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Joseph Martial Akame, Vinciane Debaille, Marc Poujol. Combined bulk-rock Lu-Hf and Sm-Nd isotopic study of Archean granitoids and mafic rocks from Sangmelima terranes (Ntem Complex, south Cameroon): Geodynamic implications. *Precambrian Research*, 2023, 392, pp.107072. 10.1016/j.precamres.2023.107072 . insu-04097151

**HAL Id: insu-04097151**

**<https://insu.hal.science/insu-04097151>**

Submitted on 16 May 2023

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# Precambrian Research

## Combined bulk-rock Lu-Hf and Sm-Nd isotopic study of Archean granitoids and mafic rocks from Sangmelima terranes (Ntem Complex, south Cameroon): Geodynamic implications.

--Manuscript Draft--

<b>Manuscript Number:</b>	PRECAM-D-22-00420R2
<b>Article Type:</b>	Research Paper
<b>Keywords:</b>	Hf isotopes; Nd isotopes; Archean granitoids; Congo Craton; Ntem Complex
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<b>Abstract:</b>	<p>Hafnium and neodymium isotopes provide robust tool tracers for tracking the mechanisms of crustal generation and differentiation, from mantle extraction to the late geological processes undergone by the studied rocks. Though these two isotope systems commonly behave similarly and define a positive correlation between <math>^{143}\text{Nd}/^{144}\text{Nd}</math> and <math>^{176}\text{Hf}/^{177}\text{Hf}</math> referred to as the "terrestrial array", they may differ in terrains having a complex geological history, such as Archean cratons. This study presents the first combined whole-rock Hf and Nd isotopic analyses of Archean mafic and felsic igneous rocks from the Sangmelima terranes in the Ntem Complex (located in the Northwest of Congo Craton (NW CC) in southern Cameroon). It mainly consists of Mesoarchean charnockites and tonalite-trondhjemite-granodiorite (TTG) suites, greenstone belts, mafic enclaves or dykes, and potassic granitoids. Mesoarchean charnockite and TTG suites have subchondritic initial <math>^{176}\text{Hf}/^{177}\text{Hf}</math> and <math>^{143}\text{Nd}/^{144}\text{Nd}</math> ratios with a mean initial (suffix (i)) <math>\epsilon^{176}\text{Hf}(i)</math> and <math>\epsilon^{143}\text{Nd}(i)</math> of <math>-3.30 \pm 1.40</math> and <math>-3.85 \pm 0.65</math> (2SD) respectively, implying the involvement of Eo- to Paleoarchean components in their petrogenesis. The bulk rock initial Hf and Nd isotope compositions of the gabbro and gabbro-norites are suprachondritic (<math>\epsilon^{176}\text{Hf}(i) = +3.4</math> to <math>+8.2</math>; <math>\epsilon^{143}\text{Nd}(i) = +0.9</math> to <math>+1.6</math>), indicate that they were derived from a depleted mantle source. Coupled with pre-existing data from the literature, these new Hf-Nd isotopic data indicate that two episodes of mantle-derived mafic magmatism in the Ntem Complex occurred during the Mesoarchean period (i.e., at <math>\sim 3.1</math> and <math>2.86</math> billion years (Ga)). Neoproterozoic dolerite dykes show broadly chondritic to supra-chondritic <math>\epsilon^{176}\text{Hf}(i)</math> and <math>\epsilon^{143}\text{Nd}(i)</math> values of <math>-0.28</math> to <math>+4.53</math> and TCHUR ages of <math>2.74 - 2.76</math> Ga, suggesting the derivation of doleritic magma from a depleted mantle source with either none or limited crustal residence time. Considering other Archean domains of the NW CC, there were multi-stage magmatism events and crustal growth events occurred at <math>\sim 3.75 - 3.31</math> Ga, <math>\sim 3.26 - 3.0</math> Ga, <math>\sim 2.92 - 2.85</math> Ga, and <math>\sim 2.75 - 2.72</math> Ga from Eoarchean to Neoproterozoic in the NW Congo Craton.</p>
<b>Suggested Reviewers:</b>	Hanika Rizo, Prof. Dr Associat Professor, Carleton University hanika.rizo@carleton.ca Hf and Nd isotopic specialiste  Martin Whitehouse, Prof .Dr. Professor, Swedish Museum of Natural History martin.whitehouse@nrm.se Archean specialist geology Research in isotope geology and bedrock geology  Kristoffer Szilas, Prof .Dr Associate Professor,, University of Copenhagen Department of Geosciences and Natural Resource Management

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<b>Response to Reviewers:</b>	

1 **Combined bulk-rock Lu-Hf and Sm-Nd isotopic study of Archean granitoids and mafic**  
 2 **rocks from Sangmelima terranes (Ntem Complex, south Cameroon): Geodynamic**  
 3 **implications.**

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10

11 **Abstract**

12 Hafnium and neodymium isotopes provide robust tool tracers for tracking the mechanisms of  
 13 crustal generation and differentiation, from mantle extraction to the late geological processes  
 14 undergone by the studied rocks. Though these two isotope systems commonly behave similarly  
 15 and define a positive correlation between  $^{143}\text{Nd}/^{144}\text{Nd}$  and  $^{176}\text{Hf}/^{177}\text{Hf}$  referred to as the  
 16 "terrestrial array", they may differ in terrains having a complex geological history, such as  
 17 Archean cratons. This study presents the first combined whole-rock Hf and Nd isotopic analyses  
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28 indicate that two episodes of mantle-derived mafic magmatism in the Ntem Complex occurred  
29 during the Mesoarchean period (i.e., at  $\sim 3.1$  and 2.86 billion years (Ga)). Neoproterozoic dolerite  
30 dykes show broadly chondritic to supra-chondritic  $\epsilon^{176}\text{Hf}_{(i)}$  and  $\epsilon^{143}\text{Nd}_{(i)}$  values of  $-0.28$  to  
31  $+4.53$  and  $T_{\text{CHUR}}$  ages of 2.74 – 2.76 Ga, suggesting the derivation of doleritic magma from a  
32 depleted mantle source with either none or limited crustal residence time. Considering other  
33 Archean domains of the NW CC, there were multi-stage magmatism events and crustal growth  
34 events occurred at  $\sim 3.75 - 3.31$  Ga,  $\sim 3.26 - 3.0$  Ga,  $\sim 2.92 - 2.85$  Ga, and  $\sim 2.75 - 2.72$  Ga  
35 from Eoarchean to Neoproterozoic in the NW Congo Craton.

36 Keywords: Hf isotopes; Nd isotopes; Archean granitoids; Congo Craton; Ntem Complex

37

## 38 1. Introduction

39 Both long-lived  $^{176}\text{Lu}-^{176}\text{Hf}$  and  $^{147}\text{Sm}-^{143}\text{Nd}$  isotope systems are robust tools for tracking  
40 the mechanisms of crustal generation and differentiation, from mantle extraction to the late  
41 geological processes it recorded through time (DePaolo, 1981; McCulloch and Bennett, 1994;  
42 Vervoort and Blichert-Toft, 1999; Salerno et al., 2021). Moreover, parent-daughter Lu/Hf and  
43 Sm/Nd ratios exhibit congruent low-pressure melting relationships as the parents, Lu and Sm,  
44 are both less incompatible compared to the daughters, Hf and Nd. This implies lower Lu/Hf  
45 and Sm/Nd ratios in the depleted mantle and, conversely, higher ratios in the crustal reservoirs.  
46 In general, the two isotopic systems are coupled and define a positive correlation between  
47  $^{143}\text{Nd}/^{144}\text{Nd}$  and  $^{176}\text{Hf}/^{177}\text{Hf}$  referred as the "terrestrial array" (Vervoort and Blichert-Toft, 1999;  
48 Vervoort et al., 2011). However, this analogous geochemical behavior of these isotopic systems  
49 may diverge, especially in ancient terrains like Archean cratons that generally have undergone  
50 a long-lasting complex geologic history (e.g., Caro et al., 2005; Rizo et al., 2011; Vervoort,

51 2014; Hammerli et al., 2019; Salerno et al., 2021). It has been suggested that the decoupling of  
52 Nd and Hf isotopes resulted from the generation of Ca- and Mg-enriched bridgmanite residues  
53 in the deep mantle during the solidification of the early magma ocean (Caro et al., 2005; Rizo  
54 et al., 2011). Recent work suggests that consumption reactions and recrystallisation of REE-  
55 bearing accessory minerals such as apatite, allanite and titanite during high-grade  
56 metamorphism may induce disturbance of the Sm–Nd and Lu-Hf isotopic systems of the bulk  
57 rock (Hammerli et al., 2014, 2019; Fisher et al., 2020; Hammerli and Kemp, 2021; Salerno et  
58 al., 2021). Thus, investigating the behaviour of both isotopic systems in the older rocks in  
59 different cratons may not only improve the understanding of the origin of the decoupling of Lu-  
60 Hf and Sm-Nd isotopic systems, but also to better constrain early Earth evolution and  
61 differentiation processes.

62 Archean cratons represent only 15% of outcropping ancient terrain worldwide (Goodwin,  
63 1981) and are the primordial archives that support our understanding of the Earth's early history.  
64 They mainly consists of the tonalite-trondhjemite-granodiorite (TTG) suite, charnockites,  
65 potassic granites (e.g., Martin, 1986) and greenstone belts which are dominantly composed of  
66 ultramafic to mafic volcanic sequences with associated sedimentary rocks (e.g., Smithies et al.,  
67 2018), variably metamorphosed up to amphibolite facies (Condie, 1994; de Wit and Ashwal,  
68 1995; Sproule et al., 2002; Furnes et al., 2015). Of granitoids, TTG suites represents the ancient  
69 archetypal juvenile felsic component of Archean terrains (Smithies, 2000; Moyen and Martin,  
70 2012 and references therein), and their decreasing occurrence in late Archean time with the  
71 progressive increase of the potassic granitoids indicate a significant geodynamic change on the  
72 Earth (Laurent et al., 2014; Halla et al., 2017; Cawood et al., 2018). Thus, studying and  
73 characterising the setting of TTG suites, their transition to potassic granitoids, and associated  
74 Archean mafic rocks is thus crucial to understand the differentiation and accretion mechanisms  
75 of Earth's earliest crust. Numerous models suggest that the TTG suites are the product of the

76 partial melting of earlier mafic rocks of the Earth's crust in subduction zone settings (Condie,  
77 1986; Martin, 1987; Moyen and Martin, 2012; Hoffmann et al., 2014). The episodic nature of  
78 TTG magmatism (i.e., at 3.80 billion years (Ga), 3.44 Ga, 3.33 Ga, 2.70 Ga) is, however, not  
79 compatible with the continuous character of modern subduction zone processes (Debaille et al.,  
80 2013; Martin et al., 2014). To add complexity, the parental magmas, presumably representing  
81 the first crust, are often no longer found on the terrestrial surface. The onset of the Archean  
82 continental crust may consequently mask a complex history of crustal reworking, concealing  
83 the nature of the most ancient crust on Earth.

84 In the present study, we performed the first combined bulk-rock Lu-Hf and Sm-Nd isotope  
85 analyses on Mesoarchean rocks from the Sangmelima terranes (Cameroon) located within the  
86 north-western margin of the Congo Craton (NW CC) (Fig.1). We aimed to characterize both  
87 the mantle sources and crustal components involved in the petrogenesis of the Archean crust in  
88 this important portion of the Congo craton. We also evaluate how much post-magmatic process  
89 affected the Hf and Nd isotope systems, and finally discuss the tectono-magmatic evolution of  
90 the NW Congo Craton with geodynamic implication of the Archean continental crustal growth  
91 models.

92 Fig.1

## 93 **2. Geological background and samples**

94 The Northwest Congo Craton (NW CC), in the west-central part of the African continent,  
95 covers the region of equatorial Africa that extends from the Republic of Congo to the Atlantic  
96 Ocean (Fig.1). It consists of three Archean domains, the Ntem Complex in southern Cameroon,  
97 the Monts de Cristal–North Gabonese Massif domain and Chaillu Massif in southern Gabon,  
98 surrounded by Proterozoic Orogenic Belts (e.g., Maurizot et al., 1986; Caen-Vachette et al.,  
99 1988; Toteu et al., 1994; Feybesse et al., 1998; Tchameni et al., 2000; Shang et al., 2004a;  
100 Chombong and Suh, 2013; Takam et al., 2009; de Wit and Linol, 2015; Thiéblemont et al.,

101 2018; Akame et al., 2020a). Eighty-five percent of the Archean basement rocks in the Ntem  
102 Complex are Mesoarchean rocks (e.g., Akame et al., 2020 end reference therein), which are  
103 dominated by ~3.15 – 2.85 Ga deformed and metamorphosed charnockites, TTGs and plutonic  
104 rocks, and minor supracrustal rocks (Tchameni et al., 2004; Chombong and Suh, 2013; Takam  
105 et al., 2009; Tchameni et al., 2010; Li et al., 2016; Akame et al., 2020a, 2021). The formation  
106 age of the greenstone belts remains poorly defined but probably falls in a period between 2.85  
107 and 3.04 Ga (Tchameni et al., 2004; Chombong and Suh, 2013). Paleoproterozoic charnockite in  
108 the Ntem Complex has been dated at  $3266 \pm 5$  million years (Ma) (SHRIMP zircon U-Pb;  
109 Takam et al., 2009). Nevertheless, this old crystallisation age of charnockites is usually  
110 considered as an inherited age (Li et al., 2016; Akame et al., 2020a). Amphibolite enclaves and  
111 gabbro dykes within the TTG suites were dated at  $2862 \pm 10$  Ma (Li et al., 2016) and  $2866 \pm 6$   
112 Ma (Akame et al., 2020a), respectively. Neoproterozoic granitoids are composed of monzogranite  
113 and syenogranite, and were generated at two main age peaks of ~2.75 Ga and ~2.68 Ga  
114 (Tchameni et al., 2000; Akame et al., 2020a). The granitic-gneissic basement and greenstones  
115 belts were intruded by Neoproterozoic dolerite dykes at 2.72 Ga (Shang et al., 2007).

116 Structural, metamorphic and geochronological data from the Ntem Complex indicate the  
117 prominence of two ductile high-grade tectonothermal events at ~2.84 – 2.82 Ga and ~2.78 –  
118 2.74 Ga (Maurizot et al., 1986; Tchameni et al., 2010; Akame et al., 2020a, 2020b, 2021). The  
119 Mesoarchean high-temperature metamorphism and the migmatization of TTGs was  
120 contemporaneous with the regional penetrative deformation (referred as D1), while the  
121 Neoproterozoic event is related to the folding of  $S_1$  regional foliation, development of  $C_2$  shear  
122 zones and minor intrusions of anatectic origin (Akame et al., 2020a, 2020b, 2021).

123 The samples analysed in this paper were collected during fieldwork in the Sangmelima  
124 granite-greenstone terrane (SGB) outcropping in the northern part of the Ntem Complex (Fig.  
125 1a). These 16 samples were previously studied by Akame et al. (2020a), who presented whole-



126 rock geochemical (Fig. 2, Table 1) and LA-ICP-MS zircon U–Pb data. Detailed field  
127 observations and a petrographic description of our sample set, including six mafic rocks (4  
128 gabbro-norites, 2 dolerites) and 10 granitoids, are provided in Akame et al. (2020a). At outcrops,  
129 Mesoarchean gabbro-norite (ca. 2866 Ma) occurs either as monolithic enclaves of centimetre to  
130 metre size (samples AJM11, AJM14, AJM17) or as larger (50–200 m) mafic dykes (samples  
131 AJM16 ~ 2866 Ma) enclosed in granitoids. The Mesoarchean granitoids include a charnockitic  
132 suite (ca. 2850 – 2912 Ma) and a TTG suite (ca. 2865–2870 Ma). Neoproterozoic high-K granites  
133 (AJM25) and dolerite dykes (ca. 2723 Ma; AJM3, AJM 6), in most cases, are intrusive  
134 veins/dykes within charnockites and TTG. Uranium-Pb dating of zircons from the charnockites,  
135 TTG and granites indicates that the granitoids of the Ntem Complex were emplaced between  
136 3050 Ma and 2750 Ma (Takam et al., 2009; Tchameni et al., 2010; Li et al., 2016; Akame et  
137 al., 2020a, 2021). In general, although there is a prominence of charnockites at ~2.91 Ga, TTG  
138 at ~2.87 Ga and granites at ~2.75 Ga, all three rock types can be found emplaced during those  
139 three Meso-Neoproterozoic events. Moreover, amphibolite and gabbro crystallisation at ~ 2.86 Ga  
140 was synchronous with the TTG magmatism in the Ntem Complex (Li et al., 2016; Akame et  
141 al., 2020a). Three samples (AJM10, AJM7, AJM16) analysed in this study are part of the series  
142 of samples on which zircon U-Pb ages have been previously determined (Akame et al., 2020a).  
143 Accordingly, the emplacement ages of the samples are also constrained.

144 Fig. 2

145 Table 1

### 146 3. Methods

147 Hafnium and Nd isotopic analyses of the 16 Mesoarchean samples of the Sangmelima area  
148 were conducted in the Laboratoire G-Time at Université libre de Bruxelles (ULB), Belgium.  
149 Around ~ 200 mg of whole rock powder of different samples were dissolved in a 1:3 mixture  
150 of subboiled concentrated HF and HNO<sub>3</sub>. After 2 days on a hot plate at 120 °C, the supernatant

151 was removed, and the residues were placed in Parr high-pressure vessels at 150 °C for 48h using  
152 fresh acids in the same proportion. All the samples were recombined, dried out and re-dissolved  
153 using 12N HCl. A 5% aliquot was removed from the initial dissolution and spiked using  $^{150}\text{Sm}$ –  
154  $^{148}\text{Nd}$  and  $^{175}\text{Lu}$ – $^{179}\text{Hf}$  spikes. Hf and Rare Earth Elements (REE) were first purified using  
155 cationic resin. Hf was collected first in 1.5N HCl and REE were eluted in 6N HCl. For both  
156 spiked and unspiked cuts, the REE were further separated using HDEHP resin by increasing  
157 the HCl molarity. For both spiked and unspiked Hf cuts, first, Fe was removed from the matrix  
158 using an anionic column in HCl 6N. Then, Hf was purified from Ti on Ln–Spec resin using a  
159 mixture of HCl 6N and  $\text{H}_2\text{O}_2$ , while Hf was collected in 4N HF. Hafnium, Lu, Sm and Nd  
160 spiked cuts and Hf and Nd unspiked cuts were analysed at ULB on the HR–MC–ICP–MS Nu–  
161 Plasma 2 coupled to an Aridus desolvator, using 0.05N  $\text{HNO}_3$  plus traces of HF for Hf  
162 measurements. Hafnium isotopic ratios were corrected for mass fractionation by internal  
163 normalisation to the value of  $^{179}\text{Hf}/^{177}\text{Hf} = 0.7325$ , and by bracketing every 2 samples with the  
164 JMC-475 standard, and the standard value of  $0.282160 \pm 0.000010$  was used for subsequent  
165 correction (Blichert-Toft, 2001). Analyses of JMC475 yielded an external reproducibility of 68  
166 ppm (2SD,  $n = 16$ ). Two analyses of the USGS BHVO-2 geostandard gave an average  
167  $^{176}\text{Hf}/^{177}\text{Hf}$  ratio of  $0.283111 \pm 9$  (2SD) in agreement with the value of  $^{176}\text{Hf}/^{177}\text{Hf} = 0.283105$   
168  $\pm 11$  reported by Weis et al. (2007). Neodymium isotope compositions were corrected for mass  
169 fractionation by internal normalisation to the value of  $^{146}\text{Nd}/^{144}\text{Nd} = 0.7219$ , and the Rennes Nd  
170 standard was measured every two samples. The accepted value of  $^{143}\text{Nd}/^{144}\text{Nd} = 0.511961$  for  
171 the Rennes standard was used for subsequent corrections (Chauvel and Blichert-Toft, 2001).  
172 The repeated measurements of the Rennes Nd Standard gave an external reproducibility of 59  
173 ppm (2SD,  $n = 19$ ). Two analyses of the BHVO-2 geostandard gave an average  $^{143}\text{Nd}/^{144}\text{Nd}$   
174 ratio of  $0.512975 \pm 10$  (2SD) in agreement with the value of  $^{143}\text{Nd}/^{144}\text{Nd} = 0.512981 \pm 10$   
175 reported by Weis et al. (2007). Spiked sample data were deconvolved following the procedure

176 of Debaille et al. (2007). Epsilon Nd and Hf values were calculated using the chondritic  
 177 (CHUR) values of  $^{143}\text{Nd}/^{144}\text{Nd} = 0.512630$ ;  $^{147}\text{Sm}/^{144}\text{Nd} = 0.1960$ ;  $^{176}\text{Hf}/^{177}\text{Hf} = 0.282785$  and  
 178  $^{176}\text{Lu}/^{177}\text{Hf} = 0.0336$  (Bouvier et al., 2008). The Nd and Hf depleted mantle model ages ( $T_{\text{DM}}$ )  
 179 were calculated using the following depleted mantle values:  $^{143}\text{Nd}/^{144}\text{Nd} = 0.513150$  and  
 180  $^{147}\text{Sm}/^{144}\text{Nd} = 0.2137$  (Goldstein et al., 1984), and  $^{176}\text{Hf}/^{177}\text{Hf} = 0.283294$  and  $^{176}\text{Lu}/^{177}\text{Hf} =$   
 181  $0.03933$  (Blichert-Toft and Puchtel, 2010). Isochrons were calculated with the ISOPLOT  
 182 program (Ludwig, 2012), using the decay constants  $\lambda^{176}\text{Lu} = 1.867 \times 10^{-11} \text{ yr}^{-1}$ ,  $\lambda^{147}\text{Sm} = 6.54$   
 183  $\times 10^{-12} \text{ yr}^{-1}$ .

#### 184 4. Results

185 Whole rock  $^{176}\text{Lu}$ – $^{176}\text{Hf}$  and  $^{147}\text{Sm}$ – $^{143}\text{Nd}$  data for the Sangmelima felsic and mafic rocks  
 186 are presented in Table 2. The  $\epsilon^{176}\text{Hf}$  and  $\epsilon^{143}\text{Nd}$  initial values (subscript i later) have been  
 187 calculated at 2900 Ma for charnockites, 2870 Ma for TTGs and 2866 Ma for gabbro-norites  
 188 (Tchameni et al., 2010; Li et al., 2016; Akame et al., 2020a). For the Neoproterozoic potassic  
 189 granite and dolerite dykes, the initial  $\epsilon\text{Hf}$  and  $\epsilon\text{Nd}$  have been calculated at 2750 Ma (Akame et  
 190 al., 2020a) and 2723 Ma (Shang et al., 2007), respectively.

191 Table 2.

192 The Lu–Hf and Sm–Nd isotope data of the charnockites define isochrons corresponding  
 193 to  $3397 \pm 88$  Ma (MSWD = 0.068,  $n = 3$ ) and  $2443 \pm 77$  Ma (MSWD = 3.8,  $n = 3$ ), respectively,  
 194 Fig. 3a-b; Tables 2). The charnockite samples show individual  $\epsilon^{176}\text{Hf}_{(i)}$  and  $\epsilon^{143}\text{Nd}_{(i)}$  values that  
 195 are all negative (from  $-4.1$  to  $-1.4$  for  $\epsilon\text{Hf}_{(i)}$  and  $-4.2$  to  $-1.9$  for  $\epsilon\text{Nd}_{(i)}$ ), except for sample  
 196 AJM20 with a very negative  $\epsilon^{143}\text{Nd}_{(i)}$  value of  $-54$  (Table 2). The  $f_{\text{Lu/Hf}}$  factor ( $f_{\text{Lu/Hf}} =$   
 197  $[(^{176}\text{Lu}/^{177}\text{Hf})_{\text{sample}} / (^{176}\text{Lu}/^{177}\text{Hf})_{\text{CHUR}}] - 1$ ) varies between  $-0.7955$  and  $-0.9272$  (average  $f_{\text{Lu/Hf}}$   
 198  $= -0.8745$ ) whereas the  $f_{\text{Sm/Nd}}$  factor ( $f_{\text{Sm/Nd}} = [(^{147}\text{Sm}/^{144}\text{Nd})_{\text{sample}} / (^{147}\text{Sm}/^{144}\text{Nd})_{\text{CHUR}}] - 1$ ) ranges  
 199 from  $0.2882$  to  $-0.6213$  with an average of  $-0.3058$  (Table 2). They yielded  $T_{\text{DM}}(\text{Hf})$  of 3317

200 to 3224 Ma (average =  $3283 \pm 6$  Ma, 2SD),  $T_{DM}(Nd)$  of 3456 to 3196 Ma (average =  $3355 \pm$   
201 370 Ma, 2SD; without sample AJM20, Table 2).

202 Fig.3

203 The Mesoarchean TTG suites, including granodiorites and tonalitic gneisses, give  $^{176}Lu-$   
204  $^{176}Hf$  and  $^{147}Sm-^{143}Nd$  isochronous ages of  $3539 \pm 220$  Ma ( $^{176}Hf/^{177}Hf_{(i)} = 0.280687 \pm$   
205  $0.000035$ , MSWD = 8.9, n = 4) and  $2661 \pm 760$  Ma ( $^{143}Nd/^{144}Nd_{(i)} = 0.508840 \pm 0.000480$ ,  
206 MSWD = 23; n = 4), respectively (Fig. 3c-b). They have individual negative values  $\epsilon^{176}Hf_{(i)}$  of  
207  $-7.4$  to  $-0.9$  and  $\epsilon^{143}Nd_{(i)}$  values of  $-3.3$  to  $-5.1$  (Table 2). Their  $f_{Lu/Hf}$  factor varies from  $-0.5803$   
208 to  $-0.9463$ , whereas  $f_{Sm-Nd}$  factors are scattered between  $-0.4283$  and  $-0.5695$  (Table 2). All  
209 Mesoarchean tonalite-granodiorite samples yield  $T_{DM}(Nd)$  of 3284 to 3513 Ma with an average  
210 of  $3366 \pm 84$  Ma (MSWD = 22; n = 6) and show  $T_{DM}(Hf)$  ages of 3172 to 3428 Ma with an  
211 average of  $3312 \pm 110$  Ma (MSWD = 42; n = 6) (Table 2). The tonalitic gneisses display higher  
212 Lu/Hf and Sm/Nd ratios (Lu/Hf = 0.0436 – 0.0989 and Sm/Nd = 0.1404 – 0.1852) than the  
213 granodiorites (Lu/Hf = 0.0126 – 0.0216 and Sm/Nd = 0.1394 – 0.1572). The 2.75-Ga-old  
214 potassic granite (sample AJM25) cutting across TTG has subchondritic initial  $^{176}Hf/^{177}Hf$  and  
215  $^{143}Nd/^{144}Nd$  ratios, with  $\epsilon^{176}Hf_{(i)} = -3.7$  and  $\epsilon^{143}Nd_{(i)} = -4.6$ . These results correspond to  
216  $T_{DM}(Hf)$  and  $T_{DM}(Nd)$  model ages of 3224 Ma and 3249 Ma, respectively (Table 2).

217 The whole rock Lu-Hf and Sm-Nd isotope data of all gabbro-norite samples define isochrons  
218 corresponding to  $3225 \pm 1400$  Ma (MSWD = 21; n = 4) and  $3324 \pm 1300$  Ma (MSWD=14; n =  
219 4), respectively, with  $^{176}Hf/^{177}Hf_{(i)}$  of  $0.28082 \pm 0.00084$  and  $^{143}Nd/^{144}Nd_{(i)}$  of  $0.508300 \pm$   
220  $0.001700$  (Fig. 3e-f; Tables 2). All samples have high  $^{176}Hf/^{177}Hf$  ratios (0.282396 – 0.283022),  
221 corresponding to positive  $\epsilon^{176}Hf_{(i)}$  values (+3.4 to +8.2) (Table 2), whereas the  $^{143}Nd/^{144}Nd$   
222 ratios range from  $\epsilon^{143}Nd_{(i)}$  values ranging from  $-2.3$  to  $+1.6$  (Table 2). They have  $f_{Lu/Hf}$  factors  
223 of  $-0.2616$  to  $+0.0320$  and  $f_{Sm/Nd}$  factor of  $-0.1969$  to  $+0.0581$ . Their ranges of  $T_{DM}(Hf)$  and  
224  $T_{DM}(Nd)$  model ages are 2634 to 3212 Ma and 3673 to 6118 Ma, respectively.

225 The Neoproterozoic dolerites have variable  $\epsilon^{143}\text{Nd}_{(i)}$  (-0.1 to +4.5) and  $\epsilon^{176}\text{Hf}_{(i)}$  (-0.3 to +16)  
226 (Table 2). However, the factors  $f_{\text{Sm/Nd}}$  and  $f_{\text{Lu-Hf}}$  show less scattered values, from -0.2935 to -  
227 0.3046 and -0.6423 to -0.7127, respectively (Table 2). Their  $T_{\text{DM}}(\text{Hf})$  of 2.27 to 3.09 Ga and  
228  $T_{\text{DM}}(\text{Nd})$  of 2.66 to 3.12 Ga model ages are younger than those of the Neoproterozoic potassic  
229 granite. Compared to the Mesoproterozoic gabbro-norites, the range of the Lu/Hf ratio is limited,  
230 between 0.068 – 0.084 and is lower (average 0.076).

231 Fig.3

## 232 5. Discussion

### 233 5.1. Post-magmatic process and its effects on Hf-Nd radiogenic isotope systematics

234 The Ntem complex underwent two major tectonothermal events between 2.84 and 2.74 Ga  
235 (Akame et al., 2020a, 2020b, 2021). Consequently, it is necessary to assess the potential effect  
236 of high-grade metamorphism and partial melting on the primary isotopic compositions of the  
237 Sangmelima igneous rocks before undertaking any petrogenetic and geodynamic interpretation.  
238 The gabbro-norite and gabbro exhibit granulite facies textures and show granulite facies  
239 assemblage of orthopyroxene, clinopyroxene, biotite, plagioclase and amphibole (Fig S1). Most  
240 of studied mafic and granitoid gabbro-norite samples show low loss on ignition (LOI) values  
241 (LOI < 3 wt%), which point to very limited post-crystallisation alteration effects. To evaluate  
242 the element mobility during metamorphism, we plotted selected trace elements (REEs, Ti, Nb,  
243 Ta, U, Y, Th, Cs, Rb, Pb, Sr and Hf) against Zr, one of the most immobile elements (Fig. 4 and  
244 Fig. S2) (Polat and Hofmann, 2003). Rare earth elements (REEs) and most of high field strength  
245 elements (HFSEs, e.g., Hf, Nb, Ta, and Ti), Sr and Y show positive correlation with Zr, and Ce  
246 anomalies are absent ( $\text{Ce}/\text{Ce}^* = 0.94 - 1.02$ ), indicating that these elements were immobile  
247 during metamorphism (Fig. S2) (Polat and Hofmann, 2003). The gabbro-norite samples have  
248 low Th ( 0.11 – 0.83ppm ) and U (0.13 – 0.29 ppm) contents and Th/U ratio (0.82 – 3.68,  
249 average = 1.76 ) similar to that of depleted MORB (Sun and McDonough, 1989), indicating  
250 that Th and U were also immobile during metamorphism. Assessment of U and Th mobility is

251 difficult in the granitoids because of Pb loss. In contrast, some major large ion lithophile  
252 elements (LILEs, e.g. Cs, Rb, Ba and Pb) and K, show variable degrees of scatter against Zr  
253 (Fig. S3), suggesting that they have been modified by post-magmatic processes. The positive  
254 Tm anomaly for AJM19 is likely related to analytical artefacts (Fig. 2c-d) (Roth et al., 2018).

255 The  $^{176}\text{Lu}$ – $^{176}\text{Hf}$  and  $^{147}\text{Sm}$ – $^{143}\text{Nd}$  isotopic data of gabbronorite samples give  
256 errorchronous ages of  $3225 \pm 1400$  Ma and  $3324 \pm 1300$  Ma, respectively (Fig. 3e-f), which are  
257 older than their emplacement age of  $\sim 2.87$  Ga (Fig. 3e-f), suggesting that either these (1)  
258 samples are from a composite source, or (2) Hf-Nd isotopic systems were reset during  
259 metamorphism. In the chondrite-normalised REE diagrams (Fig. 2e; Anders and Grevesse,  
260 1989a), most of gabbronorite samples show relatively consistent REE patterns that are similar  
261 to N-MORB ( $\text{La}_\text{N}/\text{Yb}_\text{N} = 0.82 - 0.98$ ), except for one sample (AJM17) which show a clear  
262 LREE-enrichment (high  $\text{La}_\text{N}/\text{Yb}_\text{N} = 3.2$  and low  $\text{Sm}/\text{Nd} = 0.25$ ). This may suggest that the  
263 gabbronorite did not derive from a composite source, while the enrichment in LREE of sample  
264 AJM17 could reflect the accumulation of a LREE-rich phase like monazite, apatite titanite and  
265 allanite. However, the best explanation for LREE-enrichment is secondary alteration, as LREE  
266 are more mobile than HREE. One way to evaluate a possible mixing between two sources is to  
267 plot  $^{176}\text{Hf}/^{177}\text{Hf}$  and  $^{143}\text{Nd}/^{144}\text{Nd}$  against  $1/\text{Hf}$  and  $1/\text{Nd}$  (Shafer et al., 2010; Viehmann et al.,  
268 2018; Wainwright et al., 2019), as mixing is characterised by the alignment of the samples along  
269 a line in such a plot. Figure 5 shows that  $^{176}\text{Hf}/^{177}\text{Hf}$  and  $^{143}\text{Nd}/^{144}\text{Nd}$  ratios do not correlate with  
270  $1/\text{Hf}$  and  $1/\text{Nd}$ , respectively (Fig. 5a-b), indicating that a mixing processes cannot account for  
271 the Hf–Nd errorchronous apparent ages observed for the Sangmelima gabbronorite. The L–Hf  
272 or Sm–Nd isotopic systems were possibly disturbed at the individual mineral scale. Samples  
273 AJM16 and AJM17 exhibit decoupled Hf-Nd isotopic compositions with  $\epsilon^{143}\text{Nd}_{(i)}$  value of  
274  $-1.58$  to  $-2.33$  and  $\epsilon^{176}\text{Hf}_{(i)}$  of  $+3.37$  to  $+8.25$ , and fall in the crustal contamination field (Fig.  
275 4a; Hasenstab et al., 2021). However, crustal contamination and/or assimilation commonly

276 affect(s) both Lu-Hf and Sm-Nd isotope systems in mafic magmas (Nebel et al., 2014;  
277 Hasenstab et al., 2021). We therefore consider that the negative  $\epsilon^{143}\text{Nd}_{(i)}$  of samples AJM16  
278 ( $-1.58$  at 2870 Ma) and AJM17 ( $-2.33$  at 2870 Ma) were likely resulting of post-crystallisation  
279 disturbance of the Sm-Nd isotope system. Hammerli et al. (2019) suggested that the formation  
280 or recrystallisation of REE-enriched minerals such as monazite, xenotime, apatite, allanite and  
281 epidote during metamorphism would alter the subsequent Nd isotope evolution of the rock by  
282 fractionation of their  $^{147}\text{Sm}/^{144}\text{Nd}$  ratios. Following granulite-facies mineral assemblages and  
283 geochemical evidence for LREE-phase accumulation in sample AJM17, we suggest that their  
284 Sm-Nd systematics were more easily disturbed during the 2.84–2.84 Ma metamorphic event  
285 widely reported from the Sangmelima region (Shang et al., 2004a; Akame et al., 2020a, 2021).  
286 Monazite alteration with apatite corona in granodiorites hosting gabbro (sample AJM16)  
287 (Akame et al., 2021), suggests that recrystallisation of apatite (high Sm/Nd) and other Nd-  
288 bearing phases may have likely controlled the Nd isotopic composition of this rock.  
289 Collectively, the Sm-Nd isotope system of samples AJM16 and AJM17 does not represent the  
290 primary signature of their protolith, and they were disturbed during post-crystallisation process.

291 Two dolerite samples (AJM3, AJM6) show weathering texture such as chloritisation of  
292 biotite and sericitisation of plagioclase (Fig. S2), but moderate LOI values ( $2.32 - 2.80 \text{ wt}\% <$   
293  $5$ ), and show relatively uniform REE patterns (Fig. 2a-b), and no Ce/Ce\* anomaly, indicating  
294 that REEs have not undergone significant modification (Polat and Hofmann, 2003). Sample  
295 AJM3 shows almost similar initial Nd and Hf isotopic values ( $-0.11$  and  $-0.28$ , respectively),  
296 but in contrast sample AJM6 has a very high  $\epsilon^{176}\text{Hf}_{(i)}$  value (up to  $\epsilon^{176}\text{Hf}_{(i)} = +16.09$ ) compared  
297 to the Sm-Nd isotope system with  $\epsilon^{143}\text{Nd}_{(i)}$  of  $+4.53$ . Considering that Hf might be remobilised  
298 in easily altered mafic rocks given the lack of robust phases (e.g. zircon) to anchor Hf or Lu  
299 (Hoffmann and Wilson, 2017), we suggest that the Lu-Hf system of this sample was disturbed  
300 during post-crystallisation processes. In addition, the Hf depleted model age of ca. 2270 Ma is

301 younger than the apparent emplacement age of  $2723 \pm 3$  Ma (Shang et al., 2007), and does not  
302 overlap with any known magmatic or igneous event in the Sangmelima Terrane.

303 Most of the granitoid samples yield coherent  $\epsilon^{143}\text{Nd}_{(i)}$  and  $\epsilon^{176}\text{Hf}_{(i)}$  values ranging from  
304  $-1.9$  to  $-5.1$  and  $-0.9$  to  $-7.4$ , respectively (Table 2), whereas all but one Mesoarchean  
305 charnockite (AJM20) show very negative  $\epsilon^{143}\text{Nd}_{(i)}$  of  $-54.0$  with no significant  $T_{\text{DM}}(\text{Nd})$  age.  
306 Nevertheless, there is clearly a deviation from the trend in Hf, Nd isotope versus their  
307 parent/daughter ratios diagram and both isotopic systems give different “errorchron” ages (Figs.  
308 3a-b, 3c-d). The lack of correlation between measured  $^{143}\text{Nd}/^{144}\text{Nd}$ -vs.  $1/\text{Nd}$ , and  $^{176}\text{Hf}/^{177}\text{Hf}$  vs.  
309  $1/\text{Hf}$  plots (Fig. 5a-b), suggests that the errorchrons are not an artefact of mixing (Fig. 5b), and  
310 may rather indicate the effects of different petrogenetic processes such as partial melting or  
311 melt segregation. For sample AJM20, the  $\epsilon^{143}\text{Nd}_{(i)}$  value of  $-54.0$  suggests that the Sm–Nd  
312 isotopic systematics were substantially disturbed or reset possibly due to the breakdown of less-  
313 stable REE-rich accessory phases during the Mesoarchean high grade metamorphism events.

## 314 ***5.2. Hf - Nd isotopic signature and crustal growth***

### 315 ***5.2.1. Granitoids***

316 Shang et al. (2004a) studied the geochemical and Nd–Sr isotopic signatures of the  
317 Mesoarchean felsic rocks of the Sangmelima terranes and concluded that the primary magma  
318 of both charnockite and TTG suites derived from the similar late Paleoproterozoic eclogite facies  
319 basaltic proto-crust sources with contribution of a slightly enriched proto-crust. The  
320 Mesoarchean charnockites studied here are magnesian cordilleran-type granitoids (Akame et  
321 al., 2020a), and show Sr/Y ratio similar to those of Archean low to medium pressure TTG,  
322 which probably derived from partial melting of hydrous basalts (Moyen, 2011). Following the  
323 major and trace elements composition of the Sangmelima charnockites, Akame et al. (2020a)  
324 suggested that these rocks were likely formed by high degrees of partial melting of mafic rocks.  
325 Detailed geochemical studies (Akame et al., 2020a) have suggested that most of Mesoarchean



326 Sangmelima TTG suites are low to medium pressure TTGs that were probably generated by  
327 partial melting of enriched mafic rock and/or continental arc magma, possibly in a subduction  
328 zone. Both studies emphasise a contribution of a crustal component in the formation of the  
329 Sangmelima Mesoarchean granitoids.

330 The Mesoarchean charnockites and TTG suites we analysed have subchondritic  $\epsilon^{176}\text{Hf}_{(i)}$   
331 (from  $-0.91$  to  $-7.37$  average of  $-3.3 \pm 1.4$ ,  $n = 10$ ) and  $\epsilon^{143}\text{Nd}_{(i)}$  (from  $-1.9$  to  $-5.1$ , average of  
332  $-3.85 \pm 0.65$ , 2SD;  $n = 9$ ) values (Fig. 5c-d). These subchondritic  $\epsilon^{143}\text{Nd}_{(i)}$  and  $\epsilon^{176}\text{Hf}_{(i)}$  values  
333 suggest either the involvement of significant crustal contamination or the involvement of a  
334 long-term Nd and Hf enriched component (relative to Sm and Lu) in these rocks. They reveal  
335 very similar  $T_{\text{DM}}(\text{Hf})$  ( $3.22 - 3.32$  Ga, average =  $3.28$  Ga and  $3.17 - 3.43$  Ga, average =  $3.32$   
336 Ga, respectively) and  $T_{\text{DM}}(\text{Nd})$  ( $3.19 - 3.45$  Ga, average =  $3.36$  Ga and  $3.28 - 3.51$  Ga, average  
337 =  $3.37$  Ga, respectively), indicating that a proto-crust of ca.  $3.2 - 3.5$  Ga might have existed in  
338 the Sangmelima area. Li et al. (2016) reported zircon  $\epsilon^{176}\text{Hf}_{(i)}$  values between  $-1.3$  and  $+2.0$   
339 with an average of  $+0.0$  for similar  $2.92$  Ga old charnockites from the Menguémé area, whereas  
340 a ca.  $2.87$  Ga Djoum trondhjemite displays  $\epsilon^{176}\text{Hf}_{(i)}$  between  $-2.0$  and  $-6.1$  with an average of  
341  $-3.7$ , like the Sangmelima TTG suites. However, the zircon Lu-Hf model ages of ca.  $3.3 - 3.5$   
342 Ga for the charnockite and  $3.5 - 3.8$  Ga for the trondhjemites (Li et al., 2016) are older than  
343 the whole rock  $T_{\text{DM}}(\text{Hf})$  ages ( $3.17 - 3.43$  Ga) calculated in this study. Nevertheless, the older  
344 whole-rock  $T_{\text{DM}}(\text{Hf})$  age of  $3.43$  Ga overlap with the charnockite zircon  $T_{\text{DM}}(\text{Hf})$ . It is important  
345 to note that zircon Hf model age is usually calculated using a fixed Lu/Hf value that is supposed  
346 to represent the magmatic source of the zircon-hosting rocks (Amelin et al., 1999; Bodet and  
347 Schärer, 2000; Griffin et al., 2002; Iizuka et al., 2005; Kemp et al., 2007), but both elements  
348 may significantly be decoupled during intracrustal differentiation processes involving garnet or  
349 zircon, leading to spurious zircon  $T_{\text{DM}}(\text{Hf})$  ages (Bea et al., 2018). In addition, Bea et al. (2018)  
350 suggested that using a single fixed Lu/Hf source value to calculate zircon  $T_{\text{DM}}(\text{Hf})$  ages may

351 reflect the Lu/Hf heterogeneity of the source and lead to large overestimates of zircon  $T_{DM}(Hf)$   
352 compare to whole-rock Hf or Nd model ages, notably in the case of zircons being much younger  
353 than their sources. Therefore, our new bulk-rock Lu–Hf isotope data combined with published  
354 zircon Hf data suggest the derivation of TTG magmas from an older enriched crustal reservoir,  
355 whereas the charnockite parental magmas evolved from a reservoir that was globally chondritic  
356 to enriched of Eo- to early Mesoarchean ages ( $\sim 3.2 - 3.8$  Ga).

357 Our new long-lived  $^{147}Sm-^{143}Nd$  isotopic data ( $\epsilon^{143}Nd_{(i)} = -1.9$  to  $-5.1$ ; Fig.5d) are consistent  
358 with published whole-rock  $\epsilon^{143}Nd_{(i)}$  from Sangmelima Mesoarchean granitoids (Shang et al.,  
359 2004a). The  $T_{DM}(Nd)$  model ages yield early Mesoarchean to Paleoproterozoic ages (ca.  $3.20 -$   
360  $3.52$  Ga) for the samples studied, older than the inherited zircon U-Pb ages of  $3057 \pm 7$  and  
361  $3155 \pm 11$  Ma for the Sangmelima charnockites and TTG gneisses, respectively ( Akame et al.,  
362 2020a). Pouclet et al. (2007) reported near chondritic  $\epsilon^{143}Nd_{(i)}$  values ( $-1.6$  to  $+1.3$ ) with an  
363 average of  $+0.8$  and a  $T_{DM}(Nd)$  age around  $2.92 - 3.10$  Ga for Mesoarchean charnockites from  
364 the Ntem Complex. However, these Nd model ages ( $2.92 - 3.10$  Ga) are younger than the  $3057$   
365  $- 3266$  Ma ages of the oldest magmatic zircons dated in the granitoids from the Ntem complex  
366 (Takam et al., 2009; Akame et al., 2020a). In addition, some samples analysed in these previous  
367 studies were collected near or within the Nyong Paleoproterozoic Complex. Nevertheless, by  
368 recalculating the Nd isotopic data (using Bouvier et al. (2008) values for CHUR,  $2.9$  Ga for  
369 charnockites and  $2.87$  Ga for TTG suites crystallisation ages) from Shang et al. (2004a), it  
370 appears that the Sangmelima charnockites and TTGs have  $\epsilon^{143}Nd_{(i)}$  values of  $+0.16$  to  $-5.63$   
371 (average =  $-2.14$ ) and  $+0.50$  to  $-5.07$  (average =  $-2.17$ ), respectively. Furthermore, Shang et  
372 al., (2004a) reported low  $Sr_{(i)}$  ratios ranging from  $0.70098$  to  $0.70314$  at  $2.85$  Ga for the  
373 Sangmelima charnockitic and TTG suites. As a result, a local continental crust, extracted from  
374 the mantle between  $\sim 3200$  and  $\sim 3520$  Ma, probably played a significant role in the petrogenesis  
375 of the charnockites and TTGs from the Sangmelima terranes. This conclusion is consistent with

376 results of the whole-rock  $\epsilon^{176}\text{Hf}$  average of  $-3.3 \pm 1.4$  for the same samples and consistent with  
377 a contribution of an ancient crustal component.

378 The chondritic zircon Hf signatures  $\epsilon^{176}\text{Hf}_{(i)} \sim 0$  for charnockite of Menguéme (Li et al.,  
379 2016) and near-chondritic whole-rock  $\epsilon^{143}\text{Nd}_{(i)}$  value of  $\sim +0.8$  for charnockites reported by  
380 Pouclet et al. (2007), suggest that their chondritic signatures may reflect the formation of  
381 charnockite from sources evolving from a depleted mantle component that was formed earlier.  
382 However, both data reported by Li et al. (2016) including some zircons with superchondritic  
383  $\epsilon^{176}\text{Hf}_{(i)}$  values ( $\epsilon^{176}\text{Hf}_{(i)} > 0$ ) and the positive whole-rock  $\epsilon^{143}\text{Nd}_{(i)}$  values reported by Pouclet  
384 et al. (2007) indicate that the Archean mantle of the Ntem Complex was partially depleted. The  
385 combined geochemical and isotopic (Sr, Nd, Hf and O) distinction between the charnockites  
386 and the TTG suites suggests that at least two different precursors, formed at different times,  
387 were involved in the petrogenesis of the Mesoarchean felsic rocks. The combination of the  
388 whole-rock Hf- and Nd model ages, as well as the data published by Shang et al. (2004a, re-  
389 calculated using the chondritic (CHUR) values of Bouvier et al. 2008; 2.90 Ga for charnockites,  
390 2.87 for TTG suites and 2.75 Ga for high-K granites), suggest a progressive extraction during  
391 Eo- to-Mesoarchean ages (3750 – 3050Ma) with two major peaks evidenced by whole-rock  
392  $T_{\text{DM}}$  ages of 3200 Ma and 3300 Ma of the protolith source of the Sangmelima charnockites and  
393 TTG magmas.

394 Fig.6

395 The high-K granite sample (AJM25) has higher  $\text{K}_2\text{O}/\text{Na}_2\text{O}$  (1.85), negative  $\epsilon^{176}\text{Hf}_{(i)}$  of –  
396 3.7 and  $\epsilon^{143}\text{Nd}_{(i)}$  of –4.6, consistent with their formation by partial melting of a LREE-enriched  
397 crustal source with a range in age from 3.22 to 3.24 Ga. These model ages (3.22 – 3.24 Ga) and  
398 isotopic signatures are slightly younger than those of some Mesoarchean granitoids of TTG  
399 composition (Table 2), that were formed by the melting of a mafic source. However, Na-  
400 enriched TTG do not produce melts that are sufficiently  $\text{K}_2\text{O}$ -rich to generate the high-K

401 granitoids (Watkins et al., 2007). Therefore, partial melting of relatively more K-rich  
402 components of this suite is required (eg. Patiño Douce and Beard, 1995). Our Hf-Nd isotopic  
403 data are consistent with the subchondritic Nd isotopic signatures ( $\epsilon^{143}\text{Nd}_{(i)} = -2.1$  to  $-5.0$ , re-  
404 calculated at 2.75 Ga) reported by Shang et al. (2010), and the protolith emplacement age of  
405 the Sangmelima potassic granites at ca.  $>2.9$  Ga (zircon U-Pb age ; Akame et al., 2020a).

### 406 **5.2.2. Mafic rocks**

407 Recently published zircon U–Pb dates show that most mafic rocks from the Ntem  
408 Complex were emplaced around 2866 – 2862 Ma (Li et al., 2016; Akame et al., 2020a),  
409 suggesting a significant episode of Mesoarchean mafic magmatism in this complex.  
410 Specifically, these are mafic assemblages within the Sangmelima terrane, such as the  
411 gabbro-norite-gabbro assemblage (Akame et al., 2020a), and the amphibolites hosted in Djoum's  
412 TTG (Li et al., 2016), but also older garnet-bearing amphibolite (ca. 3.1 Ga, Pb–Pb zircon  
413 evaporation age) from the Ebolowa supracrustal belt (Tchameni et al., 2004). The whole-rock  
414  $\epsilon^{176}\text{Hf}_{(i)}$  values from the four gabbro-norite and gabbro mafic samples from the Sangmelima  
415 terranes are positive, ranging from +3.4 to +8.2 with an average of  $\sim +5.9$ , which are overall  
416 close to the one of depleted mantle value of  $\sim +6.9$  (Table 2). These values are broadly similar  
417 to those of the  $\epsilon^{176}\text{Hf}_{(i)}$  values (+0.1 to +4.8) for zircons from the 2.86 Ga amphibolite enclave  
418 in the trondhjemites (Fig.5c), (Li et al., 2016). Their  $\epsilon^{143}\text{Nd}_{(i)}$  values range from  $-2.3$  to  $+4.5$ ,  
419 indeed suggesting decoupled Hf- and Nd isotopic signatures. However, as mentioned above,  
420 the Nd isotopic compositions of the samples AJM16 and AJM17 were likely disturbed by post-  
421 magmatic processes such as metamorphism/metasomatism and/or by crustal contamination.  
422 Taking this effect into account, both unaltered samples (AJM11 and AJM14) have  $\epsilon^{143}\text{Nd}_{(i)}$   
423 values of +0.9 to +1.6, which are similar to the  $\epsilon^{143}\text{Nd}_{(i)}$  values (+0.5 to +2.8) of ca. 3.1 Ga  
424 garnet-bearing amphibolites from the Ebolowa greenstone belt (Tchameni et al., 2004).  
425 Together with the published geochemical and isotopic data, we therefore suggest that the Ntem

426 Complex probably experienced two major Mesoarchean (~2.86 and ~3.1 Ga) episodes of  
427 juvenile mantle-derived mafic magmatism.

428 The whole-rock Sm–Nd and Lu–Hf data from the Neoproterozoic dolerite dykes yielded  $\epsilon^{143}\text{Nd}$   
429 and  $\epsilon^{176}\text{Hf}$  values of –0.11 to +4.53 and –0.28 to +16.09, respectively, and are interpreted to be  
430 derived from depleted mantle reservoirs. However, the near-chondritic character ( $\epsilon^{143}\text{Nd}_{(i)} = -$   
431 0.11 and  $\epsilon^{176}\text{Hf}_{(i)} = -0.28$ ) with  $T_{\text{DM}}(\text{Nd})$  of 3125 Ma and  $T_{\text{DM}}(\text{Hf})$  of 3039 Ma for sample  
432 AJM3, and positive  $\epsilon^{143}\text{Nd}_{(i)}$  and  $\epsilon^{176}\text{Hf}_{(i)}$  values with model age of 2666 Ma and 2270 Ma may  
433 suggest distinct periods of dyke emplacement. Toteu et al. (1994) and Vicat et al. (1996) have  
434 suggested the existence of Eburnian doleritic magmatism in the Ntem Complex, but there are  
435 no geochronological data to support this assumption. We therefore consider that the  
436 Sangmelima dolerites are derived from a depleted mantle source, and the Lu–Hf isotopic system  
437 of sample AJM6 was probably disturbed during post-magmatic processes.

### 438 **5.3. Geodynamic implications**

439 The Ntem Complex is one of the largest Archean domains in southern Cameroon. The  
440 statistical zircon U–Pb and monazite U–Th–Pb ages of the igneous rocks from the Ntem  
441 Complex show that the Mesoarchean (2.92 – 2.85 Ga), with two major peaks at 2920 and 2866  
442 Ma (Fig. 6), was an important period of magmatism (Takam et al., 2009; Tchameni et al., 2010;  
443 Chombong and Suh, 2013; Li et al., 2016; Akame et al., 2020a, 2021). This magmatism includes  
444 the widespread emplacement of charnockites, TTG suites and mafic igneous rocks (Li et al.,  
445 2016; Akame et al., 2020a), and the occurrence of minor dacite volcanism in the Mbalam  
446 greenstone belt (Chombong and Suh, 2013). Minor age peaks observed at 3.26 – 3.05 Ga have  
447 been interpreted as inherited ages of charnockites and TTG suites (Takam et al., 2009; Akame  
448 et al., 2020a). A few older xenocrystal zircons with ages of up to 3.3 Ga indicate the existence  
449 of early Meso- to Paleoproterozoic granitic crust in the Ntem Complex (Fig. 6). As proposed in the  
450 discussion on the Hf–Nd isotope compositions, the chondritic to subchondritic  $\epsilon^{143}\text{Nd}_{(i)}$  and

451  $\epsilon^{176}\text{Hf}_{(i)}$  of Archean granitoids from the Ntem Complex point towards the involvement of  
452 ancient paleo-crust in their petrogenesis, rather than simple oceanic crust melting. They have  
453 whole-rock Hf-Nd and zircon  $T_{\text{DM}}(\text{Hf})$  ages of  $\sim 3.9 - 3.0$  Ga (Fig. 6), which may indicate the  
454 extraction of a mafic crust (without zircon) from the mantle (at  $\sim 3.9 - 3.25$  Ga), followed by a  
455 first major recycling episode (at  $\sim 3.25 - 3.00$  Ga) that produced the first felsic crust in the  
456 region. This ancient felsic crust is emphasised by the presence of inherited zircons of  $\sim 3.25 -$   
457  $3.05$  Ga in the granitoids (Takam et al., 2009; Akame et al., 2020a), and the formation of  
458 supracrustal rocks synchronous with the juvenile mantle-derived mafic magmatism dated at  
459  $3.14 - 3.07$  Ga (Tchameni et al., 2004). The Lu-Hf and Sm-Nd isotopic data of  $2.75$  Ga high-  
460 K granite points to re-melting of Mesoarchean crustal material, whereas the  $2.72$  Ga dolerite  
461 magmas were extracted from mantle at  $\sim 2.74 - 2.76$  Ga.

462 Fig.6

463 The geodynamic setting of the Archean Ntem Complex, in which significant Mesoarchean  
464 magmatism and metamorphism occurred, remains a subject of research and discussion. The  
465 emplacement of the widespread granitoids with associated mafic rocks may occur in various  
466 environments, including subduction-related tectonic settings (Shang et al., 2004a; Pouclet et  
467 al., 2007), a hotspot beneath the lithospheric mantle related to mantle plume activity (Li et al.,  
468 2016), or a subduction-accretionary geodynamic setting (Akame et al., 2020a). The latter  
469 alternative model was recently proposed to account for the formation of different lithological  
470 assemblages observed in the Ntem Complex from  $3.2$  to  $2.75$  Ga. Isotopic data published on  
471 the Archean basement rocks of the Ntem Complex (e.g. Tchameni et al., 2004; Shang et al.,  
472 2007; Li et al., 2016; Akame et al., 2020a) show that mafic and felsic magmatism are generally  
473 synchronous from the Mesoarchean to the Neoarchean. The Mesoarchean depleted mantle-  
474 derived mafic magmatism may have provided heat to trigger partial melting of the Eo-to  
475 Paleoproterozoic mafic source for the charnockite-TTG magmatism, and also contributed to their

476 complex geochemical and isotopic compositions. Furthermore, the combined geochemical and  
477 whole-rock Sr-Nd-Hf and zircon Hf-O isotopic data suggests that the formation of Mesoarchean  
478 continental crust was episodic, with pulses of juvenile mantle-derived mafic material and  
479 crustal growth in the Ntem Complex. On the other hand, it has also been proposed that episodic  
480 crustal growth could be related to mantle plume activity (e.g. Condie, 1998), but this assumption  
481 cannot explain the large volume of arc-like felsic magmatism in the region compared to mafic  
482 magmatism. Thus, the formation of the Archean continental crust of the Ntem complex may be  
483 related to a combination of Archean accretion and subduction processes.

484       Based on the above, multi-stage magmatism, crustal reworking and growth events occurred  
485 in the Ntem Complex at  $\sim 3.90 - 3.25$  Ga,  $\sim 3.25 - 3.00$  Ga,  $\sim 2.95 - 2.85$  Ga and  $\sim 2.75 - 2.72$   
486 Ga from the Eo- to the Neoproterozoic (Fig. 6).

#### 487 **5.4. Craton scale implications**

488       The Congo Craton is one of the least studied Archean terranes in the world, and is made  
489 up of three main blocks: the Kasai Block to the South, the NE Congo and West Nile blocks to  
490 the Northeast of D.R. Congo (Bomu Complex), and the Ntem-Chaillu block (Cameroon,  
491 Gabon) (Thiéblemont et al., 2018). Available U–Pb zircon ages indicate that the Congo Craton  
492 was formed mostly between ca. 3.4 and 2.5 Ga (eg. Cahen et al., 1976; Toteu et al., 1994;  
493 Mayaga-Mikolo, 1996; Feybesse et al., 1998; Takam et al., 2009; Maier et al., 2015; Tchameni  
494 et al., 2010; Li et al., 2016; Akame et al., 2020a; Turnbull et al., 2021). However, neither  
495 spatially resolved Sm–Nd or Lu–Hf isotope studies have been conducted on the Kasai and  
496 Bomun terranes. Consequently, the only possible regional comparison within the Congo Craton  
497 for the rocks of the Ntem Complex is with those of the Monts de Cristal and Chaillu terranes of  
498 Gabon.

499 In the Monts de Cristal region of northern Gabon, charnockites and TTG gneisses were  
500 dated between 3186 and 3120 Ma (Rb/Sr whole-rock ages; Caen-Vachette et al., 1988).  
501 However, both U–Pb zircon ages and Nd isotopic data for these rocks are not yet available.  
502 Recently, Thiéblemont et al. (2018) reported the compilation of zircon U–Pb ages and Nd  
503 isotopic data from Archean granitoid rocks exposed alongside the north Gabon domain and the  
504 Chaillu Complex. Zircon extracted from these rocks yielded crystallisation ages from ca. 2.92  
505 Ga to ca. 2.55 Ga with two major magmatic events at ca 2.92 – 2.85 Ga (TTG and charnockites  
506 plutonism) and ca 2.8 – 2.6 Ga (Neoproterozoic magmatic episode), like those of the Ntem  
507 Complex. In addition, most of the Mesoarchean felsic rocks show a chondritic to sub-chondritic  
508  $\epsilon^{143}\text{Nd}_{(i)}$  signature with  $T_{\text{DM}}(\text{Nd})$  ages old as 3.2 Ga (de Wit and Linol, 2015b; Thiéblemont et  
509 al., 2009, 2018). However, the zircon Hf isotopic composition of the 2.89 Ga TTGs from Ivindo  
510 Basement Complex (Republic of Congo) shows positive  $\epsilon\text{Hf}(t)$  values of +1.22 to +4.55 and  
511 3.23 – 3.02 Ga  $T_{\text{DM}}(\text{Hf})$  model ages (Gatsé Ebotehouna et al., 2021), which suggest the  
512 existence of a depleted mantle domain underlying the Ivindo Basement Complex. Moreover,  
513 these super-chondritic  $\epsilon^{176}\text{Hf}$  values differ from the subchondritic values measured in the ca.  
514 2.86 Ga zircon and whole rocks from the Ntem Complex TTG suites (Fig. 5c). We suggest that  
515 the NW Congo likely developed from Paleoproterozoic mafic protoliths that underwent  
516 considerable reworking and juvenile accretion additions during the Mesoarchean. Near Mitzié  
517 in the Monts de Cristal Complex, meta-kimberlite dykes were emplaced at 2848 – 2862 Ma  
518 (zircon U–Pb LA-ICP-MS ; Henning et al., 2003), which suggests a Mesoarchean age for  
519 ultramafic–mafic magmatism in the north Gabon domain. These Mesoarchean kimberlite  
520 intrusions were synchronous with the dacitic volcanism and mafic magmatism at 2862 – 2883  
521 Ma in the Ntem Complex (Chombong and Suh, 2013; Li et al., 2016; Akame et al., 2020a). The  
522 Neoproterozoic ultramafic–mafic intrusion from the Monts de Cristal Complex (North Gabon  
523 terrane) studied by Maier et al. (2015) yielded crystallization ages of ca. 2765 – 2775 Ma with



524 positive  $\epsilon^{143}\text{Nd}_{(i)}$  values (+0.1 to +1.8) and  $T_{\text{DM}}(\text{Nd})$  ages of 2.96 – 3.06 Ga. While, the  
525 Neoproterozoic anatectic granitoids are sub-chondritic, with  $\epsilon\text{Nd}_{(i)}$  values ranging from –1 to –7  
526 (Thiéblemont et al., 2018), indicating the reworking of older felsic crustal rocks. The time for  
527 the Bélinga greenstone belt formation (North Gabon domain) is defined between  $2918 \pm 7$  and  
528  $2868 \pm 9$  Ma by U–Pb based on detrital zircons (Thiéblemont et al., 2009, 2018). The ca 2.84  
529 – 2.82 Ga and ~ 2.75 Ga magmatic-metamorphic events in the north Gabon domain  
530 (Thiéblemont et al., 2009, 2011, 2018) have also been reported in the Sangmelima area (Akame  
531 et al., 2020a, 2021). Overall, according to the current geochronological and isotopic data, it is  
532 evident that the entire Ntem-Chaillu block or NW Congo Craton experienced the same  
533 geodynamic evolution.

534 From above, we conclude that minor Paleoproterozoic crust existed in the NW Congo Craton  
535 with the generation started at ~ 3.5 Ga likely by melting of mafic crust extracted from the mantle  
536 at ~ 3.8 –3.3 Ga (Fig. 6). Finally, all the NW Congo craton Archean underwent important  
537 episodes of crustal generation and reworking from the Mesoarchean to Neoproterozoic.

## 538 6. Conclusions

539 We present the first coupled whole-rock  $^{176}\text{Lu}$ – $^{176}\text{Hf}$  and  $^{147}\text{Sm}$ – $^{143}\text{Nd}$  dataset for  
540 Archean felsic and mafic rocks from the NW Congo Craton. Sixteen mafic and felsic igneous  
541 rocks were collected in the Sangmelima terranes, located in the northern part of the Ntem  
542 Complex, southern Cameroon. The mafic rocks include 2.87 Ga gabbro-norites and late 2.72 Ga  
543 dolerite dykes. The granitoids include 2.91 Ga charnockites, 2.87 Ga TTG suites, and 2.75 Ga  
544 high-K granites. Some isotopic data suggest post-crystallisation modification of the whole-rock  
545 Sm–Nd isotopic system during the Mesoarchean tectonothermal events. All the Mesoarchean  
546 charnockite and TTG samples we analysed show negative  $\epsilon^{176}\text{Hf}_{(i)} = -0.9$  to  $-7.4$  and  $\epsilon^{143}\text{Nd}_{(i)}$   
547  $\epsilon^{143}\text{Nd}_{(i)} = -1.9$  to  $-5.1$  with average Hf and Nd depleted mantle model ages of ~ 3.30 Ga and 3.37 Ga,  
548 respectively. The Hf–Nd isotopic composition ( $\epsilon^{176}\text{Hf}_{(i)} = -3.7$  and  $\epsilon^{143}\text{Nd}_{(i)} = -4.6$ ) of high-K  
549 granite (2.75 Ga) is consistent with derivation by re-melting of an enriched crustal source at ~

550 3.2 Ga. The depleted  $\varepsilon^{176}\text{Hf}_{(i)}$  and  $\varepsilon^{143}\text{Nd}_{(i)}$  values (+3.4 to +8.2 and +0.9 to +1.6, respectively)  
551 in the Sangmelima Mesoarchean gabbro-norites highlight a depleted mantle source. The whole  
552 set of the geochronological and isotopic data in the Ntem Complex and other domains of NW  
553 Congo Craton like Monts de Cristal and Chaillu Complex in Gabon and the Ivindo Basement  
554 Complex (Republic of Congo) revealed that minor Paleoproterozoic crust existed in the NW Congo  
555 Craton with the generation starting at  $\sim 3.5$  Ga likely by melting of mafic crust extracted from  
556 the mantle at  $\sim 3.8 - 3.3$  Ga. Subsequently, all Archean basement domains underwent  
557 considerable reworking and juvenile accretion additions during the Mesoarchean. Collectively,  
558 the Archean crustal accretion is characterised by multi-stage magmatism, crustal reworking,  
559 and growth events that occurred at  $\sim 3.90 - 3.30$  Ga,  $\sim 3.25 - 3.0$  Ga,  $\sim 2.95 - 2.85$  Ga and  
560  $\sim 2.75 - 2.72$  Ga in the NW Congo.

#### 561 **Acknowledgements**

562 We are grateful to Sabrina Cauchies for trace element measurements and isotope purification,  
563 and to Jeroen de Jong for Nd and Hf isotopes measurements on the Nu-Plasma II at ULB.  
564 We also want to acknowledge Prof. Tony Kemp for his editorial handling, Dr. Jonas Tusch and  
565 an anonymous reviewer for their positive and constructive comments which greatly helped to  
566 improve the final version of the manuscript. We also thank Dr. Fabien Humbert for her help  
567 and constructive suggestions. JMA thanks the grant “Prix de Meurs-Francois”, ULB,  
568 Fédération Wallonie-Bruxelles, and *MOPGA 2022 Visiting Fellowship Program for Young*  
569 *Researchers* for current support. VD thanks the EoS project ET-Home and FRS-FNRS for  
570 support.

571

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844 **Figure captions**

845 **Figure 1.** a) Regional geological map of the Ntem Complex, and thrust contact with the Pan-  
846 African fold belt in Southern Cameroon modified after Maurizot et al. (1986). b) Simplified  
847 geological map of the Sangmelima region and sample locations.

848 **Figure 2.** (a-c) N-MORB and primitive-mantle normalised trace-element patterns and (b-d)  
849 chondrite-normalised REE patterns for the mafic rocks and granitoids. The trace elemental data  
850 are from Akame et al. (2020a). Primitive mantle and NMORB values are from Sun and  
851 McDonough, (1989) and chondrite values are from Anders and Grevesse (1989)

852 **Figure 3.** Lu-Hf and Sm-Nd isochron diagrams for Sangmelima igneous rocks.

853 **Figure 4.** a)  $\epsilon\text{Hf}_{(i)}$  vs.  $\epsilon\text{Nd}_{(i)}$  from Sangmelima igneous rocks. Two mafic samples (AJM16 and  
854 AJM17) are decoupled in their  $\epsilon\text{Hf}_{(i)}$  and  $\epsilon\text{Nd}_{(i)}$  composition, which can be explained by crustal  
855 contamination or secondary alteration due to metamorphism. b) Binary diagrams of Zr versus  
856 selected elements for the whole-rock samples.

857 **Figure 5.** (a) Measured  $^{176}\text{Hf}/^{177}\text{Hf}$  vs.  $1/\text{Hf}$  and (b) measured  $^{143}\text{Nd}/^{144}\text{Nd}$  vs.  $1/\text{Nd}$ . (c)  $\epsilon\text{Hf}_{(i)}$   
858 vs. age (Ma) and (d)  $\epsilon\text{Nd}_{(i)}$  vs. age (Ma) diagrams for the igneous rocks.

859 **Figure 6.** A timeline of Ntem Complex crustal evolution processes during Archean. Zircon U-  
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865 2004a, 2010; re-calculated from Pouclet et al., 2007) and the whole rock Nd-Hf model ages  
866 calculated in this study. Zircon Hf model ages are from Li et al. (2016)

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869 **Tables.**

870 **Table 1.**

871 Major (wt% ) and trace (ppm) element contents of Sangmelima igneous rocks

872

873 **Table 2.**

874 Bulk-rock Sm–Nd and Lu–Hf isotope data for granitoids and mafic rocks of Sangmelima area

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### Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

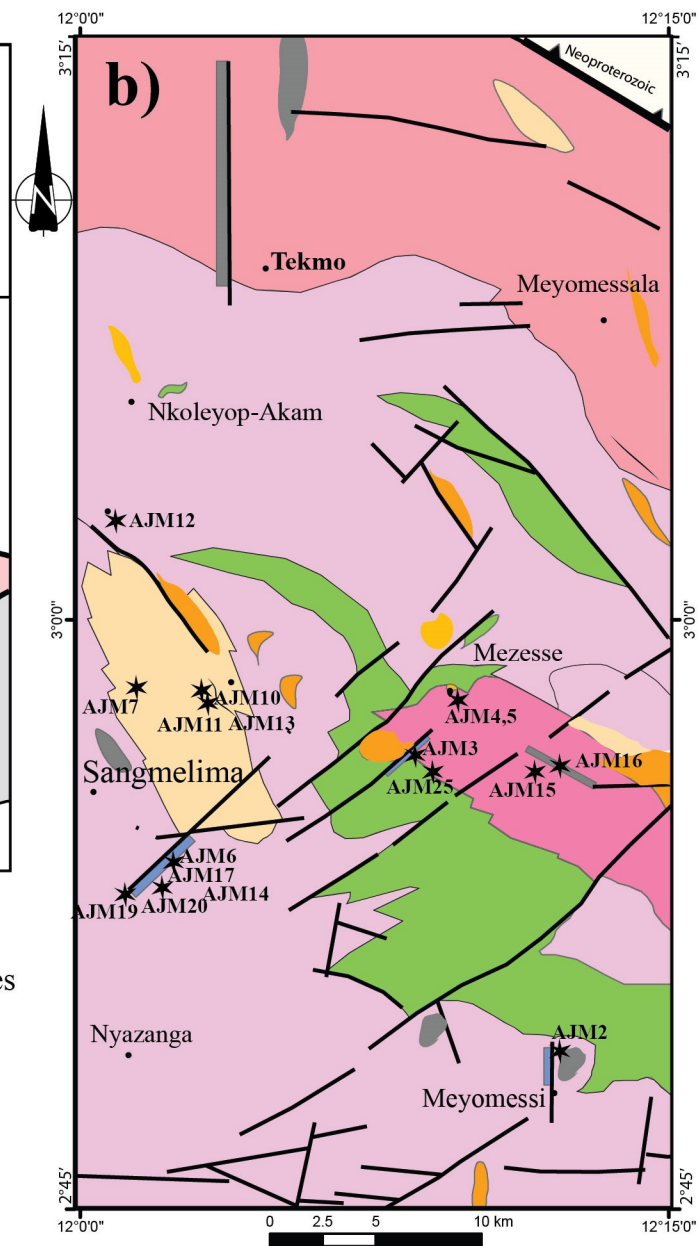
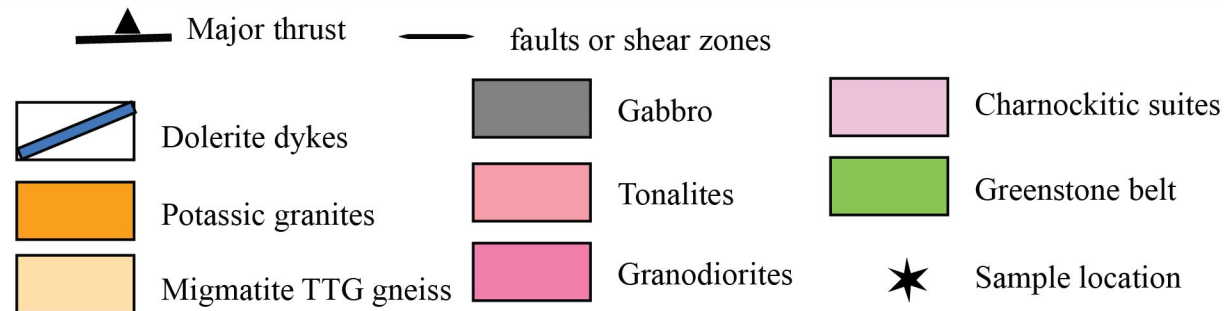
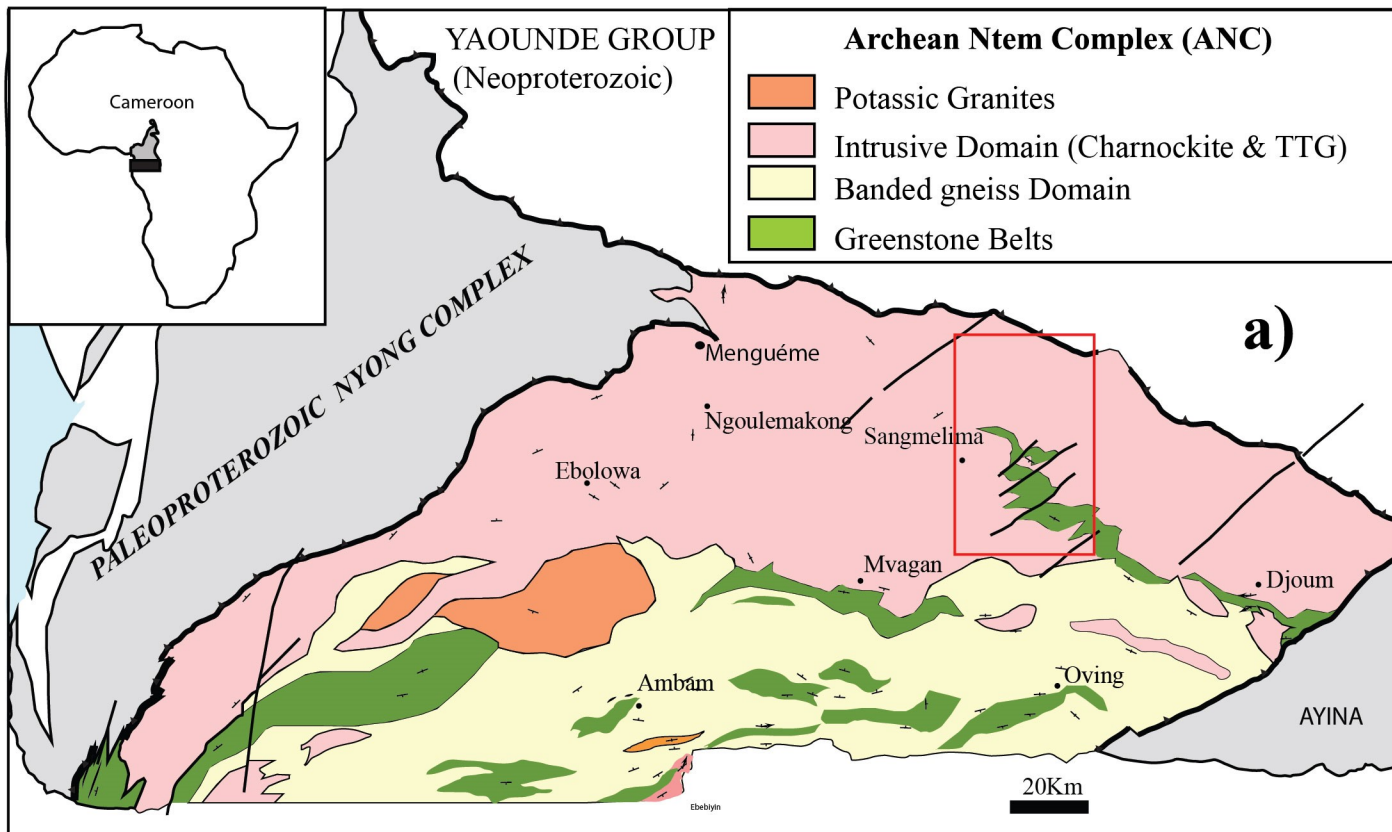
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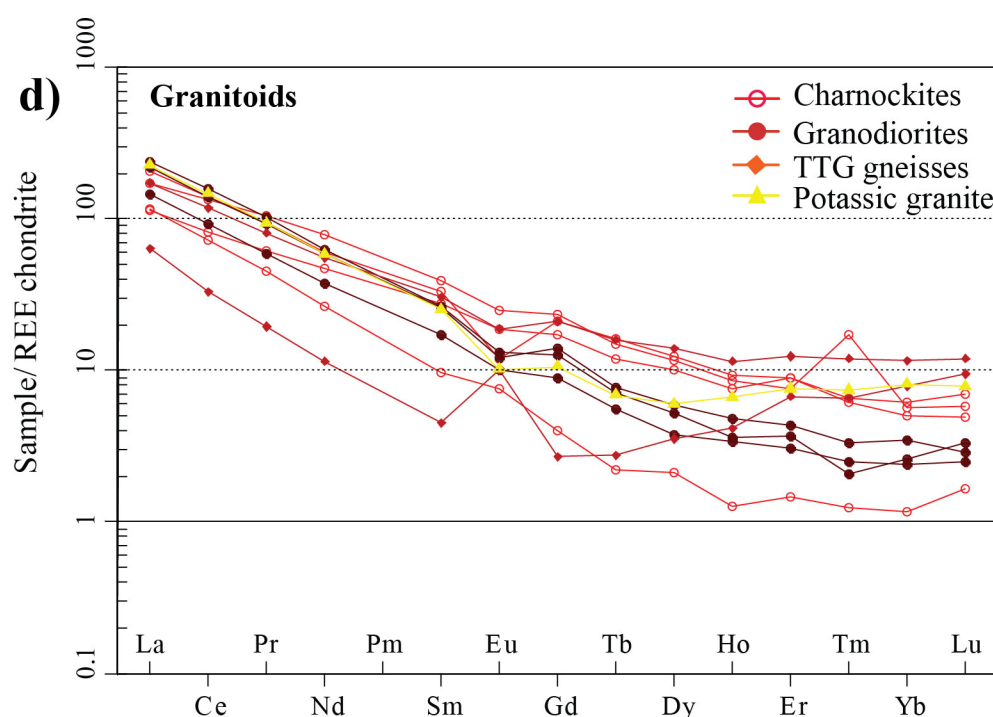
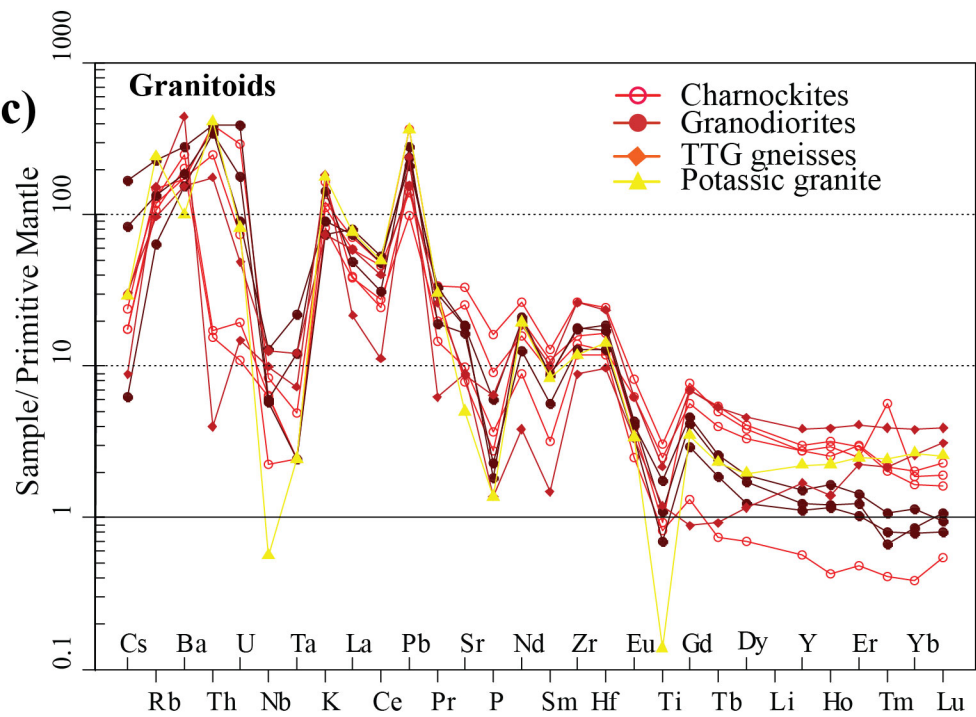
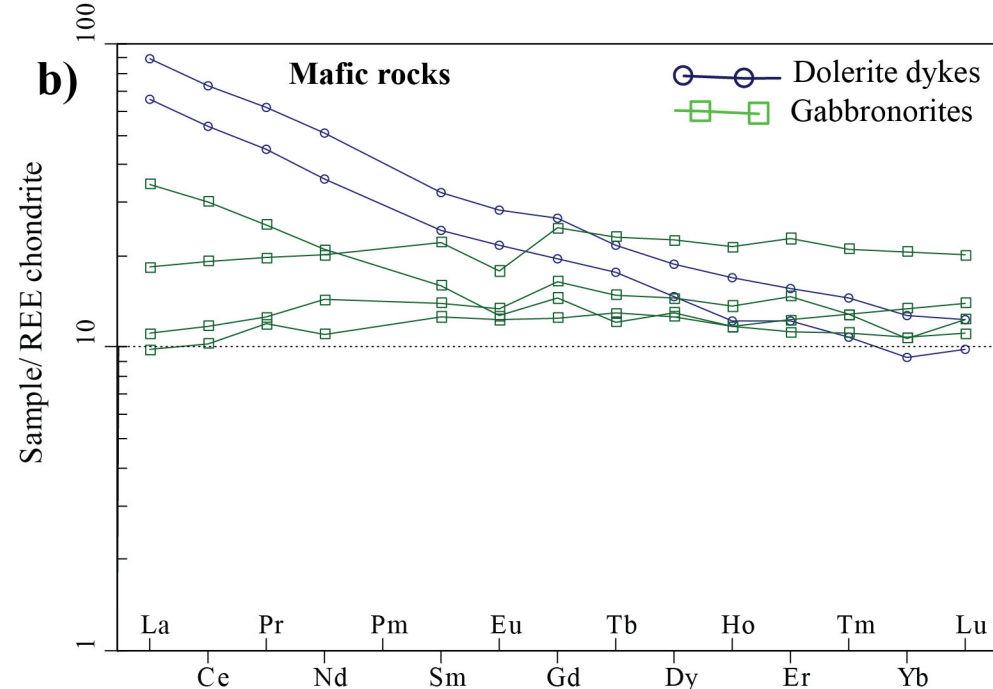
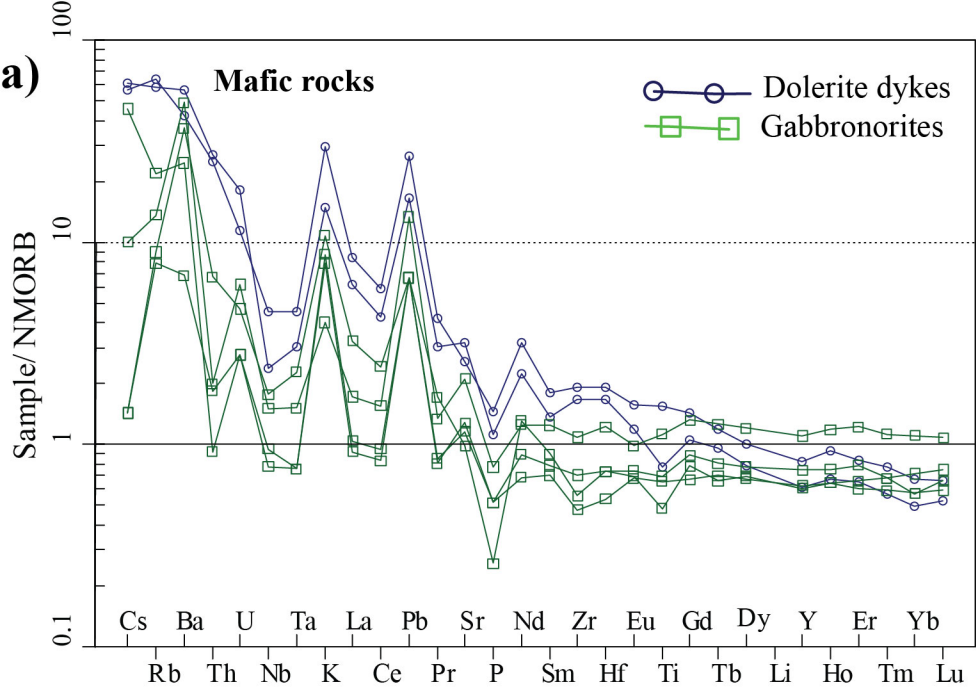
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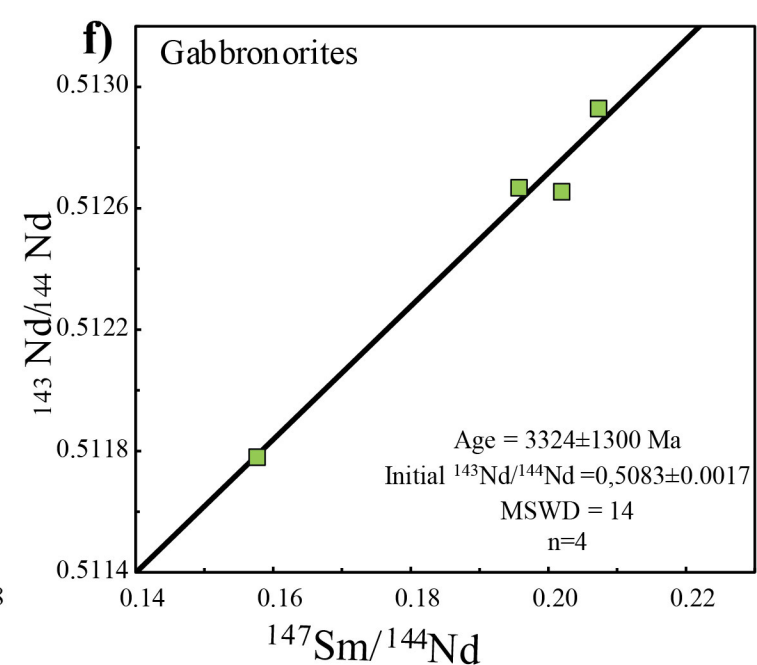
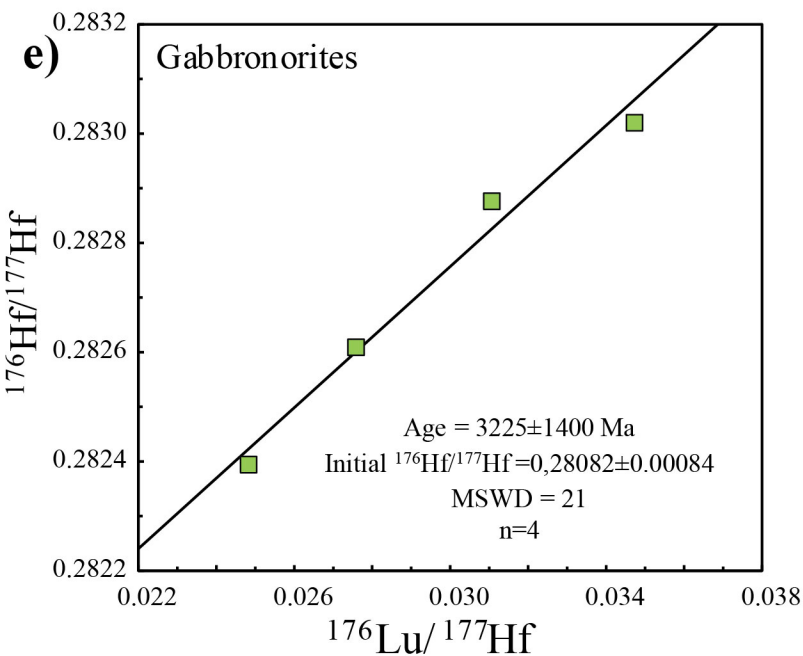
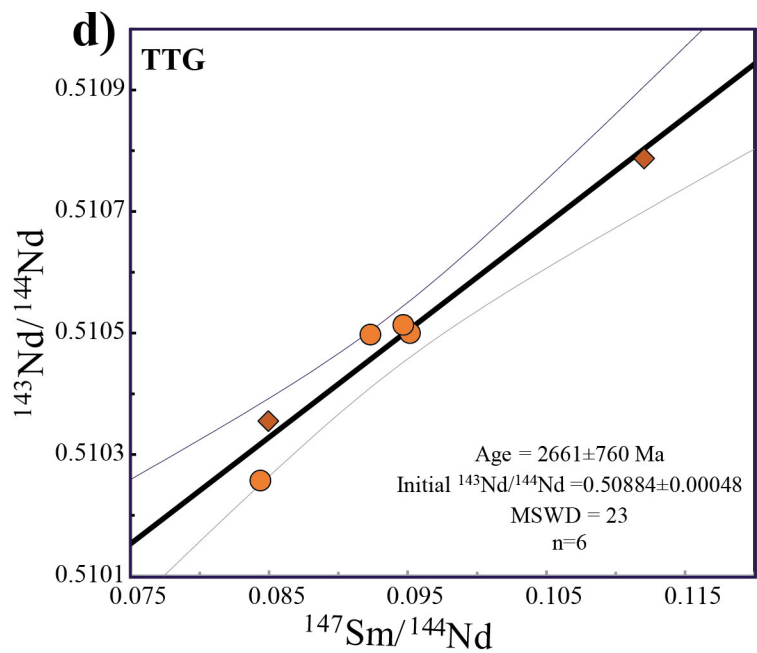
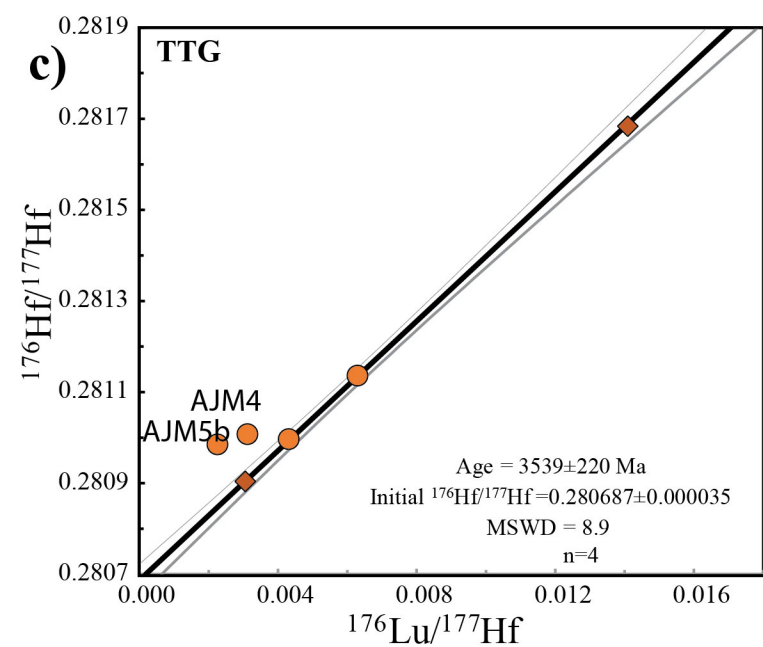
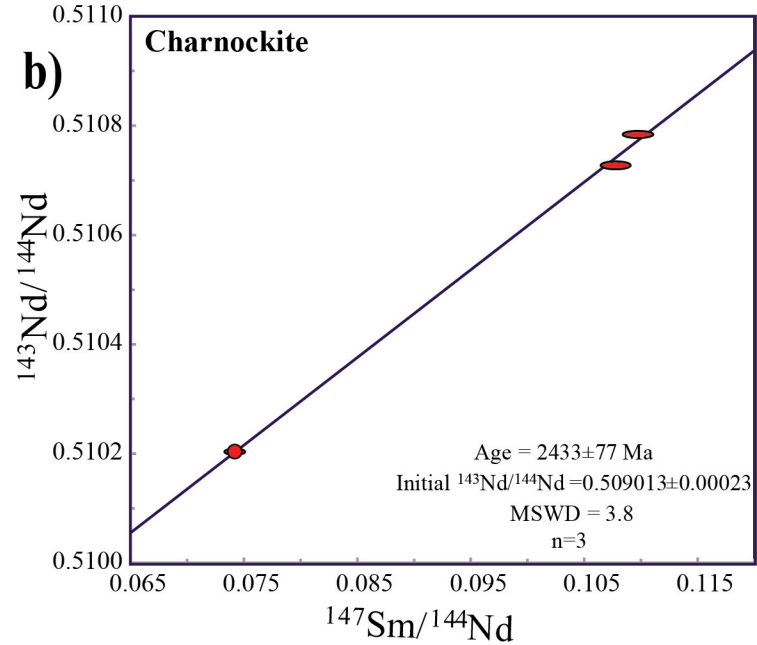
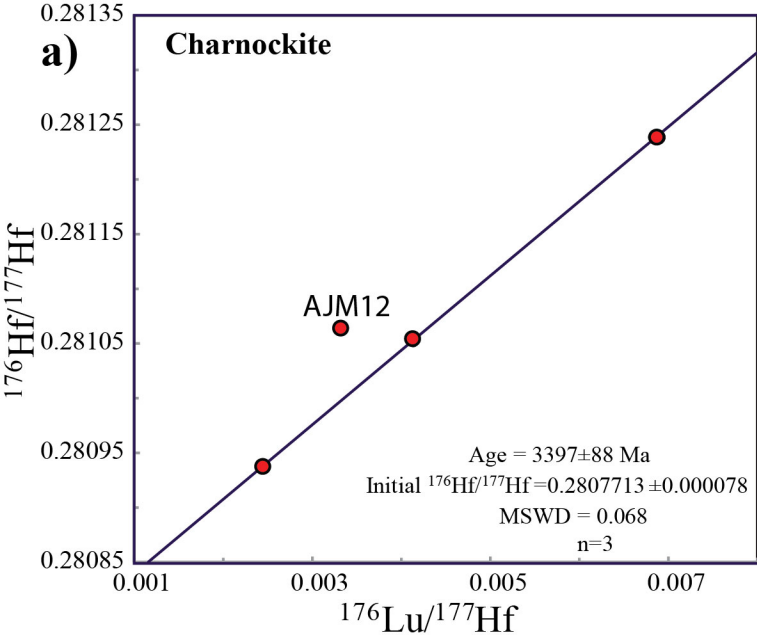
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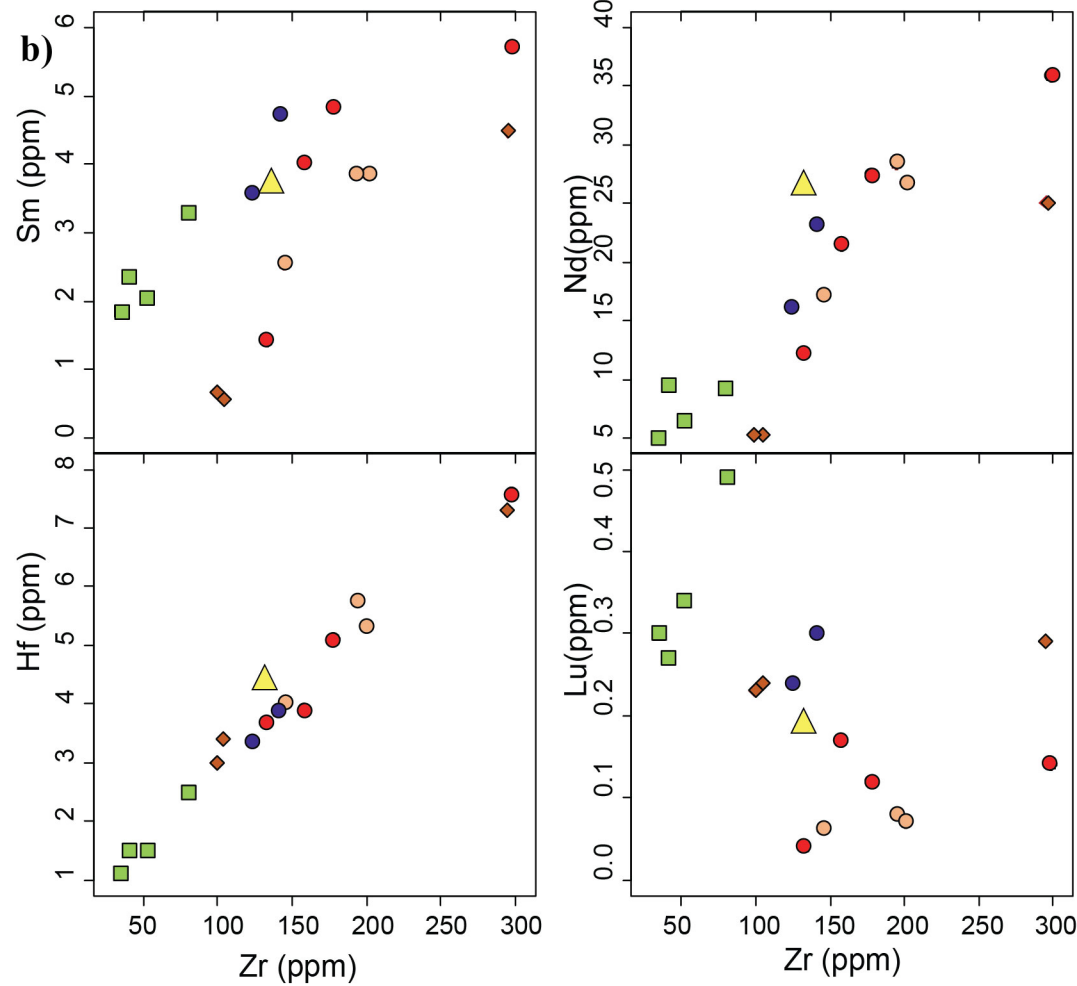
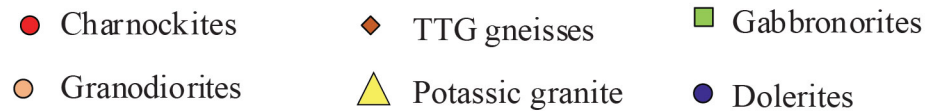
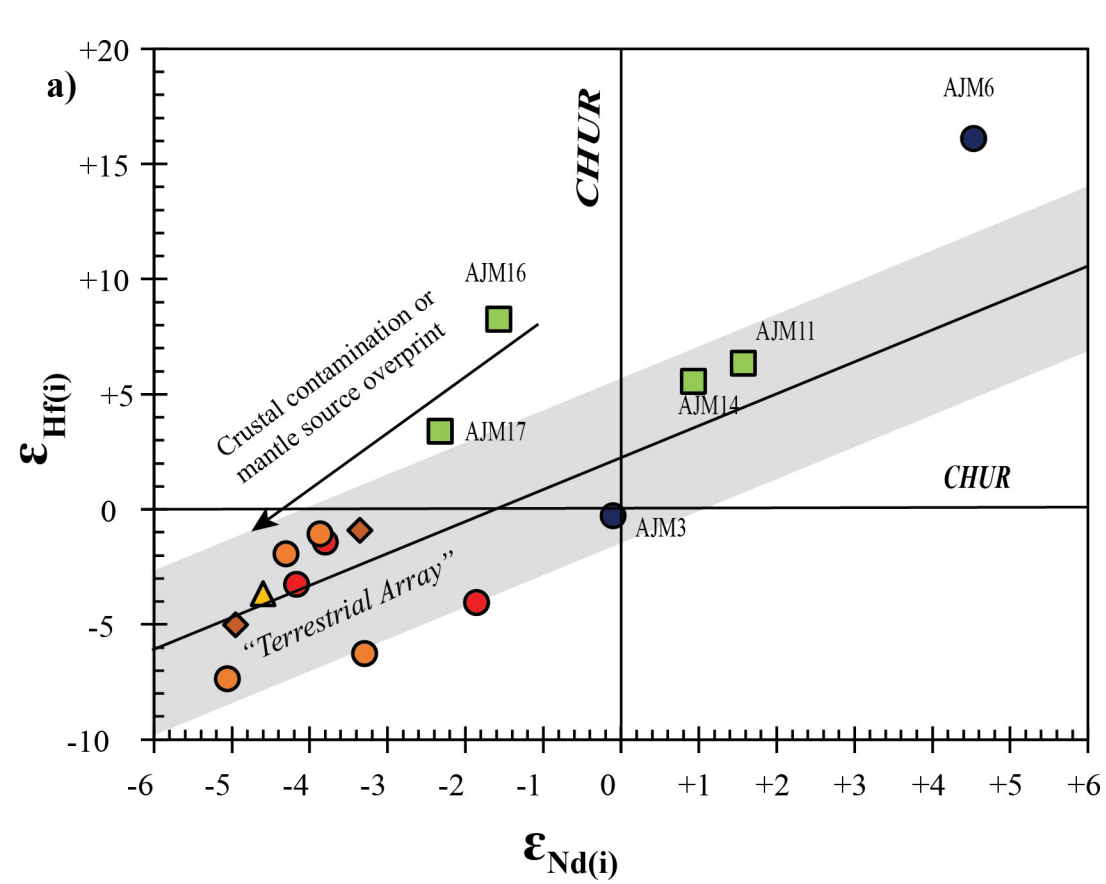
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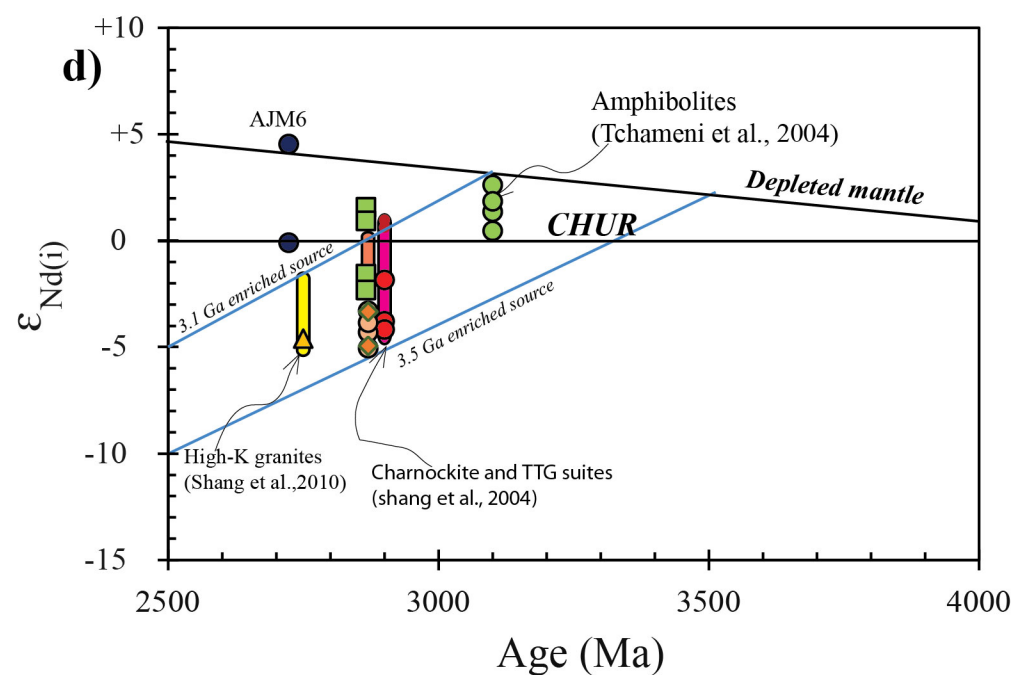
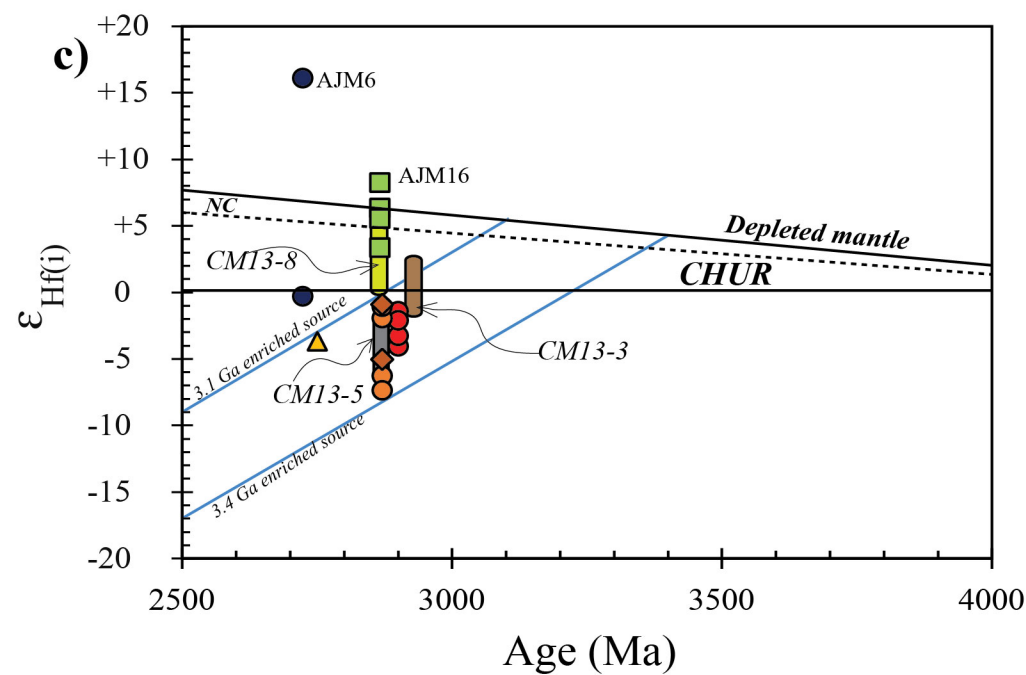
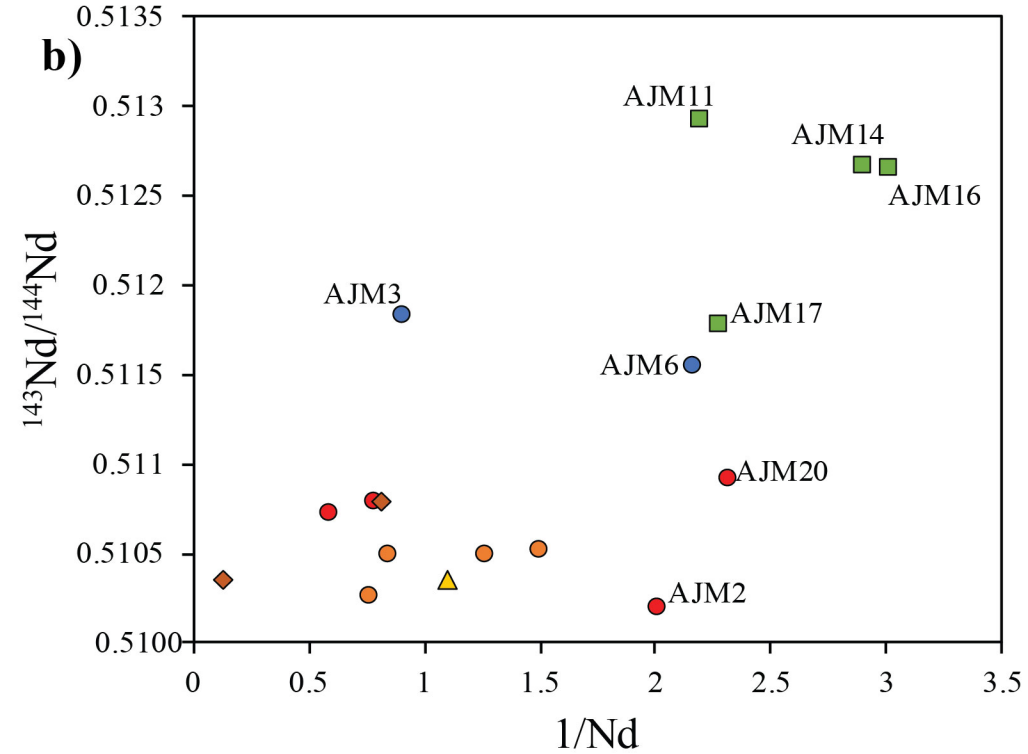
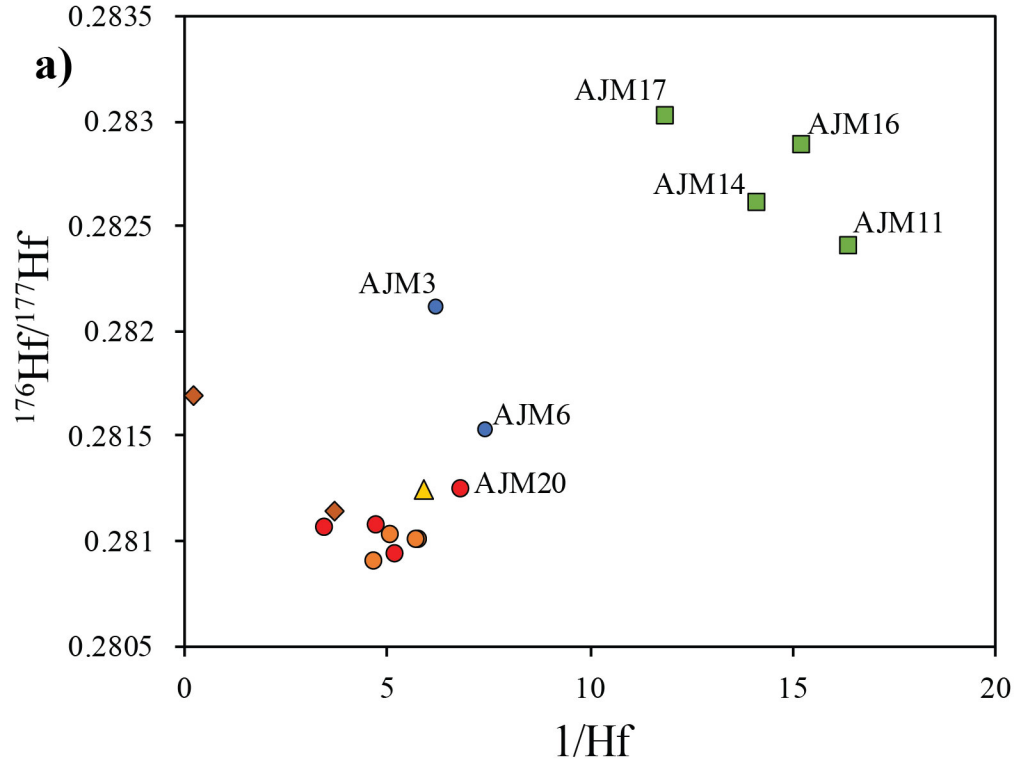






● Charnockites   ● Granodiorites   ◆ TTG gneisses   ■ Gabbroonrites





● Charnockites    ◆ TTG gneisses    ● Granodiorites    ▲ High-K granite    ● Dolerites    ■ Gabbro-norites

