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Glacially influenced provenance and Sturtian affinity revealed by detrital zircon U–Pb ages from sandstones in the Port Askaig Formation, Dalradian Supergroup

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Abstract: The Cryogenian 'Sturtian' snowball Earth glaciation (*c.* 717–658 Ma) likely had a major role in shaping continental landscapes and biotic radiations of the late Neoproterozoic Era. However, an incomplete sedimentary record and inadequate syn-glacial age constraints make Cryogenian studies challenging. We present detrital zircon U–Pb ages for >2000 zircons from 11 sandstone samples taken at <200 m stratigraphic resolution throughout the Port Askaig Formation, a *c.* 1.1 km thick glaciogenic succession within the Dalradian Supergroup of Scotland and Ireland. Eight new maximum depositional age constraints, including a key constraint on deglaciation (<662.7 ± 7.8 Ma), support lithostratigraphic and stable isotope evidence that suggests the Port Askaig Formation preserves a relatively complete record of the global 'Sturtian' glaciation. An increasing contribution from Archean and Paleoproterozoic detritus to the sandstones through the lower *c.* 500 m of the Port Askaig Formation likely reflects the progressive glacial unroofing of the previously buried Lewisian Gneiss terrane. Archean and Paleoproterozoic grains then become scarce in the uppermost *c.* 300 m of the formation, which we attribute to glacial modification of the Laurentian continental margin landscape during the waning stage of 'Sturtian' glaciation. The disruption to sediment transport pathways caused by this modification, and evidenced by the detrital zircon data, points to partially warm-based 'Sturtian' ice sheets that were, to some degree, dynamic.

Supplementary material: Supplementary figures and data are available at https://doi.org/10.6084/m9.figshare.c.7301043

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The 'Sturtian' glaciation is the first of two 'snowball Earth' glaciations that characterize the Cryogenian Period, when ice is predicted to have extended to the equator where it persisted for millions of years. The so-called 'snowball Earth' hypothesis has been continuously refined by geochronological studies, which show that the initiation and termination of the older 'Sturtian' and younger 'Marinoan' glaciations were globally synchronous, lasting from *c.* 717 to *c.* 658 Ma and from *c.* 645 to *c.* 635 Ma, respectively (Hoffman *et al.* 2017 and references cited therein). The dynamism of 'snowball Earth' ice sheets is contested, with early models predicting a shut-down of the hydrological cycle and the prevalence of cold-based ice sheets with minimal sub-glacial erosion (e.g. Hoffman and Schrag 2002). By contrast, others argue that the vast extents and durations of the Cryogenian glaciations must have been coupled with significant glacial erosion, shaping the continental landscape in unprecedented ways (White 1972; Keller *et al.* 2019; McDannell *et al.* 2022; Segessenman and Peters 2023).

The glaciogenic Port Askaig Formation is a suspected 'Sturtian' succession deposited in the Dalradian Supergroup of Scotland and Ireland during the break-up of the supercontinent Rodinia (Spencer 1971; Anderton 1985; Prave *et al.* 2023). The Port Askaig Formation preserves an alternation of glaciogenic diamictites and non-glacial sandstones and is one of the thickest (up to 1.1 km) and most complete records of Cryogenian glaciation (Spencer 1971; Ali *et al.* 2018). The thickness of the Port Askaig Formation, alongside sedimentological evidence for glacial advances and retreats throughout its deposition, are at odds with suggestions of the persistence of ubiquitous cold-based ice sheets throughout the 'Sturtian' (e.g. Donnadieu *et al.* 2003). The consequences of the glacial processes associated with such an extensive, long-lasting and possibly dynamic glaciation are currently ill-defined.

Spencer (1971) previously alluded to a shift in provenance in the Port Askaig Formation, noting a gradual stratigraphic change from predominantly intrabasinal to extrabasinal clasts within its diamictites. This was later quantified by Ali *et al.* (2018). An upwards change in the major and trace element composition of the matrix of the Port Askaig Formation diamictites has also been recorded (Panahi and Young 1997). Cawood *et al.* (2003) noted a shift in the stratigraphic record of U–Pb age populations of detrital zircons in the middle of the Dalradian Supergroup, just prior to the deposition of the Port Askaig Formation (Cawood *et al.* 2003; Johnson *et al.* 2016; Olierook *et al.* 2020). Specifically, Archean and late Paleoproterozoic to early Mesoproterozoic detritus emerged and seemingly persisted for the remainder of the Dalradian (Cawood *et al.* 2003).

The timing and causes behind these provenance shifts remain unclear. Cawood *et al.* (2003) attributed the 'sudden' exposure of Archean material to the modification of the basin's sediment routing system. The uplift of Archean cratons during the rifting of Rodinia (Cawood *et al.* 2007; Johnson *et al.* 2016), the removal of a topographic barrier (Spencer and Kirkland 2016) or the compartmentalization of basins yielding unique sediment dispersal pathways (Olierook *et al.* 2020) have also been suggested. Modern and recent ice sheets are known to cause significant continental denudation and alter sediment routing systems (e.g. Herman *et al.* 2013; Ghienne *et al.* 2018; Alley *et al.* 2019). However, the part that glaciation may have played in altering the detrital zircon provenance of the Dalradian Supergroup has not yet been explored.

The detrital zircon U–Pb age spectra of a total of five samples from the glaciogenic Port Askaig Formation and correlative Irish boulder beds have been analysed previously by Cawood *et al.* (2003) and Chew *et al.* (2009, 2019). The age populations present were matched with known Laurentian source rocks and the provenance during glaciation was interpreted to be similar to non-glacial intervals within the Dalradian Supergroup (Cawood *et al.* 2003; Chew *et al.* 2009, 2019). A Laurentian provenance was also suggested by both Loewy *et al.* (2003) and Evans *et al.* (1998) based on the lead isotope composition of 12 granitic clasts and the crystallization ages of two granitic clasts entrained with the Port Askaig Formation diamictite, respectively.

This paper presents detrital zircon U–Pb data from >2000 zircons in 11 metasandstone (referred to as sandstone in the following text) samples taken at a <200 m stratigraphic resolution throughout the Port Askaig Formation and underlying Garbh Eileach Formation. The Port Askaig Formation lacks any useful direct age constraints and has therefore been correlated with both the ‘Sturtian’ (Prave 1999; McCay *et al.* 2006; Prave *et al.* 2009; Fairchild *et al.* 2018) and ‘Marinoan’ (Rooney *et al.* 2011; Moles and Selby 2023) glaciations within the Cryogenian. Only one of the five samples across three previous detrital zircon studies yielded Cryogenian zircons, providing a maximum depositional age (MDA) constraint of 687 ± 12 Ma on the uppermost part of the Port Askaig Formation (Chew *et al.* 2019).

This study aimed initially to build on the work of Chew *et al.* (2019) by using high zircon yields to constrain meaningful MDAs and confirm (or invalidate) a ‘Sturtian’ affinity for the Port Askaig Formation. We also used the detrital zircon age spectra of the sandstones throughout the Port Askaig Formation to track possible landscape evolution and discuss how glacial processes may have impacted sediment dispersal pathways to the Dalradian basin during this time. Investigating the provenance of the sandstones in the Port Askaig Formation may yield key insights into how ice sheets were shaping the landscape against a backdrop of supercontinent break-up during the ‘Sturtian’ glaciation.

Geological setting

The Dalradian Supergroup is made up of five main groups, which are, from oldest to youngest, the Grampian, Appin, Argyll, Southern Highland and Trossachs groups (Stephenson *et al.* 2013; Tanner *et al.* 2013). Dalradian rocks can be traced across the Grampian Highlands in Scotland, as well as in parts of Northern Ireland and Donegal, Ireland (Fig. 1). The succession has an apparent total thickness of *c.* 25 km; however, strong lateral facies variations,

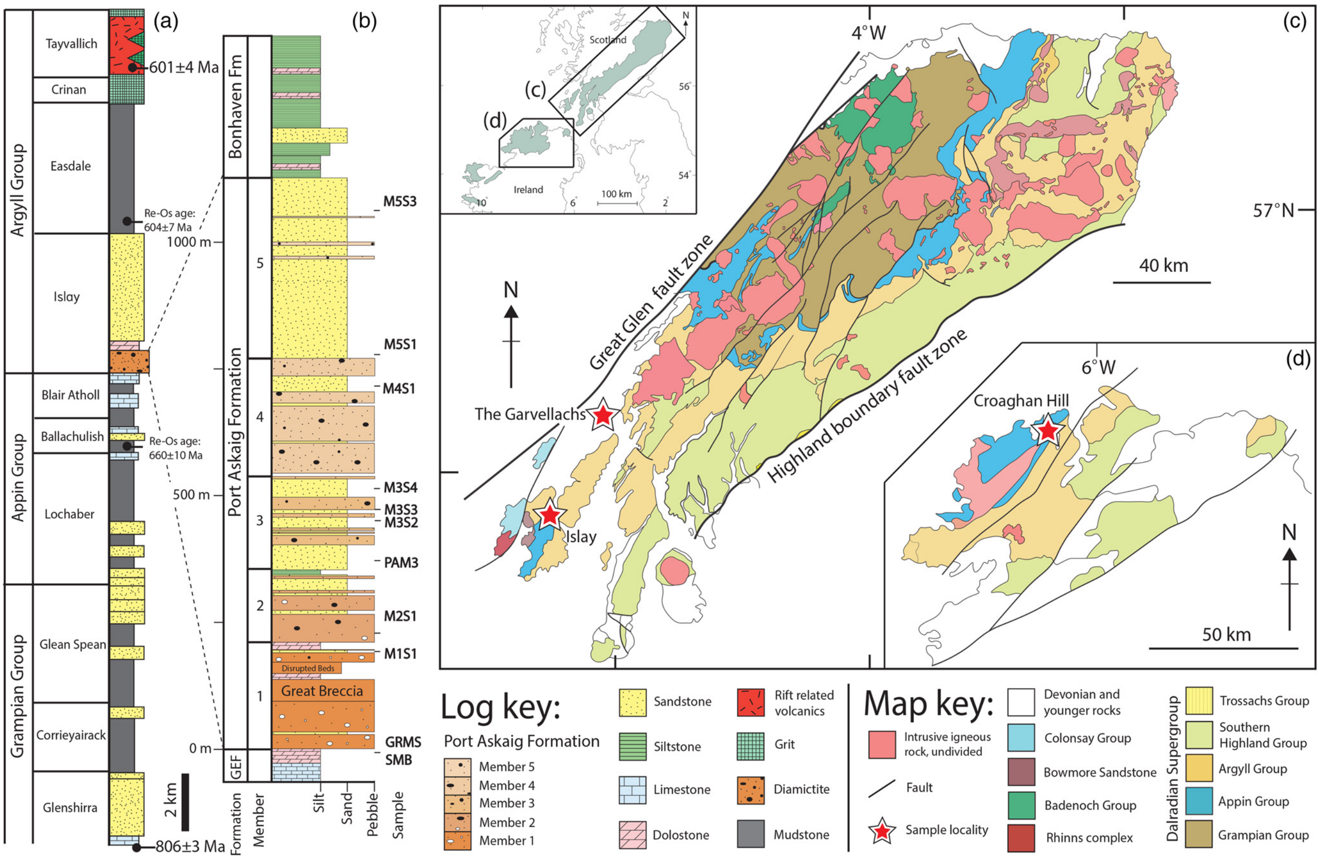

Fig. 1. Synthesis of the geological background of the Port Askaig Formation. (**a**) Generalized stratigraphy of the lower part of the Dalradian Supergroup (Stephenson *et al.* 2013). The older geochronological constraint shown on the lithostratigraphic log of the Dalradian is derived from a U–Pb age from deformed rocks beneath the Grampian Group (Noble *et al.* 1996) and the young constraint is from a U–Pb zircon age from rift-related volcanics at the top of the Argyll Group (Dempster *et al.* 2002). Two Re–Os ages are also shown for the Appin Group Ballachulish Slate Formation (Rooney *et al.* 2011) and Easdale Group Ben Eagach Schist Formation (Moles and Selby 2023). The validity of these being depositional ages is discussed in the text. (**b**) A stratigraphic column from the Port Askaig Formation (Ali *et al.* 2018). The approximate stratigraphic positions of the sampled horizons are shown. (**c**) Simplified geological map of the Dalradian Supergroup in Scotland (Shetland Isles not shown). (**d**) Simplified geological map of the Dalradian Supergroup in Donegal, Ireland. Source: parts (c) and (d) modified after Thomas *et al.* (2004).

intrabasinal unconformities and a migrating depocentre mean that a single continuous succession of this thickness is absent in the Dalradian (Stephenson *et al.* 2013; Leslie *et al.* 2024). Although the Dalradian Supergroup underwent greenschist to amphibolite facies metamorphism during the mid-Ordovician Grampian Orogeny (*c.* 470–460 Ma), it commonly retains sedimentary structures and primary textures in outcrop.

A maximum age constraint for the Dalradian Supergroup is provided by an 806 ± 3 Ma pegmatite intruded into the basement rocks of the Badenoch Group that does not cross-cut the overlying Grampian Group (Noble *et al.* 1996). The age of the upper part of the Dalradian is constrained by a U–Pb zircon age of 601 ± 4 Ma for a tuff in the Tayvallich Volcanic Formation of the upper Argyll Group (Dempster *et al.* 2002).

The Port Askaig Formation

The glaciogenic Port Askaig Formation marks the base of the Argyll Group and sits stratigraphically near the middle of the Dalradian Supergroup (Fig. 1). The most complete and best exposed outcrops of the Port Askaig Formation can be found on the Garvellach archipelago and the Isle of Islay in the Argyll region of Scotland, as well in parts of north and south Donegal in Ireland (Fig. 1). Throughout the Dalradian, the thickness of the Port Askaig Formation varies strongly. At its thickest on Islay and the Garvellach Islands it measures *c.* 1.1 km in total (Ali *et al.* 2018), however, it thins extensively to the NE and SW of this region. The unusually expanded thickness of the Port Askaig Formation in the SW of Scotland implies that it was deposited in a unique depocentre at that time.

The Port Askaig Formation consists of a total of 48 metadiamictite beds (henceforth referred to as diamictite), as well as sandstones, minor siltstones and dolostone interbeds. The formation is divided into five members, termed Members 1–5 (Kilburn *et al.* 1965; Spencer 1971), and records 28 glacial episodes, 25 periglacial episodes and 23 non-glacial episodes (Ali *et al.* 2018). Sedimentological evidence for a glaciated environment begins just below the Port Askaig Formation, in the upper part of the Garbh Eileach Formation (Fairchild *et al.* 2018). The Garbh Eileach Formation is a *c.* 70 m thick carbonate succession that is apparently unique to the Garvellach Islands, where it shares a demonstrably conformable contact with the Port Askaig Formation (Fairchild *et al.* 2018). By contrast, on Islay, the Port Askaig Formation rests unconformably on the Lossit Limestone, which is argued to be of Tonian age due to the presence of ‘molar-tooth’ calcite microspar (Fairchild *et al.* 2018), a carbonate fabric that is scarce to absent in post-Tonian strata (Shields 2002). The upper boundary of the Port Askaig Formation is marked by the mixed carbonate–siliciclastic Bonahaven Formation, which lacks the characteristics of a typical cap carbonate sequence (McCay *et al.* 2006). Evidence for the termination of glaciation is therefore difficult to pin-point within the Port Askaig Formation. Members 4 and 5 have the fewest interpreted glacial episodes and glaciation had waned dramatically by Member 5, which is composed of rare thin diamictite horizons within thick non-glacial sandstones (Fig. 1; Ali *et al.* 2018).

The diamictites of the Port Askaig Formation show evidence for both grounded ice and glaciomarine environments, whereas the sandstones are almost all non-glacial and interpreted as deltaic and shallow marine tidal deposits with variable textural and bedform characteristics that relate to minor changes in water depth (Spencer 1971; Eyles 1988; Ali *et al.* 2018). Sandstone makes up *c.* 35–40% of the Port Askaig Formation and is present in every member of the succession, evidencing the frequent advance and retreat of ice throughout deposition of the formation (Ali *et al.* 2018).

The lack of direct age constraints for large parts of the Dalradian means that the Port Askaig Formation has been correlated with both the older ‘Sturtian’ (Prave 1999; Prave *et al.* 2009; Ali *et al.* 2018; Fairchild *et al.* 2018) and younger ‘Marinoan’ glaciations (Rooney *et al.* 2011; Stephenson *et al.* 2013; Moles and Selby 2023). Support for a ‘Marinoan’ age (*c.* 645–635 Ma) assignment comes predominantly from Re–Os ages (Rooney *et al.* 2011; Moles and Selby 2023). A Re–Os age of 659.6 ± 9.6 Ma was derived from the Ballachulish Slate Formation, which lies stratigraphically below the Port Askaig Formation in the Appin Group (Rooney *et al.* 2011) and a Re–Os age of 604.0 ± 7.2 Ma was derived from diagenetic pyrite in the Ben Eagach Schist Formation in the middle part of the Argyll Group (Moles and Selby 2023). Within this framework, the Port Askaig Formation could only correlate with the global ‘Marinoan’ glaciation. However, the behaviour of the Re–Os system in metamorphosed sediments is unclear, with Rooney *et al.* (2014) stating that these age constraints require additional supporting evidence. Furthermore, the uncertainty on the Re–Os age of the Ben Eagach Schist Formation overlaps with the age of the overlying Tayvallich Volcanic Formation (Fig. 1), leaving open the possibility that it may be a diagenetic rather than a depositional age.

Evidence for correlating the Port Askaig Formation with the global ‘Sturtian’ glaciation is centred on combined litho- and chemo-stratigraphic evidence. For example, the strontium and carbon isotope compositions of the Garbh Eileach Formation match the isotope composition of the late Tonian ocean worldwide (Prave *et al.* 2009; Sawaki *et al.* 2010; Fairchild *et al.* 2018). This is supported by the discovery of the ‘molar-tooth’ calcite in the Lossit Limestone that constrains these strata to the pre-Cryogenian (Fairchild *et al.* 2018). Furthermore, the presence of iron formations within the Port Askaig Formation (Spencer 1971) is thought to be exclusive to rocks of ‘Sturtian’ age (Macdonald *et al.* 2010). The Dalradian in Ireland is also host to glaciogenic deposits younger than the Port Askaig Formation, which, when calibrated against the global carbon and strontium isotope curves, suggests that the Port Askaig Formation represents the earlier ‘Sturtian’ phase of the Cryogenian (McCay *et al.* 2006). Although the balance of evidence would appear to favour a ‘Sturtian’ assignment for the Port Askaig Formation, there is still no consensus (e.g. Moles and Selby 2023).

Sample collection and analytical methods

Eleven sandstone samples were collected across three locations in the Scottish and Irish Dalradian belt (Fig. 1 and Table 1). We chose to only analyse the zircon populations of sandstones in the hope that changes in the detrital zircon U–Pb spectra could be related to landscape evolution throughout the glaciation, rather than the different glacial settings of the diamictites. Seven samples spanning the Garbh Eileach Formation and Members 1–3 of the Port Askaig Formation were collected from the Garvellach Islands, Scotland (samples SMB, GRMS, M1S1, M2S1, M3S2, M3S3 and M3S4), three samples spanning Members 4 and 5 of the Port Askaig Formation were collected from Croaghan Hill, North Donegal, Ireland (samples M4S1, M5S1 and M5S3) and a single sample was collected from Member 3 of the Port Askaig Formation on Islay, Scotland (sample PAM3). The geographical locations of the samples are given in the Supplementary materials.

Mineral separation, imaging and analysis were completed at the London Geochronology Centre, University College London. Zircon separates were prepared from *c.* 5 kg of sample using heavy liquid and magnetic separation techniques. The separates were mounted in epoxy and polished and then the zircons were analysed using an Agilent 7900 laser ablation inductively coupled plasma mass spectrometer. A 25 µm spot operating at 10 Hz and *c.* 2.2 J cm^{-2} fluence was used. Isotopic ratios were reduced with GLITTER 4.4.2 software (Griffin *et al.* 2008) using Plešovice zircon (Sláma *et al.* 2008) as a primary age standard and GJ-1 (Jackson *et al.* 2004) and 91 500 (Wiedenbeck *et al.* 2004) as secondary age standards, yielding average ages of 598.5 ± 2.78 Ma (MSWD 1.3, $n = 22$) and

1054.5 ± 5.9 Ma (MSWD 1.3, $n = 17$), respectively. A NIST SRM612 glass was used as a compositional standard for uranium and thorium concentrations. Concordia ages were calculated as the maximum likelihood intersection between the concordia line and the error ellipse of ^{207}Pb/^{235}U and ^{206}Pb/^{238}U ratios (Ludwig 1998). The discordance cut-off was set at −2.0/+5.8 (Vermeesch 2021*a*). Zircon age data handling, kernel density estimation, plotting and finite mixture model calculations were performed using IsoplotR 5.3 (Vermeesch 2018). The complete dataset is available in the Supplementary data.

Cathodoluminescence imaging of selected grains was used to investigate representative zircon characteristics in the sample set, including magmatic and metamorphic events. The various stages of the zircon's growth (e.g. core and rim) were then analysed via inductively coupled plasma mass spectrometry and are shown in the Supplementary materials.

Zircon age populations

The detrital zircon ages of the 11 sandstone samples display a nearly continuous distribution from the Paleoarchean to the Cryogenian (3.6–0.66 Ga) (Fig. 2). There is often a dominant cluster of Stenian (1.2–1.0 Ga) ages, with Stenian zircons present in all samples in different proportions, as well as emerging Orosirian (2.0–1.8 Ga) and Neoarchean (2.8–2.5 Ga) populations, and mostly an absence of values from the Rhyacian to Siderian interval (2.5–2.05 Ga). Concordia diagrams show an outstanding number of concordant ages (74–90%), demonstrating the minor influence of metamorphic overgrowth and multiple domains in the zircon structures (Supplementary materials). A higher degree of concordance is noticeable in the Garbh Eileach Formation samples as well as in the upper section of the Port Askaig Formation. Discordant ages in the lower and middle section of the Port Askaig Formation align along a discordia line between Paleoproterozoic and Stenian ages, testifying to possible sourcing from reworked old terranes. The central ages and proportions of the dominant detrital zircon age clusters within each sample are displayed in Table 1, along with the age of each sample's youngest single grain (YSG).

Garbh Eileach Formation (samples SMB and GRMS)

In the pre-glacial Garbh Eileach Formation, Stenian ages form the most common population (32–43%), together with 12–13% Ectasian (1.4–1.2 Ga) and 10–12% Tonian (1.0–0.72 Ga) populations. A main age peak centred at 1030–1020 Ma likely relates to Grenville–Sveconorwegian magmatism (Gower and Krogh 2002; Cawood and Pisarevsky 2017). Minor Orosirian age clusters at *c.* 1900 and 1800 Ma are present in both samples, while Statherian zircons are present mostly in sample SMB. Archean ages are less abundant and younger in sample GRMS than in sample SMB (peak centred on 2710 v. 2620 Ma). The YSGs in samples GRMS and SMB measure 907.6 ± 8.8 Ma (disc = 14.0) and 740.3 ± 12.5 Ma (disc = 2.9), respectively.

Port Askaig Formation Members 1 and 2 (samples M1S1 and M2S1)

The age distribution changes sharply passing into the sandstones in the lower members of the Port Askaig Formation. In particular, the Stenian peak becomes less prevalent (19–14%) and older (1150–1080 Ma). The dominant ages shift from Mesoproterozoic (51% in sample M1S1) to Paleoproterozoic and Archean (63% in sample M2S1). In the lower sample, one Calymmian (*c.* 1.5 Ga) and one Statherian (*c.* 1.75 Ga) age peak are also significant (19–22%), while Ectasian and Archean populations are minor. Zircon ages in the upper sample (M2S1) are instead largely Statherian to Orosirian

Table 1. U–Pb youngest single grain, maximum likelihood age (after Vermeesch 2021a) and the peaks of detrital zircon ages identified in each studied sample

	Sample	Height (m)	YSG (Ma)	2σ	MLA (Ma)	2σ	Cryo	2σ	%	Ton	2σ	%	Sten	2σ	%	Ect	2σ	%	Cal	2σ	%	Sta	2σ	%	Oro	2σ	%	NeoA	2σ	%	PaleoA	2σ	%
Port Askaig Formation	M5S3	1080	662.7	7.8	692.2	8.8	662.7	7.8	1				1003.7	2.6	33	1002.2	3.0	30	1418.7	2.2	18	1726.3	2.6	16				2619.2	7.7	3			
	M5S1	770	673.5	6.7	694.3	7.6	673.5	6.7	1				1026.0	0.9	69	1319.4	2.0	20				1687.5	3.6	9				2664.0	11.5	2			
	M4S1	700	906.5	10.3	919.3	7.2							1039.1	1.5	35	1274.1	1.8	30				1779.8	2.5	21				2658.9	3.9	14			
	M3S4	500	689.8	8.7	707.6	10.1	689.8	8.7	1				1133.5	1.5	34							1747.9	1.7	47				2713.4	3.7	17	3313.6	11.8	2
	M3S3	420	1009.7	10.6	1023.1	10.9							1092.4	3.3	8	1394.4	4.0	8							1833.1	2.2	35	2715.3	2.3	48	3509.6	22.8	1
	M3S2	400	686.5	8.3	696.0	10.1	698.0	5.9	2				1099.2	1.8	27				1491.6	2.6	20				1820.2	2.7	26	2728.2	3.3	26			
	PAM3	310	703.5	8.8	715.2	9.0	710.8	8.4	1				1043.0	1.6	27	1387.3	2.7	21							1809.2	2.6	27	2681.6	3.2	22	3494.0	8.1	3
	M2S1	230	686.9	10.3	706.9	12.2	686.9	10.3	1	957.3	3.3	10	1142.3	2.9	14				1457.0	3.4	14				1834.3	2.3	42	2702.0	4.1	20			
	M1S1	180	697.5	9.8	721.3	11.3	697.5	9.8	1				1076.9	1.8	26				1433.8	2.1	29	1761.0	2.3	32				2721.2	4.4	13			
Garbh Eileach Formation	GRMS	−20	907.6	8.8	922.8	7.6							1020.2	1.6	36	1184.2	2.4	26	1431.2	3.8	12				1860.2	3.3	14	2621.9	4.5	12			
	SMB	−25	740.3	12.5	793.8	14.4							1036.0	1.6	36	1241.1	2.4	19	1567.2	2.8	17				1906.0	3.6	13	2717.3	4.0	15			

MLA, maximum likelihood age; youngest single grain.Central age, standard deviation and proportions are calculated with IsoplotR 5.3 (Vermeesch 2018), implementing the discrete mixture modelling algorithms of Galbraith and Laslett (1993). Mixture modelling proportions do not represent the dataset results, but rather estimate a model for the population proportion and their central age. Height or stratigraphic thickness is relative to the contact between the Garbh Eileach and Port Askaig Formations. Divisions of geological time were taken from the International Chronostratigraphic Chart 2023/24: Cry, Cryogenian (0.72–0.635 Ga); Ton, Tonian (1.0–0.72 Ga); Ste, Stenian (1.2–1.0 Ga); Ect, Ectasian (1.4–1.2 Ga); Cal, Calymmian (1.6–1.4 Ga); Sta, Statherian (1.8–1.6 Ga); Oro, Orosirian (2.05–1.8 Ga); NeoA, Neoarchean (2.8–2.5 Ga); PaleoA, Paleoarchean (3.6–3.2 Ga).

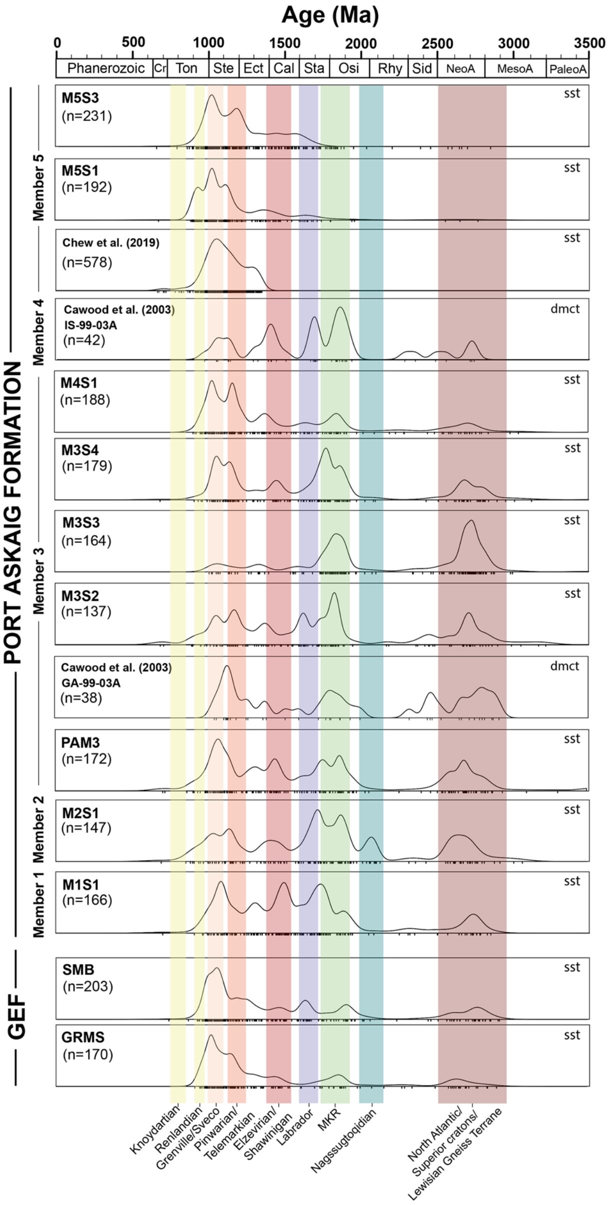

Fig. 2. Kernel density estimation of zircon age populations for the analysed sandstone samples, as well as a Member 3 and 4 diamictite from Cawood *et al.* (2003) and a single Member 5 metasandstone from Chew *et al.* (2019). Only concordant U–Pb ages are shown. Samples are in the relative stratigraphic order. Colour swaths represent possible zircon source regions/orogenic events (see Rivers 1997; Krabbendam *et al.* 2017; Lebeau *et al.* 2020). Divisions of geological time were taken from the International Chronostratigraphic Chart 2023/24: Cr, Cryogenian; Ton, Tonian; Ste, Stenian; Ect, Ectasian; Cal, Calymmian; Sta, Statherian; Osi, Orosirian; Rhy, Rhyacian; Sid, Siderian; NeoA, Neoarchean; MesoA, Mesoarchean; PaleoA, Paleoarchean. MKR, Makkovik–Ketilidian–Rhinnian; GEF, Garbh Eileach Formation; sst, sandstone; dmct, diamictite.

(both 18%), matched with 6% of Rhyacian zircons (nine ages centred at 2085 Ma). One extremely old zircon was found in sample M1S1 of Eoarchean age (3816.8 ± 22.4 Ma disc = 1.3). Two Cryogenian ages of 697.5 ± 9.8 Ma (disc = 3.5) and 686.9 ± 10.3 Ma (disc = 0.6) are also present.

Port Askaig Formation Member 3 (samples PAM3, M3S2, M3S3 and M3S4)

The zircon ages in sandstones in Member 3 of the Port Askaig Formation are remarkably different from those beneath and above as the proportions drift towards older age domains up to a maximum of 84% Paleoproterozoic and Archean ages in sample M3S3. This trend is paired with a decline in Stenian ages, which are only dominant in the lower and upper portion of the section (sample PAM3, 20%; sample M3S4, 24%) and an older age peak (1135–1045 Ma). The Orosirian cluster steadily increases up to 24% before decreasing again at the top of the section and is centred at 1825 Ma. Peaks from the Ectasian and Calymmian populations are stochastically distributed between the samples, whereas a Statherian aged peak is consistently present. Archean ages increase from 20 to 45% and decrease again at the top of the succession, displaying a consistent central age of 2700 Ma, with minor Mesoarchean to Eoarchean ages. Cryogenian zircons are present in all Member 3 samples, aside from sample M3S3 (Table 1). The diamictite sample GA-99-03A included in Cawood *et al.* (2003) is assigned to Member 3 of the Port Askaig Formation and was collected from just below sample M3S2 on the Garvellach Islands. The detrital zircon U–Pb spectra of this diamictite is indistinguishable from the sandstone samples of Member 3 shown in this study (Fig. 2).

Port Askaig Formation Members 4 and 5 (samples M4S1, M5S1 and M5S3)

A sharp increase in Tonian and Mesoproterozoic zircon ages and a decrease in Paleoproterozoic and Archean ages occur in the sandstones in Members 4 and 5 of the Port Askaig Formation. The Stenian population reaches its maximum prominence (sample M5S1, 48%), reverting to a younger age peak (1020 Ma), and is associated with abundant Tonian (16–23%) and Ectasian (13–15%) ages. Calymmian and Orosirian populations are present in samples M5S3 and M4S1, respectively, but generally late Mesoproterozoic to Paleoproterozoic ages disappear. Similarly, the Archean cluster declines from 11 to 2% and consists only of Neoarchean ages. Two Cryogenian ages of 673.5 ± 6.7 (disc = 1.6) and 662.7 ± 7.8 (disc = −1.7) are also present in samples M5S1 and M5S3, respectively.

The diamictite sample IS-99-03A in Cawood *et al.* (2003) was collected from Port Askaig on Islay and can be assigned to Member 4. This sample shares similar zircon ages to sample M4S1 from this study, albeit with an early Paleoproterozoic population present. The single sandstone sample included in Chew *et al.* (2019) was taken from Croaghan Hill, Ireland, at roughly the same stratigraphic level as M5S1. As with the Member 5 sandstone samples from this study, their sample yielded dominant Stenian and Ectasian zircon populations, with a mostly absent Archean population (Fig. 2).

‘Sturtian’ age constraints within the Port Askaig Formation

Detrital zircons are useful for defining MDAs in successions that lack biostratigraphy or zircon-bearing volcanic rocks. The YSG within a detrital population can provide an MDA for the specific horizon within a sedimentary sequence and has shown its utility in ‘Sturtian’ successions globally (Lloyd *et al.* 2023 and references cited therein). Of the 11 samples analysed in this study, eight samples yielded grains with U–Pb ages that fall within the time interval of the ‘Sturtian’ glaciation and/or provide a useful MDA constraint on deposition (Table 1). No grain younger than the onset of the Sturtian glaciation (*c.* 717 Ma) are present in 373 concordant ages from the underlying Garbh Eileach Formation, which has an MDA of 740.3 ± 12.5 Ma based on the YSG across two samples. Similarly, only a single grain from the stratigraphically youngest sample (M3S5) yielded a U–Pb zircon age with an uncertainty that overlaps the termination of the ‘Sturtian’ glaciation (*c.* 658 Ma).

Five of the YSGs from the Port Askaig Formation provide relatively precise and distinct ages that can be used to infer sedimentation post-dating the horizon that yielded the grain (i.e. the MDAs). Based on the YSG from the underlying Garbh Eileach

Formation, an MDA of 740.3 ± 12.5 Ma can be inferred for the Port Askaig Formation and glacial onset. This age constraint is consistent with other geochronological constraints, which suggest that the onset of the 'Sturtian' occurred synchronously at *c.* 717 Ma (Hoffman *et al.* 2017 and references cited therein). An MDA of 697.5 ± 9.8 Ma can be assigned to the top of Member 1 of the Port Askaig Formation at a stratigraphic height of *c.* 180 m. An MDA horizon of 686.9 ± 10.3 Ma constrains the base of Member 2 of the Port Askaig Formation at a stratigraphic height of *c.* 230 m. Additionally, two possible MDA horizons of 673.5 ± 6.7 and 662.7 ± 7.8 Ma are inferred for the base (*c.* 770 m) and upper part (*c.* 1080 m) of Member 5 of the Port Askaig Formation, respectively.

The Cryogenian is generally not considered to be a time of major felsic magmatism in Laurentia and hence no specific magmatic source has been identified for the YSG ages shown in Figure 3. This may suggest that they were derived from airborne tuffaceous fallout, rather than exhumed igneous bodies. Despite this, significant zircon-bearing anorogenic rocks associated with the break-up of Rodinia have been identified across the eastern margin of Laurentia (McClellan and Gazel 2014), which may have fed into the Dalradian basin at the time of Port Askaig deposition. The low count of the youngest detrital grains recorded in each sample may reflect the small size of these magmatic sources and/or be due to the dilution of the grains in large sediment routing systems.

The YSG is susceptible to two forms of negative bias. The first type of bias is a statistical effect whereby extreme values (in this case, the minimum value) of continuous distributions, such as the normal distribution, drift to ever smaller numbers with increasing sample size. Given a sufficiently large sample, this phenomenon may cause the YSG to be younger than the actual depositional age. A second form of negative bias is a geological effect that arises when a sample has undergone partial lead loss. The maximum likelihood age (MLA) of Vermeesch (2021*b*), which is based on an algorithm of Galbraith and Laslett (1993) reduces or eliminates the statistical bias. However, it does not address the lead loss problem. Importantly, the MLA equals the YSG when the youngest tail of a detrital distribution is sparsely sampled and the youngest date is several standard errors removed from the second youngest date. Whether to trust such MLA estimates is a matter of geological, not statistical, debate.

The YSG constraints shown in Figure 3 appear to be robust because they consistently young upwards and adhere to the currently accepted chronology of pre-'Sturtian' and post-'Sturtian' non-glacial strata. Such results are difficult to reconcile with either lead loss or statistical errors. Additionally, the MDA from sample M5S3 defines a horizon close to the last glaciogenic deposit (diamictite) within the Port Askaig Formation. This age (662.7 ± 7.8 Ma) is in good agreement with other global age constraints for 'Sturtian' deglaciation (Lloyd *et al.* 2023 and references cited therein), further validating the YSG approach in this instance.

The consistent younging of YSG values up-section (Fig. 3) implies that the grains crystallized near the time of sedimentation and so their ages may be close to the true depositional age of their respective sandstones (Rossignol *et al.* 2019). Given that the proposed MDA horizons span the duration of the 'Sturtian' glaciation (*c.* 717–658 Ma), this would support the sedimentological evidence for stratigraphic completeness in the Port Askaig Formation (Ali *et al.* 2018). Alternatively, if a stratigraphic break were present within the Port Askaig Formation, we propose that it might be located within Member 1 (0–200 m). The MDA horizon at the top of Member 1 suggests a relatively slow depositional rate of 6–18 m Myr^{-1} across the initial *c.* 200 m of strata, assuming that the base of the Port Askaig Formation can be correlated with the global onset of 'Sturtian' glaciation (*c.* 717 Ma). By contrast, the MDA constraints suggest a faster depositional rate of 19–36 m Myr^{-1} for Members 2–5 (see Supplementary materials).

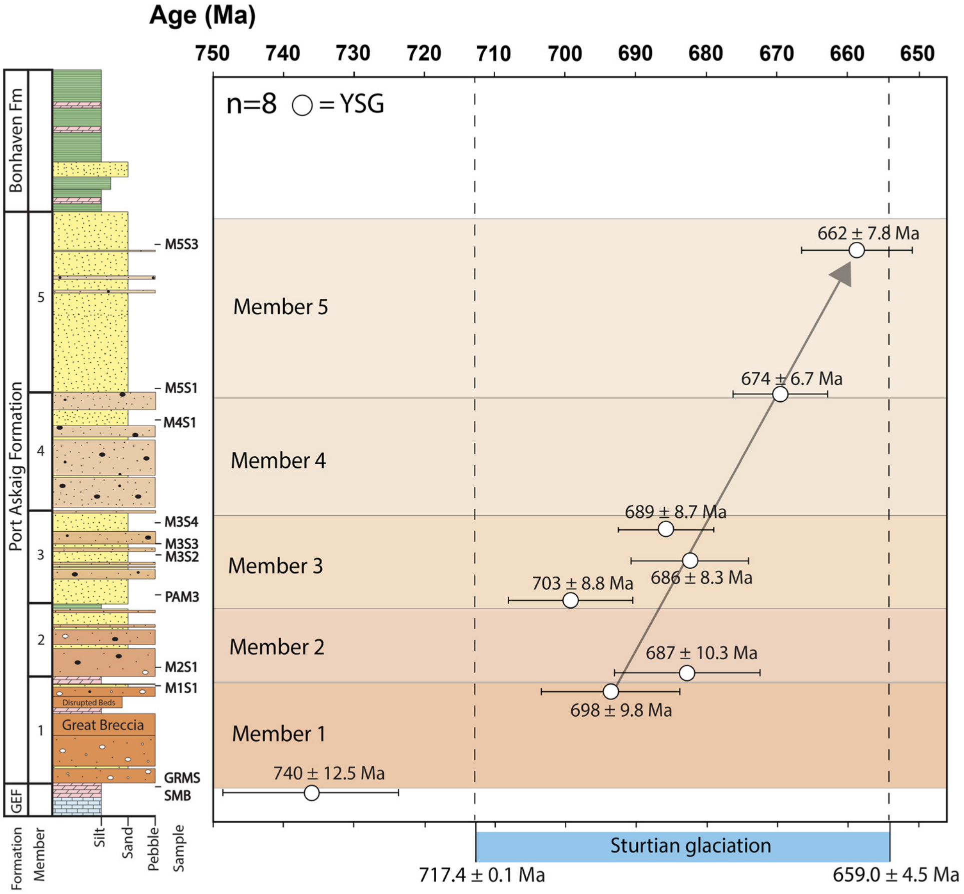

Fig. 3. Youngest single grain ages from eight samples from this study that yielded grains with U–Pb ages that fall within the time interval of the 'Sturtian' glaciation or potentially provide a useful maximum depositional age shown relative to their stratigraphic height. The youngest single grain age from the other three samples are not shown because they are greater than the current maximum age constraint for the Dalradian Supergroup (i.e. >806 ± 3 Ma; Noble *et al.* 1996). The 1100 m thick Port Askaig Formation and 70 m Garbh Eileach Formation stratigraphic column from Figure 1 is shown for reference. The horizontal bars span the different members of the Port Askaig Formation. The youngest single grain ages young up-section (arrow) and fall within the expected framework of pre-glacial and syn-glacial strata for the 'Sturtian' glaciation (717–658 Ma). YSG, youngest single grain.

Overall, the geochronological framework of this study's detrital zircon age spectra strengthens the current geochemical and lithostratigraphic evidence (Fairchild *et al.* 2018) that correlates the Port Askaig Formation with the 'Sturtian' glaciation, rather than the younger 'Marinoan' glaciation of the Cryogenian Period.

A glacially influenced provenance during deposition of the Port Askaig Formation

Glacial unroofing of basement source regions

The broad detrital zircon U–Pb age spectra of the sandstones we have measured in the Port Askaig Formation can be matched with previous studies (Cawood *et al.* 2003; Banks *et al.* 2007; McAteer *et al.* 2010; Strachan *et al.* 2013) and confirm a Laurentian provenance for the Dalradian Supergroup. A summary of the proposed Laurentian craton provenance age ranges can be seen in Figure 2. It is unclear whether the detritus of the Dalradian Supergroup is composed of first-cycle sediments sourced from the more distal Laurentian interior or reworked sediments from the proximal Tonian successions in Scotland. The Dalradian Supergroup is the youngest of three megasequences (Megasequence 3) that make up the Scottish Highlands (Stephenson *et al.* 2013; Olierook *et al.* 2020; Krabbendam *et al.* 2022). The pre-960 Ma Wester Ross Supergroup and the *c.* 950–900 Ma Loch Ness Supergroup (formerly the 'Torridonian' and the 'Moine') make up Megasequences 1 and 2, respectively, and were likely situated to the north of the Dalradian basin at the time of its deposition (Krabbendam *et al.* 2022). The detrital zircon U–Pb age spectra of the Wester Ross and Loch Ness supergroups (Krabbendam *et al.* 2022) share similarities with the Dalradian Supergroup (Fig. 4) in that Proterozoic grains from 1.9 to 0.9 Ga are present consistently throughout, albeit in variable proportions. The sediments of the Wester Ross and Loch Ness supergroups are thought to have formed a widespread blanket across the Scottish Highlands in early Tonian times (Stewart 2002; Krabbendam *et al.* 2017) and would be easily eroded and reworked by fluvial and/or glacial erosion. This would have allowed these proximal sedimentary successions to become major sources to the Port Askaig Formation and the Dalradian Supergroup. A high chemical index of alteration in the lower diamictites of the Port Askaig Formation, typical of reworked material, supports this (Panahi and Young 1997).

Archean grains are mostly absent in the Grampian Group of the Dalradian (Cawood *et al.* 2003; Banks *et al.* 2007) and are also scarce within the older Wester Ross and Loch Ness supergroups (Krabbendam *et al.* 2022). By contrast, Archean grains emerge by the late Appin Group and increase in proportion from the Garbh Eileach Formation to Member 3 of the Port Askaig Formation, alongside late Paleoproterozoic to early Mesoproterozoic grains (Fig. 2). Most of the Archean grains in the sandstones of the Port Askaig Formation fall between 2.8 and 2.6 Ga, which matches Lewisian Gneiss terrane ages (Friend and Kinny 2001; Kelly *et al.* 2008; Love *et al.* 2010). The detrital age spectrum from Members 1 to 3 of the Port Askaig Formation also becomes increasingly similar to the *c.* 1.2 Ga Stoer Group (Fig. 4), which was predominantly sourced from the basement Lewisian Gneiss Complex (Lebeau *et al.* 2020).

The 'Sturtian' glaciation represents a period of up to *c.* 58 Ma of widespread ice cover. The alternations of glacial diamictite and non-glacial sandstone throughout the Port Askaig Formation (Fig. 1) record glacial retreat and re-advance cycles within this time. It therefore seems unlikely that cold-based ice sheets prevailed throughout the entire 'Sturtian', but rather that large polythermal ice sheets existed that caused significant erosion during periods of warm-based ice (e.g. Donnadieu *et al.* 2003). The presence of Archean grains in the Port Askaig Formation therefore presents glacial erosion as a plausible mechanism by which the blanket of the Wester Ross and Loch Ness supergroups was cut through (unroofed) and the underlying Lewisian Gneiss eroded. Glaciation tends to concentrate in fjords and overdeepenings, progressively

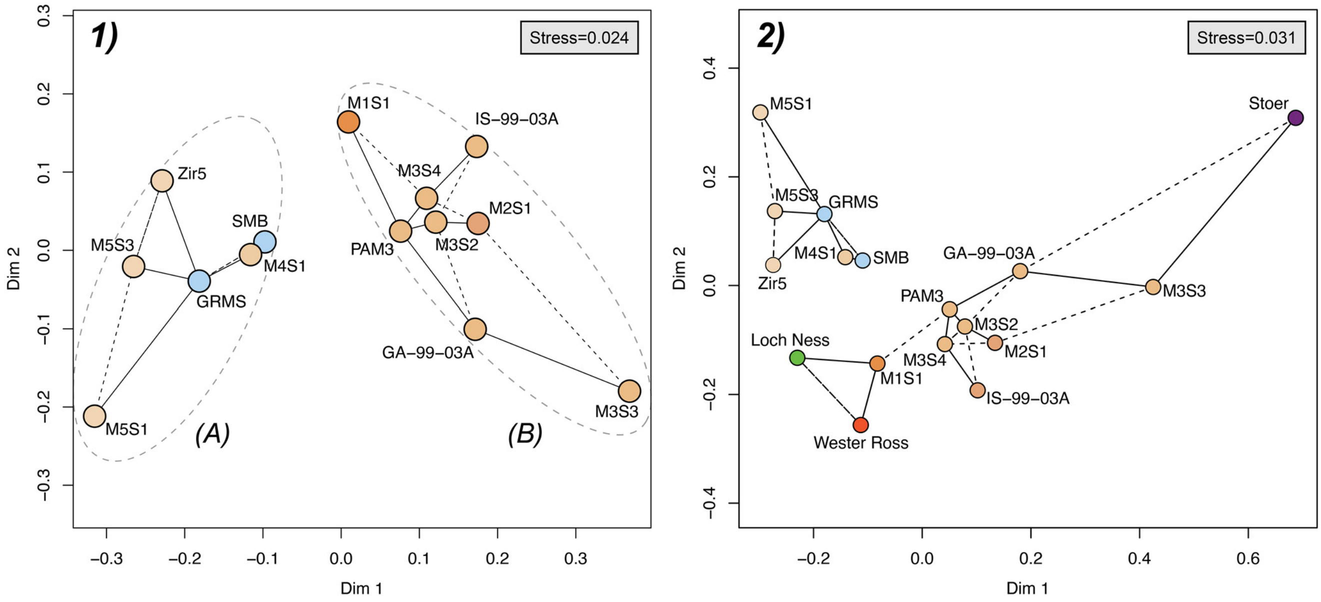

Fig. 4. Multidimensional scaling plots using the Kolmogorov–Smirnov statistic as a dissimilarity metric (Vermeesch 2013). The axis scales are dimensionless and have no physical meaning. The closer the samples plot together, the more similar their detrital zircon age spectra. A solid line links the nearest neighbour and a dashed line the second nearest. The goodness of fit is evaluated using the 'stress' value of the configuration (0.2 = poor; 0.1 = fair; 0.05 = good; see Vermeesch 2013, table 1). (1) Comparing 11 sandstone samples from the Garbh Eileach Formation and 'Sturtian' Port Askaig Formation (this study), alongside a Member 3 and 4 diamictite from Cawood *et al.* (2003) and Member 5 sandstone from Chew *et al.* (2019). Two groups are statistically highlighted: group A (Members 1, 2 and 3 of the Port Askaig Formation), where there is sedimentological evidence for relatively more frequent and longer lived glacial episodes than group B (Garbh Eileach Formation and Members 4 and 5 of the Port Askaig Formation). (2) Comparing the detrital zircon U–Pb data from the Garbh Eileach Formation and Port Askaig Formation with the Tonian Wester Ross and Loch Ness supergroups (Krabbendam *et al.* 2022 and references cited therein) and the Stoer Group, sourced predominantly from the Lewisian Gneiss terrane (Lebeau *et al.* 2020).

widening and deepening them with time (Egholm *et al.* 2017). The increasing Archean proportion of the detrital age spectra throughout Members 1–3 of the Port Askaig Formation may reflect this process (Fig. 2). Furthermore, the chemical index of alteration of the diamictites decreases up-section in the Port Askaig Formation (Panahi and Young 1997), indicating a shift from mature, reworked sediment to more immature source terranes, thus strengthening the case for glacial unroofing. Overall, it seems probable that glaciation was the primary mechanism influencing up-section changes in the detrital zircon spectra of the sandstones.

Post-glacial provenance

The distribution of Archean and late Paleoproterozoic to early Mesoproterozoic grains in the sandstones through the Port Askaig Formation presents a conundrum. The proportion of these grains gradually increases from Members 1 to 3, before decreasing through to Member 4 and becoming entirely absent in Member 5. If these grains are present due to the unroofing of Archean and late Paleoproterozoic to early Mesoproterozoic source regions, why are these grains not present in the samples from the final 300 m of Port Askaig Formation strata?

Member 5 records the waning phase of 'Sturtian' glaciation with less frequent and thinner glacial units (diamictites) (Fig. 1) and this is consistent with YSG ages that suggest deposition within the final <13 ± 6.7 Myr of the accepted 'Sturtian' interval (Fig. 3). The change in provenance, shown by the absence of Archean and late Paleoproterozoic grains from Member 5, suggests a significant modification of the landscape by the 'Sturtian' glaciation.

The complete erosion of the Archean and late Paleoproterozoic to early Mesoproterozoic source regions by the time of Member 5 is unlikely given the time frame available between glacial onset and retreat, but also because detritus that spans this time interval returns within the detrital zircon record higher up in the Dalradian Supergroup (Cawood *et al.* 2003). It is also unlikely that the source regions were separated from the basin by tectonics or a topographic barrier because no sedimentological evidence for an unconformity exists between Members 4 and 5 (Ali *et al.* 2018). We therefore regard it to be more probable that glacial erosion significantly changed sediment pathways, altering the post-glacial provenance of the Dalradian.

Several plausible scenarios exist that can explain the change in sediment routing on Laurentia following peak 'Sturtian' glaciation. First, the incised fjords and overdeepenings, which likely unroofed the Lewisian Gneiss and other Archean rocks, may have eroded below base level, creating intracontinental basins with the potential to trap large volumes of sediment (Preusser *et al.* 2010; Cook and Swift 2012; Egholm *et al.* 2017). Deep incisions of this kind are prevalent in modern and recently glaciated environments (e.g. Larson and Schaetzl 2001; Preusser *et al.* 2010), but have also been found in association with 'Sturtian' glacial deposits in South Australia (Mitchell *et al.* 2019). Second, a subtle change in ice dynamics may have led to the rerouting of ice streams and therefore a different provenance for the Dalradian post-glaciation. The rerouting and cessation of ice streams is a common occurrence across Antarctica (e.g. Fahnestock *et al.* 2000). Third, the scarcity of Archean and late Paleoproterozoic detritus in Member 5 may be due to a reconfiguration of drainage divides in a post-glacial landscape. Glaciations are known to transport material across fluvial drainage divides, reorganizing the entire drainage architecture, which are then 'shut-off' again as the ice disappears. Such processes are commonly documented in Mid- to Late Pleistocene glaciations across northern Eurasia (see Panin *et al.* 2020). Overall, the proposed change in sediment routing on Laurentia during the waning of the 'Sturtian' further highlights the dynamism of this glaciation.

Conclusions

Sandstones from the uppermost Tonian Garbh Eileach Formation and early Cryogenian glaciogenic Port Askaig Formation are characterized by detrital zircon populations ranging from *c.* 3700 to 660 Ma, which can all be attributed to Laurentian source rocks.

The large number of zircons analysed per sample within this study has revealed five MDA constraints for the Port Askaig Formation. The youngest single concordant zircons within each sample young upwards and their ages span the currently accepted time interval of the 'Sturtian' glaciation. This evidence suggests that the young ages are not the product of lead loss or statistical errors associated with the large sample set, but confirm that the deposition of the Port Askaig Formation spanned most of the 'Sturtian' ice age. A key MDA constraint (662.7 ± 7.8 Ma), which is close to the last sedimentological evidence for glaciation within the Port Askaig Formation, overlaps with other global geochronological constraints for 'Sturtian' deglaciation, further validating the youngest single zircon constraint.

Major stratigraphic changes in the detrital zircon ages of the sandstones in the Port Askaig Formation reflect major changes in source provenance and/or sediment routing and thus suggest major changes in the source landscape. An increased input of Archean and late Paleoproterozoic to Mesoproterozoic grains across the Garbh Eileach Formation and initial *c.* 500 m of the Port Askaig Formation (Members 1–3) is interpreted as reflecting the progressive glacial unroofing of the Lewisian Gneiss terrane that was previously blanketed by the Tonian Wester Ross and Loch Ness supergroups. The then near-complete disappearance of the Archean and late Paleoproterozoic to Mesoproterozoic grains in the uppermost *c.* 350 m of the Port Askaig Formation, as glaciation appears to wane, is attributed to the glacial modification of sediment dispersal pathways, which trapped or diverted detritus from these older, recently unroofed source regions. The changes in transport pathways suggested by the zircon evidence show that 'Sturtian' ice sheets were at least partially warm-based and dynamic to some degree.

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Data availability

All data generated or analysed during this study are included in this published article (and if present, its Supplementary information files).

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