CI [35]. The zircon crystallization temperature was determined by the Ti-in-Zrn thermometer [36].

4. Results

4.1. Zircon Characterization

Around fifty zircon grains 100 to 500 µm in maximum dimension were separated from sample 101d. Figure 3 shows a representative subset of the whole fraction, which has been selected for analytical examinations. The majority of the zircons (the main population) exhibit an elongated shape, with aspect ratios (length to width) varying from 2–3 to 5–6. These zircons are characteristically dark to almost
Abundant alteration zones within the zircons appear as vermiform domains of variable thickness (5–20 µm) and are confined mainly to the margin of the grains (e.g., spots 33, 36, 37, 39, and 42; Figures 3 and 4). The alteration zones are evident in CL imaging as black bands with light grey rims (Figure 3). In BSE mode, the alteration zones are easily distinguished by a dark grey color against the light grey background of unaltered zircon (Figure 4). Occasionally, these zones are recognized in the grain interior as well (e.g., an elongated grain with spots 36 and 37; Figure 4) or encircle the zircon cores. At some examples the alteration zones affect the zircon cores, where they are represented by veinlets of submicron thickness.

Figure 3. CL images of zircons from the pegmatite vein (sample 101d). Circles indicate the SIMS analytical spots with their numbers corresponding to those in Tables S1 and S2.

Figure 4. BSE images of zircons from the pegmatite vein (sample 101d). Circles indicate the SIMS analytical spots with their numbers corresponding to those in Tables S1 and S2.
In twelve zircon grains from the main population, inherited (xenocrystic) cores 20 to 50 μm across have been recognized. The cores are rounded to ovoid in shape. In CL imaging, the cores commonly display a darker central part and a thin light grey fringe not exceeding 10 μm (Figure 3). The cores are clearly visible in BSE (spots 11 and 13, Figure 4) and transmitted-light images (Figure 5a) as well. Most of the cores are penetrated by thin veinlets that are dark in BSE images, most likely microcracks.

Another variety of zircons is represented by ten rounded to subrounded grains not exceeding 150 μm in diameter. In transmitted light, all zircons from this population are distinguished by an adamantine lustre (Figure 5b). Their predominant color in CL is light grey, whereas dark grey is less common (spots 3, 29, 29re, Figure 3). In the light grey in CL zircons’ well-defined sectorial (mosaic) zoning patterns transitional into fir-tree zoning have been established (spots 28, 32 and 27, 31, Figure 3); these are also suggested to be present in dark grey in CL grains from this population, but are barely visible. Most of these zircons display a thin (≤5–10 μm) brightly-luminescent margin (Figure 3). In rare cases, an additional external rim of variable thickness is observed as well (up to 30 μm; spot 4 in Figure 3).

![Figure 5](image-url) Transmitted-light images of zircons from the pegmatite vein (sample 101d). (a) dark-CL pegmatite zircons (main population); (b) zircons with sectorial (mosaic) in CL zoning.

### 4.2. Zircon U-Pb Geochronology

A summary of zircon U-Pb-O isotope systematics is provided in Table S1. Zircons from the main population yielded a weighted mean age of 1890 ± 2 Ma based on 14 in situ analyses (MSWD = 1.4; Figure 6a). These zircons show a high U content (1091–9253 ppm, mean 3856 ppm). Th varies from 3 ppm to 46 ppm, with a mean of 20 ppm, while Th/U ratio is extremely low from 0.001 to 0.008, with a mean of 0.005.

The inherited cores in the main zircon population failed to provide a concordant age. The values of the $^{207}\text{Pb} / ^{206}\text{Pb}$ age for the cores range from 1973 to 2694 Ma, with a discordance value varying from 0.7% to ~63%. Four spots (5, 7, 17 and 19) made it possible to construct a discordia plot with a lower intersection, which, due to a high value of MSWD = 30 and a significant error in the value of an upper intersection, cannot be considered satisfactory. Nevertheless, the upper intersection of this discordia with the concordia at ~2713 Ma corresponds to the discordant age of the light grey sectorial in CL zircons, which will be considered below. U content varies from 40 ppm in a core bright in CL (spot 21) to 2043 ppm in the darkest one (spot 19). Th abundance lies within 7–203 ppm. Th/U in the inherited cores is higher than in the main population (0.05–0.39 with a mean of 0.19).
The light grey sectorial in CL zircons (spots 28, 32 and 27, 31) and a dark grey in the CL zircon (spot 3) gave a concordant age of 2743 ± 10 Ma (MSWD = 0.054; Figure 6b). The light grey sectorial in CL zircons (spots 27, 28, and 31, 32) have low contents of U (mean 27 ppm) and Th (mean 9 ppm), while Th/U in these varies from 0.28 to 0.49, with a mean of 0.36. The zircon dark grey in CL (spot 3) exhibits increased levels of U (216 ppm) and Th (79 ppm), compared to the light grey in CL grains, but the same value of Th/U (0.37).

Two sectorial zircons showing darker CL (spots 29 and 30) have not been included in the concordant group due to the presence of discordance (~3% and ~8%, respectively). The 207Pb/206Pb ages for these zircons (2655 ± 9 Ma and 2345 ± 8 Ma, respectively) fall below the concordant determinations in proportion to the value of discordance. For the considered grains, a higher content of U (108 and 157 ppm, respectively) and Th (32 ppm and 52 ppm, respectively) was established. In the meanwhile, the Th/U ratios (0.30 and 0.43, respectively) are similar to those in the concordant zircon group.

4.3. Zircon Oxygen Isotope Composition

The δ18O value of the main zircon population varies from 6.07‰ to 8.33‰, with a mean of 6.72‰ ± 0.64 (SD). For the zircon cores, a generally higher value of δ18O was established, varying from 6.69‰ to 8.81‰, with a mean of 7.35‰ ± 0.55 (SD). For the grains’ sectorial in CL, δ18O values lie between 6.18‰ and 7.90‰. A mean δ18O value of 7.13‰ ± 0.57 (SD) for this zircon population is closer to the inherited zircon cores rather than to the remaining zircon interior of the main population.

4.4. Zircon Trace Element Composition

Zircon trace element composition and REE distribution patterns appear in Table S2 and Figure 7, respectively. The REE patterns of the main zircon population are subdivided into three groups. The first group (spots 2, 10, and 41; Figure 7a) is characterized by a strongly fractionated distribution of REE with a regular increase from light to heavy REE (LuN/LaN 15,293–39,223; LuN/GdN 342–465). The total LREE content is low (mean 2.1 ppm). A positive Ce anomaly (Ce/Ce* averages 13.6) and a moderately negative Eu anomaly (Eu/Eu* averages 0.44) were revealed in zircons of this group. The average content of U is 3126 ppm, while Hf is relatively high at the level of 17,850 ppm. Y is within the normal range (mean 1360 ppm; [37,38]). Based on the distribution of REE, as well as Ti (mean 1.7 ppm) and Th (mean 15.9 ppm) abundances, zircons of this group correspond to zircons from the veins of ceramic pegmatites cutting Salma eclogites in the Kuru-Vaara deposit area in the northwestern BMB (Figure 7a; [18,20]). Ca and Sr are within the normal range (e.g., [38]). The temperature value calculated by the Ti-in-zircon thermometer [36] for zircons of this group averages ~600 °C, which is generally consistent with the temperature of pegmatite formation [39].
The second group of zircons (spots 14, 16, and 26; Figure 7a) differs from the first group in showing a higher LREE content (mean 13.7 ppm) and a less fractionated LREE distribution (Sm\textsubscript{N}/La\textsubscript{N} averages 1.9). Moreover, here both Ce and Eu anomalies are significantly subdued (mean 1.9 and 0.64, respectively). However, the fractionation of HREE is similar to that of the first group (Lu\textsubscript{N}/Gd\textsubscript{N} 240–344). U and Th slightly increase compared with the first group (mean 4311 and 19.8, respectively). Hf and Ti show a modest increase as well (mean 19,400 ppm and 3.0 ppm, respectively); Ti abundances correspond to the zircon formation temperature of \~650 °C [36]. Y, Ca and Sr are still within the normal range.

The third group of zircons (the remaining spots in Figure 7a) demonstrates the highest LREE abundances among the three groups up to 2203 ppm with a mean of 721 ppm. As in the second group, the REE spectra are almost horizontal in the LREE region (Sm\textsubscript{N}/La\textsubscript{N} averages 3.8). However, the slope of the HREE distribution becomes less steep (Lu\textsubscript{N}/Gd\textsubscript{N} averages 61.5). A positive Ce anomaly is markedly subdued (mean 2.3), as well as a negative Eu anomaly (mean 0.91). The content of Th and U in this group of zircons remains roughly at the level of the two other groups (mean 14.7 and 3961 ppm, respectively), whereas Hf content is increased to a mean value of 22,196 ppm. Note that an abnormally high Hf concentration was recorded at two spots (36,548 ppm at spot 12 and 42,267 ppm at spot 24). Y varies broadly, but is generally higher than in zircons of the two other groups (mean 2320 ppm). A characteristic feature of the third group is a significant increase in the content of other non-formula elements (Ca, Sr, and Ti). The average concentration of Ca is 529 ppm, which is more than an order of magnitude greater than in the first and second groups. The abundances of Sr (mean 34.8 ppm) and Ti (mean 54.2 ppm) are proportionally increased as well.

Of significant interest is the composition of the alteration zones, which are sharply distinguished by a dark grey color in BSE imaging. The most striking feature is an anomalously high REE content in zircons of this type (5191–84,107 ppm, mean 31,593 ppm; Figure 7b). Hence, the average REE abundances in the alteration zones are one to two orders of magnitude higher than those in the main zircon population where these alteration zones are developed. The REE patterns of the alteration zones (Figure 7b) are almost flat at the entire range (Lu\textsubscript{N}/La\textsubscript{N} averages 6.7). A positive Ce anomaly is significantly reduced (Ce/Ce\textsuperscript{*} averages 2.9); however, at two spots with a higher REE content (spots 36 and 37), the positive Ce anomaly is more prominent than at the other three spots. The magnitude of a negative Eu anomaly remains the same (Eu/Eu\textsuperscript{*} averages 0.68) as in the unaltered zircons. The concentrations of Th (mean 52.4 ppm), U (mean 5141 ppm), and Hf (mean 20,661 ppm) generally correspond to the content of these elements in the unaltered zircons. Variations in the Ti content remain approximately the same compared with the unaltered zircons (17 to 61 ppm). However, the content of other non-formula elements is anomalously high in this zircon population. Y correlates positively with HREE and attains 29,598 ppm with a mean of 11,177 ppm. Ca varies from 1646 to 9914 ppm, with a mean of 5120 ppm. Sr content averages 340 ppm, which is an order of magnitude higher than in the unaltered zircons of the main population.

It should be noted that only a half of twelve initially observed zircon cores have been analyzed for the content of trace elements, because during re-polishing of epoxy mounts before this type of analysis the zircon cores had significantly decreased in area. In terms of the distribution of REE, a single large (~50–60 μm; spot 13) core showing no signs of alteration significantly differs from the others (Figure 7c). Its REE pattern exhibits moderate fractionation with a positive slope from light to heavy REE (Lu\textsubscript{N}/La\textsubscript{N} = 984). The total REE content is low (64.4 ppm), of which 62.3 ppm are HREE. A moderately pronounced positive Ce anomaly (3.59) and negative Eu anomaly (0.78) were revealed. Compared to the main zircon population, Y content in this core is noticeably lower (88.2 ppm), Th content is slightly increased (44.2 ppm), while the abundance of U, by contrast, is decreased to 280 ppm, which results in an increase of the Th/U ratio to 0.16. The content of non-formula elements (Ca, Ti, Sr) is within the normal range [37,38]. The temperature value calculated for this core is \~860 °C [36].
with the unaltered core (spot 13). Ti varies from 13 to 96 ppm (mean 57 ppm), which is similar to Ti (Figure 7d). Only at spot 32 is the REE distribution spectrum distinguished by a high LREE content.

Th population is low, not exceeding 67 ppm and 247 ppm, respectively, with Th in this zircon that are crosscut by this vein ([11,12]; Figure 7d). A prominent positive Ce anomaly (mean 7.43) and a relatively high REE content (mean 2096 ppm) and a flattening of patterns were recorded (Figure 7c). Lu/Nd averages 984. A positive Ce anomaly is virtually absent (mean 3.1). An Eu anomaly varies in sign; at spot 19 it is positive (Eu/Eu* = 1.37), whereas other spots exhibit a moderately negative Eu anomaly (mean 0.71). U content, in comparison with the unaltered core (spot 13), increases by about 3 times, whereas Th abundance does not fundamentally change, which results in a decrease of the Th/U ratio to a mean value of 0.07. Hf concentration averages 13,930 ppm. In these cores, the content of Y (up to 2000 ppm on average) and non-formula elements significantly increases in comparison with the unaltered core (spot 13). Ti varies from 13 to 96 ppm (mean 57 ppm), which is similar to Ti content in the alteration zones. Ca and Sr increase to mean values of 317 and 23.5 ppm, respectively.

In the remaining five zircon cores with varying degrees of alteration (spots 11, 17, 19, 21, and 23), concentrations of Th (mean 52.4 ppm), U (mean 5141 ppm), and Hf (mean 20,661 ppm) generally correspond to the content of these elements in the unaltered zircons. Variations in the Ti content remain subdued (mean 2.3), as well as a negative Eu anomaly (mean 0.91). The content of Th and U in this group significantly reduced (Ce/Ce* averages 2.9); however, at two spots with a higher REE content (spots 36 and 37), the positive Ce anomaly is more prominent than at the other three spots. The magnitude of a negative Eu anomaly remains the same (Eu/Eu* averages 0.68) as in the unaltered zircons. The content of Y (up to 2000 ppm on average) and non-formula elements is anomalously high in this zircon population. Y correlates positively with approximately the same compared with the unaltered zircons (17 to 61 ppm). However, the content of other non-formula elements (Ca, Sr, and Ti). The average concentration of Ca is 529 ppm, which is more than an order of magnitude higher than in zircons of the two other groups (mean 2320 ppm). A

Figure 7. Zircon REE distribution patterns normalized to CI chondrite [35]. (a) dark-CL pegmatite zircons (main population). The characterization of zircon groups is given in the text. The grey field shows the composition of zircons from the pegmatites cutting Salma eclogites [18,20]; (b) dark grey in BSE imaging alteration zones in zircons; (c) inherited zircon cores; (d) zircons with sectorial (mosaic) in CL zoning. The grey field shows the composition of zircons cores from the eclogite boudin cut by the pegmatite vein [11,12].
(mean 11,472 ppm). Y content ranges from 39 to 190 ppm (mean 99 ppm). The concentration of other non-formula elements (Ca, Ti, and Sr) is generally low, rarely exceeding the first tens of ppm. The temperature value determined by the Ti-in-zircon thermometer [36] averages 714 °C.

5. Discussion

5.1. Inter-Element Relationships

Binary plots depicting inter-element relationships in the examined zircons are given in Figure 8. The zircons show a strong positive correlation between the contents of Y and REE (Figure 8a). In the lower left part of the Y-REE diagram, a group of analyses is distinguished, representing the sectorial in CL zircons, as well as the large unaltered core (spot 13). The remaining zircon cores and dark-CL grains from the main population, including the alteration zones, form a single linear trend (Figure 8a). Within this region, Y and REE consistently increase from ~1000 to ~10,000 ppm along a 1:1 correlation. Two zircon spots (36 and 37), located in a strongly altered central part of an elongated zircon grain (Figure 4), occur separately in the upper right part of the trend, showing extremely high abundances of Y (29,598 and 18,565 ppm) and REE (84,107 and 54,135 ppm, respectively).

Figure 8. (a–f) Co-variation diagrams showing the composition of the studied zircons. 1—dark-CL pegmatite zircons (main population); 2—dark grey in BSE alteration zones in zircons; 3—inherited zircon cores; 4—zircons with sectorial (mosaic) in CL zoning.
The incorporation of Y and REE (mainly HREE) into the zircon structure is commonly ascribed to the coupled xenotime-type substitution (Y, REE)\(^{3+}\) + P\(^{5+}\) = Zr\(^{4+}\) + Si\(^{4+}\) [40]. Such an isomorphic scheme suggests a proportional increase in the content of Y and HREE on the one hand, and P on the other. Indeed, this holds true for zircons from the Ichetju (Middle Timan) ore deposit, whose P content attains 41,830 ppm [41]. For the zircons studied here, however, an increase in P content falls behind compared to an increase in Y and REE (Figure 8b). Even where a total content of Y and REE exceeds 100,000 ppm, P reaches only ~800 ppm. It is possible that, in this case, the xenotime-type substitution is of subordinate significance, whereas the prevailing isomorphic mechanism involves the incorporation of hydrogen, i.e., H\(^+\) + (REE, Y)\(^{3+}\) = Zr\(^{4+}\) [42]. Based on the distribution of Y and P, the compositions of the sectorial in CL zircons, as well as of some zircon cores, fall separately from the other zircon varieties in the region of relatively low Y and P concentrations (Figure 8b).

A comparison of LREE and HREE abundances in the zircons shows that an increase in LREE content is an order of magnitude greater than an increase in HREE (Figure 8c). Here again, the compositions of the sectorial in CL zircons, as well as of the unaltered core (spot 13), are separated from the main population. In these zircons, the concentrations of LREE and HREE do not exceed 100 ppm (Figure 8c).

Sr abundance in zircon, as a rule, is an order of magnitude lower than the level of Ca (e.g., [22]). In the zircons studied here, Ca \(\leq 100\) ppm and Sr \(\leq 1\) ppm have been recorded only in the sectorial in CL zircons, as well as in the unaltered core at spot 13 (Figure 8d). For the remaining grains, a strong positive correlation between the content of these two non-formula elements is observed. The compositions of the main population, alteration zones, and the majority of the zircon cores form an almost continuous trend, with two spots (36 and 37) being located separately in the upper part, which also plot away in the other binary diagrams described above due to very high amounts of trace elements (Figure 8a–c).

A positive correlation between Ti and Ca in the zircons is less pronounced (Figure 8e). It is accepted that Ti in the vast majority of terrestrial zircons does not exceed 20 ppm [43]. It is this range of Ti (0–20 ppm) that was used for the calibration of the Ti-in-zircon thermometer [36]. The sectorial in CL grains, some unaltered cores, and about a half of the main zircon population fall well into this interval, whereas other grains display high Ti abundances up to 224 ppm. The median Ti content for the entire zircon separate is 17 ppm. The alteration zones display no correlation between Ti and Ca (Figure 8e).

The main zircon population and alteration zones on the one hand, and the species sectorial in CL and inherited cores on the other hand, are critically different in U abundances, as well as in the Th/U ratios. These two populations compose, in Figure 8f, two distinct fields. Th/U for the zircons with sectorial zoning and zircon cores lies mostly in the range of 0.01–0.2, whereas the main population and alteration zones display much lower Th/U values mostly in the range of 0.001–0.01.

5.2. Anomalous Level of Non-Formula Elements in Zircon: The Role of Fluids Rich in Incompatible Elements

A part of the zircons studied here demonstrate extremely high concentrations of non-formula elements, especially in the alteration zones. While zircons enriched in Hf, U, and Th are well-known, significant deviations in the contents of Y and REE are less common. As a rule, Y content in zircon lies in the range of 10–5000 ppm and REE content is commonly from 100 to 2500 ppm [37]. However, a series of studies established a prominent increase in the content of non-formula elements in zircon from several global occurrences. An extremely high REE abundance of ~98,200 ppm was reported in zircons from metasomatites of the unique Oklo uranium deposit (the Republic of Gabon, Equatorial Africa) [44]. A high content of Y (up to 4.2 wt% \(Y_2O_3\)) and other non-formula elements (Mg, Al, Ca, and Fe) was revealed in altered zircons from metasedimentary rocks of the Dalradian complex (Scotland) [45]. An abnormally high content of trace elements (Y up to 61,874 ppm, REE up to 27,667 ppm, and Nb up to 7976 ppm) was recorded in rims and alteration zones of zircons formed from a fluid-saturated syenite melt at the late magmatic stage of the formation of the Zr-Y-REE Azov deposit (Ukrainian shield) [46,47]. Zircons from the Thor Lake REE deposit (Canada) show REE content up to ~74,000 ppm and Y abundance up to ~31,500 ppm; these are also high in Nb and
In zircons from the Ichetju polymineralic ore deposit (Middle Timan, North-East European part of Russia), for which a high-temperature hydrothermal genesis is assumed, an unusually high content of Y and REE was also established—up to 96,240 and 104,578 ppm, respectively [41]. On the Fennoscandian shield, an anomalously high REE abundance was recorded in zircons from metasites of the Kontokki dike complex, Kostomuksha structure (29,800 ppm, [49]), lamproites of the Panozero sanukitoid complex, Central Karelia (55,300 ppm, [50]), and quartz syenites in the central part of the North-Karelian greenstone belt (89,100 ppm, [22]).

In the above examples of zircons from rocks of various composition and age, high levels of non-formula elements occur in local domains and alteration zones that are characteristically dark in BSE mode. All these zircons are united by the influence of fluids enriched in incompatible elements (such as Y, REE, and high field strength elements). HFSE are commonly not mobile during magmatic and metamorphic processes, but exert a strong role in the processes of fluid metasomatism ([51] and references therein). An indicator of the fluid effect on zircon is also an increased content of other non-formula elements (Ca, Sr, and Ba) [52,53]. Ca content of ~100 ppm can be considered as a formal threshold for determining whether or not zircon has been affected by fluids [52]. As is evident from Figure 8, the alteration zones in the zircons examined here (red data points) are highly enriched in non-formula incompatible elements, which indicates a pronounced impact of late-stage fluids on the zircon trace-element geochemistry.

In the main zircon population, including the alteration zones (the first and second groups of data points, Figure 8), Th content is low (mean 23 ppm). Meanwhile, U content in these zircons is relatively high with a mean value of 4150 ppm, which leads to a sharp decrease in the Th/U ratio (mean 0.005). Such an extremely low Th/U is typical for hydrothermal or metasomatized zircons, though it is not an unequivocal discriminant [54,55]. However, note that for zircons without any signs of hydrothermal-metasomatic alterations from the veins of ceramic pegmatites that cut Salma eclogites (Kuru-Vaara, northwestern BMB), the mean Th/U is much higher (0.02), whilst U content does not exceed 1000 ppm [18,20].

For only three spots of the main zircon population, the REE distribution spectra turned out to be close to the spectra of zircons from the Kuru-Vaara pegmatites (spots 2, 10, and 41, Figure 7a), whereas others show REE distribution spectra similar to zircons that have undergone metasomatic or hydrothermal processing. In the La-(Sm/La)_N scatter plot characterizing the degree of LREE fractionation in zircons [56–58], the main zircon population, alteration zones, and a part of the inherited cores fall within or near the fields of porous and hydrothermal zircons formed as a result of intensive fluid processing (Figure 9a). However, almost all the sectorial in CL varieties, the large unaltered core (spot 13), and those zircons from the main population whose REE distribution spectra coincide with those typical for the zircons from the Kuru-Vaara pegmatites (spots 2, 10 and 41, Figure 7a), belong to the field of unaltered magmatic zircons. Likewise, in the U-Ca scatter plot, also designed to differentiate between unmodified zircons, porous, and fluid-altered zircons [58,59], the main zircon population and alteration zones belong chiefly to the fields of porous and altered zircons, whereas almost all the sectorial in CL grains and inherited cores fall into, or immediately near, the field of unaltered zircons (Figure 9b).

All these lines of evidence on the peculiarities of the zircon composition allow us to conclude that the main zircon population from the pegmatite vein is indeed pegmatitic, but appears to have crystallized from an evolving fluid-saturated media that provided a progressively increasing supply of LREE, Y, Sr, Ca, and Ti (from Group 1 to Group 3, see Figure 7a). Subsequently, these zircons underwent an irregular autometasomatic overprint by a fluid extremely rich in incompatible elements, most likely related to the latest stages of the pegmatite vein formation. Meanwhile, the inherited zircon cores and varieties with sectorial zoning show distinct optical properties, morphology, and chemistry and thus are regarded to have been captured as xenocrysts; these are further considered at some length below.
5.3. Granulite-Derived Zircons

The average Th/U ratio for the sectorial in CL zircons and zircon cores is 0.17, which enters within the range of Th/U ratios in metamorphic zircons [60,61]. A common feature of metamorphic zircons is the “smoothing” of geochemical characteristics typical of magmatic ones, e.g., Th/U in metamorphic zircons, as a rule, does not exceed 0.2, while a negative Eu anomaly and a positive Ce anomaly are less prominent [60,62]. However, it should be noted that it is not always possible to discriminate between magmatic and metamorphic zircons based only on the distribution of REE and other trace elements ([63,64] and references therein). It has been found that the composition of granulite zircon, often in equilibrium with an anatectic melt, does not significantly differ from that of magmatic zircon with respect to a wide range of trace elements, such as Y, Hf, and P [60,65,66]. According to some authors, such zircon can be considered “magmatic” (e.g., [67]). The REE distribution in granulitic zircon appears to depend on the composition and volume of a melt [68].

Among metamorphic zircons, varieties from eclogites are sharply distinguished by composition and show several general patterns, i.e., abnormally low Th and Th/U ratio, as well as a low content of the entire spectrum of REE, low Y, U, and P, yet an increased abundance of Hf [12,60]. Therefore, for zircons from rocks of amphibolite-facies metamorphism and eclogites, differences in composition between zircon domains corresponding to magmatic protolith (usually cores) and metamorphism (usually rims) are often obvious. By contrast, for zircons from granulites, especially in cases where there are no coexisting cores and rims to be compared, compositional data is insufficient to draw a conclusion on their origin and it is necessary to utilize data on zircon morphology and its internal characteristics in CL as well (e.g., [69,70]).

As a rule, granulite zircons differ from other zircon varieties by their optical properties; these grains are colorless to light pink with an adamantine lustre. The isometric or rounded shape of granulitic zircon grains, resembling a soccer-ball, is also noted in many publications (e.g., [65,71,72]). Therefore, based on the composition, morphology, and optical characteristics of the sectorial in CL zircon population studied here, we consider these zircons as corresponding to granulite-facies metamorphism [69,70,73,74]. These zircons are regarded to have been captured from metabasites that are distributed in the form of dikes and boudins within the region. The inherited cores share compositional similarities with the zircons sectorial in CL and thus have been likely derived as xenocrysts from metabasites as well.

5.4. Age Constraints

For the moment, there are several coinciding age estimates accumulated by the authors for the Gridino-type eclogites, provided by both rock-forming and accessory minerals. Garnets from two eclogite boudins of the Gridino region yielded Lu-Hf ages of 1937 ± 8 Ma (sample 102) and 1892 ± 10 Ma (sample 108) [21]. Similar age estimates of 1911 ± 11 Ma (sample T1106D9) have
been reported for garnets from an eclogitized dike in the same region; these values, within the error, coincide with the age of zircons from this dike (1887 ± 13 Ma; [15]). Note that a close Lu-Hf age value of 1.96–1.92 Ga, interpreted as the age of prograde eclogite metamorphism, has been recently determined by Yu et al. [8] for garnets from eclogite boudins (three samples) and from an eclogitized dike (one sample) within the Gridino area.

Previous U-Pb datings of zircons from the eclogite boudin cut by the pegmatite vein on Stolbikha Island (sample 102) yielded two age estimates: a concordant value of 2702 ± 25 Ma for the zircon cores and a lower intersection of the discordia at 1878 ± 18 Ma for the zircon rims that appear bright in CL [11,12]. Since these zircon rims demonstrated geochemical characteristics typical of eclogitic zircons, their age was attributed to represent the timing of eclogite-facies metamorphism [12].

Hence, a comprehensive isotope-geochemical investigation of rock-forming eclogite minerals (garnet and clinopyroxene, Sm-Nd and Lu-Hf techniques; [15,21]) and zircons (U-Pb method; [11,12]), accompanied by a study of the distribution of REE and other trace elements in zircons [11,12], allows us to determine the age of the eclogite metamorphism in the Gridino region at ~1.9 Ga. This age value is in good agreement with the age determinations for other eclogite occurrences within the BMB—Salma eclogites in the northwestern BMB [18–20], as well as Keret Archipelago eclogites [75,76] and Krasnaya Guba eclogites [77] in the central BMB.

The direct traces of eclogite metamorphism in the zircons from the pegmatite vein examined here are barely discernable. It is likely that submicron brightly-luminescent rims, which are present in the captured granulite zircons and border the pegmatite zircon cores (Figure 3), reflect the eclogite metamorphism. These are the rims of this shape and CL-intensity that are a diagnostic feature for eclogite zircons, taking into account their trace-element composition as well [12]. However, the thickness of these rims renders it impossible to perform in situ isotope-geochemical studies without overlapping the primary ion beam onto other domains. Nevertheless, taking into account the distribution of REE in the main population of zircons from the pegmatite vein, the age of 1890 ± 2 Ma yielded by these zircons is interpreted as the age of the pegmatite vein injection. This value generally corresponds to the age of the BMB eclogites and unambiguously constrains their upper age limit based on the field relations between the vein and boudin. Hence, the crystallization of the pegmatite appears to have occurred after the eclogitization within a geologically short time span. More specifically, the obtained age values and temperature estimates (~600 °C) for the pegmatite vein injection indicate that the generation of melts parental for the pegmatite was likely related to the retrograde metamorphic stage under the amphibolite-facies conditions [9,78]. This retrograde stage dated at 1890–1870 Ma [75,76,79,80] was characterized by an intensive partial melting (migmatization). Note that the outer rims of zircon grains from both intensively migmatized gneisses and amphibolites of the Chupa Sequence (BMB) yielded the same age value (1890 Ma; [80]) as obtained here for the pegmatitic zircons of the main population.

The age value of 2710 ± 15 Ma (SIMS) determined for zircons from Chupa paragneisses was interpreted as the age of collision metamorphism that involved high-pressure granulite facies [81]. A similar age value was obtained for zircons from migmatites in the Chupa paragneisses (2710 ± 15 and 2706 ± 14 Ma, LA-ICP-MS, [82]). Zircons from enderbites cutting gneisses on and nearby Pongoma Island (BMB) afforded an age of 2728 ± 21 Ma (TIMS), which can also be considered as the age of the granulite metamorphism within the BMB [83–85]. TIMS approach was also used to establish the age of various types of zircons from fine-grained garnet-biotite gneisses, likely formed at the early stages of the BMB formation. It was shown that an age value of 2691 ± 15 Ma corresponds to the migmatization process under high pressure conditions at the collisional stage of the BMB formation [86]. The age of another type of zircon, 2788 ± 4 Ma, coincides with the age of early high-temperature moderate-pressure metamorphism [86]. The examples referred to above indicate a relatively widespread occurrence of the high-temperature (granulitic) metamorphic event with an age of ~2.7 Ga within the BMB.

The age of zircon cores from the eclogite boudin cut by the pegmatite vein (~2.7 Ga) has been interpreted previously as the age of a magmatic eclogite protolith [11,12,17]. The deviation of the geochemical characteristics of these zircons from those typical of magmatic zircons has been attributed...
to partial recrystallization of the cores during eclogitization [12]. Here, the obtained new data on the age of the zircons with a typical granulitic appearance and sectorial zoning in CL allow us to reconsider this interpretation and suggest the age of ~2.7 Ga (documented both in the eclogite boudin for zircon cores and in the pegmatite vein cutting this boudin for captured zircons) as the age of the granulite metamorphism, also recorded in the adjacent BMB regions [81–85].

The analysis of existing geochronological data on the eclogite protoliths in the BMB showed that the main group of protoliths is 2.5–2.4 Ga in age [14]. For the Krasnaya Guba coast area and Pezhostrov Island, the age of the protoliths was determined at 2.2 Ga. Less frequently than the Paleoproterozoic, the Neoarchean age of the protoliths was recorded—2.88 Ga for Salma eclogites and 2.79 Ga for the Gridino region [14]. For the age of 2.7 Ga there are no similar determinations provided in the literature. Hence, the interpretation of this value as the age of the Archean granulite metamorphism leaves open the question of the age of the magmatic eclogite protolith on Stolbikha Island, Gridino area, which is yet to be constrained.

6. Conclusions

1. The zircons from the pegmatite vein that cuts the eclogite boudin on Stolbikha Island, Gridino area, are represented by two populations. The main population, which is pegmatite-derived, is dark (almost black) in CL. About a third of these zircons contain inherited cores captured from metabasites during the pegmatite injection. The second population has a typical granulitic appearance with sectorial (mosaic) zoning in CL and, similarly to the cores, is interpreted to have been captured from metabasites.

2. A U-Pb age of 1890 ± 2 Ma for the main zircon population is the age of the pegmatite injection. The vein formation was linked to the retrograde amphibolite-facies metamorphism and occurred within a geologically short time span after the eclogitization (~1.9 Ga). The age of 2743 ± 10 Ma obtained for the sectorial in CL zircons is interpreted as the age of the granulite-facies metamorphism within the BMB. Similarly, one should interpret the age of zircon cores from the eclogite boudin cut by the pegmatite vein (2702 ± 25 Ma, [11,12]).

3. The zircon populations show overlapping δ18O values of 6.1–8.3‰, 6.7–8.8‰, and 6.2–7.9‰ for the pegmatitic zircons, inherited cores and granulitic varieties, respectively. This data suggests that the influence of fluids at the final stage of the pegmatite formation on the zircon oxygen isotope composition has been insignificant.

4. The effect of late-stage fluids, though, has been very pronounced with regard to trace element behavior in the zircons. During the interaction with fluids, the abundances of non-formula elements in the alteration zones increased by more than an order of magnitude, i.e., REE, Y, Ca, Sr, and Ti increased up to ca. 84,100, 29,600, 9900, 670, and 60 ppm, respectively. The REE spectra of the altered zircons are flattened and exhibit subdued Ce and Eu anomalies.

5. Our data provide new constraints on the timing of eclogite metamorphism within the BMB and document a new occurrence of zircon showing an extreme enrichment in a series of non-formula elements.

Supplementary Materials: The following are available online at http://www.mdpi.com/2076-3263/10/5/197/s1, Table S1: Zircon U-Pb-O SIMS data (sample 101d), Table S2: Trace element composition of zircons (sample 101d).

Author Contributions: S.G.S., A.V.B., and V.V.T. conducted the fieldworks and V.V.T. picked up the pegmatite sample; S.G.S. wrote a major portion of the manuscript draft and analyzed the zircon grains at the IGG CAS; X.-H.L. and Q.-L.L. provided methodology; L.I.S. and D.I.R. contributed to visualization and editing. All authors provided input to the evaluation and discussion of the data and to the final version of this report. All authors have read and agreed to the published version of the manuscript.

Funding: The study was funded by the National Key R&D Program of China grant No. 2016YFE0203000, the National Natural Science Foundation of China grant No. 41673018 and the Russian Foundation of Basic Research project No. 18-55-53022. This study was conducted under state contracts No. 0153-2019-0002 (Institute of Precambrian Geology and Geochronology of Russian Academy of Sciences) and No. 0218-2019-00058 (Institute of Geology Karelian Research Centre of Russian Academy of Sciences). D.I.R. acknowledges the state assignment project of IGM SB RAS.
Acknowledgments: We thank Olga Galankina for assistance with the BSE images of zircons, and Aleksey Melnik, Xiao-Xiao Ling, Jiao Li and Guo-Qiang Tang for their help with U-Pb-O isotopic analyses. Evgeny Potapov and Sergey Simakin assisted with measuring of zircon trace element compositions. Two anonymous reviewers provided valuable comments that improved the quality of the report.

Conflicts of Interest: The authors declare no conflict of interest.

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