Coupling of the atmosphere and sediment melts across the Archean-Proterozoic transition

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Article

Keywords: Great Oxygenation Event, sulfur isotopes, oxygen isotopes, Earth system

Posted Date: August 17th, 2020

DOI: <https://doi.org/10.21203/rs.3.rs-55162/v1>

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Abstract

The Archean-Proterozoic transition marks a time of fundamental geologic, biologic, and atmospheric changes to the Earth system, including oxygenation of the atmosphere (termed the Great Oxygenation Event; GOE), and the emergence of continents above sea-level. The impacts of the GOE on Earth's surface environment are imprinted on the geologic record, including the attenuation of mass-independent fractionation of sulfur isotopes (S-MIF). Temporally overlapping geologic and geochemical observations (e.g. a change in oxygen isotope ratio of sediment melts) imply the widespread subaerial emergence of continents was coeval with atmospheric oxygenation. Here we present triple sulfur isotope ratios in pyrite and oxygen isotope ratios in garnet and zircon in a global suite of Archean and Proterozoic sediment-derived granitoids. These crustal melts record an increase in average $^{18}\text{O}/^{16}\text{O}$ isotope ratio and a disappearance of S-MIF in the Paleoproterozoic. The coupled behaviour of sulfur and oxygen isotope signatures imply a potential causal link between the emergence of continents and atmospheric oxygenation at ~ 2.3 Ga.

Introduction

The oxygenation of the Earth's atmosphere and oceans irreversibly changed many major biogeochemical cycles (e.g. Fe, S, Mn) ¹ and provided the base for a highly efficient aerobic metabolism that allowed the development of complex life ². During the Great Oxygenation Event (GOE) (c. 2.3 Ga) atmospheric oxygen increased from $<0.001\%$ of the present atmospheric level (PAL) ³ to 10-40% PAL ⁴, or perhaps much higher approaching 100% PAL ⁵, and references therein. The mechanism that led to atmospheric oxygenation remains controversial. Proposed scenarios for oxygenation include 1) an increase in O_2 production (i.e. through the emergence of oxygenic photosynthesis) ⁶, 2) a decrease in O_2 consumption (e.g. through changing redox state of volcanic gases, increased burial of organic carbon, or decreased pyrite weathering) ^{7,8}, and 3) a combination of both processes (through enhanced oxygenic photosynthesis combined with increased carbon burial) ⁹. One source of dispute is the timing of the invention of oxygenic photosynthesis; suggestions range from >3.7 Ga ¹⁰ to immediately preceding atmospheric oxygenation ⁶. Previous studies imply that the widespread emergence of continents above sea-level is temporally correlated with atmospheric oxygenation ¹¹. Furthermore, it has been proposed that the subaerial emergence of continents may have led to a flux of life-essential nutrients into the ocean supporting a boost in photosynthetic activity ¹².

Multiple sulfur isotopic signatures are a sensitive tracer for atmospheric oxygen levels ¹³. Some sulfides and sulfates in metasedimentary rocks deposited prior to the GOE display mass-independent fractionation of sulfur (S-MIF), whereas those deposited after the GOE display almost wholly mass-dependent fractionation (S-MDF) ^{13,14}. It has been postulated that S-MIF was generated in the atmosphere through ultraviolet photolysis of gas molecules ¹⁵. The establishment of an ozone shield as a consequence of atmospheric oxygenation led to blocking of UV radiation and attenuation of the

photolysis of volcanic sulfur species ¹³. In addition, enhanced oxidative weathering of sulfides and the formation of oceanic sulfates stimulated the activity of sulfur-metabolizing bacteria supporting the generation of S-MDF signatures ¹⁶. Recent studies show that fluctuations in atmospheric oxygen level are also captured in the igneous rock record; namely, through recycling of S-MIF in the crust ^{17,18}, and a change in oxygen fugacity of strongly peraluminous granites ¹⁹.

Broadly coeval with atmospheric oxygenation, the average oxygen isotope ratio of global felsic magmas (recorded by zircon $\delta^{18}\text{O}$) increases ²⁰. The oxygen isotopic composition of a magma is sensitive to the recycling of supracrustal material ²¹. Igneous zircon in high-temperature equilibrium with the mantle has remarkably homogenous d^{18}O values which average $5.3 \pm 0.6\text{‰}$ ²² relative to standard mean ocean water (VSMOW). Supracrustal material (e.g. sedimentary and volcanic rocks), in contrast, has a wide range of d^{18}O values ($\sim 0\text{--}40\text{‰}$) with most reservoirs being elevated in d^{18}O relative to the mantle ²³. Hence, assimilation of supracrustal material commonly results in magmas with elevated d^{18}O compared to primitive mantle-derived magmas. An increase in average $\delta^{18}\text{O}$ of zircon in the Paleoproterozoic has been recognized for over a decade ²⁰. Contrasting models have been suggested to account for this increase in $\delta^{18}\text{O}$; 1) the formation of high $\delta^{18}\text{O}$ sediments (and sediment-derived melts) potentially linked to enhanced subaerial weathering and erosion related to the emergence of continents above sea-level ^{24,25}, and 2) enhanced crustal recycling associated with the onset of collisional tectonics ²⁶. However, high- $\delta^{18}\text{O}$ sediment melts do not show a concomitant depletion in radiogenic ^{176}Hf , as would be expected if the increase in $\delta^{18}\text{O}$ was related to enhanced supracrustal recycling ²⁵. Therefore, a change in sediment oxygen isotope composition potentially associated with the subaerial emergence of continents seems to be the likely driver for the Paleoproterozoic increase in average zircon $\delta^{18}\text{O}$ ²⁵.

We explore a potential link between the emergence of continents above sea-level and atmospheric oxygenation through a coupled analysis of proxies for atmospheric oxygen level and sedimentary recycling in Archean to Mesoproterozoic sediment-derived granitoids (i.e. granitoids that partially or wholly derive from the partial melting of metasediments). Here we present pyrite multiple sulfur isotope ratios in tandem with zircon and garnet oxygen isotope ratios for a global sample set. The combination of these proxies allows us to provide new insights into the coupled behaviour of geodynamic, biogenic, and atmospheric evolution.

Results

Bulk-rock geochemistry and mineralogy

Samples include granitoids comprised of quartz + alkali feldspar + plagioclase $\pm$ biotite in varying proportions, and contain one or more peraluminous indicator minerals, such as garnet or muscovite. The samples for which bulk-rock geochemical data is available are strongly peraluminous with an aluminium saturation index (ASI) ≥ 1.1 (defined as molecular $\text{Al}/[\text{Ca} - 1.67\text{P} + \text{Na} + \text{K}]$). For comparison ACNK values (defined as molecular $\text{Al}/[\text{Ca} + \text{Na} + \text{K}]$) are also reported in Table 2. With the exception of sample 15K-2,

ASI and ACNK yield identical values to the first decimal place. The mineralogy of all samples is summarized in Table 1. Bulk rock major element concentrations are given in Table 2. Thin section photomicrographs can be found in supplementary Figure A4.

Geochronology

Magmatic crystallization ages of the 30 sediment-derived granitoids in this study are Neoarchean to Mesoproterozoic, ranging from 2664 ± 45 Ma to 1447 ± 50 Ma. For nine of these samples the magmatic crystallization age was determined in this study (as zircon concordia, upper intercept, or weighted mean $^{207}\text{Pb}^*/^{206}\text{Pb}^*$ ages). For nine samples zircon U-Pb SIMS ages were determined in previous studies²⁵. For the remaining 12 samples for which no or only metamict zircon was extracted, preferred ages use robust published dates from the same batholith or are estimates based on the age of proximal magmatism. A detailed description of the geochronology is given in Appendix A; a summary is given in Table 3. Single-spot zircon U-Pb results can be found in supplementary Table B1.

Zircon and garnet oxygen isotope geochemistry

Zircon $\delta^{18}\text{O}$ values range from $4.93 \pm 0.82\text{‰}$ to $11.41 \pm 1.00\text{‰}$. Similarly, garnet $\delta^{18}\text{O}$ values range from $7.06 \pm 0.09\text{‰}$ to $11.50 \pm 0.12\text{‰}$. Note, that no garnet was extracted from the two samples with the lowest zircon $\delta^{18}\text{O}$ values (19GH9, and 19GH11B). Zircon records a lower range of $\delta^{18}\text{O}$ values for samples with crystallization ages >2.3 Ga ($2\sigma=0.5\text{‰}$, number of samples=3) as compared to samples with crystallization ages <2.3 Ga ($2\sigma=4.1\text{‰}$, $n=12$), whereas the range of garnet $\delta^{18}\text{O}$ stays approximately the same (>2.3 Ga garnet: $2\sigma=1.2\text{‰}$, $n=10$; <2.3 Ga garnet: $2\sigma=1.1\text{‰}$, $n=10$). On average $\delta^{18}\text{O}$ values increase post-2.3 Ga; from 7.15‰ to 9.08‰ in zircon, from 7.22‰ to 10.24‰ in garnet, and from 7.19‰ to 9.30‰ combining the data of both zircon and garnet. Equilibrium fractionation of oxygen isotopes between zircon and almandine-rich garnet is small at temperatures typical for granitoid melts ($<0.1\text{‰}$ at temperatures >650 °C)²⁷. The garnet-zircon pairs of all samples indicate oxygen isotopic equilibrium (Figure 1). Two samples (17FIN02 and 17FIN04A) yield heterogeneous single spot zircon $\delta^{18}\text{O}$ values ($2\sigma > 3\text{‰}$) and are interpreted to reflect secondary signatures²⁸. This is further supported by CL images revealing that some areas in some zircon grains are affected by metamictization (supplementary Figure A2). Therefore, the oxygen isotopic composition recorded by garnet from these two samples provides the best estimate of their parental magma $\delta^{18}\text{O}$. Data tables with single spot O isotopic data are given in the supplementary Table D1, a summary of weighted means is given in Table 3.

Sulfur isotope geochemistry

Pyrite grains are euhedral to sub-euhedral, chemically homogenous, and largely free of inclusions and intergrown phases. BSE images of representative pyrite grains for each sample are given in the supplementary Figure A3. Pyrite $\delta^{34}\text{S}$ values range from $-13.33 \pm 0.36\text{‰}$ to $9.72 \pm 0.99\text{‰}$, but are mostly (11 out of 13 samples) between -4‰ and 4‰ . Single spot pyrite $\delta^{34}\text{S}$ and $\Delta^{33}\text{S}$ values cluster tightly around discrete values for each sample, and seemingly define single populations (Figure 2). Four

samples show pyrite $\Delta^{33}\text{S}$ outside of the S-MDF range (Figure 2). These samples include three ~ 2.7 Ga granites from the Superior province that exhibit positive $\Delta^{33}\text{S}$ values of $0.13 \pm 0.06\text{‰}$ to $0.18 \pm 0.05\text{‰}$, and a ~ 2.5 Ga granite from the North China Craton with negative $\Delta^{33}\text{S}$ value of $-0.29 \pm 0.12\text{‰}$. Data tables with single spot sulfur isotopic data are given in the supplementary Table C1, a summary of weighted means is given in Table 3.

Discussion

The presence of aluminous mineral phases and/or $\text{ASI} \geq 1.1$ strongly suggest that the studied granitoids were derived from the partial melting of metasedimentary protoliths^{29,30}. The partial or entire derivation of these granitoids from metasedimentary protoliths is in accord with previous interpretations of the regional geology^{31–34}. Zircon and garnet tend to preserve a record of the oxygen isotope composition of their parental melt due to slow intracrystalline diffusion rates of oxygen in these minerals^{35,36}. Given the small equilibrium fractionation (smaller than the analytical uncertainty) of oxygen isotopes between almandine-rich garnet and zircon at temperatures typical for granitoid melts²⁷ (Figure 1) $\delta^{18}\text{O}$ values recorded by garnet and zircon, respectively, in the sediment-derived granitoids of this study are directly comparable. The range and average $\delta^{18}\text{O}$ values of sediment-melts increases post-2.3 Ga (Figure 3), in accord with previous studies that interpret the rise of average $\delta^{18}\text{O}$ to be the result of continental emergence and a concomitant change in sediment composition^{24,25}. Low $\delta^{18}\text{O}$ values in sediment melts (equal or lower than the mantle value of $5.3 \pm 0.6\text{‰}$;²², like in 19GH9 and 19GH11B, are interpreted to reflect hydrothermal alteration of the protolith³⁷. It has been proposed that interaction of a melt with hydrothermal meteoric water – a mechanism that requires emergent land area – may be the process responsible for such low $\delta^{18}\text{O}$ melts³⁷. The emergence of continents at ~ 2.4 Ga is supported by changes to geochemical proxies at this time, such as an increase in seawater $^{87}\text{Sr}/^{86}\text{Sr}$ ³⁸, a decrease in shale $\Delta^{17}\text{O}$ ^{24,39}, as well as an increase in subaerial large igneous province volcanism¹¹.

Pyrite-bearing sediment-derived granitoids with crystallization ages > 2.3 Ga yield non-zero $\Delta^{33}\text{S}$ values, whereas those younger than 2.3 Ga uniformly show $\Delta^{33}\text{S}$ of 0‰. Although the $\Delta^{33}\text{S}$ values of the > 2.3 Ga granitoids are small compared to those in pre-GOE sedimentary rocks (up to $\sim 12\text{‰}$)¹⁴, the magnitude of non-zero $\Delta^{33}\text{S}$ values observed here is too large to be the result of S-MDF processes alone (Figure 2). Hence, the S-MIF signatures in the > 2.3 Ga pyrite-bearing granitoids record the recycling of sedimentary sulfur species formed under the anoxic pre-GOE atmosphere, as has been demonstrated for S-MIF carrying strongly peraluminous granites in previous studies¹⁸. Three of the > 2.3 Ga granitoids of this study show positive $\Delta^{33}\text{S}$ values, one sample shows a negative $\Delta^{33}\text{S}$ value. The pre-GOE pyrite record is skewed towards positive $\Delta^{33}\text{S}$ values¹⁴. Positive $\Delta^{33}\text{S}$ anomalies are commonly found in Archean sedimentary pyrite that may have formed from reduced sulfur species (e.g. S_8 aerosols) produced through photodissociation in the oxygen-poor atmosphere^{13,40}. Based on the dominance of negative $\Delta^{33}\text{S}$ values in Archean barites, it has been posited that oceanic sulfates (formed from oxidized sulfur

species produced through photodissociation; e.g. SO_4 aerosols) carry the complementary negative $\Delta^{33}\text{S}$ signatures required by isotopic mass balance¹³. However, recent studies report positive $\Delta^{33}\text{S}$ anomalies in Archean oceanic sulfate⁴¹. The inconsistent sulfur isotopic budget is a yet unsolved scientific problem, and the complementary negative $\Delta^{33}\text{S}$ reservoir remains cryptic.

The low $\delta^{34}\text{S}$ value ($\sim -13\text{‰}$) of pyrite in sample SP-17-43 from the Wenasaga Lake batholith, Superior province, could derive from the contribution of organic sulfur. Microbial sulfate reduction commonly produces sulfides that are strongly depleted in the heavy ^{34}S isotope⁴². The S-MIF signature in this sample ($\Delta^{33}\text{S} = 0.16 \pm 0.04\text{‰}$) indicates atmospheric influence. Therefore, the recorded sulfur isotopic composition of this sample may be the result of mixing between two reservoirs (i.e. atmospheric and microbial sulfur). A similar scenario has been suggested to be responsible for the negative $\delta^{34}\text{S}$ values and positive $\Delta^{33}\text{S}$ values of pyrite in the upper Mount McRae shale in the Hamersley Basin, Western Australia⁴³. Alternatively, the low $\delta^{34}\text{S}$ value in this sample could arise during fractionation between S^{2-} in the melt and H_2S in a vapor phase (produced through metamorphic devolatilization) as documented for other samples from the Superior Craton¹⁸.

The youngest sediment-derived granitoid of this study that shows S-MIF and oxygen isotopic signatures respectively implying an anoxic atmosphere and widely submerged continents (sample 18IM19) has a crystallization age of 2478 ± 18 Ma. The oldest sample (19GH11B) showing only S-MDF and O isotopic signatures indicating respectively an oxygenized atmosphere and emergent continents yields a crystallization age of 2188 ± 20 Ma. The evolution of oxygen and sulfur isotopic signatures appear to be coupled in the samples of this study. However, the data is inconclusive for the ~ 300 Myr interval between the youngest S-MIF and the oldest S-MDF sample. The preserved rock record between 2.4 and 2.2 Ga is sparse^{44,45} making it challenging to fill this temporal gap. If the evolution of sulfur and oxygen isotopic signatures in sediment-derived melts is truly coupled, this would provide strong evidence for a temporal, and potentially causal connection of the emergence of continents and atmospheric oxygenation.

According to Warke et al. (2020)⁴⁶ atmospheric oxygenation preceded widespread, potentially global, glaciations. This conclusion is based on sedimentary formations on the Fennoscandian shield, in which the transition from S-MIF to S-MDF-bearing strata occurs below the diamictite member of the formations linked to Makganyene glaciation⁴⁶. This temporal relationship between atmospheric oxygenation and widespread glaciations rules out models in which Proterozoic ice houses facilitated the invention of oxygenic photosynthesis⁴⁷, and hence, calls for an alternative driver for the rise of O_2 . Numerical models demonstrate the strong influence of subaerial continental emergence on phosphorous fluxes to the oceans¹². Elevated, emerged continental crust and associated enhanced erosion would increase the supply of life-essential nutrients into the ocean, increasing that available for oxyphotobacteria^{9,12}. It has been suggested that a similar mechanism involving the erosion of elevated continental crust has initiated the explosive radiation of animal-life during the Late Ediacaran to Early Cambrian periods (575-510 Ma; 'Cambrian explosion')⁴⁸. A flush of nutrients associated with enhanced erosion may have led to an

increase in O₂ production and provided a favourable environment for the explosive diversification of life⁴⁹. Similarly, an increased nutrient flux (e.g. phosphorous) to the oceans associated with the Paleoproterozoic subaerial emergence of continents may have fuelled a boost in oxygenic photosynthesis (either in terms of radiation or bioproductivity)⁵⁰. A shift in ocean nutrient availability from phosphorous to iron limiting may have resulted in ecological conditions favourable for oxygenic photoautotrophs over anoxygenic photoautotrophs⁵¹. Furthermore, increased erosion and sediment supply from the elevated continents would facilitate the burial of organic carbon leading to diminished O₂ consumption⁹.

Potential causes of the Paleoproterozoic subaerial emergence of continents are a ~2.4-2.2 Ga tectono-magmatic slowdown^{44,45}, or the effect of dynamic topography⁵² during the ~2.45 Ga rifting of Superior⁵³. The proposed ~2.4-2.2 Ga lull in mantle activity and concomitant reduced plate-velocities⁴⁴ and mid-ocean ridge activity⁵⁴ could have led to cooling and thickening of the oceanic lithosphere causing subsidence of the ocean floor and a drop in eustatic sea-level^{54,55}. Mantle upwelling during the ~2.45 Ga rifting of Superior⁵³ and associated dynamic topography would likely have led to continental uplift and high freeboard⁵².

The implication of our study is that the subaerial emergence of continents constitutes a potential driver for ecological changes that fuelled oxygenic photosynthesis, ultimately leading to a change in redox state of the Earth's atmosphere and oceans.

Materials And Methods

Sample context

Samples of this study derive from various localities within the Superior, North China, West African, and East European Cratons, and the Yavapai province (USA). A list of all samples including location is given in Table 1.

Superior Craton

The Superior Craton forms the Archean core of the Canadian Shield and can be divided into the Western and Eastern Superior Province, which are further subdivided into 17 distinct tectonic terranes³¹. (Meta-)granitoid- and greenstone-dominated terranes evolved independently from 3.7-2.75 Ga, followed by accretionary events associated with the trapping of sedimentary basins and high-temperature metamorphism, leading to the formation of a coherent Superior Craton by 2.60 Ga³¹. Peraluminous granites are widespread within the Superior Craton, and their occurrence is described in detail by⁵⁶.

North China Craton

The North China Craton comprises Archean to Paleoproterozoic basement overlain by Mesoproterozoic to Cenozoic cover sequences³². Recent publications subdivide the Precambrian basement of this region into three tectonic domains; Archean to Proterozoic Western and Eastern Blocks and a central Paleoproterozoic collisional Trans-North China Orogen³². The Western Block comprises the Yinshan Block in the north and the Ordos Block in the south. The Neoarchean Yinshan Block comprises tonalite-trondhjemite-granodiorite (TTG) gneisses and minor supracrustal rocks that were metamorphosed at ~2.5 Ga⁵⁷.

Baoulé-Mossi domain, West African Craton

The Paleoproterozoic (~2.3-2.0 Ga) Baoulé-Mossi domain in the southern portion of the West African Craton comprises sedimentary basins and volcanic/volcaniclastic rocks that are intruded by multiple generations of granitic rock³³, and references therein. The emplacement of felsic intrusions across the Baoulé-Mossi domain is associated with the 2.2-1.8 Eburnean Orogeny⁵⁸. No agreement has been reached upon the tectonic model for the Paleoproterozoic evolution. Proposed scenarios include plume-related, subduction-related, and continental collision-related models³³, and references therein.

Ukrainian Shield, East European Craton

The Ukrainian Shield is a region of exposed Archean and Proterozoic crust within Samaria in the southwestern part of the East European Craton. The Ukrainian Shield is comprised by several tectonic blocks separated by suture zones described in detail by⁵⁹. High grade metamorphism and associated magmatism occurred in multiple domains of the Ukrainian Shield at ~2.8 and 2.1-2.0 Ga and may reflect an active-margin setting⁵⁹.

Svecofennian domain, Baltic Shield, East European Craton

The Svecofennian Orogeny in Finland between 1.91 and 1.87 Ga involved accretion of island arc complexes and older crustal fragments to the Archean basement of the Karelian Craton³⁴. In southernmost Finland, ~2.0-1.8 Ga old crust was intruded by sediment-derived granites at 1.84-1.83 Ga associated with high-temperature, low-pressure granulite facies metamorphism and migmatization⁶⁰. This complex, referred to as the late Svecofennian granite-migmatite zone, includes the Sulkava, West Uusumäki, and Turku areas⁶¹. Granitic material is abundant in the Turku area. Mostly, these magmas occur as garnet and cordierite bearing leucosome in magmatic metapelites³⁴.

Yavapai province

The Yavapai province (or Colorado province) south of the Wyoming Craton comprises ~1.79-1.66 Ga volcanic-plutonic suites and sediments that are interpreted to have formed in a convergent margin setting⁶². These rocks experienced multiple deformational episodes associated with metamorphism and

plutonism between 1.71 and 1.62 Ga described in detail by Hoffman, (1988). The deformational episodes were followed by two pulses of calc-alkaline to alkaline magmatism at 1.50-1.42 Ga and 1.40-1.34 Ga ⁶³.

Sample ID	Locality	Latitude	Longitude	Lithology	Mineralogy	Reference
17FIN01	Svecofennian domain	60.498	22.262	Grt migmatite	Grt+Qz+Afs+Pl+Bt	this study
17FIN02	Svecofennian domain	60.470	22.368	Grt migmatite	Grt+Qz+Afs+Pl+Bt	this study
17FIN03B	Svecofennian domain	60.425	22.382	Grt migmatite	Grt+Qz+Afs+Pl+Bt	this study
17FIN04A	Svecofennian domain	60.461	22.176	Grt migmatite	Grt+Qz+Afs+Pl+Bt	this study
17FIN05B	Svecofennian domain	60.546	22.128	Grt migmatite	Grt+Qz+Afs+Pl+Bt	this study
17FIN06	Svecofennian domain	60.482	22.018	Grt migmatite	Grt+Qz+Afs+Pl+Bt	this study
19GH11B	West African Craton	-1.609	5.140	Bt Ms granite	Ms+Qz+Afs+Pl+Bt	this study
19GH9	West African Craton	-1.377	5.340	Bt Ms granite	Ms+Qz+Afs+Pl+Bt	this study
19GH3	West African Craton	-1.156	5.160	Grt granite	Grt+Qz+Afs+Pl+Bt	this study
18IM19	North China Craton	40.693	109.641	Grt granite	Grt+Qz+Afs+Pl+Bt	Liebmann et al. 2020a
18IM20	North China Craton	40.709	109.643	Grt granitoid	Grt+Qz+Afs+Pl+Bt	Liebmann et al. 2020a
18IM23D	North China Craton	40.811	110.258	Grt granite	Grt+Qz+Afs+Pl	Liebmann et al. 2020a
18IM25C	North China Craton	41.182	109.479	Ms Bt granite	Ms+Qz+Afs+Pl(+Bt)	Liebmann et al. 2020a
18IM11B	North China Craton	40.604	112.500	Grt granitoid	Grt+Qz+Afs+Pl+Bt	Liebmann et al. 2020a
18IM12B	North China Craton	40.839	112.565	Grt quartz rich granitoid	Grt+Qz+Afs+Pl	Liebmann et al. 2020a
18IM13C	North China Craton	41.084	110.924	Grt granite	Grt+Qz+Afs+Pl+Bt(+Ms)	Liebmann et al. 2020a
18IM15B	North China Craton	40.994	110.947	Grt granitoid	Grt+Qz+Afs+Pl	Liebmann et al. 2020a
18IM3	North China Craton	40.848	113.921	Grt granite	Grt+Ms+Qz+Afs+Pl(+Bt)	Liebmann et al. 2020a
15K-2	Ukrainian Shield	50.980	28.680	Granite	Qz+Pl+Bt	this study
CO-17-8	Yavapai province	39.776	105.780	Bt Ms granite	Ms+Qz+Afs+Pl+Bt	Bucholz and Spencer, 2019
SP-16-20a	Superior Craton	49.815	93.020	Bt Ms granite	Ms+Qz+Afs+Pl+Bt	Bucholz et al. 2018
SP-16-2b	Superior	47.661	92.939	Bt Ms granite	Ms+Qz+Afs+Pl+Bt	Bucholz

	Craton					et al. 2018
SP-16-34	Superior Craton	49.848	92.694	Bt Ms Grt granite	Ms+Qz+Grt+Afs+Pl+Bt	Bucholz et al. 2018
SP-17-43	Superior Craton	50.741	93.209	Bt Ms Grt granite	Ms+Qz+Grt+Afs+Pl+Bt	this study
SP-17-33	Superior Craton	50.284	94.495	Bt Grt granite	Grt+Bt+Qz+Afs+Pl	this study
SP-17-38	Superior Craton	49.863	93.771	Ms Grt granite	Grt+Ms+Qz+Afs+Pl	this study
SP-17-82	Superior Craton	51.200	92.383	Ms Grt granite	Grt+Ms+Qz+Afs+Pl	Bucholz and Spencer, 2019
SP-17-71	Superior Craton	50.994	92.328	Ms Grt granite	Grt+Ms+Qz+Afs+Pl	Bucholz and Spencer, 2019
SP-17-13	Superior Craton	48.906	89.213	Bt Grt granite	Grt+Qz+Afs+Pl+Bt	this study
SP-17-50	Superior Craton	50.847	92.105	Bt Ms Grt granite	Grt+Ms+Qz+Afs+Pl+Bt	this study

Table 1. Summary of sample locations, lithologies, and mineralogy. Minerals in parentheses are accessory phases.

Mineral abbreviations: Qz, quartz; Afs, alkali feldspar; Pl, plagioclase; Grt, garnet; Ms, muscovite, Bt, biotite.

Sample ID	Major element concentrations (reported as oxide wt %)													Reference	
	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	MnO	LOI	Total	ASI		
19GH11B	73.78	0.08	14.74	0.55	0.15	1.25	3.39	5.63	0.01	0.01	0.22	99.81	1.1	1.1	this study
19GH9	73.23	0.11	15.67	1.06	0.28	2.91	5.02	1.14	0.03	0.02	0.63	100.09	1.1	1.1	this study
19GH3	73.16	0.05	16.96	0.33	0.14	0.91	4.36	2.34	0.08	0.01	1.31	99.64	1.5	1.5	this study
17FIN01	74.00	0.04	13.38	2.13	0.28	0.26	2.05	7.40	0.11	0.02	0.58	100.26	1.1	1.1	this study
17FIN02	61.87	0.85	17.24	7.95	2.94	1.18	2.45	3.84	0.06	0.03	1.41	99.81	1.7	1.7	this study
17FIN03B	60.20	0.82	17.37	8.76	2.41	2.15	3.73	3.53	0.07	0.08	0.69	99.81	1.3	1.3	this study
17FIN04A	71.01	0.23	14.58	3.56	0.95	1.62	3.02	4.40	0.08	0.07	0.49	100.01	1.2	1.2	this study
17FIN05B	56.62	0.89	18.71	8.63	3.91	1.42	2.63	5.86	0.08	0.04	1.24	100.02	1.4	1.4	this study
17FIN06A	64.84	0.66	15.99	6.92	2.21	1.62	2.92	3.45	0.07	0.05	1.16	99.89	1.4	1.4	this study
17FIN06B	70.66	0.06	15.00	3.88	0.72	1.47	3.14	4.28	0.10	0.04	0.71	100.06	1.2	1.2	this study
18IM19	72.13	0.31	14.05	3.51	1.41	1.27	2.49	3.68	0.06	0.05	0.73	99.69	1.4	1.4	this study
15K-2	62.29	0.57	17.36	5.97	1.76	3.23	5.15	2.19	0.33	0.06	0.90	99.81	1.1	1.0	this study
18IM11B	61.93	1.06	18.04	6.58	2.18	2.88	2.80	3.58	0.07	0.06	0.22	99.38	1.3	1.3	Liebman et al. 2020a
18IM12B	69.05	0.14	14.73	5.97	1.90	2.85	2.32	1.03	0.03	0.13	0.77	98.92	1.5	1.5	Liebman et al. 2020a
18IM13C	70.74	0.33	14.84	3.06	0.87	3.04	3.29	1.73	0.10	0.01	1.00	99.01	1.2	1.2	Liebman et al. 2020a
18IM15B	65.23	0.58	15.48	7.01	2.12	3.52	2.84	0.96	0.09	0.09	1.34	99.25	1.3	1.3	Liebman et al. 2020a
18IM19	72.13	0.31	14.05	3.51	1.41	1.27	2.49	3.68	0.06	0.05	0.73	99.69	1.4	1.4	Liebman et al. 2020a
18IM20	63.52	0.26	17.78	6.13	1.81	2.63	3.50	3.05	0.07	0.07	0.85	99.69	1.3	1.3	Liebman et al. 2020a
18IM23D	71.76	0.11	14.05	2.83	0.79	0.60	2.01	6.51	0.12	0.04	0.50	99.32	1.2	1.2	Liebman et al. 2020a
18IM25C	74.42	0.06	13.17	0.95	0.17	0.95	2.71	5.80	0.02	0.00	0.89	99.15	1.1	1.1	Liebman et al. 2020a
18IM3	77.57	0.37	10.88	3.01	0.87	1.03	2.23	2.72	0.05	0.05	0.68	99.45	1.3	1.3	Liebman et al. 2020a
SP-16-20a	74.14	0.10	13.97	0.92	0.27	0.65	2.70	6.62	0.12	0.01	0.35	99.86	1.1	1.1	Bucholz et al. 2018
SP-16-2b	72.83	0.18	14.50	1.33	0.31	1.11	4.05	4.58	0.07	0.03	0.53	99.53	1.1	1.1	Bucholz et al. 2018
SP-16-34	75.30	0.08	14.30	0.73	0.25	0.84	2.80	5.00	0.05	0.02	0.61	99.97	1.3	1.3	Bucholz et al. 2018
SP-17-71	75.31	0.05	14.11	0.71	0.06	0.60	5.45	1.58	0.11	0.17	0.97	98.08	1.3	1.3	Bucholz and Spencer, 2019
SP-17-82	75.45	0.05	14.27	0.44	0.01	0.32	4.85	3.74	0.02	0.08	0.74	99.18	1.2	1.2	Bucholz and Spencer, 2019
CO-17-8	70.54	0.39	14.37	2.95	0.49	1.24	2.56	5.78	0.28	0.03	1.25	99.58	1.3	1.3	Bucholz and Spencer, 2019

Table 2: Bulk rock major element concentrations. The aluminium saturation index (ASI) is calculated as molecular Al/(Ca - 1.67P + Na + K); ACNK is calculated as molecular Al/(Ca + Na + K).

Sample ID	Locality	Age $\pm 2\sigma$ Ma	zircon		garnet		pyrite				Reference
			$\delta^{18}\text{O}$ [‰]	2 σ	$\delta^{18}\text{O}$ [‰]	2 σ	$\delta^{34}\text{S}$ [‰]	2 σ	$\Delta^{33}\text{S}$ [‰]	2 σ	
17FIN01	Svecofennian domain	1850 ¹⁾	-	-	10.39	0.20	1.54	0.99	-0.01	0.08	this study
17FIN02	Svecofennian domain	1896 $\pm$ 26	8.78	3.22	9.87	0.24	-0.87	0.90	-0.02	0.07	this study
17FIN03B	Svecofennian domain	1824 $\pm$ 13	9.32	1.16	9.48	0.51	-0.09	0.88	0.01	0.08	this study
17FIN04A	Svecofennian domain	1850 ¹⁾	10.37	3.46	10.02	0.10	0.85	1.41	0.01	0.06	this study
17FIN05B	Svecofennian domain	1836 $\pm$ 11	10.52	0.77	10.30	0.11	1.61	0.55	-0.01	0.06	this study
17FIN06	Svecofennian domain	1815 $\pm$ 16	10.20	0.61	10.28	0.11	1.58	0.93	-0.01	0.07	this study
19GH11B	West African Craton	2188 $\pm$ 20	5.34	0.83	-	-	-3.71	1.51	-0.05	0.07	this study
15K-2	Ukrainian Shield	2144 $\pm$ 28	8.86	0.81	-	-	9.72	0.99	-0.04	0.06	this study
CO-17-8	Yavapai province	1447 $\pm$ 50	8.74	0.61	-	-	3.31	0.26	-0.02	0.05	this study
SP-16-20a	Superior Craton	2654 $\pm$ 24	-	-	-	-	0.60	0.56	0.13	0.06	this study
SP-16-2b	Superior Craton	2664 $\pm$ 45	-	-	-	-	1.79	0.61	0.18	0.05	this study
SP-17-43	Superior Craton	2690 ²⁾	-	-	7.06	0.09	-13.33	0.36	0.16	0.04	this study
18IM19	North China Craton	2478 $\pm$ 18	7.21	0.58	7.50	0.41	-1.26	1.29	-0.29	0.12	this study, Liebmann et al. 2020a
18IM20	North China Craton	2374 $\pm$ 48	7.39	0.70	7.22	0.05	-	-	-	-	this study, Liebmann et al. 2020a
18IM11B	North China Craton	1901 $\pm$ 17	10.8	0.90	10.77	0.14	-	-	-	-	this study, Liebmann et al. 2020a
18IM12B	North China Craton	1929 $\pm$ 29	9.74	1.16	10.05	0.12	-	-	-	-	this study, Liebmann et al. 2020a
18IM3	North China Craton	1917 $\pm$ 70	11.41	1.00	11.52	0.12	-	-	-	-	this study, Liebmann et al. 2020a
18IM25C	North China Craton	2493 $\pm$ 28	6.86	0.89	-	-	-	-	-	-	this study, Liebmann et al. 2020a
18IM13C	North China Craton	2536 $\pm$ 13	8.12	0.70	-	-	-	-	-	-	Liebmann et al. 2020a
18IM15B	North China Craton	2530 $\pm$ 60	8.57	0.42	-	-	-	-	-	-	Liebmann et al. 2020a
18IM23D	North China Craton	2453 $\pm$ 11	6.31	0.63	-	-	-	-	-	-	Liebmann et al. 2020a
19GH9	West African Craton	2183 $\pm$ 14	4.93	0.82	-	-	-	-	-	-	this study
19GH3	West African Craton	2180 ¹⁾	-	-	9.72	0.09	-	-	-	-	this study
SP-16-34	Superior Craton	2654 $\pm$ 24	-	-	7.99	0.10	-	-	-	-	this study
SP-17-33	Superior Craton	2650 ¹⁾	-	-	7.85	0.13	-	-	-	-	this study
SP-17-38	Superior Craton	2650 ¹⁾	-	-	6.93	0.11	-	-	-	-	this study
SP-17-82	Superior Craton	2650 ¹⁾	-	-	6.06	0.12	-	-	-	-	this study
SP-17-71	Superior Craton	2650 ¹⁾	-	-	6.92	0.09	-	-	-	-	this study
SP-17-13	Superior Craton	2660 $\pm$ 10 ³⁾	-	-	7.15	0.10	-	-	-	-	this study

SP-17-50 Superior Craton 2650 ¹⁾ - - 7.55 0.09 - - - - this study

Table 3. Summary of O and S geochemistry, and crystallization ages. Isotopic data is given as weighted average. ¹⁾ expected age (see appendix A), ²⁾ age from ⁸⁷, ³⁾ age from ⁸⁸.

Declarations

A

Supplementary Information (pdf file), including supplementary information on geochronology, supplementary Figures A1-A6, and supplementary Tables A1-A4.

B

Supplementary Table B1 (spread sheet), zircon U-Pb data.

C

Supplementary Table C1 (spread sheet), pyrite S isotope data.

D

Supplementary Table D1 (spread sheet), zircon and garnet O isotope data.

Author contributions

JL, CJS, CLK, CEB designed the research or contributed to designing the research. JL, CJS, CEB, LS collected the samples. JL, CLK, CEB, XPX, LM, NK carried out the analyses. JL, CJS, CLK, CEB contributed to interpreting the results. JL wrote the manuscript. All authors provided comments on the manuscript.

Acknowledgements

U-Pb geochronology analytical work at the John de Laeter Centre, Western Australia was enabled by NCRIS via AuScope. Oxygen and sulfur isotope analytical work at the Guangzhou Institute of Geochemistry was supported by the open fund from the State Key Laboratory of Isotope Geochemistry, GIGCAS (SKLabIG-KF-18-04). The authors acknowledge the facilities, and the scientific and technical assistance of Microscopy Australia at the Centre for Microscopy, Characterisation & Analysis, The University of Western Australia, a facility funded by the University, State and Commonwealth Governments.

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Figures

Figure 1

Zircon $\delta^{18}\text{O}$ vs garnet $\delta^{18}\text{O}$ color-coded by crystallization age of sediment-derived igneous rocks. Isotherms at 650°C and 1200°C are after 27. Oxygen isotope data is shown as weighted averages. Single spot results of O isotope analysis are given in the appendix. Error bars are shown at 2 σ level. Note that large error bars for zircon $\delta^{18}\text{O}$ are due to heterogeneity in these samples interpreted to be related to secondary processes (discussed in the text).

Figure 2

Single spot $\delta^{34}\text{S}$ vs. $\Delta^{33}\text{S}$ of sediment-derived igneous rocks. Error bars for $\Delta^{33}\text{S}$ are shown at 1σ level. Errors of $\delta^{34}\text{S}$ at 2σ level are smaller than the symbols. Grey area marks the range of $\Delta^{33}\text{S}$ values that can be produced through mass-dependent fractionation processes 83. Single spot results of sulfur isotope analysis are given in the appendix.

Figure 3

Oxygen and sulfur isotopic data vs. crystallization age of sediment-derived igneous rocks. Timing of atmospheric oxygenation 84 is shown as blue bar. Legend on the right applies to both parts of the figure. (A) Zircon and garnet $\delta^{18}\text{O}$ vs. crystallization age of sediment-derived igneous rocks. Oxygen isotope data is shown as weighted averages. Grey bars mark average $\delta^{18}\text{O}$ (as recorded by zircon and garnet) pre- and post-2.3 Ga, respectively. Single spot results of O isotope analysis are given in the appendix. Error bars for oxygen isotope data and age are 2σ . Zircon oxygen isotope data from Suomussalmi and Helanshan are from 1)85, and 2)86. (B) Pyrite $\Delta^{33}\text{S}$ vs. crystallization age of sediment-derived igneous rocks shown as violin plots; white circles mark medians. Black bar marks $\Delta^{33}\text{S} = 0$ ‰.

Supplementary Files

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