



## Research Article

# Widespread poly-metamorphosed Archean granitoid gneisses and supracrustal enclaves of the southern Inukjuak Domain, Québec (Canada)



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## ABSTRACT

The ~12,000 km<sup>2</sup> Inukjuak Domain in northern Québec is part of the Archean Minto Block in the northwestern Superior Province of Canada. Eoarchean (ca. >3800–3780 Ma) rocks of the Nuvvuagittuq supracrustal belt (NSB) are the best known occurrence of otherwise abundant <1 m to km-scale supracrustal enclaves dispersed throughout the Innuksuac Complex. The supracrustals are dominantly amphibolites, with subordinate intermediate-, mafic- and ultramafic schists, quartzo-feldspathic (trondhjemitic and granodioritic) sills, dikes and sheets, banded iron-formations and quartz-pyroxene±magnetite rocks, and (detrital) fuchsite quartzites. Supracrustal assemblages are in turn hosted by variably deformed granite-granitoid gneisses metamorphosed to amphibolite facies. Locally, retrogression is expressed as pervasive chloritization and development of jaspilite box veinings. This metamorphic history precludes preservation of original fragile microfossil shapes. Despite its importance as one of the few terranes to host Eoarchean supracrustal assemblages, limited geochronology was previously available for rocks beyond the ~8 km<sup>2</sup> NSB. Here, we report new major-, minor-, and trace-element geochemistry and metamorphic petrology coupled with U–Pb zircon geochronology, from rocks within and surrounding the NSB. These include the little-studied but volumetrically significant Voizel suite gneisses. Results show that intra-NSB fold belt rocks of the Central Tonalitic Gneiss (CTG) preserve mainly ca. 3650 Ma zircons. Beyond the NSB, the Voizel gneisses – previously considered contemporaneous with the CTG – are instead about 100 Myr younger (~3550 Ma). Tonalitic (ortho)gneisses at the margin of the NSB were previously assigned a ca. 3650 Ma age, and the surrounding Boizard suite gneisses may be about 2700 Ma. We find the Boizard rocks contain inherited zircon cores up to ca. 3700 Ma, with younger overgrowths dated at ca. 2700 Ma. A tonalitic gneiss that transects another highly deformed supracrustal enclave north of the NSB – dubbed the Ukaliq Supracrustal Belt – yields maximum concordant zircon ages of 3653 ± 16 Ma (2σ). Detrital zircons from Ukaliq and Nuvvuagittuq quartzites and quartz-biotite schists define a maximum age of ca. 3780 Ma. No indication of U–Pb zircon ages older than about 3800 Ma exist in this terrane from our geochronological surveys. New reconnaissance sampling of gneisses to the west of the NSB fold belt yielded more zircon-bearing rocks with ages of ca. 3760 Ma. This discovery of more pre-3700 Ma rocks beyond the NSB outcrops calls attention to the existence of hitherto unidentified but widespread scattered occurrences of Eoarchean and Paleoarchean rocks throughout the region.

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## 1. Introduction

The world's oldest rocks of demonstrably volcano-sedimentary origin comprise the Archean “supracrustal belts”; these typically occur as irregularly deformed and metamorphosed enclaves and rafts with broad spectrum of sizes captured in ancient granite-granitoid gneiss terranes that are in turn separated unconformably from the surrounding felsic cratonic basement rocks (e.g., de Witt and Ashwal, 1997). Although such bodies tend to be bounded by shear zones, examples also exist of Archean crustal enclaves that preserve weathering horizons and basal conglomerates, and quartzites, which unconformably rest on old tonalite-trondhjemite-granodiorite (TTG) gneisses (e.g. Bleeker, 2002). Ranging in size from centimeter-scale to tens of kilometers in outcrop area, the various Eoarchean (3850–3600 Ma) supracrustals thus far documented are dominantly amphibolitic. Because of their widely documented poly-phase metamorphic histories, it is a misnomer to refer to these – either at the well-known Isua locality in West Greenland (Nutman et al., 1996), the paragneisses of the Saglé Harbor rocks of Labrador (Schiette et al., 1989), or for that matter in the Inukjuak region in northern Québec described herein – as “greenstone belts” (cf. O’Neil et al., 2007). That is because *without known exception*, all Eoarchean rocks experienced at least one (or more commonly, several) peak mid- to upper-amphibolite (or in some cases granulite facies) metamorphic events with associated recrystallization(s) and several episodes of deformation before undergoing locally highly variable retrogression (e.g. Cates and Mojzsis, 2006, 2009; Manning et al., 2006; Nutman et al., 2007). It is owing to the intense structural juxtaposition of Eoarchean supracrustal rocks with TTG gneisses in the Paleoarchean, followed by terrane assembly, that all such rocks are so strongly modified. As such, none of these rocks are “greenstones” in the strict sense, nor do they hold much potential for preservation of ultrafine-scale primary sedimentary features such as fragile microfossil shapes (Bridgwater et al., 1981; Whitehouse et al., 2019; cf. Dodd et al., 2017). In spite of the fact that all of the oldest (>3.7 Ga) supracrustal rocks are undoubtedly modified due to their long residence times in the crust, they nevertheless constitute the only direct sources of evidence – albeit strongly debated – for the early biological evolution of our planet (e.g., Schidlowski, 1988; Mojzsis et al., 1996; Rosing, 1999; Nutman et al., 2016). We come back to significance of this metamorphic history and what it means in the context of origin of life studies in Sections 4.1 & 4.5.

It is worth emphasizing that these rocks are so important to our understanding of the nature of the “Early Earth” as a terrane separate from the famous Itsaq Gneiss Complex in southern West Greenland (Nutman et al., 1996) that it makes sense to assess an ancient complex history through enhanced understanding of the gneisses that encompass them. For instance, exploration of surrounding granitoid gneisses can provide minimum ages for the supracrustals where unambiguous field relations exist on cross-cutting dikes, sheets and sills. Extensive surveys of the surrounding granitoids and their ages can also point to thermal peaks in the (poly-)metamorphic history endemic to these terranes that can be recapitulated in thermochronological investigations of the supracrustal rocks themselves.

One such locality of ancient rocks is the ca. 3750–3780 Ma Nuvvuagittuq supracrustal belt (NSB) in northern Québec (Fig. 1) (O’Neil et al., 2007, 2019). These were discovered in 2001 during a regional geological and geochemical survey sponsored by the Provincial Government of Québec (Simard et al., 2003; Berclaz et al., 2003; David et al., 2002; Stevenson and Bizzarro, 2005). It should be emphasized that the NSB is merely the best known of what are at least a dozen or so km-scale enclaves in the southern part of the vast (~12,000 km<sup>2</sup>) Inukjuak Domain recognizable from aerial photographs available from the offices of Natural Resources Canada and thus mappable at the regional scale (Fig. 2). Collectively, the assorted supracrustals in the Inukjuak Domain comprise what is termed the Innuksuac Complex (Cates and Mojzsis, 2007, 2009 and references therein).

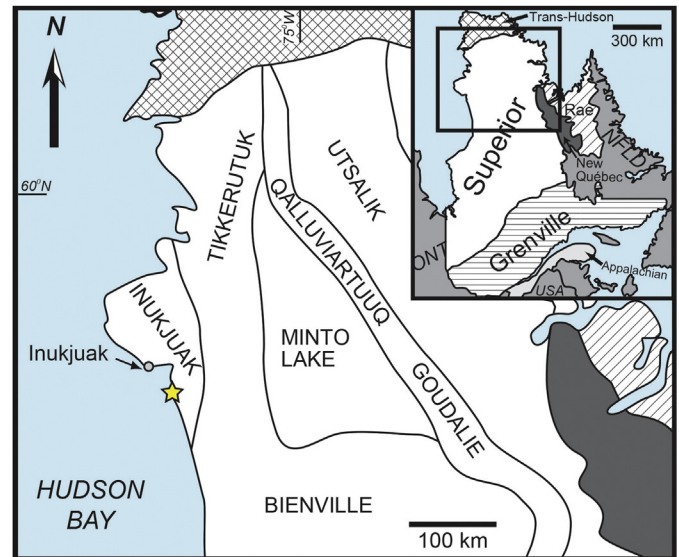


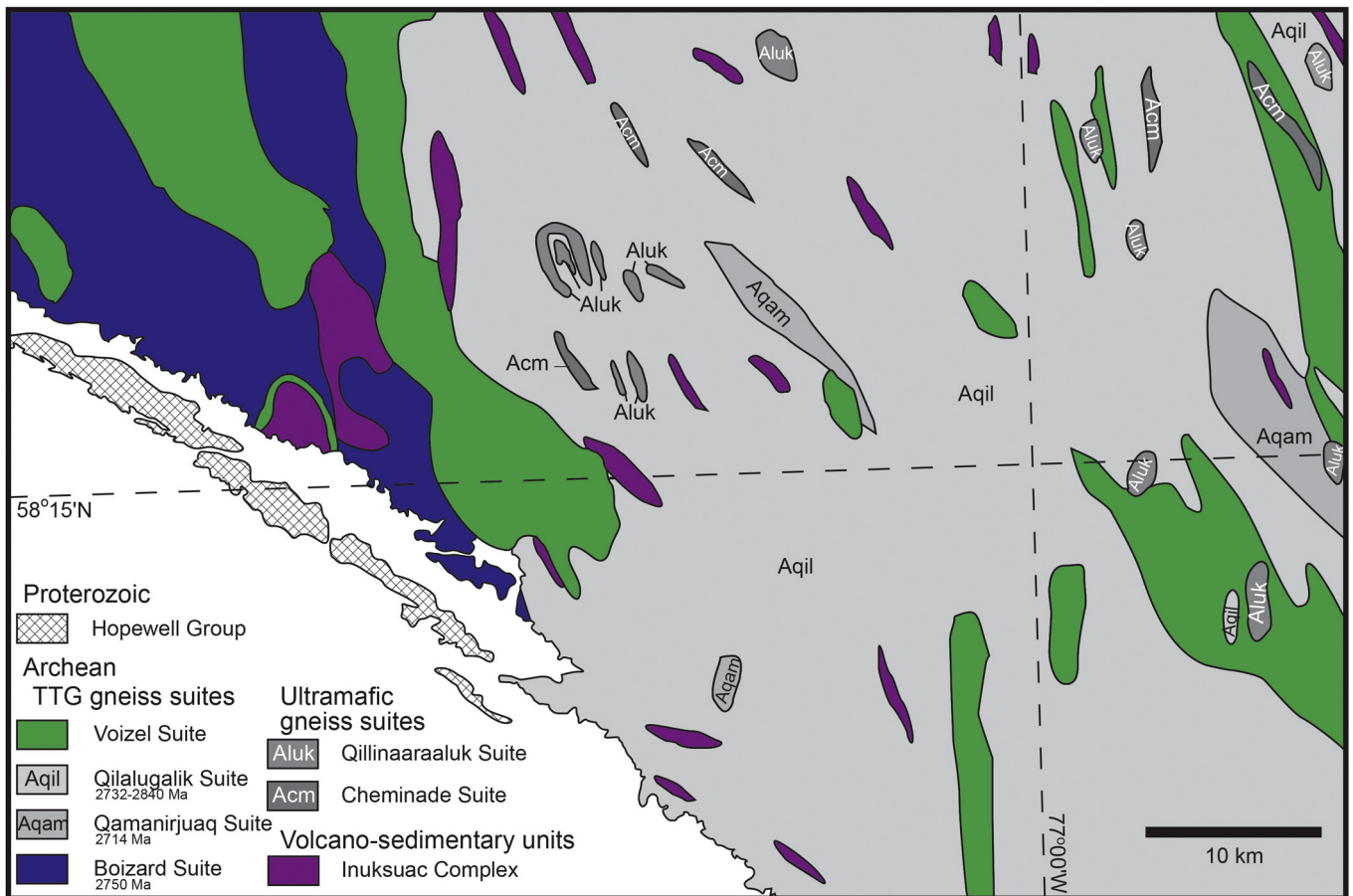
Fig. 1. Simplified map of the northwestern Superior Province, with the location of the town of Inukjuak (gray dot) and the study area (yellow star) highlighted.

To enhance our understanding of this history, we present new field relations, metamorphic petrographic analysis, geochemical data, and U–Pb zircon geochronology for a selection of rocks within and beyond the NSB that include the little-studied TTG gneisses and associated supracrustal rocks from the nearby Ukaliq supracrustal belt (USB) that was introduced in Caro et al. (2017).

## 2. Geologic background

### 2.1. Innuksuac complex supracrustal rocks – Nuvvuagittuq locality

Reviews of the geology of the Nuvvuagittuq supracrustal belt (NSB) are provided elsewhere (O’Neil et al., 2007, 2019 and references therein); a short summary is presented here. The NSB and neighboring supracrustals rocks that compose the Innuksuac Complex are found within the Inukjuak Domain, which in turn is one of several large deformed belts (e.g., Tikkertuk/Bienville; Minto Lake; Qalluviartuuq/Goudalie; Utsalik) that collectively make up the Minto Block in the northeast Superior province. The Inukjuak Domain is volumetrically dominated by Neoproterozoic plutonic suites with subordinate rocks of Paleo- to Eoarchean age (Fig. 3; Simard et al., 2003). The ~8 km<sup>2</sup> outcrop area of the NSB has garnered much interest because of the verified discovery of anomalous deficits in <sup>142</sup>Nd/<sup>144</sup>Nd vs. Bulk Silicate Earth (O’Neil et al., 2008; Roth et al. (2013)). Debate continues over the interpretation of these data (e.g. Cates et al. (2013); Roth et al. (2013); Guitreau et al. (2013); Caro et al. (2017), but there is broad agreement that the anomalous <sup>142</sup>Nd/<sup>144</sup>Nd values must represent evidence for an early (pre-4300 Ma) separation of the terrestrial silicate reservoirs during the short (half-life = 103 Myr) activity of now-extinct <sup>146</sup>Sm (Roth et al., 2013). One school of thought is that the <sup>142</sup>Nd/<sup>144</sup>Nd vs. Sm/Nd data, in conjunction with some ancient Sm–Nd model ages, indicate that formation times for certain protoliths to the NSB may be 4300–4400 Ma (O’Neil et al., 2008, 2012, 2019). This interpretation has been challenged, however, by separate studies, and the topic is not discussed further here; instead we refer the reader to data and discussion in Cates et al. (2013), Roth et al. (2013), Guitreau et al. (2013) and Caro et al. (2017). Despite their obvious importance, the NSB rocks specific to the “Porpoise Cove” locality are the only ones mapped at the appropriate detail (i.e., 1:100 scale) to understand a complex and



**Fig. 2.** Regional geologic map (after Simard, 2003) showing the geological context of the ancient supracrustal remnants of the Innusuaq Complex. The study area is outlined by the rectangle (see Fig. 3). Rock suites that have not been substantially characterized are shown in gray.

protracted metamorphic history (Cates and Mojzsis, 2009; Cates et al., 2013; cf. O'Neil et al., 2007; Darling et al., 2013).

As has been reviewed elsewhere (Cates and Mojzsis, 2007; O'Neil et al., 2019), the Nuvvuagittuq supracrustals are dominantly composed of (Ca-poor; cummingtonite-rich) basaltic amphibolites. Other rocks include layered ultramafic schists that comprise some rocks with geochemical signatures akin to komatiites (Frank et al., 2016; Touboul et al., 2014), finely-banded quartz magnetite±pyroxene±amphibole rocks (s.s. banded iron-formations (BIF); Dauphas et al. 2007; Mloszewska et al. 2012), massive Ca-Fe-Mg silicate “quartzites” (Mloszewska et al., 2013), and quartz-biotite schists of probable detrital origin (Bell et al., 2018; Cates et al., 2013; Cates and Mojzsis, 2007; Darling et al., 2013; David et al., 2009; O'Neil et al., 2007; Simard et al., 2003). Cates et al., 2013 proposed that the cummingtonite amphibolite rocks owed their Ca-poor compositions to albitization of pyroclastic debris at time of formation around 3.8 Ga. These authors further argued that the colloquial term “faux amphibolite” endorsed by O'Neil et al. 2009 and others is an unfortunate obfuscation of geologic data. Amphibolitic rocks of such composition are have been noted elsewhere in Eorchean terranes including from southern West Greenland (e.g. sample GR00114 of Manning et al., 2006).

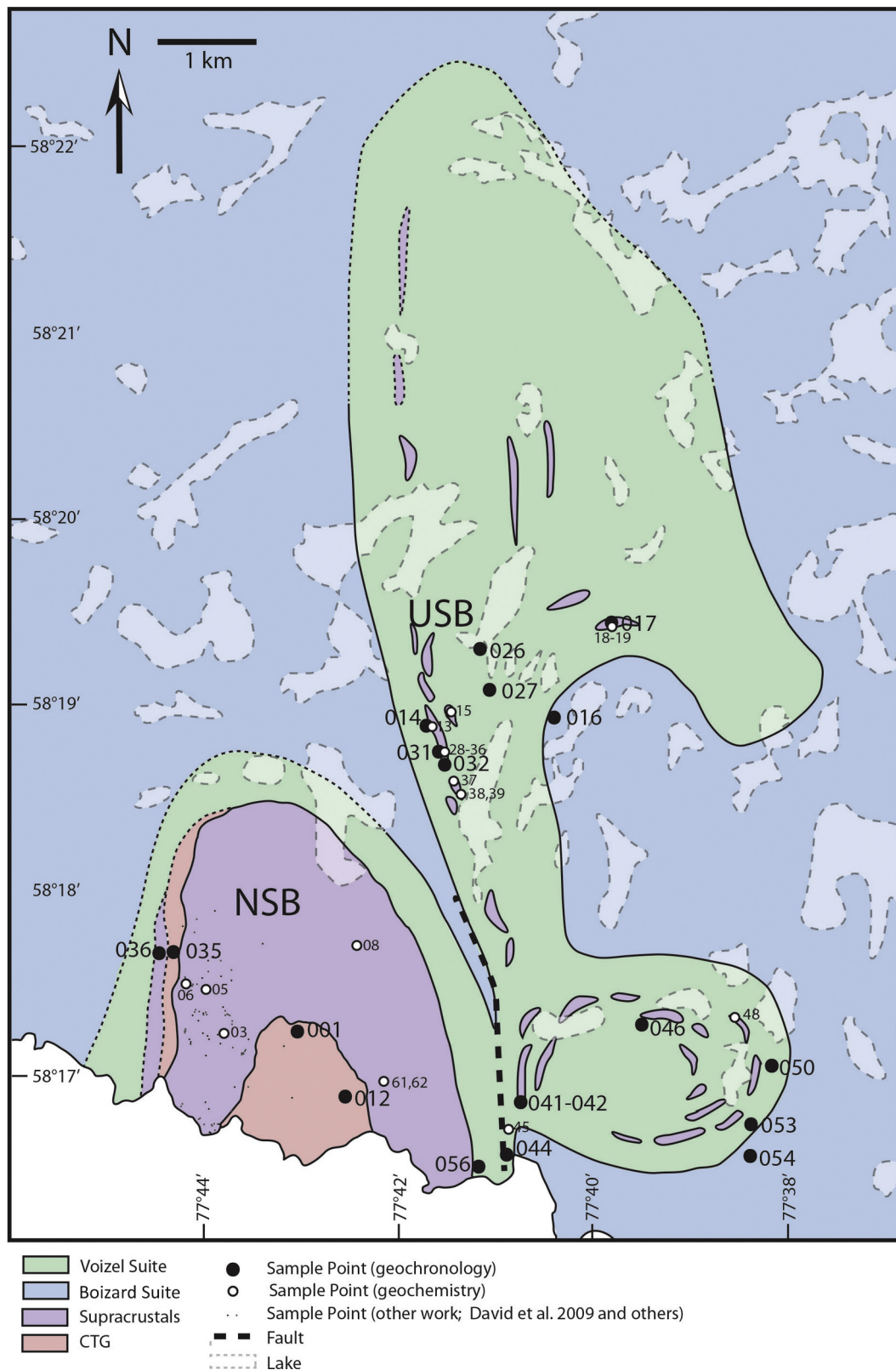
Furthermore, it has been detailed that detrital igneous zircons in fuchsite (Cr-muscovite rich) quartzites and quartz-biotite schists, along with ages for igneous zircons from trondhjemitic gneiss sheets – which in some places structurally transect the supracrustal sequences – show that the NSB is probably not older than ca. 3800 Ma (Cates et al. 2007, 2009, 2013). Even after nearly a thousand published U–Pb zircon ages, very few pre-3900 Ma ages have thus far been identified

in the NSB (Bell et al., 2017, 2018). Yet, like many other aspects of the geology of the Nuvvuagittuq rocks, the interpretation that these detrital zircons bracket the maximum age of the NSB at ca. 3800 Ma has also been disputed (Darling et al., 2013; O'Neil et al., 2019).

## 2.2. Innusuaq complex supracrustal rocks beyond the NSB

A large number of scattered small pods and lenses (<1–10 m), as well as larger (>1 km) supracrustal rafts and enclaves have been provisionally noted within the Inukjuak Domain; the largest of these were tentatively mapped from aerial reconnaissance surveys supplemented by field studies in the early 2000s (Berclaz et al. 2003). In several aspects, an emulsion-like assortment of locally mappable supracrustal rocks locked within poly-phase granitoid gneisses is not unlike the outcrops of the so-called “Akilia association” found southwards of the Isua area and throughout the Itsaq Gneiss Complex of southern West Greenland (McGregor and Mason, 1977), as well as the lesser-known “Manfred Complex” rocks of the Murchison region of Western Australia (Kinny et al., 1988). Furthermore, much like the type-locality on Akilia (island) for the Akilia association (Manning et al., 2006; Nutman et al., 2002) and other Akilia rocks such as on Innersuartaq in the southern Faeringhavn terrane (Cates and Mojzsis, 2006 and references therein), the Ukaliq rocks are the only other Innusuaq supracrustals in the area beyond Nuvvuagittuq that have mapped in the detail required to understand the field relationships (Caro et al., 2017). Similarly, unlike the large (and arguably coherent) Isua and Nuvvuagittuq belts, those of the Ukaliq, Akilia and Manfred supracrustal enclaves are mostly present in outcrop as a scattered set of





**Fig. 3.** Local geologic map showing the main lithologies discussed herein. Samples described in this work are represented by large black dots, additional samples with geochemical data are represented by small white dots, and samples from other studies are represented by small gray points (see Supplementary Information in [Cates et al., 2013](#) for reported data tables).

volumetrically small ( $\leq 1 \text{ km}^2$ ) and dominantly mafic to ultramafic enclaves, surrounded and broken up by strongly foliated plagiogranitoid gneisses.

Specific to the Inukjuak Domain, the ancient gneisses which host the supracrustals are attributed to the Voizel suite (Avoi; [Simard et al., 2003](#)). Field relations show that the Voizel suite rocks are further



enclosed and disrupted by a pinkish granitoid with only weakly developed tectonic fabric, termed the Boizard suite (*Aboz*). The Boizard rocks were provisionally assigned an age of  $2750 \pm 5$  Ma in published reports from the Ministère des Ressources naturelles, de la Faune et des Parcs (Provincial government of Québec). Evidence exists that the Boizard rocks intrude some supracrustal enclaves in places, for example a pegmatitic dike with the same age was described previously described within the NSB (Simard et al., 2003). The large number of metamorphic zircon growth ages at about 2700 Ma in the NSB imply that the emplacement of the Boizard suite led to regional metamorphism of the Innuksuac complex that variably retrogressed the earlier (Paleoarchean) amphibolite facies metamorphism (Cates et al., 2013; Cates and Mojzsis, 2007, 2009; O'Neil et al., 2019).

### 3. Sample collection and analytical methods

#### 3.1. Field relationships

Representative samples of the various rocks associated with the NSB, USB and surrounding terrane (Fig. 4) were collected by us during the course of regional-scale studies in our 2005, 2012, and 2014 field seasons. A subset of these samples was chosen for geochemical analysis based on their apparent lack of weathering and homogeneity at the hand-specimen scale; a further subset was set aside based on perceived likelihood of producing abundant zircons for geochronology.

#### 3.2. Petrography

Four representative samples were prepared into thin sections and subsequently investigated using standard optical microscopy and back-scatter electron (BSE) images to illustrate the textural relations, as well as by electron microprobe analysis. These samples were specifically

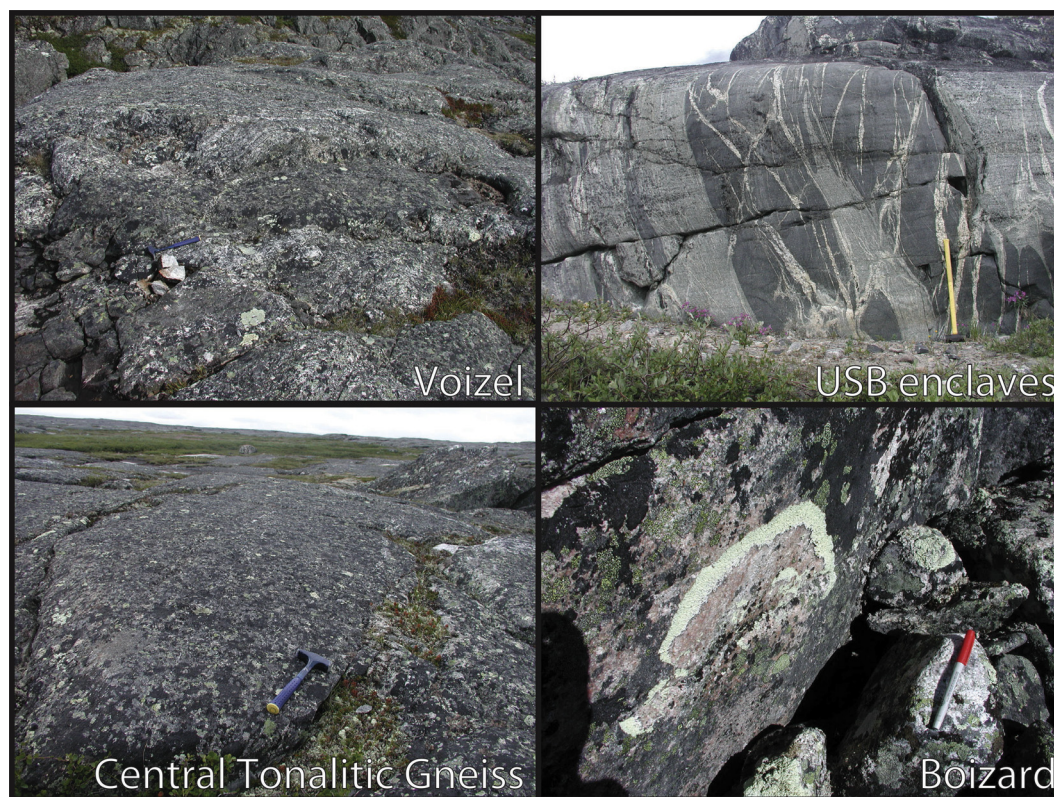
chosen for thermobarometry work and provenance studies; see Sections 4.1, 4.5, 4.6).

#### 3.3. Whole-rock geochemistry

Samples for whole-rock geochemical analyses were crushed to fine powders at the Rock Preparation Facility in the Department of Geological Sciences at the University of Colorado, Boulder. Dedicated alumina mortars were pre-cleaned with washed quartz sand and conditioned with small amounts of sample before powders were made of the main sample masses. Care was taken to avoid contact with metal hammers, saws, and other metal dividing apparatus that could compromise future isotopic or trace metal analytical work. Splits from homogenized powders were subdivided for major-, minor-, and trace element geochemistry and approximately 40% by mass for each was reserved for archival purposes. Whole-rock geochemical analyses were performed at the CRPG SARM Facility in Nancy (France). Samples for which only geochemical data are presented herein were also analyzed at this Facility.

#### 3.4. Ion microprobe U—Pb zircon geochronology

A fraction of those samples targeted for geochronology, based on visual inspection of mineralogy, documented zirconium contents from the geochemical analyses as well as geologic setting, was set aside for zircon extraction. Whole-rock samples were crushed and sieved to  $<350 \mu\text{m}$  to yield an appropriate size range of zircons. Sieved aliquots were then passed through two stages of standard heavy liquid separations (tetrabromoethane and methylene iodide) with intermediate washings in reagent-grade acetone by ultra-sonication before final washings in acetone and DI water baths. After drying, strongly ferromagnetic minerals and metal filings fractions were removed with a hand-magnet, followed by separations using a Franz magnetic

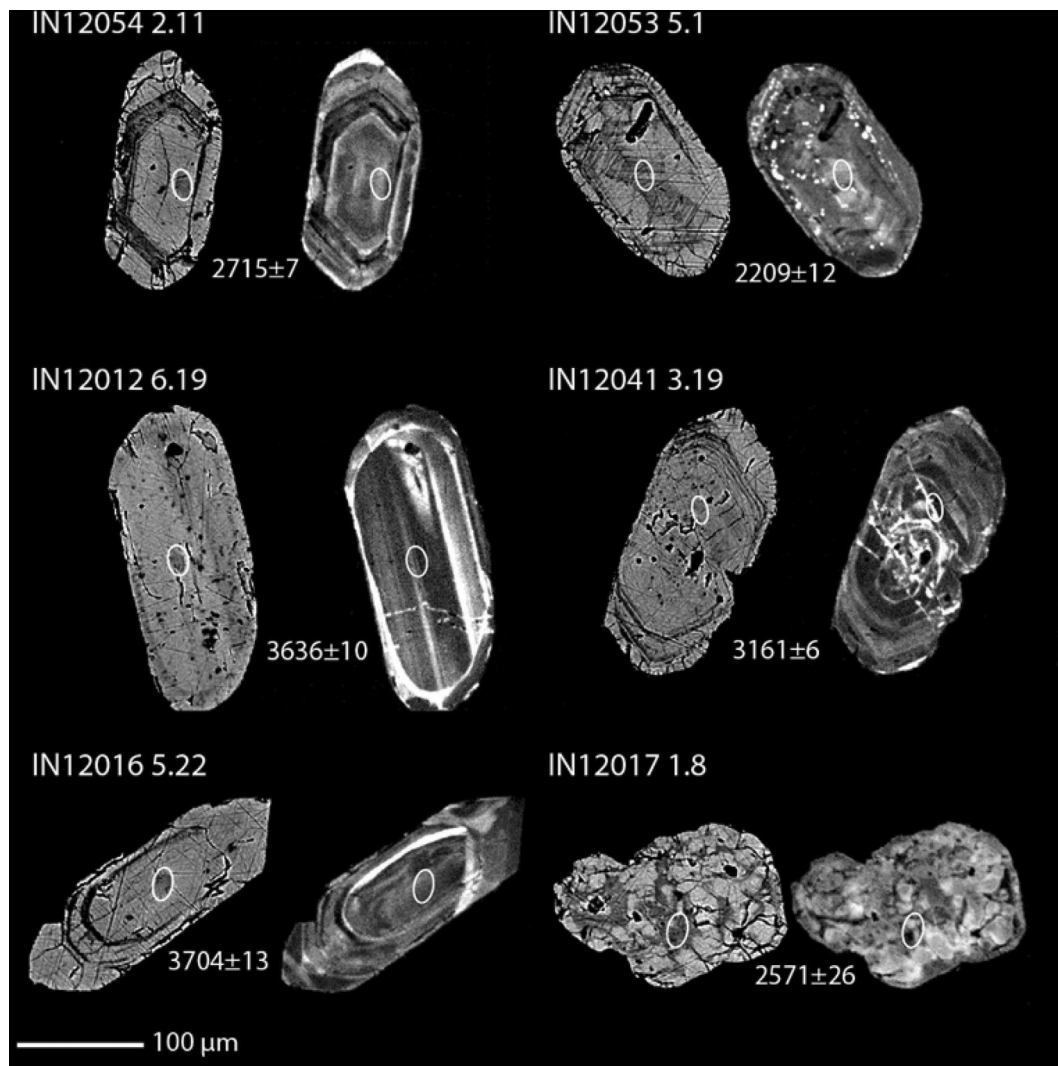


**Fig. 4.** Field photographs of the main lithologies discussed in this work. The Voizel suite is represented by sample IN12041 (hammer for scale, ~30 cm); Ukaliq enclaves are exemplified by sample IN12042 (as a white gneiss that cross-cuts amphibolite; 1 m hammer for scale); an example of the Central Tonalitic Gneiss is from sample IN12012 (hammer for scale); and the Boizard suite is represented by IN12054 (pen for scale, 10 cm). (For interpretation of the colors in this figure, the reader is referred to the web version of this article.)

separator. Individual zircon grains from the least magnetic Franz fraction were then handpicked under a binocular microscope, placed as oriented arrays (common long-axis directions) on double-sided adhesive tape and cast in 2.52 cm-diameter molds with Buehler® epoxide resin along with a halo of grain fragments from zircon standard AS-3 (Black et al., 2003; Cates and Mojzsis, 2009; Paces and Miller, 1993). A variety of zircon sizes and morphologies from each aliquot was chosen so as to diminish sampling bias. The mounts were cured in a 60 °C oven for at least 48 h prior to the first polishing step. The sample mounts were then polished to brilliance in stages down to 0.25 µm alumina to expose approximate grain centers depending on the grain sizes of the cast fraction. Transmitted and reflected optical micrograph mount maps were produced to facilitate identification and navigation on the mounts. Paired BSE (back-scattered electron) and CL (cathodoluminescence) images (e.g., Fig. 5; see also Supplementary Figs. S1) were also acquired for all zircons analyzed for U—Pb to guide primary ion beam position.

Immediately prior to ion microprobe analysis, the zircon mounts were ultrasonically bathed in a 1 N HCL solution for several minutes to reduce common Pb contamination, rinsed in ultrapure water, air dried and then sputter-coated with ~100 Å of Au to facilitate conductivity.

All U—Pb zircon ion microprobe data were acquired using the UCLA Cameca *ims1270* high-resolution secondary ion mass spectrometer under standard operating conditions (e.g., Cates et al., 2013). An abridged description of the methods is provided here: a ~8 nA O<sub>2</sub><sup>+</sup> primary ion beam was defocused to a ~25 µm diameter spot, and the ion probe was operated at a mass resolving power of ~6000 to exclude molecular interferences. Oxygen flooding to a pressure of  $3.2 \times 10^{-5}$  Torr in the analysis chamber was used to increase Pb ion yields (Schuhmacher et al., 1994). Prior to sample (unknown) analysis, a calibration curve was established using the zircon standards 91,500 (for set-up) and AS-3 (embedded with the unknowns). Ages for unknown zircons were determined by comparison with a working curve defined by multiple measurements of zircon standard AS3 that yields concordant <sup>206</sup>Pb/<sup>238</sup>U and <sup>207</sup>Pb/<sup>235</sup>U ages of  $1099.1 \pm 0.5$  Ma. Raw machine data were reduced using the program ZIPS© (C. Coath, University of Bristol). Geochronological data are presented using the Isoplot software package (Ludwig, 2003) by generating Tera-Wasserburg plots (<sup>207</sup>Pb/<sup>206</sup>Pb vs. <sup>238</sup>U/<sup>206</sup>Pb) that yield discordia, and weighted average ages. Weighted average <sup>207</sup>Pb/<sup>206</sup>Pb ages were calculated when a sample had a population of zircons that overlaps concordia. We use Tera-Wasserburg plots



**Fig. 5.** Back-scattered electron and cathodoluminescence paired images of representative zircon grains reported in this work (a–f). (a) Example of a euhedral, igneous zircon; note rim with bright CL response. (b) Example of an igneous core with an overgrown metamorphic rim. (c) Example of a rounded, inherited core surrounded by an igneous overgrowth. (d) Example of a zircon with a texture typical of metamictization. (e) Zircon with a geochronology spot that has overlapped a fracture. (f) Zircon with a texture that suggests intense metamictization and/or hydrothermal alteration. Please see Supplementary Figs. S1 for the complete documentation of these samples.



for ancient zircons to take advantage of the large differences in  $^{207}\text{Pb}/^{206}\text{Pb}$  ratios that are not so well expressed in the conventional Wetherill Concordia plot.

### 3.5. Zircon trace-element geochemistry

All zircon rare earth element ( $[\text{REE}]_{\text{zirc}}$ ) analyses reported herein were measured on the UCLA Cameca *ims1270* ion microprobe following the approach described by Schmitt and Vazquez (2006). Analysis spots were  $\sim 25\ \mu\text{m}$  in diameter and carefully positioned to overlap previous U–Pb geochronology analysis points to provide internally-consistent data sets. The REE intensities were normalized to  $^{30}\text{Si}^+$  and corrected for interfering oxides. The trace element content of NIST standard glass 610 was used to determine the analytical sensitivity. Instrument stability was monitored by regular and frequent bracketed measurements on standard zircon 91,500.

## 4. Results

### 4.1. Petrographic analysis

#### 4.1.1. Metamorphism of Innuksuac complex supracrustals

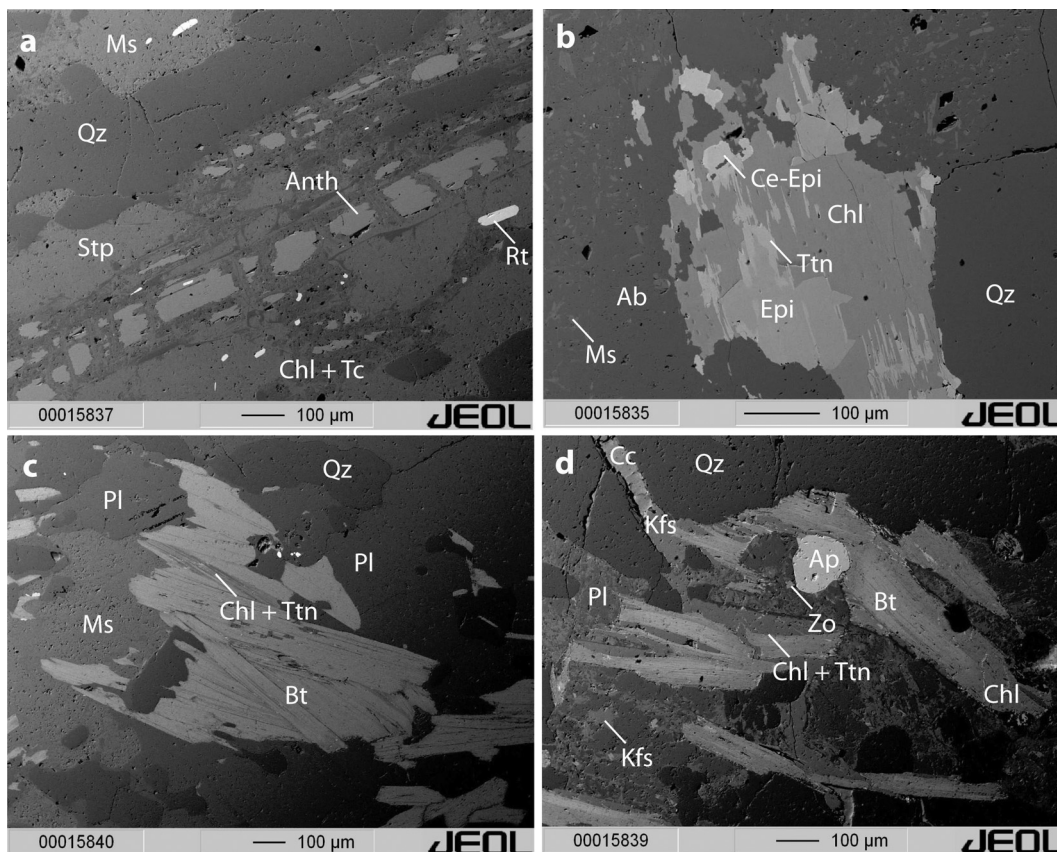
A suite of characteristic NSB and USB samples were earmarked for petrographic analysis; compositional descriptions of these samples are provided in the next section (4.2). Ukaliq sample IN14032A is provisionally interpreted by us as a (mafic) matrix-hosted “meta”-conglomerate (Dauphas et al., 2007; Caro et al., 2017). Although

quartz is subordinate in this rock, we interpreted it to have a conglomerate protolith based on the mineralogy and association of strongly s-deformed quartz “clasts” that are similar to samples described in the so-called “Porpoise Cove” locality (e.g. Darling et al. 2016). Thin section studies also revealed the presence of rounded zircon grains interpreted by us to be detrital. The main mineral assemblage of this sample is anthophyllite + muscovite + quartz + rutile + zircon (Fig. 6a). In retrogression, former biotite is now completely replaced by stilpnomelane (a common phyllosilicate in ferruginous sediments such as metagreywackes), and anthophyllite is replaced by chlorite + talc.

Sample IN12016 is a granite from the Boizard rocks. The mineral assemblage is chlorite + epidote + albite + K-feldspar + muscovite + titanite + quartz (Fig. 6b). Chlorite + epidote + titanite replace former biotite. Muscovite + epidote also grow within albite porphyroblasts. Epidote shows different contrast values in the BSE images indicative of variable REE contents. The Ce-rich epidote (allanite) probably replaces former monazite grains (e.g. Finger et al., 1998).

Sample IN14036 is a gray gneiss collected to the west of the NSB and contains the mineral assemblage biotite + plagioclase + muscovite + quartz + zircon (Fig. 6c). Biotite shows retrogressive replacement by the assemblage chlorite + muscovite + titanite.

Sample IN12044 is a Voizel tonalite sample and contains the mineral assemblage biotite + K-feldspar + plagioclase + quartz + apatite (Fig. 6d). It is evident from this sample that retrogression led to the formation of the secondary assemblage chlorite + titanite + epidote replacing biotite and plagioclase. Calcite also occurs in small veins.



**Fig. 6.** Back-scatter electron images illustrating the textural relationships in four representative samples discussed in the text. (a) Meta-conglomerate sample **IN14032A** shows the mineral assemblage anthophyllite (Anth) + muscovite (Ms) + stilpnomelane (Stp) + quartz (Qz) + rutile (Rt). Anthophyllite is replaced by the assemblage chlorite + talc (Chl + Tc). (b) Boizard suite granite sample **IN12016** contains the mineral assemblage chlorite (Chl) + epidote (Epi) + titanite (Ttn) + albite (Ab) + quartz (Qz). (c) The gray gneiss sample **IN14036** shows the mineral assemblage biotite (Bt) + muscovite (Ms) + plagioclase (Pl) + quartz (Qz). Biotite is replaced by chlorite + titanite (Chl + Ttn). (d) The Voizel tonalite sample **IN12044** contains the mineral assemblage biotite (Bt) + K-feldspar (Kfs) + plagioclase (Pl) + quartz (Qz) + apatite (Ap). Biotite is replaced by chlorite + titanite (Chl + Ttn) and albite contains small zoisite (Zo) grains.



#### 4.1.2. Secondary jaspilite in the Nuvvuagittuq rocks

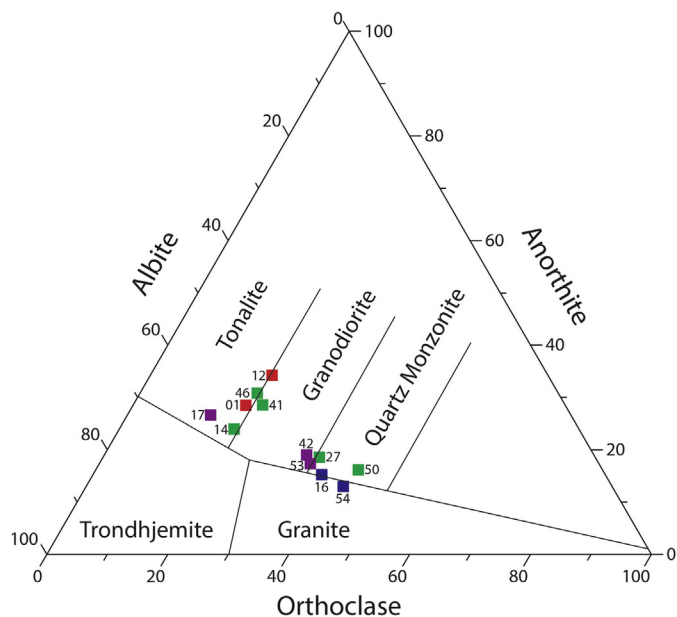
Later hydrothermal metamorphism in parts of the NSB – exemplified in the samples described in Sec. 4.1.1. – also led to re-hydration and locally extensive formation of structurally discordant (and locally as box work veinings) jaspilite (Supplementary Figs. S2). This process is described in detail elsewhere for other similarly metamorphosed banded iron formations (e.g. Czaja et al., 2018). It is evident given the metamorphic history and the obvious later (discordant, post-deformation) emplacement of the jaspilites, that the alleged microfossil features claimed by Dodd et al. 2017 could not be primary to the rocks.

### 4.2. Whole-rock geochemistry

#### 4.2.1. Central Tonalitic Gneiss

The largest outcrop of the Central Tonalitic Gneiss (CTG) is found near the geometric center of the NSB. Field observations show that rather than constituting a single mass of homogenous tonalitic gneiss, outcrops of the CTG instead comprise diverse interleaved granitoid gneisses and biotite-rich bodies (including possible paragneisses) with highly variable grains sizes; all are foliated in a general N-S direction (Supplementary Fig. S3). Disrupted and strongly recrystallized enclaves of rocks that superficially resemble banded iron-formations (at least in mineralogy), which show evidence for extreme quartz mobility and schlieren are also found locally. Although difficult to assess, it is possible that this BIF was originally part of the Nuvvuagittuq supracrustal succession before becoming engulfed by these intrusive granitoids. Previous works (O'Neil et al., 2007, 2019) proposed that the CTG was genetically related to the enclosing gneisses of the NSB (i.e. Voizel suite) but with no supportive evidence from geochronology.

The three CTG gneiss samples investigated (see sec. 4.3.1) here plot compositionally on the border between tonalite and granodiorite on the conventional TTG classification diagram (Fig. 7; Barker, 1979). These have typical (felsic) granitoid REE patterns with chondrite-normalized LREE enrichments around 100, and HREE enrichment of about 10, although the sample collected somewhat outside the main lobe of the

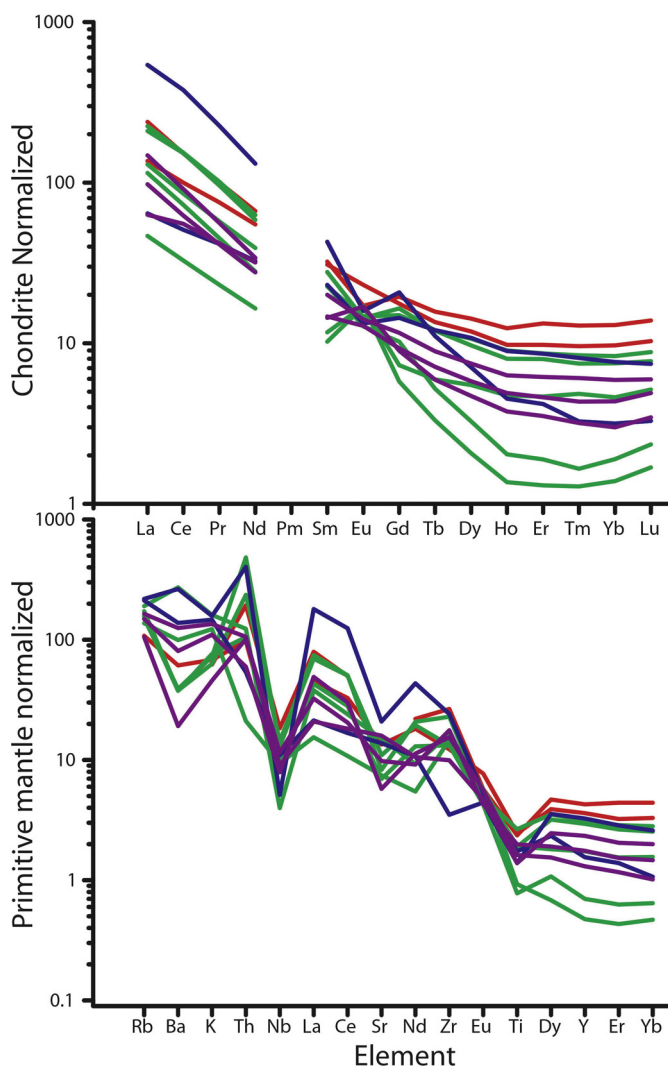


**Fig. 7.** Classification of samples based on normative anorthite, albite, and orthoclase compositions. Fields from Barker (1979). CTG samples are red, Voizel samples are green, Boizard samples are blue, and supracrustals and USB enclaves are purple. Numbers refer to the last two-digits of the sample identifiers in Supplementary Table S1.

NSB (IN14035) is less enriched in REEs than the other more ‘interior’ samples (IN05001, IN12012). These latter two interior samples have weak negative Eu anomalies (0.66 and 0.95) whereas sample — 035 shows a weak positive Eu anomaly (1.07; Fig. 8). On a multi-element plot, all three show typical granitoid patterns with strong negative Nb anomalies (all have Nb/Nb\* = 0.13) and moderate Ti anomalies (0.48 to 0.67). Samples IN05001 and IN14035 also have positive Zr anomalies, with values of 1.92 and 1.45, respectively (all whole-rock geochemical data are reported in Supplementary Table S1).

#### 4.2.2. Voizel suite

In outcrop, the coarse-grained Voizel (Avoi) rocks appear gray or white, depending on the degree of weathering (Supplementary Fig. S4). They have a pronounced gneissic texture with a foliation trend that conforms to the structural outline of the NSB and USB as seen in comparative field and aerial photographs, as well as regional-scale Google-Earth images. Unlike at the NSB, the Avoi rocks disrupt and enclose the USB into 1–10 m-scale enclaves except for a few rare cases. For this study, eight Avoi samples were chosen for geochemical analysis. Three come from the upper (northern) lobe of the Ukaliq locality and within the mass of Voizel gneisses (IN12014, IN12026, and



**Fig. 8.** (top) Chondrite-normalized (Anders and Grevesse, 1989) REE plot; (bottom) Primitive Mantle-normalized (McDonough, 1992) multi-element plot. Color coding of the samples as in Fig. 7 (Supplementary Table S1).

IN12027), and a further five samples come from the lower (southern) USB lobe (IN12041, IN12044, IN12046, IN12050, and IN12056; Fig. 3). These rocks plot in the tonalite, trondhjemite, granodiorite, and quartz monzonite fields on the TTG classification diagram (Fig. 7). On an REE plot, the samples have typical TTG gneiss patterns, as above with the CTG. Samples IN12050 and IN12027 fall in the quartz monzonite field and have large positive Eu anomalies, with  $\text{Eu}/\text{Eu}^*$  values of about 2. Samples IN12050 and IN12014 have relatively low concentrations of REE overall, and are depleted in HREE relative to other samples. Overall, Avoi samples show typical Archean igneous felsic patterns with strong negative Nb anomalies ( $\text{Nb}/\text{Nb}^*$  values between 0.05 and 0.51) and moderate Ti anomalies ( $\text{Ti}/\text{Ti}^*$  values between 0.28 and 0.70).

#### 4.2.3. Boizard suite

Two samples of Boizard suite rocks were selected for analysis; the rocks are coarse grained, pink, K-feldspar-bearing, have little to no fabric (Supplementary Fig. S5), and plot in the granite and quartz-monzonite fields (Fig. 7). The HREE enrichments for these samples are around 10 (normalized to chondrite) and both exhibit negative Eu anomalies ( $\text{Eu}/\text{Eu}^*$  values between 0.50 and 0.71). On a multi-element plot, the Avoi samples have typical Archean igneous felsic patterns with strong Nb anomalies ( $\text{Nb}/\text{Nb}^*$  values between 0.29 and 0.02) and moderate Ti anomalies ( $\text{Ti}/\text{Ti}^*$  values between 0.48 and 0.49). The Boizard suite dominates the landscape of the Innuskuac Complex. Its rocks enclose the Voizel suite and these in turn envelope the ancient gneisses hosting supracrustal rocks.

#### 4.2.4. Ancient granitoid gneisses from the Ukaliq locality

In places, rare, thin (0.1–1 m-scale) variably banded gray TTG gneisses can be seen to crosscut the various amphibolitic USB enclaves in the Ukaliq locality. Field relations suggest that these gneisses pre-date the dismemberment of what was a previously a coherent supracrustal belt or belts (*sensu lato*) via extensive intrusions of Avoi granitoids accompanied by deformation (Supplementary Fig. S6). Three of these crosscutting gneissic sheets were sampled for analysis, and all show typical Archean igneous felsic patterns with chondrite normalized LREE enrichments similar to the other gneisses described above. These samples have positive and negative Eu anomalies ( $\text{Eu}/\text{Eu}^*$  values between 0.89 and 1.45). The three gneissic samples also have strong negative Nb anomalies ( $\text{Nb}/\text{Nb}^*$  values between 0.14 and 0.17) and moderate Ti anomalies ( $\text{Ti}/\text{Ti}^*$  values between 0.45 and 0.63).

#### 4.2.5. Candidate volcano-sedimentary protoliths from Ukaliq

The largest, best-preserved ~50–100 m rafts of Ukaliq supracrustals comprise alternating bands of variably recrystallized BIF and/or quartz  $\pm$  amphibole  $\pm$  pyroxene  $\pm$  magnetite rocks; massive quartzite with relict banding that may include faint cross-bedding structures, possible graded bedding and/or stretched clasts; fine grained, strongly banded amphibolitic gneisses; massive to layered ultramafic schists; and sheets or dikes of trondhjemitic gneiss (Fig. 9). The northernmost extension of the Ukaliq supracrustal belt found in our field studies (Caro et al., 2017) is represented here by a coarse-grained quartzite (sample IN14032A), the protolith of which is interpreted by us as a quartz-pebble metaconglomerate within a broad sequence of quartzitic rocks of possible detrital origin as also found in the NSB (Cates and Mojzsis, 2007, 2009; Cates et al., 2013; Dauphas et al., 2007; Darling et al., 2013).

#### 4.2.6. Sample IN14036 – An orphaned member of the CTG?

This gray gneiss is similar in appearance to the CTG gneisses but comes from the west of the main NSB outcrops (Fig. 3; Supplementary Fig. S7). The sample also has a positive Eu anomaly ( $\text{Eu}/\text{Eu}^* = 1.08$ ), a strong negative anomaly ( $\text{Nb}/\text{Nb}^* = 0.12$ ), and a moderate Ti anomaly ( $\text{Ti}/\text{Ti}^* = 0.68$ ). We return to this sample later, owing to the fact that the geochronology revealed it to be older than the CTG gneisses.

### 4.3. U–Pb zircon geochronology

#### 4.3.1. Central Tonalitic Gneiss

The three CTG samples with geochemical data were prepared for geochronological analysis. Following on the previous descriptions (see sec. 4.1.1), one of these was collected from the internal northwestern margin of the NSB (IN05001), another was collected from the internal westernmost limit of the NSB (IN12012), while the third was collected at the external western limit of the NSB (IN14035).

For sample IN12012 the weighted mean age ( $^{207}\text{Pb}/^{206}\text{Pb}$ ) of the 11 most concordant grains (chosen grains are within 10% of concordia) is  $3652 \pm 14$  Ma (Fig. 10). These zircons tend to mostly be zoned cores surrounded by thin rims; the rims are too thin to measure by conventional ion microprobe techniques (Supplementary Fig. S1). Retrospective imaging of younger age spots which do not plot within 10% of concordance show that they come from altered cores filled with fractures. For sample IN05001, the weighted mean age of the 14 grains that cluster on the concordia line is  $3487 \pm 15$  Ma (Fig. 10). Zircons extracted from this sample are considerably more fractured and filled with inclusions than those of sample IN12012, but some preserve oscillatory zoning around cores. For sample IN14035, a total of 24 spots were analyzed on 24 grains. The weighted mean age of the grains that cluster on the concordia line (within 10% concordance) is  $3647 \pm 10$  Ma (all geochronological data are reported in Supplementary Table S2). It is evident that samples IN12012 and IN14035, although spatially separated, have well-constrained ages that are essentially identical within error.

#### 4.3.2. Voizel suite

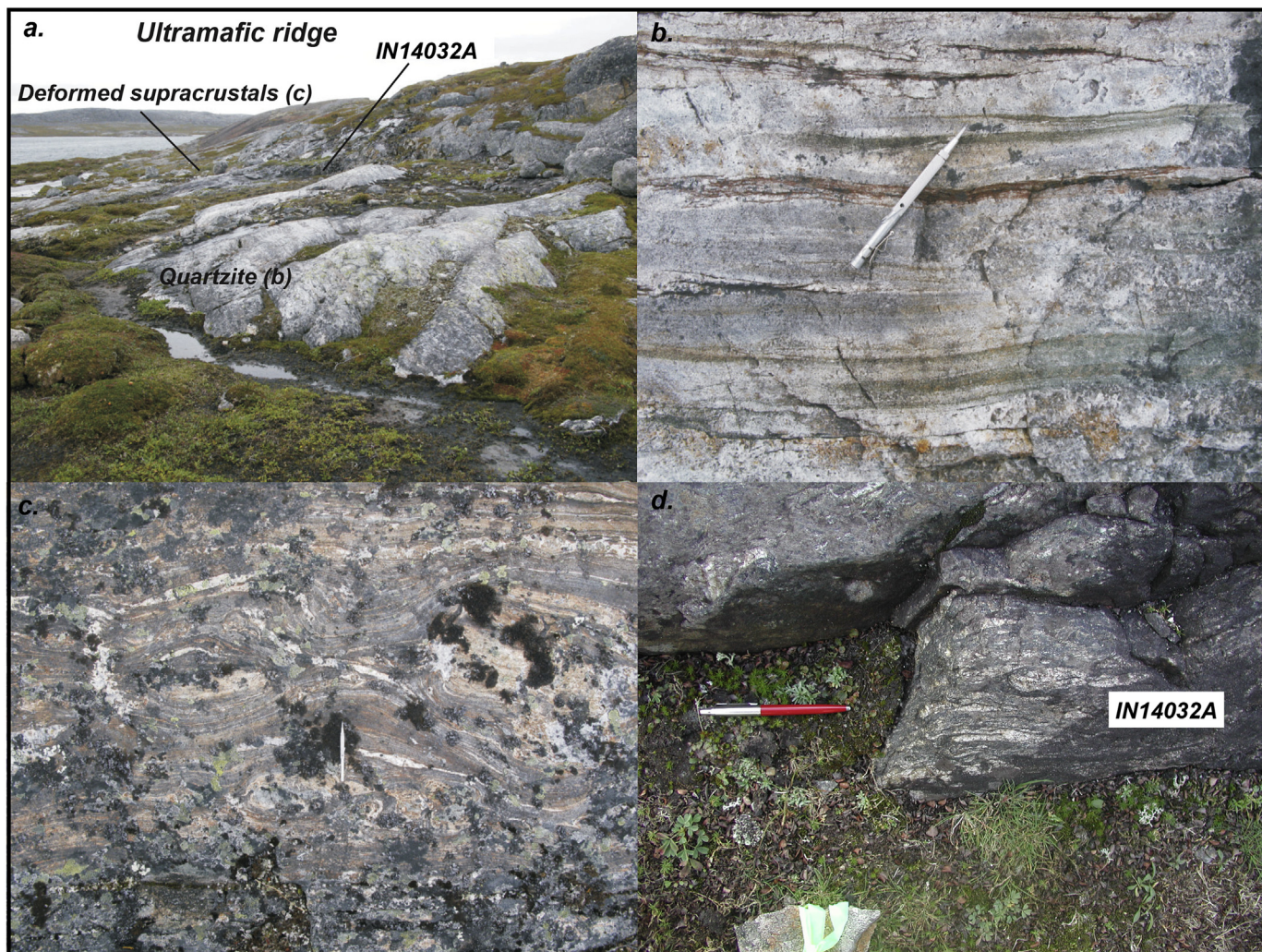
Seven samples from the Voizel suite with whole-rock geochemistry data were selected for geochronology. Three samples are from the northern lobe of the USB (IN12014, IN12026, and IN12027) whereas four samples are from its southern lobe (IN12041, IN12044, IN12046, and IN12050). Sample IN12014 gives an upper intercept age of 3550

$\pm 52 / -44$  Ma (MSWD = 4.7) based on assessment of 10 analyses on 10 zircons. Two zircon grains upon which there are four analyses that were not included in the collective age determination yield a weighted mean  $^{207}\text{Pb}/^{206}\text{Pb}$  age of  $1871 \pm 86$  Ma. These grains were highly discordant but trace along a horizontal axis on the Tera-Wasserburg plot, suggestive of recent lead loss (Fig. 11). Sample IN12026 gives an upper intercept age of  $3481 \pm 84$  Ma (MSWD = 1.9) with a large scatter around the discordia. Sample IN12027 yields a weighted mean  $^{207}\text{Pb}/^{206}\text{Pb}$  age from the four most concordant zircons of  $3519 \pm 16$  Ma. The southern USB lobe samples have ages that overlap these within error (with a skew towards slightly older ages). Sample IN12041 has an upper intercept concordia age of  $3550 \pm 120$  Ma (MSWD = 11.6). IN12044 has an upper intercept age of  $3554 \pm 46$  Ma (MSWD = 1.1) from analyses that plot on discordia. Sample IN12046 has an upper intercept concordia age of  $3483 \pm 140$  Ma (MSWD = 11.6) from analyses that are spread around the discordia. A weighted average age of  $3515 \pm 140$  Ma comes from analyses of the most concordant grains (within 3% of concordia) and are typically from larger grains.

Sample IN12050 comes from the easternmost margin of the Voizel suite, near the contact with the Boizard suite, and has an upper concordia intercept age of  $3437 \pm 170$  Ma (MSWD = 17). Although there are multiple upper intercept ages of ca. 3550, no analyses from individual Voizel suite zircons are this old. Instead, the oldest individual spot ages (ca. 3530) are from the cores of euhedral to subhedral zircons with younger overgrowths, whereas the younger population ages are exclusively from zircon rims (Supplementary Figs. S1).

With regards to the high degree of scatter in some of the upper concordia intercept ages reported here (with high MSWD), such values are not uncommon in polycyclic metamorphosed early Precambrian terranes (e.g. Corfu, 2013).





**Fig. 9.** (a) Light-colored quartz-rich rocks below the belt of ultramafic boudins in the northern Ukaliq supracrustal belt. Significantly, the quartz-rich rocks appear to show a progression from detrital quartzites to meta-cherts with magnetite-bearing laminae higher up. Sample location of IN14032A is annotated in the figure. (b) Close-up view of the quartz-rich rocks of probable detrital origin (i.e. quartzite proper). Note subtle aspects of the relict layering suggesting both grading and truncations. The greenish color is probably due to calc-silicates. (c) Mesoscopic view of highly deformed amphibolites and supracrustal gneiss, with strong boudinage of the more competent layers. Gneissosity has been pulled into the boudin necks and folded, whereas quartz-feldspar veins and pods fill discontinuities and areas of strong local extension. (d) Quartz-biotite schist at the base of the ultramafic body (IN14032A; labelled) for which detrital zircon U–Pb geochronology was attempted. These various outcrop-scale structures illustrate the structural style of the Ukaliq supracrustal belt, with the boudins being analogous to the larger ultramafic bodies in the wider area, and the quartz veins representing late Neoproterozoic granitic pegmatites that intruded late during the boudinage forming deformation. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)

#### 4.3.3. Boizard suite

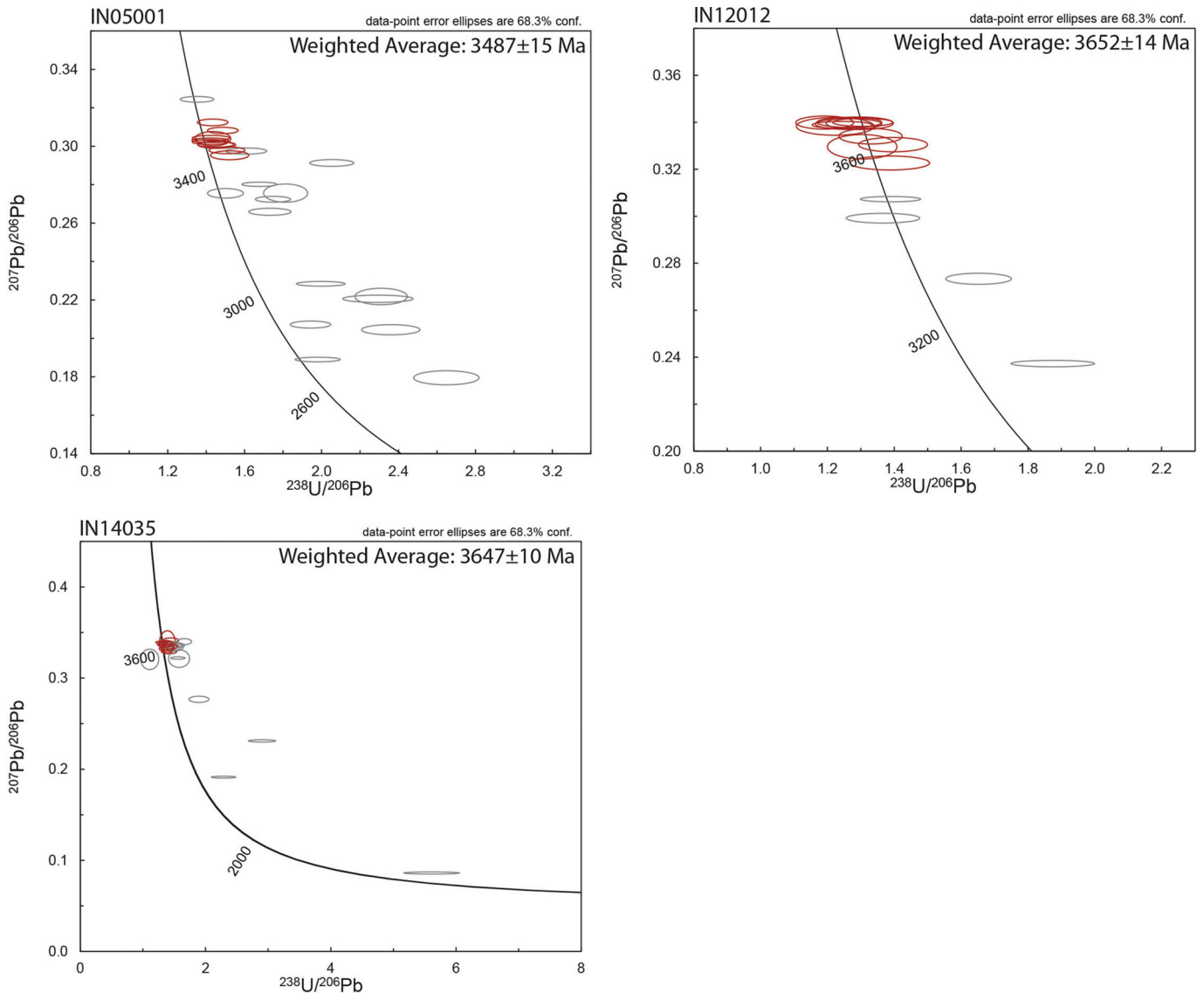
Two samples from the Boizard suite with whole-rock geochemistry were selected for geochronology. One was collected to the east of the northern lobe of the USB (IN12016), and one was taken to the south of the lower lobe (IN12054; Fig. 3). Sample IN12016 has a weighted mean  $^{207}\text{Pb}/^{206}\text{Pb}$  age of  $2706 \pm 19$  Ma from a total of 6 grains that cluster on or close to concordia (Fig. 12). Several old domains – as cores surrounded by zoned igneous overgrowths – were recognized from zircons within this sample with  $^{207}\text{Pb}/^{206}\text{Pb}$  dates up to ca. 3700 Ma; Supplementary Figs. S1, Supplementary Table S1). Other zircons with dates older than 3000 Ma also come from core regions of the grains, although in images some of these appear altered and fractured. Young, highly discordant dates come from zircons with altered cores that appear to be either metamict or filled with fractures and inclusions. Four highly discordant zircon grains give a weighted mean  $^{207}\text{Pb}/^{206}\text{Pb}$  age of  $1175 \pm 340$  Ma, but these trace along a horizontal axis on the Tera-Wasserburg plot, suggestive of recent lead loss and should be considered estimates. Sample IN12054 has a weighted mean  $^{207}\text{Pb}/^{206}\text{Pb}$  age of  $2720 \pm 20$  Ma from a total of 8 analyses that cluster on or near

concordia. The oldest date (and not, therefore necessarily age) in this sample,  $3431 \pm 6$  Ma, comes from a grain with a core surrounded by a younger rim ( $2921 \pm 9$  Ma). We find that the grains from the younger but still concordant population have euhedral to anhedral habits.

#### 4.3.4. Structurally intrusive gneisses to the Ukaliq supracrustal belt enclaves

Four samples collected from structurally invasive gneisses transecting enclaves within the various USB sites were selected for geochronology. Two of these were taken from the northern lobe (IN12017 and IN12031) and two were taken from the southern lobe (IN12042 and IN12053). Sample IN12017 gives a concordia upper intercept  $^{207}\text{Pb}/^{206}\text{Pb}$  date (not necessarily ‘age’) of  $3598 + 44 / - 37$  Ma, owing to the fact that the majority of analyses come from discordant zircons (Fig. 13). Some of the younger, discordant zircons in this sample have complex textures typical of hydrothermal growth or metamictization (Corfu et al., 2003) whereas the oldest zircon in this sample ( $3653 \pm 8$  Ma) comes from a core with a thick, zoned overgrowth (Supplementary Figs. S1). Sample IN12031 has two populations, with the older population more concordant which yields a weighted mean  $^{207}\text{Pb}/^{206}\text{Pb}$  age





**Fig. 10.** U–Pb zircon geochronology expressed in Tera-Wasserburg plots for Central Tonalitic Gneiss samples (IN05001, IN12012, IN14035); gray ellipses not included in the weighted average  $^{207}\text{Pb}/^{206}\text{Pb}$  age.

of  $3505 \pm 5$  Ma. The younger population lies on the discordia with the older population with an upper intercept at  $3496 \pm 35$  Ma. Sample IN12042 also gives an upper intercept at  $3492 \pm 190$  Ma from a cluster of the most concordant grains, but the oldest analysis in this sample comes from a 30% discordant grain with a  $^{207}\text{Pb}/^{206}\text{Pb}$  date of  $3694 \pm 9$  Ma. Sample IN12053 seems to have three populations, all of which are discordant. The youngest, most discordant grains yield a weighted average  $^{207}\text{Pb}/^{206}\text{Pb}$  date of  $2292 \pm 120$  Ma and come from either zircon rims or metamict cores (Supplementary Figs. S1). One spot on a zircon with 25% concordance gave a weighted average  $^{207}\text{Pb}/^{206}\text{Pb}$  date of  $3362 \pm 19$  Ma. The oldest population comprises two grains with  $^{207}\text{Pb}/^{206}\text{Pb}$  ages of  $3582 \pm 9$  Ma and  $3694 \pm 19$  Ma (Supplementary Table S1). These grains are euhedral to subhedral, and the oldest age domains correspond to zircon core surrounded by rims that have spongy textures typical of hydrothermal alteration (e.g., Hoskin and Schaltegger, 2003).

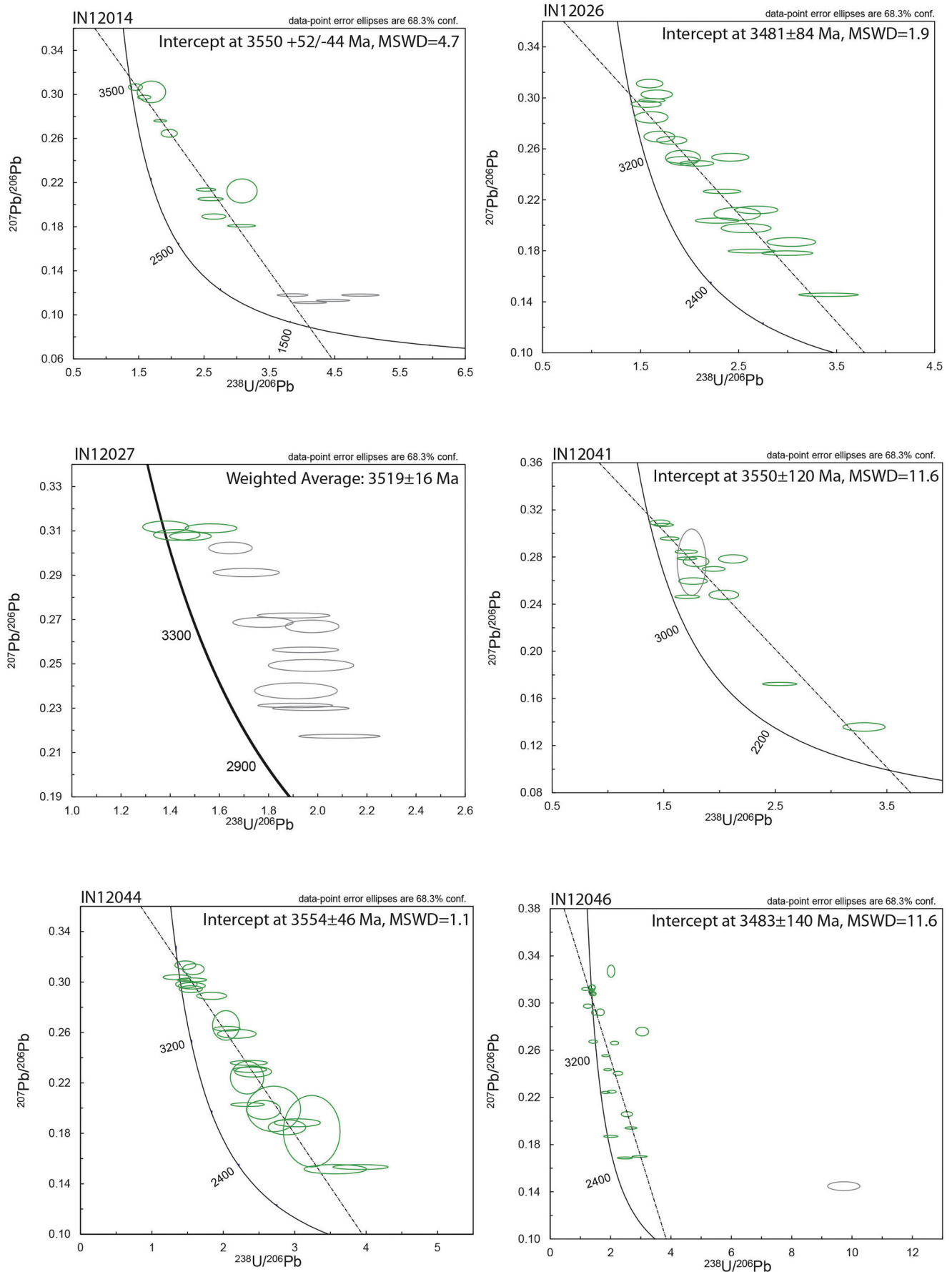
#### 4.3.5. Igneous and sedimentary protoliths in the Ukaliq supracrustal belt enclaves

A quartz-biotite schist sample IN14032A interpreted to be a ‘meta-conglomerate’ based on field relations, petrology (see sec. 4.2) and

resemblance to other rocks from the Porpoise Cove locality of the NSB (e.g., Dauphas et al., 2007; Cates et al., 2013), was discovered in the upper lobe of the Ukaliq belt. This rock is within a succession of quartzites, amphibolites, and ultramafic rocks (Fig. 8d; Caro et al. 2017) surrounded by Voizel gneisses. This sample has several different zircon populations the oldest of which is interpreted here to be of detrital origin (see sec. 5.3); the oldest of these clusters around a weighted average  $^{207}\text{Pb}/^{206}\text{Pb}$  age of  $3736 \pm 5$  Ma (Fig. 13). Younger, smaller, populations cluster around ca. 3650 Ma and ca. 2700 Ma. It is important to note that these zircon U–Pb age populations are defined by spot analyses with less than 5% discordance. This sample has a group of highly reversely discordant grains, with ages between  $3185 \pm 28$  Ma and  $3440 \pm 29$  Ma.

#### 4.3.6. Granitoid gneiss sample IN14036 from outside the NSB

Gray gneiss sample —036 was collected to the west of the westernmost limb of the NSB (Supplementary Fig. S7), approximately 150 m west of the outcrop of possible CTG-related sample IN14035 (Fig. 3). Sample —036 is similar in appearance to other TTGs analyzed herein (in particular samples IN12012 and IN12044), but yields a much older age. It has an upper concordia intercept  $^{207}\text{Pb}/^{206}\text{Pb}$  age of  $3760 \pm 17$  Ma, with a weighted mean age, comprising only the least discordant



**Fig. 11.** U–Pb zircon geochronology for Voizel suite samples investigated in this work; gray ellipses not included in weighted mean or upper intercept age calculations.

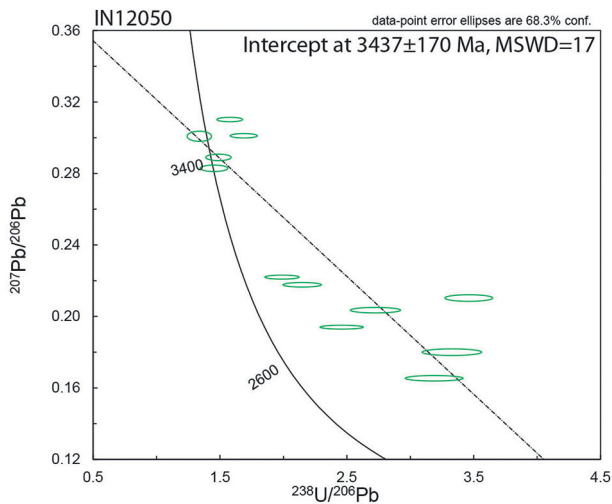


Fig. 11 (continued).

grains, of  $3750 \pm 7$  Ma. Zircons extracted from this sample generally plot on concordia, with only a few analyses defining the discordia (Fig. 13).

#### 4.4. Zircon Trace-element Geochemistry

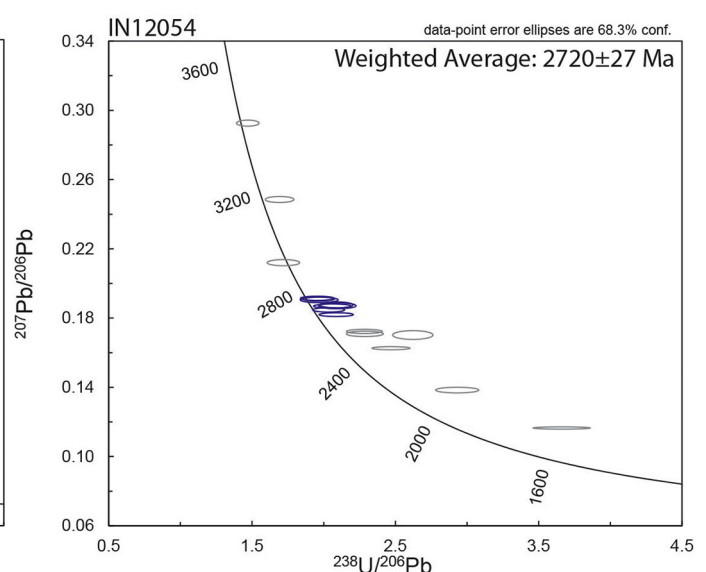
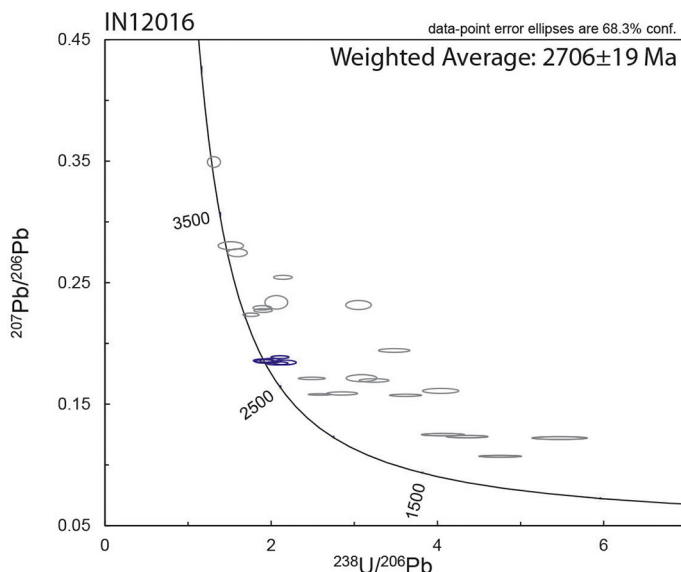
A routinely used means to determine whether a particular zircon age population is original to its host rock (e.g. Manning et al., 2006; Cates and Mojzsis, 2009; Cates et al., 2013; Mojzsis et al. 2014; Reimink et al. 2014), is the lattice strain model for ionic compatibility between the composition of a mineral (in this case, zircon) and a melt of the composition of the host rock (Blundy and Wood, 1994; Taylor et al., 2015). In those rocks with multiple zircon age populations, application of this model can help to determine the most likely age of the rock under the assumption that the original melt and native, age-representative zircons, formed in equilibrium. The assumption requires that the composition of the rock hosting the minerals in question represents the last melt composition (Onuma et al., 1968). As described elsewhere (Taylor et al., 2015) the model takes advantage of the chemical similarities of the trivalent rare earth elements, where the main controlling factor for how

easily these ions are incorporated into growing minerals is the predictable change in ionic radius with increasing atomic number from the lanthanide contraction. It has long been known that HREEs are more compatible in zircon than LREE because their ionic radii approach that of  $Zr^{4+}$  (0.84 Å in eight-fold coordination). Thus, the distribution of REEs in zircon is controlled by the composition of the melt, and the  $REE_{Zr}/REE_{Rock}$  should plot as a parabola for a zircon that crystallized in a melt with the composition of the whole rock representative of that melt (e.g. Trail et al., 2007; Mojzsis et al., 2014 and references therein).

Partition coefficients were determined for each zircon in our database ( $REE_{Zr}/REE_{WR}$ ) and fitted using the Blundy and Wood (1994) lattice strain model. The best fit parabola (described by its  $R^2$  value) for the exponential function was then calculated iteratively over 1000 model fits to the data. The higher the  $R^2$  value, the better the model fit of the data to the lattice strain model and consequently the more likely it is that the zircon REE chemistry comports with the whole-rock composition. In this work, we selected a subset of concordant zircons from the Voizel suite and CTG granite-granitoid gneisses for REE analysis (results in Fig. 14a; data reported in Supplementary Table S3) and lattice-strain modeling in order to refine our estimate(s) of the most likely crystallization age(s) for these two units. Three concordant zircons from the Voizel rocks (IN12027 and IN12046) and four concordant zircons from CTG sample IN12012 and one from IN05001 were set aside for REE analyses. The results of our analysis show that only the oldest zircons in this particular analysis have statistically good fits ( $R^2 \geq 0.9$ ) with their host-rock REE compositions (Fig. 14b), which we interpret to represent the crystallization age.

#### 4.5. Zr-in-rutile geothermometry

The temperature dependence of Zr incorporation into rutile grown in equilibrium with quartz and zircon yields a widely useful single-mineral geothermometer (Tomkins et al., 2007; Watson et al., 2006; Zack et al., 2004). Petrography of the four samples selected for Zr-in-rutile geothermometry (Sec. 4.2) revealed an older high-grade mineral assemblage and a texturally later secondary mineral assemblage as previously shown in Fig. 6. Owing to the pervasive retrogression of the samples (complete replacement of biotite, plagioclase) it was not possible to constrain P-T conditions using phase equilibrium calculations; instead this geothermometer provides a constraint on temperature (T) by analyzing rutiles from the purported metaconglomerate sample

Fig. 12. Boizard suite U–Pb zircon geochronology; gray ellipses not included in weighted mean  $^{207}Pb/^{206}Pb$  age.



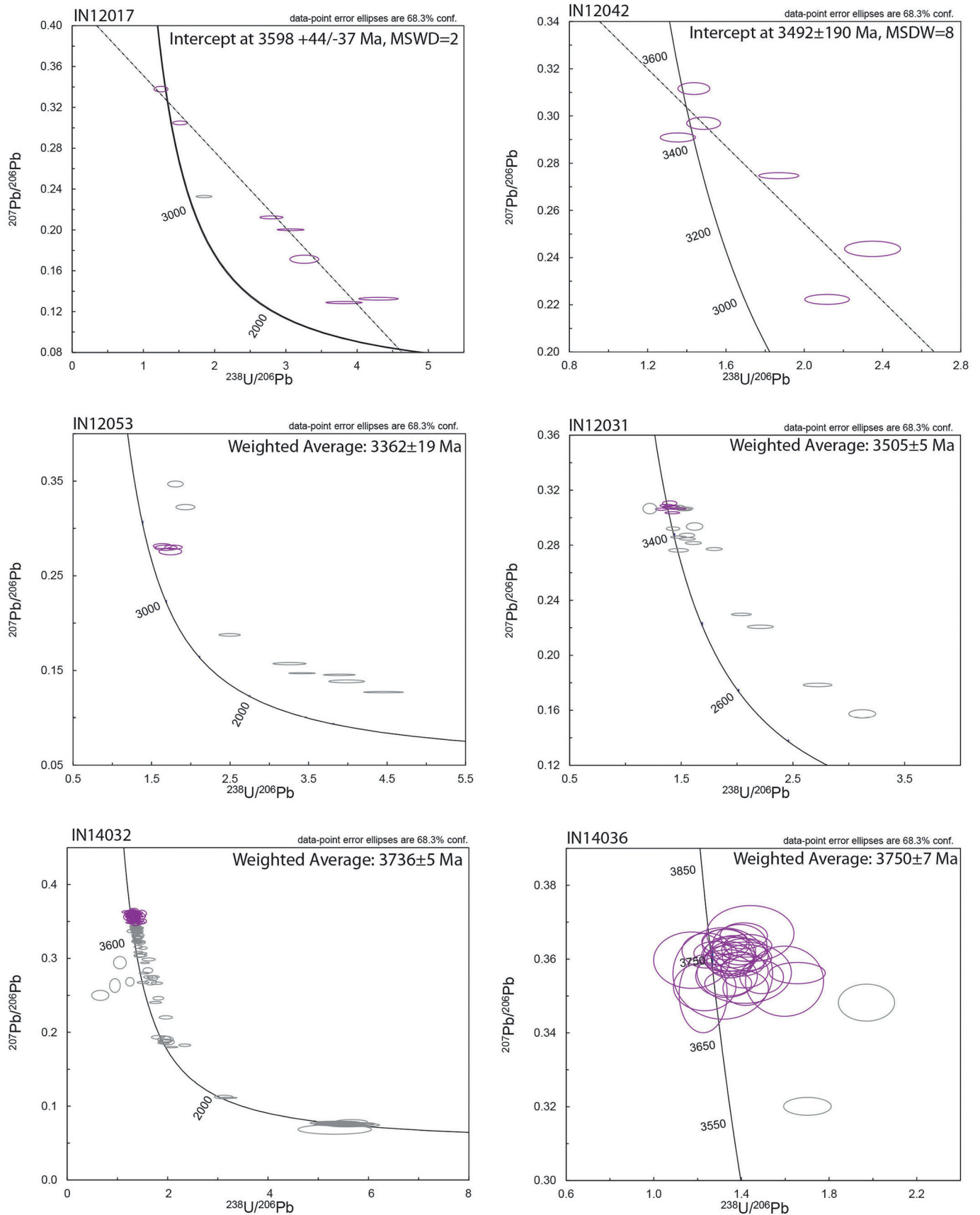
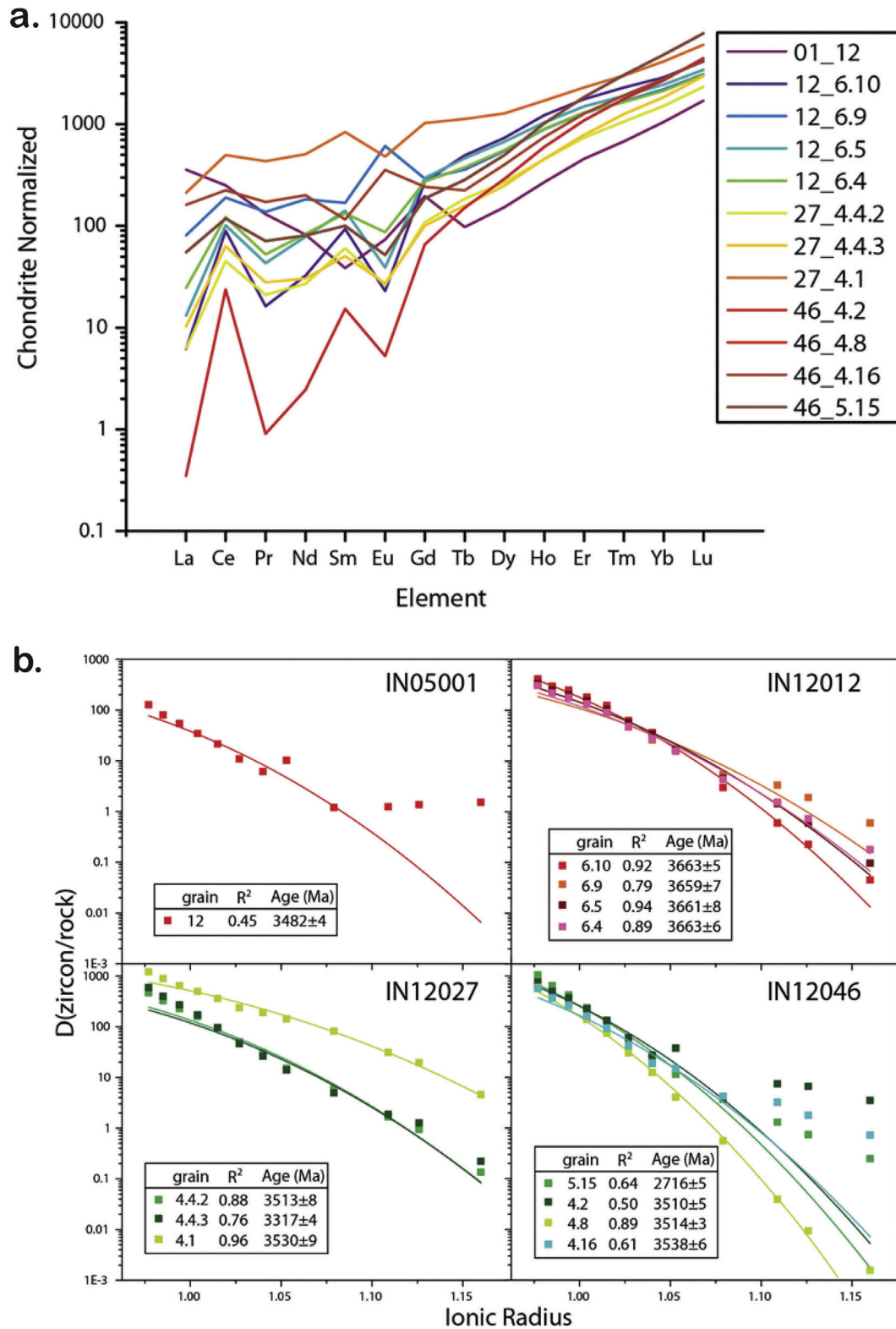


Fig. 13. U–Pb zircon geochronology for various supracrustal (NSB and USB) and USB intrusion samples; gray ellipses not included in weighted mean or upper intercept age calculations.



**Fig. 14.** (a.) REE data plotted for zircons used in the lattice-strain partitioning modeling work described herein. (b) Onuma diagrams of selected zircons from TTG and Voizel samples showing R<sup>2</sup> fitting values (see Mojzsis et al. 2014 and references therein). Colors correspond to the calculated partition values for a particular grain using its host rock, and the model best-fit line.

IN14032A. A total of 41 rutile analyses were performed by electron microprobe at the Institute of Mineralogy and Petrography, University of Innsbruck (Austria); Zr contents range from 407 ppm to 914 ppm (Supplementary Table S4). No differences between core and rim analyses

were found. The calibration of Tomkins et al. (2007) yielded T of 660–730 °C at 0.6 GPa and calculated mean T is 670 °C ± 40 °C (2σ). Due to the lack of precise pressure (P) constraints an arbitrary P of 0.6 GPa was chosen, which is in agreement with the observed mineral

assemblage in sample IN14032A. The calculations also show that P has only minor influence on the estimates presented here since mean T varies only from 660 °C at 0.4 GPa to 680 °C at 1 GPa (Triebold et al., 2012). We find that these results are in good agreement with the earlier study of Cates and Mojzsis (2009) for the NSB rocks, which used the conventional garnet–biotite and plagioclase–hornblende geothermometry.

#### 4.6. Rutile crystal chemistry as provenance indicator

Geochemical discrimination studies of specific detrital minerals can be used in provenance characterization. The widespread occurrence of rutile in medium to high-grade metamorphic rocks as well as sedimentary rocks, and its high mechanical and chemical stability during weathering, transport and diagenesis make rutile a prime-candidate for such work (Triebold et al., 2012). It was shown that Cr and Nb abundances can effectively be used to distinguish between metamorphosed mafic and pelitic lithologies; Fe content also serves as an indicator of metamorphic origin since metamorphic rutile contains generally >1000 ppm Fe (Zack et al., 2004). Rutiles investigated in this study have Cr contents ranging from 300 to 2400 ppm and Nb contents ranging from 570 to 1900 ppm. It is also noteworthy that Fe contents range from 300 to 3900 ppm, and most analyses exceed 1000 ppm. According to the chemical classification of Triebold et al. (2012) most of our rutile analyses suggest a metapelitic origin with only 7 rutile analyses indicative of origin from metamorphism in the mafic rock.

### 5. Discussion

#### 5.1. Geology, age and origin of Voizel suite and Central Tonalitic gneisses

Although long considered part of the same suite, new evidence shows that the Voizel and CTG instead belong to separate generations of TTG gneisses within the Inukjuak Domain. Their geochemical properties are similar: all the felsic gneisses analyzed here yield multi-element plot patterns that conform with classic Archean continental crust signatures and have negative Nb and Ti anomalies (Fig. 8b; Rudnick and Fountain, 1995). Both units are primarily tonalitic gneisses with about 70 wt% SiO<sub>2</sub> and moderate concentrations of alkalis (NaO + CaO + K<sub>2</sub>O ~9.0 wt%). The Voizel suite rocks, however, are more depleted in REEs (particularly the HREEs) than those from the CTG, and two Voizel suite rocks that plot in the quartz monzonite field as opposed to the tonalite field (Fig. 7) also have positive Eu anomalies. Gneissic rocks with these types of features (low REE concentrations, positive Eu anomalies) in ancient (Hadean–Archean and younger) metamorphic terranes have been interpreted as leucosomes during migmatite formation (e.g. Sawyer, 2008; Cates et al., 2013; Mojzsis et al. 2014). Although these small geochemical differences could be the result of internal variability, our preferred interpretation is that the geochronological differences between these two suites point to two distinct emplacement events. The Voizel suite is ca. 3550 Ma old based on upper concordia intercept ages, even if it has no individual zircon analysis with an age above 3538 ± 6 Ma (IN12046). The zircon with the best fit for the lattice strain model is among the oldest of the zircons (Sec. 4.4), with an age of 3530 Ma and 99% concordant (IN12027). In contrast to the Voizel suite samples, the CTG sample IN12012 has abundant zircons with ages up to 3663 ± 5 Ma, and the zircon with the best fit for the lattice strain model is 3661 ± 8 Ma. The ca. 3660 Ma cores are consistent with igneous growth and are interpreted as the maximum age of the CTG. Thus, the age of the CTG as determined by the upper intercept of the discordia on the Tera–Wasserburg plot, is the age of sample IN12012, which is 3652 ± 14 Ma. This age is about 100 million years older than the previously (albeit provisionally) assigned values for the Voizel suite (O’Neil et al., 2007, 2019 and references therein). Furthermore, David et al. (2009) presented geochronological data for a single mylonitized sample from the northwest of the margin of the NSB,

which was resampled by us as IN14035, and our data are in agreement. As IN14035 gives a weighted mean <sup>207</sup>Pb/<sup>206</sup>Pb zircon age of ca. 3650, this rock is most likely related to the CTG rather than to the more widely distributed and volumetrically more important Voizel suite.

#### 5.2. Nature of the Boizard suite gneisses

We find that the Boizard suite is both geochemically and geochronologically distinct from the other granitoid gneisses described within the Inukjuak Domain. It is a meta-granite and lacks the foliation observed in the Voizel and CTG outcrops. Zircons from the Boizard suite cluster mostly around ca. 2700 Ma, with some degree of inheritance. These older, inherited ages all come from grains with rounded cores, some of which are surrounded by igneous rims with strong oscillatory zoning. The inherited cores of the Boizard suite zircons include ages previously seen in the NSB (Cates et al., 2013), which suggests that the granites were extensively contaminated by zircons from the supracrustals during emplacement.

#### 5.3. Candidate Innuksuac complex supracrustal rocks beyond the NSB

In contrast with the Nuvvuagittuq outcrops, the Ukliaq rocks are more strongly deformed into a set of relatively small disconnected mafic to ultramafic enclaves with very minor (<1% of total outcrop) metasedimentary components. The three TTG gneisses sampled for this study were selected because they were mapped to cross-cut USB amphibolites. Results of our U–Pb zircon geochronology show that they are generally younger than those found in the ca. 3780 Ma Nuvvuagittuq outcrops further to the southwest (Cates and Mojzsis, 2007). Zircons from the oldest Ukliaq supracrustal belt enclave samples mostly yield an upper intercept age of ca. 3600 Ma (sample IN12017, Fig. 13), although the oldest and still highly concordant individual grain in this particular sample is ca. 3650 Ma. These ages might be interpreted as evidence that modification and dismemberment of the USB was contemporaneous with the emplacement of the CTG. Associated granitoid gneisses, which preserve the oldest age in this sample is tonalitic with less than 200 ppm Zr (Supplementary Table S1), thus making inheritance an unlikely source of the oldest grains. The other two supracrustal gneiss samples are granodioritic. Sample IN12053, with a zircon population of ca. 3360 Ma also has two older grains at ca. 3700 Ma and ca. 3580 Ma, but these grains are discordant (~30%). This sample has low Zr concentration (111 ppm), again arguing against inheritance, and all of these analyses come from zircon cores with oscillatory zoning. Thus an absolute age of this unit cannot be established, though the older analyses likely best represent the sample. Sample IN12053 also has a population of ca. 2700 Ma zircons, which is unsurprising due to the proximity of the enclave to the contact between the Voizel suite and Boizard suite. This age has not been observed in any of the other enclave samples analyzed in this study.

The quartz–biotite schist we provisionally assigned as a quartz-pebble “metaconglomerate” sample IN14032A has a minimum age of formation of 3550 Ma, as determined by field relationships with the surrounding Voizel suite. If the different disrupted USB rafts are related to one another, then the oldest age of the felsic enclaves sets the minimum age at 3600 Ma. The oldest zircon in this sample, at 3771 Ma, if interpreted as such gives a minimum age of the detrital source material (Supplementary Table S2; Fig. 13). A small zircon population in this sample the same age of the CTG further supports the idea that the dismemberment of the USB commenced at 3650 Ma, which led to partial re-setting of the ca. 3770 Ma zircons (Cates et al., 2013; Cates and Mojzsis, 2009). This could also suggest that the USB incorporated detritus after the emplacement of the CTG, but not since that time, and instead the quartz–biotite schist has a maximum age of 3650 Ma.

Sample IN14036 was collected outside of the limits of the NSB. However, since its age of ca. 3760 is the same as other samples collected from the NSB (Cates et al., 2013; Cates and Mojzsis, 2007), it is likely related



to the supracrustals in some way. This particular sample could represent an enclave of the NSB within the surrounding TTGs, suggesting that the NSB is not as cohesive as previously thought, or that the section of CTG that separates this sample from the rest of the belt is intruding the NSB, which extends further west than previously thought. It is also possible that the USB exists as a set of enclaves within the Voizel, and this sample represents the westernmost expression of the USB as well as a maximum age for the supracrustals. Further field studies to document further Eoarchean rocks in the northern Inukjuak Domain are needed to distinguish between these possibilities.

## 6. Conclusions

Poly-metamorphosed TTG gneisses of the Inukjuak domain were emplaced over multiple generations spanning a billion years. Our results from detailed U–Pb zircon geochronology of samples from both the Nuvvuagittuq and Ukalik supracrustal belts reveal multiple generations of zircon growth, spanning from ca. 3780 to 2700 Ma. These intrusive events, distributed over space and time, disrupted, deformed and metamorphosed the various supracrustal enclaves of the Innuksuac Complex and are recorded as episodes of metamorphic zircon growth from samples collected from within the enclaves as well as in the oldest TTG gneisses (Cates and Mojzsis, 2007; Cates et al., 2013; cf. Darling et al., 2013).

The results of our study can be summarized as follows:

1. Lattice strain partition modeling differentiates between different zircon age populations based on compositional parameters, and suggests both crystallization ages and neof orm zircon growth events for many of the studied units. Based on this combined approach, the age of the CTG can now be resolved to ca. 3650 Ma. The previously undated Voizel suite is about 100 million years younger, with a maximum age of approximately 3550 Ma. The Boizard suite, emplaced at ca. 2700 Ma, experienced widespread xenocrystic contamination during its emplacement and carries inherited zircons from all older suites in the terrane, including those from the NSB. Disruption and partial structural dismemberment of the Ukalik supracrustal belt (with a maximum age of ca. 3770 Ma) may be contemporaneous to the timing of the formation of the CTG. This age relationship may provide a link to the neighboring ca. 3750–3780 Ma NSB (Cates et al., 2013; Cates and Mojzsis, 2007). Sample IN14036 represents a new expression of an old TTG gneiss to the north and west, but further field study is needed to determine whether it represents an outcrop of the NSB or USB, or something else entirely.
2. Petrological investigations of select samples show pervasive retrograde metamorphism of the mineral assemblages. Our new Zr-in-rutile thermobarometry results underscore the history of amphibolite to greenschist facies metamorphism that accompanied poly-phase deformation events throughout the southern Inukjuak region. It is apparent that none of the Eoarchean Innuksuac supracrustal rocks escaped a variable history of retrograde metamorphism and deformation.
3. No confirmed microfossils older than about 3500 Ma exist, probably because all known Eoarchean rocks of sedimentary protolith – including those of the Inukjuak Domain – are both metamorphosed, deformed and variably recrystallized. In our view, the claims of Dodd et al. 2017 that microfossil shapes including “hematite tubes and filaments” are found in secondary jaspilite veins from Nuvvuagittuq are no exception. Indeed, many of the structures they describe including associations of hematite, quartz and magnetite are documented as non-biological features (e.g. Figueiredo et al., 2008). The geologic history described herein precludes preservation of any fine-scale primary structural features, either “stromatolites” or purported fragile microfossil shapes hosted in jaspilite box-work veinings (Taylor, 2011). Despite such shortcomings, the Inukjuak Domain supracrustal rocks are a unique complementary repository to the only other large (~3000 km<sup>2</sup>) domain of Eoarchean rocks known, those of the Itsaq Gneiss Complex in West Greenland. Investigations of the Inukjuak Domain have only scratched the surface of a region that was not recognized for its enormous antiquity until recently (2001–2002). To date, the most exciting rocks reported from the Inukjuak region are those that are also easily accessible by boat, but otherwise the vast inland localities can only be spot-checked here and there, with many more apparent from aerial photographs and satellite images.
4. That some rocks from the area indeed include paragneisses with protoliths ascribable to detrital “quartzites” and “conglomerates” means that they could preserve one of the oldest if not the oldest unconformities thus far recognized. For instance, quartz-rich sedimentary rocks appear to overly ca. 3650 Ma TTG basement in several places in the Nuvvuagittuq and Ukalik outcrops. Also, although data reported herein are largely of a reconnaissance nature on a rather extensive and well-selected suite of “reconnaissance samples” it is striking to note that there are no zircon grains older than about 3800 Ma despite thousands of zircon analyses since 2005. This result in of itself casts serious doubts on the interpretation that the Inukjuak Domain, including the Nuvvuagittuq Supracrustal Belt, preserves a formational history that goes back half a billion years older (to ca. 4300 Ma; cf. O’Neil et al., 2007, 2019).
5. The ca. 3.8 Ga Innuksuac Complex provides a hitherto unexpected windfall in the study of early Earth materials by providing a new, more global perspective beyond that of the Isua, Akilia, Manfred and Saglék Harbor rocks. The ~12,000 km<sup>2</sup> Inukjuak Domain is unquestionably an important resource that adds to our understanding of the physical, chemical, (proto-)biological and geodynamical state of the Earth’s surface at the Hadean–Eoarchean transition around 3850–3800 million years ago.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.lithos.2020.105520>.

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