

# **Discovery of a Devonian mafic magmatism on the western border of the Murzuq basin (Saharan metacraton): Paleomagnetic dating and geodynamical implications**

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**Keywords**: Basic magmatism, Isotopic (K-Ar, U-Pb) dating, paleomagnetic dating, 33 Devonian, Murzuq craton, Saharan metacraton 34

#### 35

#### 36 **Abstract**

Intraplate deformation is most often linked to major stress applied on plate margins. 37 When such intraplate events are accompanied by magmatism, the use of several dating 38 methods integrated within a multidisciplinary approach can bring constraints on the age, 39 nature and source mobilized for generating the magma and in turn on the nature of the 40 intraplate deformation. This study focuses on the large gabbro Arrikine sill (35 km in 41 extension) emplaced within the Silurian sediments of the western margin of the Murzuq 42 43 cratonic basin in southeastern Algeria. Its emplacement is dated during the early Devonian (415-400 Ma) through the determination of a reliable paleomagnetic pole by comparison with 44 45 the Gondwana Apparent Polar Wander Path (APWP). This age can be correlated with deep phreatic eruptions before Pragian time thought to be at the origin of sand injections and 46 47 associated circular structures in Algeria and Libya. For the sill, the K-Ar age of  $325.6 \pm 7.7$  Ma is related to a K-rich aplitic phase that has K-enriched by more than 20% the Devonian gabbro. Laser-ICP-MS U-Pb method dates only inherited zircons mostly at c. 2030 49 50 Ma with additional ages at c. 2700 Ma and younger ones in the 766-598 Ma age range. The Arrikine sill is a high-Ti alkaline gabbro having the geochemical composition of a hawaiite 51 52 akin to several intraplate continental and oceanic provinces, including the contemporaneous Aïr ring complexes province in Niger, but also to the Mauna Loa volcano in Hawaii. This 53 peculiar composition akin to that of the contemporaneous Aïr province is in agreement with a 54 55 lower Devonian age for the Arrikine sill.

The lower Devonian Arrikine sill emplacement is related to a "Caledonian" 56 57 transtensive reactivation of the western metacratonic boundary of the Murzuq craton. This 58 event also generated in the Saharan platform the so-called "Caledonian unconformity" of regional extension, the Aïr ring complexes and magmatic rocks that produced sand injections. 59 It could be related to rifting of the Hun terranes that occurred at the plate margin to the north 60 (Stampfli and Borel, 2002, Blackey, 2008 and references therein). The mid-Carboniferous (c. 61 326 Ma) reactivation corresponds to Variscan compression on NW Africa generating aplitic 62 fluids, but also to the major "Hercynian unconformity" of regional extension. The generation 63 of the Arrikine magma is attributed to partial melting through adiabatic pressure release of 64 uprising asthenosphere along tectonically reactivated mega-shear zones, here bordering the 65 relictual Murzuq craton enclosed in the Saharan metacraton. 66

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#### 68 **1. Introduction**

The Murzuq basin located in central North Africa, in Algeria, Libya and Niger (Fig. 1) 69 is a key area within the Saharan metacraton, being located on a relictual cratonic nucleus, the 70 Murzuq craton (Liégeois et al., 2013). It is one of the largest cratonic sedimentary basins of 71 the Saharan platform, filled by different Phanerozoic series covered by a very large sandy 72 desert, the Murzuq edeyen or erg. On its western border, we discovered a very large sill of 73 mafic rocks interbedded in the Silurian sedimentary series belonging to the so-called Tassilis 74 series. The sedimentary sequences unconformably rest on the late-Neoproterozoic Pan-75 African basement of the Tuareg Shield (Fig. 2). Known post-Pan-African magmatism in the 76 77 In-Ezzane area is limited to Cenozoic lava flows  $(2.86 \pm 0.07 \text{ Ma}; \text{Y}$ ahiaoui et al., 2014). Around the Murzuq basin, several large Cenozoic volcanic areas are known: Hoggar, Tibesti, 78 Libyan basaltic fields (Liégeois et al., 2005; Fezaa et al., 2010). On the southwestern border 79 80 of the Murzuq basin in Niger (between  $11^{\circ}53'$  E/22°27' N and  $12^{\circ}04'$  E/  $21^{\circ}53'$  N), Paleozoic dolerites are mapped on the "Carte géologique du Sahara central" (Lelubre et al., 81 1962). These dolerites seem to be interbedded between Silurian and Devonian sedimentary 82 sequences and correspond to a very large volume of mafic rocks (the biggest outcrop is 83 indicated to be 90 km long on this map). Several hundred kilometers far to the west and 84 85 southwest of In-Ezzane, Lower Carboniferous basic intrusions (Djellit et al., 2006; Derder et al., 2006) in the Tin Serririne basin and Devonian ring complexes in Aïr region (Black et al., 86 1967, Moreau et al., 1994) have been evidenced. In addition, Moreau et al. (2012) suggested 87 that three areas with "sand injections", which generated circular structures along the Murzuq 88 basin boundaries during lower Devonian, could be related to deep phreatic eruption before 89 Pragian times (Lower Devonian, 407-410 Ma). One of these structures is very close to the sill 90 in the In-Ezzane area (Fig. 1A). We note that the In-Ezzane circular structures proposed to be 91 92 meteoritic impacts (Bonin et al., 2011; Reimold and Koeberl, 2014) are located more to the 93 West, within the Ordovician sandstones.

Such an intraplate magmatism is a key event for understanding the geodynamical 94 evolution of the Saharan platform. The aim of this paper is to date this event and to determine 95 96 its different implications.

97

#### 98 **2. Geological setting**

The Murzuq Basin is the principal feature of Central Sahara. This basin lies across 99 Libya, Algeria and Niger. From a geomorphological point of view, this intracratonic 100 101 sedimentary basin can be described as an enormous flat hollow of approximately 900 km in

102 diameter from Libya in the north to Niger in the south. This basin is filled by different 103 Phanerozoic series covered by a very large sandy desert (the "Murzuq edeyen or erg"). The 104 main morphological units of this basin consist of plateaus, oueds and sand dunes cover. The Tassili-n-Ajjer (Algeria) and Djado (Niger) uplifted plateaus constitute the southwestern and 105 southern margins of this basin. Primary geological formations dip gently from the flanks 106 towards the basin centre in a concentric pattern. In the In Djerane and Tadrart areas (Figs. 1 107 and 2), the different Paleozoic series (Cambro-Ordovician, Silurian, Devonian, Carboniferous 108 109 and Permian – Henry et al., 2014) outcrop in several trenches of oueds, which produce 110 remarkable rock cliffs (Fig. 2).

To the west, the Murzuq craton is bordered by its metacratonic margin, corresponding 111 to the Djanet terrane, made of greenschist Ediacaran sediments (Fezaa et al., 2013) crosscut 112 by 575-545 Ma old high-level plutonic bodies, batholiths, plutons and dyke swarms, (Fezaa et 113 114 al., 2010). Further west, on the other side of a major NW-SE trending shear zone, the Edembo 115 terrane resulted from a stronger intracontinental metacratonic reactivation that generated amphibolite-facies migmatitic gneisses and aluminous granitoids during the same period (c. 116 568 Ma; Fezaa et al., 2010). This intracontinental reactivation (575-545 Ma 117 118 metacratonization) occurred to the east of the Raghane mega-shear zone (western boundary of 119 the Saharan metacraton; Abdelsalam et al., 2002) in a region previously stable, that was covered by sediments deriving from the more western Tuareg Shield in the 595-575 Ma time 120 span (Fezaa et al., 2010). These metacratonic reactivations occurred mostly along pre-existing 121 Eburnean (c. 2 Ga) lithospheric-scale terrane boundaries, along the Murzuq craton, of 122 123 probable Archean age (Fezaa et al., 2010).

These metacratonic zones were reactivated during the Mesozoic, with deposition of 124 several thousand meters of sediments in troughs, and during the late Cenozoic, with the 125 emplacement of the In-Ezzane basaltic field (Liégeois et al., 2013). The Phanerozoic 126 127 sediments of the Murzuq basin are located to the East, partly resting on the Murzuq 128 metacratonic margin, the Djanet terrane.

The Arrikine area consists of a monoclinal structure dipping 6° toward E to ENE. This 130 structural orientation gradually changes. The Murzuq geological map (Geological Map of Libya, 1985) shows that its borders, including Cretaceous levels, were uplifted and slightly 131 tilted. Very locally, folding has been observed in the In-Ezzane area (Amenna et al., 2014), 132 133 probably related to far-field effects of the Hercynian orogeny (Haddoum et al., 2001), again 134 reactivating the metacratonic structures.

In this area, we discovered a large sill of mafic rocks (maximum observed NNW-SSE 135 136 outcrop extension of about 35 km) interbedded in Silurian sediments (Fig. 2). The current 137 description of the local stratigraphy (Freulon, 1964; Bellini and Massa, 1980; Legrand, 2000; Ghienne et al., 2013), together with the published maps of this area, do not mention any 138 139 occurrence of mafic rocks.

The sill intruded the upper member of the *Oued In Djerane Formation*, mainly made 140 of shales. This upper member displays signs of a more marine environment than the 141 underlying levels, but in its top part, presents facies that are more and more regressive 142 (Legrand, 2000). It is overlain by the detritic *Acacus Formation*. The host-rocks constituting 143 144 the roof of the sill are affected by a contact metamorphism.

145

#### 146 **3. Petrography and mineralogy**

The Arrikine sill (Fig. 2) has a maximum thickness of c. 250 m and is dipping gently 147 to the ENE similarly to the host-rocks series. The Silurian host-rocks, both at the floor and 148 149 roof of the sill, show local folding due to the intrusion. Farther from the intrusion, in the Ordovician series, deformations become brittle. The sill is constituted of a pyroxene-rich 150 151 gabbro. On its borders, it is light gray in color due to a strong weathering, while in the other parts, it is dark in appearance and not or slightly altered. These dark-facies rocks are medium-152 153 grained gray-green gabbros. The main mineral is plagioclase appearing as large lengthened intertwined laths. The mafic mineral assemblage is dominated by granular clinopyroxene. 154 155 Minor constituents are thin laths of biotite associated with oxides, few small quartz grains and relatively large granules of opaque minerals. Weathering renders plagioclase brownish in 156 color and is at the origin of the incomplete replacement of some pyroxene by chlorite. 157

Microprobe analyses (Table 1) confirm that the magmatic plagioclase is a labrador 158  $(Ans<sub>55</sub>)$ , the pyroxene a diopside, the presence of biotite and apatite and that the oxides are 160 ilmenite and magnetite. The biotite is particularly rich in TiO<sub>2</sub> (c. 7%), which is also a 161 characteristic of the biotite from the Iskou ring complex in Aïr (Léger, 1985). These analyses also show that Fe-Mg minerals are often altered but above all, that an aplitic component is 162 present and is represented by small spots of K-feldspar, with diverse K compositions ranging 163 from 2 wt % to 15 wt %. This K enrichment is accompanied by a Ba-enrichment (Table 1) 164 165 developed within acid plagioclase  $(An_{13-17})$ , two minerals that cannot be in equilibrium with 166 the gabbroic magma. The development of this phase enriched in K and Ba, has consequences  $167$  for the whole rock geochemistry and its K-Ar dating (see below).

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#### 169 **4. Isotopic dating**

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#### 171 *4.1 K/Ar geochronology*

Analyses were done by one of us (H.B) at the K-Ar dating platform of UMR 6538 173 "Domaines Océaniques" CNRS, UBO at Brest.

174 *4.1.1 Analytical procedure* 

175 This sample of gabbro (AF-ALG/Dj-Site58 from site 58 – Fig. 2) was also considered 176 for dating on a whole-rock fraction made of grains  $0.3$  to  $0.15$  mm in size, prepared after hand-crushing and sieving of the solid rock, then cleaned using ultrapure water. One aliquot 177 of grains was powdered in an agate grinder. Then two independent dissolutions of 80 mg of 178 179 powdered sample were realized in two well-sealed synthetic bottles using 4  $cm<sup>3</sup>$  of 180 hydrofluoric acid at a temperature of  $80^{\circ}$ C for 8 hours. K content of the two HF solutions was 181 determined by Atomic Absorption Spectrometry after dilution (1/2500).

A second aliquot of grains was reserved for under vacuum extraction of all gases by 182 heating in a molybdenum crucible, using a high frequency generator. Cleaning of active gases 183 from the whole extracted gases was realized during and after their extraction by gettering on 184 hot titanium sponge contained in three quartz traps successively activated when their 185 186 temperature was decreasing from  $800^{\circ}$ C to the ambient one during 10 minutes. The final step 187 was the ultra-purification using an Al-Zr SAES getter.

188 Isotopic composition of argon and concentration of  ${}^{40}Ar_R$  were measured in a stainless steel mass spectrometer with a 180° geometry. Isotopic dilution was realized during the fusion 189 190 step, using precise concentrations of  $38$ Ar buried as ions in aluminium targets (Bellon et al., 1981).

A huge degasing of grains was observed and measured for the two first extractions 192 from 0.4 to 0.3 g of sample and led to a long and difficult purification of argon; consequently 193 it was less easy to reduce the effects of signal evolution through time . Two complementary 194 195 extractions of gases from significantly lighter weights  $(0.07 \text{ and } 0.06 \text{ g})$  have permitted to restore better conditions for purification and for isotopic analysis. 196

197 *4.1.2 Data* 

Resulting data of the four extractions and isotopic analyses are presented in Table 2. 198 Ages are calculated using Steiger and Jäger's (1977) constants and errors following the 199 200 equation of Cox and Dalrymple  $(1967)$ .

201 *4.1.3 Interpretation* 

202 A mean age of  $325.6 \pm 7.7$  Ma results from the analyses, i.e. a Serpukhovian age (from uppermost Visean to upper Bashkirian, if taking error margins into account). The Silurian age 203 of the host sedimentary series questions the significance of the K-Ar age particularly when 204 205 considering the huge degassing of samples under vacuum that may indicate the effects of 206 secondary processes that affected the Arrikine sill.

Such a result points to an event occurring during the Variscan period. Therefore, it is 207 legitimate to raise the question whether a geochemical remobilization of the sill, leading to a 208 potassium and barium enrichment in the plagioclases (Table 1), has occurred. This alteration 209 210 could have been promoted by cryptocirculations of fluids (supposed hot but not exactly 211 known) that may have reset the K-Ar system by complete external diffusion of radiogenic 212  $^{40}$ Ar. Following such a hypothesis, the 325.6 Ma age marks the time to which the gabbro 213 should have been rejuvenated during the Variscan evolution.

214

#### 215 *4.2 U-Pb geochronology*

#### 216 *4.2.1 Analytical procedure*

Zircons were separated from 4 kg of rocks and were subsequently processed according 217 218 to standard technique (e.g. Bosch et al., 1996), including magnetic separation and careful hand-picking under a binocular microscope. U-Pb dating on zircon was performed by laser 219 220 ablation sector field ICP-MS at Geosciences Montpellier (France). The ICP-MS (ThermoFinnigan Element XR high resolution) was coupled to a Lambda Physik CompEx 221 102 excimer laser generating 15 ns duration pulses of radiation at a wavelength of 193 nm. 222 223 Data were acquired at low resolution  $(\Delta M/M= 300)$  in the fast electrostatic scan mode. Signals were measured in pulse counting mode using 3 points per peaks and a 20% mass 224 225 window. A pre-ablation step consisting of 7 pulses was applied to clean the sample surface. 226 The acquisition step began with 15 s of gas blank measurement followed by 45 s of laser 227 ablation at a frequency of 4 Hz with a spot size of 26  $\mu$ m and an energy density of 10 J/cm<sup>2</sup>. 228 Measured isotopic ratios were monitored with reference to the G91500 zircon standard with a 229 <sup>206</sup>Pb/<sup>238</sup>U ratio of 0.17917 and a <sup>207</sup>Pb/<sup>206</sup>Pb ratio of 0.07488 equivalent to ages of 1062 Ma 230 and 1065 Ma, respectively (Wiedenbeck et al., 1995). The standard was measured four times 231 each five unknowns in a sequence of 2 standards, 5 unknowns and 2 standards (see Bosch et al., 2011 for details). Analytical data are plotted and ages calculated using the IsoplotEx 232

233 program (Ludwig 2000). Analyses in Table 3 and in the Concordia plots are  $\pm 1\sigma$  errors and 234 uncertainties in ages quoted in the text are given at  $\pm 2\sigma$ .

235 *4.2.2 Results* 

Only 17 zircons were recovered, which is not so surprising in a basic rock. They 236 237 display a large range of shapes and colors, indicating they do not form a homogeneous population. Most grains are subhedral to rounded, translucent and colorless to pink in color. 238 Although some grains have euhedral shapes, the variety of shapes and the occurrence of 239 240 rounded grains indicate that most grains may have been snatched from the country rocks, 241 which possibly include a detrital component. Reported in the Concordia diagram (Fig. 3), analyzed grains display a wide range of ages, from Neoproterozoic to Archean, ranging from, 242 243  $598\pm14$  Ma to 2707 $\pm$ 38 Ma (2 $\sigma$ , see Table 3). The main population is Paleoproterozoic with a 244 mean age of around 2030 Ma  $(n= 7)$ . The Neoproterozoic population is dominated by a coherent group of four grains plotting concordantly at *c.* 682 Ma, while other grains are 245 concordant at 766, 632 and 598 Ma. Lastly the recovered zircon population includes a 246 247 homogeneous Archean component dated at *c*. 2700 Ma.

#### 248 *4.2.3 Interpretation*

The Arrikine sill cannot be older than the intruded Silurian sediments The dated 249 zircons are therefore xenocrysts and not new minerals crystallized within the sill. This agrees 250 251 with its low-silica geochemical composition and high temperature of crystallization, 252 conditions during which the zircon saturation is very rarely attained (Watson and Harrison, 1983; Boehnke et al., 2013). The inherited zircons can have been incorporated in the Arrikine 253 254 magma by incorporating (1) unmelted zircons from the source, (2) zircons from the basement 255 during its ascent or (3) zircons from the Ediacaran or Paleozoic sediments during its final 256 emplacement, these possibilities being not mutually exclusive.

Incorporating unmelted zircon crystals from the source is unlikely, again because such 257 magmas are far from zircon saturation and are thus efficient to dissolve zircon (Watson and 258 Harrison, 1983). Moreover, the source of the Arrikine gabbro (the mantle or the lower mafic 259 260 crust) itself most probably bears very few zircons for the same reason.

261 The ages of the inherited zircons in the Arrikine gabbro  $(0.6, 0.63, 0.68, 2.03, 0.67, 2.07)$ Ga) match those of the detrital zircons from the Ediacaran sediments of the Djanet terrane, 262 263 just to the west and probably present at depth below the Arrikine area (Fezaa et al., 2010). 264 However, 2.7 Ga zircons are, strictly speaking, not present in these Ediacaran sediments (only 265 2.65 and 2.8 Ga old zircons are present). In addition, it is noteworthy that they bear other age

peaks, especially at 0.7, 1.9 and 2.5 Ga, which are not present in the Arrikine sill. The source 266 267 of these sediments is Central Hoggar, especially its closest eastern part, explaining that some samples have a proximal immature source with few detrital zircon ages (Fezaa et al., 2010; 268 2013). So, the Ediacaran sediments could have provided some of the inherited zircons in the 270 Arrikine sill, but other sources should be found.

The detrital zircons from the Tassilis Ordovician sandstones just to the West 271 (Linnemann et al., 2011) display much more numerous age peaks. These sediments are 272 considered as deriving from sources located all over West Africa and even Amazonia 273 274 (Linnemann et al., 2011). However, the 2.7 Ga age is systematically absent. Incorporation of 275 detrital zircons from these Ordovician mature sediments is thus not favored. The same 6 conclusion can be reached for the Silurian series in which the Arrikine sill intruded. Its source 27 277 is also distal and it is made of shales whose fine granulometry does not favor the presence of 278 zircons.

The basement is not outcropping in the Easternmost Hoggar. The oldest known rocks 279 are the Ediacaran sediments, metamorphosed in the greenschist facies (Djanet terrane) or 280 281 migmatized in the amphibolite facies (Edembo terrane), intrusive and subvolcanic rocks being younger (Fezaa et al., 2010). Ediacaran magmatic rocks could have had extracted material but 282 few old ages have been measured in their zircons. The Tin Amali dyke swarm for example 283 284 provided a 2090  $\pm$ 40 Ma age (Fezaa et al., 2010) for an inherited component. This age is not present in the detrital zircon age spectrum of the Ediacaran sediments and is likely to be 285 related to the age of one basement component. More information can be obtained from the Nd 286 287 isotopes. The mean  $T_{DM}$  model ages for the various magmatic bodies (batholith, pluton, and dyke swarm in the 575-545 Ma age range) fall in the 1.6-1.8 Ga age interval (Fezaa et al., 288 2010). This points to a Paleoproterozoic and/or Archean basement in the area. More to the west, but still to the east of the Raghane shear zone that constitutes the western boundary of 290 291 the Saharan metacraton, the basement of the Aouzegueur terrane (the third Eastern Hoggar 292 terrane; Fig. 1A) has been dated at  $1918 \pm 5$  Ma (LA-ICP-MS U-Pb zircon age; Nouar et al., 2011). This basement is intruded by various granitoids close to the Raghane shear-zone at c. 790 Ma, c. 590 and c. 550 Ma (Henry et al., 2009; Nouar et al., 2011). All these ages do not 294 correspond to the inherited zircons dated in the Arrikine sill. 295

So few informations are available on the basement below the Arrikine sill. Nd isotopes 296 indicate that the basement is Paleoproterozoic and/or Archean in age and an inherited zircon 297 298 point to an age of  $2090 \pm 40$  Ma. On the other side, the Arrikine inherited zircons have two 299 well-defined old ages at  $2698 \pm 22$  Ma (3 zircons) and  $2028 \pm 15$  Ma (7 zircons) with no other

zircons older than 1 Ga. These zircons can be considered as having been snatched from the 300 underlying basement, but in very small quantities due to the low viscosity of this magma, and 301 not from the superficial sediments that bear many ages but these ones. On the other hand, the 302 303 vounger inherited zircons, at  $682 \pm 7$  Ma (4 zircons),  $632 \pm 10$  Ma (1 zircon) and  $598 \pm 15$  Ma (1 zircon) could be sourced from the Ediacaran sediments that derived from the erosion of 304 Neoproterozoic units close to the Raghane shear zone. 305

The Arrikine sill, through its inherited zircons, is thus currently the best probe for 306 getting information on the basement of the Murzuq craton, at least of its western boundary, 307 indicating the existence of Archean (c 2.7 Ga) and Paleoproterozoic (c. 2.03 Ga) lithologies at 308 309 depth. On the other hand, due to the absence of magmatic zircons from the sill itself, the zircon dating was not suitable to date emplacement of the Arrikine sill. 310

311

#### 312 **5. Paleomagnetic dating**

#### 313 *5.1 Sampling and analysis procedure*

The sampling sites for paleomagnetism are located near the oued Arrikine (Fig. 2). 314 110 standard paleomagnetic samples, taken using a portable gasoline-powered drill and 315 316 oriented with magnetic and sun compasses, were collected from the 12 sites (sites 50 to 61 on Fig. 2) in the gabbro (including one site in the weathered gray facies on the sill border and 317 another one within a late dyke). 9 samples were chosen in one site (site 49 on the Figure 2) of 318 Silurian host-rocks affected by contact metamorphism close to the roof of the sill. One to 319 320 three specimens were cut from each core in order to have additional specimens for pilot studies and rock magnetic analysis. Prior to any demagnetization analysis, the specimens were 321 stored in a zero field for at least one month, in order to reduce possible viscous components. 322 The Natural Remanent Magnetization (NRM) of the specimens was measured using a JR5 323 spinner magnetometer (AGICO, Brno). Several pilot specimens from each site were subjected 324 325 to a stepwise alternating field (AF) demagnetization up to 100 mT or thermal (TH)  $326$  demagnetization up to  $670^{\circ}$ C in order to characterize the components of magnetization. Following combined demagnetization process, some other specimens were subjected to 327 328 combined AF and TH treatments. The directions of the magnetization components were plotted on orthogonal vector plots (As and Zijderveld, 1958; Wilson and Everitt, 1963; 329 Zijderveld, 1967) and the remaining vectors and vectorial differences of the magnetization on 330 equal-area projections. The direction of the different components was computed using 331 principal component analysis (Kirschvink, 1980). Fisher (1953) statistics was used to 332 determine the mean directions. 333

334

### 335 *5.2. Rock magnetism*

The NRM has a relatively high intensity in dark facies (mean 3.2 A/m) compared to 336 337 the weathered site close to the sill border (mean 2.7  $10^{-2}$  A/m) and the late dyke (mean 5.3  $10^{-2}$  A/m). AF demagnetization yields a relatively regular decrease of magnetization intensity. For most samples, magnetization decrease during thermal demagnetization (Fig. 4) is 339 progressive (IZ647, IZ668) or steep above 400-450°C treatment up to about 530-560°C 340 (IZ654). These maximum blocking temperature values suggest the presence of Ti-poor 341 342 titanomagnetite as the principal mineral carrier. In few samples (Fig. 4), a strong increase of 343 the magnetization for about 400 $^{\circ}$ C is followed by a sharp decrease around 450 $^{\circ}$ C up to about 530-560°C (IZ718, IZ729). No increase appears during AF demagnetization of these samples, for which complementary studies of the magnetic properties have to be performed and will be 345 346 the subject of an independent paper.

Thermomagnetic curves (low-field magnetic susceptibility as a function of 347 348 temperature) have been measured for representative samples by heating in air using CS2–3 349 oven and KLY3 Kappabridge (AGICO, Brno). For the gray weathered facies, during heating, 350 the partial cooling curves are often reversible with the heating ones (Fig. 5 - IZ696). In such 351 cases, irreversibility of the thermomagnetic curves appears only after heating at the highest  $352$  temperatures. During heating at temperatures higher than  $400^{\circ}$ C, a decrease of susceptibility is observed, giving Curie temperature of about 550°C. This confirms that the principal 353 magnetic carrier is a Ti-poor titanomagnetite. However for some samples, a slight decrease 354  $f_{\text{55}}$  for temperatures higher than  $580^{\circ}$ C points out the presence of a small amount of hematite besides the titanomagnetite (Fig. 5 - IZ696). In the dark facies, the sample thermomagnetic 356 curves are not reversible since 400 to 500°C heating, highlighting a relatively complicated 357 mineralogical alteration during heating at higher temperature (Fig. 6 IZ716). 358

359 Hysteresis loops were obtained for small cores (cylindrical samples of about 3 cm<sup>3</sup>) using a laboratory-made translation inductometer within an electromagnet capable of reaching 360 1.6 T. The loops show that the remanent coercive force determined from several 361 362 representative samples is low (around 30 mT) and that a partial saturation of the 363 magnetization is at least reached between approximately 0.2 to 0.4 T (IZ678, IZ725), confirming the presence of a low coercivity component (like Ti-poor titanomagnetite). For 364 weathered gray facies, susceptibility is very low and almost linear hysteresis loops have been 365 obtained, indicating largely dominating effects of the paramagnetic minerals. For dark facies 366 (Fig. 6), a much higher susceptibility is associated with a clear dominance of ferrimagnetic 367

368 minerals. For these sites, Day plot (Day et al., 1977) indicates pseudo single-domain (PSD)  $369$  grain size for the titanomagnetite (Fig. 6).

- In conclusion, Ti-poor titanomagnetite is the main magnetic carrier in the non-370 weathered gabbro. It is also present in the weathered gray facies, but in much lower amount 371 372 and associated with few hematite.
- 373

#### 374 *5.3. Paleomagnetic analyses*

375 TH or AF or mixed AF-TH demagnetizations (Fig. 7) were performed on all samples. Very similar Characteristic Remanent Magnetization (ChRM B) data have been obtained 376 377 from all methods, in addition to a viscous component A. During the demagnetization process, three different main kinds of evolution were observed for gabbro sites: 378

379 - The first type, obtained on half the samples, is illustrated by a single stable magnetic 380 direction. Figures 7a, b and g show examples of this behavior obtained from an AF method (IZ659), thermal process (IZ 644) or mixed AF-TH (IZ660A). In this case, after elimination 381 382 of the viscous component A, the analysis of the magnetization shows that the linear segments 383 towards the origin on Zijderveld (1967) plots, define the ChRM B.

384 - The second type of behavior shows magnetic directions evolving first along a great circle 385 (remagnetization circle), which indicates superimposition of the unblocking temperature (or 386 unblocking fields) spectra for at least two remanent magnetization components. This 387 evolution always reaches a ''stable end point'' direction yielding the ChRM B. This case is 388 illustrated on the figures 7c and d for the AF method (IZ 709) and the thermal method (IZ 389 739). Here also, similar results are obtained by the two method of demagnetization. The 390 secondary component at the origin of the variation along the great circles cannot be properly 391 identified. It could correspond to a Cenozoic overprint (Henry et al., 2004; see also for 392 example Hargraves et al., 1987), but other origins cannot be rejected by lack of constraints.

393 - The third case (Figs. 7e, f) displays similar variation than the first case, except that, during the thermal treatment, a component, with the same direction as the main magnetization but 394 395 with opposite polarity, appears at c.  $400^{\circ}$ C and progressively disappears at higher temperature (from c 500°C), yielding a well-defined ChRM magnetization. During AF demagnetization, 396 this intermediate component of opposite polarity is not present. The final ChRM directions 397 obtained for both demagnetization processes are the same. This additional component is 398 399 therefore related to a mineralogical alteration due to heating. These samples will be the subject of a complementary analysis about magnetic properties. 400

The ChRM B obtained from samples with these three behaviors was isolated between 401 402 200 and 500 °C for the lowest temperatures and 400–560 °C for the highest ones (thermal process), and between 10-70 mT for the lowest blocking field and 60 and 102 mT for the 403 404 highest ones (AF demagnetization).

Results from the sites sampled in weathered facies on the sill border (14 samples) and 405 406 within a late dyke (12 samples) are mostly not reliable. The obtained results from all the other samples are homogeneous (Table 4). The mean direction of this ChRM B computed from 84 407 408 specimens (from 12 sites) has the following directions:  $D = 355.4^{\circ}$ ,  $I = -38.4^{\circ}$ ,  $k = 38$ , 409  $\alpha_{95} = 2.5^{\circ}$  and  $D = 350.7^{\circ}$ , I = -38.6°, k = 38,  $\alpha_{95} = 2.5^{\circ}$ , before and after dip correction, 410 respectively (Table  $4 - Fig. 8$ ). This result is similar to that obtained from the mean direction 411 of the 10 sites (two sites with less than 3 ChRMs being not considered):  $D = 355.5^{\circ}$ ,  $I = -38.8^\circ$ ,  $k = 86$ ,  $\alpha_{95} = 5.2^\circ$  and  $D = 350.6^\circ$ ,  $I = -38.9^\circ$ ,  $k = 86$ ,  $\alpha_{95} = 5.2^\circ$ , before and after dip 413 correction, respectively (Fig. 8).

The paleomagnetic data obtained from the site of Silurian host-rocks affected by 414 415 contact metamorphism have similar characteristics with neighboring ChRM orientation  $(N=9)$ 416 samples,  $D = 358.6^\circ$ ,  $I = -42.8^\circ$ ,  $k = 402$ ,  $\alpha_{95} = 2.6^\circ$  and  $D = 349.5^\circ$ ,  $I = -41.7^\circ$ ,  $k = 402$ ,  $\alpha_{95} = 2.6^{\circ}$ , before and after dip correction, respectively) but with lower magnetization 8 intensity. Other sites, taken in the Ordovician and Silurian rocks far from the sill (sites 43 to 41 419 48 on Fig. 2), yield only weak Cenozoic remagnetization of normal and reversed polarity (see 420 Henry et al., 2004), totally overprinting any possible previous earlier magnetization. That is then not the case for the site affected by contact metamorphism. The samples from this site 421 422 carried a much more stable magnetization than the other sites. That is attributed to an effect of the contact metamorphism generated by the sill. This kind of positive contact test strongly 423 argues for a primary magnetization of the gabbro. 424

The corresponding paleomagnetic pole determined from the sill data is defined by 425 426  $\lambda = 43.6^{\circ}$ S,  $\phi = 17.0^{\circ}$ E, K = 88 and A<sub>95</sub> = 4.7° and  $\lambda = 42.8^{\circ}$ S,  $\phi = 22.9^{\circ}$ E, K = 88 and  $A_{95} = 4.7^{\circ}$ , before and after bedding correction, respectively.

428

#### 429 *5.4 Paleomagnetic dating of the Arrikine sill*

A gabbro magnetization age younger than the post-Cretaceous tilting of the formations 430 is unrealistic when comparing the Arrikine poles with the known Apparent Polar Wander Path 431 432 - APWP - (Fig. 9) and data can only be used after applying a bedding correction. The so corrected Arrikine pole (Fig. 9) corresponds to the Gondwana APWP segment for the 400 Ma 433 434 period of Derder et al., 2006, Amenna et al., 2014 and Henry et al., 2015 and to that of

Torsvik et al., 2012 (overlapping of confidence zones) for the 410 Ma mean pole. In the latter, 435 the uncertainty on the paleomagnetic mean pole age is not taken into account while it 436 corresponds to a single pole (Aïr intrusives, Hargraves et al., 1987), which can explain the 437 438 difference existing with the former one. Indeed, integration of such uncertainties in the first APWP adds a weighted effect for poles having a large age uncertainty and thus covering 439 several windows in time, smoothing the curve. This is particularly critical for periods affected 440 by strong changes of the APWP drift such as the 400-410 Ma period (Fig. 9). Geologically, 441 the Arrikine sill is post-Silurian and pre-Cretaceous. The Arrikine pole is very far from all 442 mean paleopoles from Middle Devonian to Present times, especially for Carboniferous times. 443 It is also far from Silurian mean paleopoles (Fig. 9). By contrast, the Arrikine pole is 444 consistent with Early Devonian poles of Gondwana: Mt. Leyshon Devonian dykes in 445 Australia (Clark, 1996), Snowy River Volcanics in Australia (Schmidt et al., 1987), Herrada 446 447 Member, Sierra Grande in Argentina (Rapalini and Vilas, 1991) and Aïr alkaline ringcomplexes in Niger (Hargraves et al., 1987). In particular, the coincidence of the Arrikine 448 449 pole with this last pole  $(\lambda = 43.4^{\circ}S, \phi = 8.6^{\circ}E, K = 50 \text{ and } A_{95} = 6.2^{\circ})$  is remarkable. The 450 angular difference between these paleomagnetic poles  $(10.7^{\circ})$  is of the order of what can be 451 ascribed to the secular variation. In both the Arrikine sill and the Aïr alkaline ring-complexes, the paleomagnetic directions have the same single polarity. So the Aïr ring-complex age of 452  $453$  407 $\pm$ 8 Ma (Moreau et al., 1994) can be considered as a good approximation of the age of the 454 Arrikine sill, which can be fixed to the 400-415 Ma age interval, i.e. at the base of the Emsian, from mid-Lochkovian to mid-Emsian (Cohen et al., 2013), thus globally Lower 455 Devonian. 456

The loop of the Gondwana APWP between the Late Ordovician (pole 450 Ma) and 457 Late Devonian (370 Ma) was not generally deemed as very reliable. The Aïr and Arrikine 458 coherent mean is "unique" and supports the existence of this loop that has to be considered for 459 460 the paleocontinental reconstructions for this period.

461

#### 462 **6. Geochemistry**

Only one analysis (sample AF-ALG/Dj-Site58 from site 58 – Fig. 2) is available and 463 listed in Table 5. However, its geochemical signature (Fig. 10) is sufficiently diagnostic to 464 allow a proper geochemical characterization of the Arrikine sill and to deduce constrained 465 466 conclusions about its origin. All oxides are considered in column  $a^*$  as deduced from the geochemical analysis by ICP and in column b\* are recalculated to 100% on an anhydrous 467 basis. Oxide % are always wt. %, unless otherwise stated. The Arrikine sill is a fine-grained 468

69 gabbro but (see below) its chemistry is similar to intraplate alkali basaltic rocks in continental 4 flood basalt (CFB) and ocean island basalt (OIB) provinces. So, in order to make the 470 comparison more efficient, geochemical diagrams dedicated to volcanic rocks and volcanic 471 nomenclature are applied here, which is justified by the subvolcanic character of the sill and 472 by the difficulty of counting a mode on this kind of rock. 473

474 The Arrikine gabbro having 45.99% SiO<sub>2</sub> and 5.79% Na<sub>2</sub>O+K<sub>2</sub>O, on an anhydrous basis, belongs to the alkali series, being an alkali gabbro. It has the composition of a 475 476 trachybasalt (Le Bas et al. 1986), more precisely of an hawaiite (wt%Na<sub>2</sub>O-2 > wt%K<sub>2</sub>O), i.e. a sodic trachybasalt (Le Maitre et al., 2002). The depleted MgO content (4.1%) corresponds 477 to typical hawaiite (3 - 5.5 % MgO; MacDonald and Katsura, 1964) as is the absence of 478 chromite (Cr: 3 ppm; Ni: 39 ppm), a characteristic of hawaiite (Cr: 5-150 ppm; Ni: 5-80 ppm; 479 Nelson and Carmichael, 1984 and references therein), in agreement with the differentiated 480 481 character of hawaiites. Immobile trace elements confirm that the Arrikine gabbro has the 482 composition of an alkali basalt with  $Nb/Y = 0.85$  and  $Zr/TiO<sub>2</sub>*0.0001 = 0.0064$  (diagram not shown; Winchester and Floyd, 1977). Low concentration of Ni, Cr and absence of positive Eu 483 anomaly do not point to a significative cumulative component. Even the very low Cr content 484 485 in Arrikine gabbro points to a differentiated character.

486 In addition, with  $TiO_2 = 5.01\%$ , the Arrikine gabbro belongs to the high-Ti series 487 (TiO<sub>2</sub>  $>2\%$ ); it has the composition a high-Ti alkali basalt. CIPW norm points to 60% plagioclase (An30), 11% K-feldspar, 9% pyroxene, 6% olivine, 2% apatite and 11% Fe-Ti 488 oxides (Table 5) in rough agreement with the petrography except that the plagioclase is acid 489 (actually Labrador) and the absence of biotite, not computed in the norm. This norm is in 490 agreement with a composition of a high-Ti hawaiite. The important difference in the An 491 content of the plagioclase can be ascribed to the late "aplitic" phase determined by the 492 493 microprobe measurements to be acid oligoclase (An13-17). The An30 value determined by the norm suggests that this aplitic phase was quite pervasive at least in the analyzed sample. 494 This implies that concerned elements (i.e. K, Ba but also Rb, Sr) must be used with caution 495 496 when the original magma is concerned as well as the K-Ar age for which we have calculated (method based on an initial age of 410 Ma given by paleomagnetic considerations and the 497 rejuvenated result at 326 Ma) a K enrichment of up to 20 %. 498

499 The contemporaneous (407  $\pm$  8 Ma; Moreau et al., 1994) large province of alkaline 500 ring-complexes of Aïr bears also high-Ti gabbros, especially the large Ofoud ring-complex 501 where they are associated with numerous layers and/or lenses of titanomagnetite up to 10 m 502 thick (Karche and Moreau, 1978) and the smaller Abontorok ring-complex, associated with

503 massive anorthosites (Brown et al., 1989). These gabbros are of uniform grain size (2-7 mm) 504 and massive in texture and titanomagnetite and ilmenite can make up as much as 10 percent 505 of the rock (Hush and Moreau, 1982). The Aïr ring-complex province is intrusive along the 506 western boundary of the Saharan metacraton (Liégeois et al., 1994, 2013) as a consequence of 507 its reactivation during early Devonian (Moreau et al., 1994). The Arrikine gabbro is compared 508 to the available analysis from Abontorok ring complex (Brown et al., 1989) and to several intraplate continental high-Ti alkali basalt or gabbro provinces: the Trias rift-related from SW 509 510 China, similar to the high-Ti basalts from the Emeishan LIP (Zhang et al., 2013), the 511 Ordovician Suordakh and Cambrian Kharaulakh mafic rocks from Siberia (Khudoley et al., 2013) or the 1.5 Ga Yangtse mafic province (Fan et al., 2013). We added also for comparison 512 513 the intraplate high-Ti potassic basalts from the Virunga (Rogers et al., 1998).

514 When compared to these high-Ti alkali basalts from different settings and different 515 ages, the Arrikine gabbro has globally a similar pattern (Fig. 10). Compared to Mauna Loa 516 volcano in Hawaii (Wanless et al., 2006), the Arrikine gabbro displays a subparallel REE pattern, being slightly more enriched, especially in LREE (Fig. 3a). Similarly, its pattern in 517 518 the spidergram (Fig. 10b) is parallel, only very slightly enriched in HFSE, more enriched in LILE especially in Ba. High-Ti basalts have a largely preponderant mantle origin by contrast 519 520 to low-Ti basalts (e.g. Deckart et al., 2005), explaining similar compositions in the oceans and 521 in the continents. This characteristic renders, however, this kind of magmas relatively rare 22 within the continents, which is an important fingerprint of the Arrikine sill. The spatially close 5 523 Aïr high-Ti gabbros (Abontorok ring complex; Brown et al., 1989) display REE patterns close 524 to that of Arrikine (Fig. 10a) except for Eu, probably due to a plagioclase cumulative pattern 525 (Aïr ring-complexes comprise massive anorthosites - Brown et al., 1989; Demaiffe et al., 1991). Abontorok spidergrams are also close to that of Arrikine, the latter being on the most 526 enriched side. This can be related to its low Cr content suggesting a differentiated character, 527 528 the differentiation being able to concentrate incompatible elements. However, in the Arrikine 529 gabbro Rb, Ba and K appear particularly enriched; these enrichments can be related to the 530 presence of the late metasomatic aplitic phase (See K-Ar section; Table 5) probably favored 531 by the sedimentary environment. This is particularly visible when compared with the Suordakh high-Ti basalt from Siberia (Khudoley et al., 2013) in a primitive mantle-532 normalized graph (Fig. 11): the Arrikine basalt is very similar to the Suordakh basalts but 533 534 with concentrations in K as high as the richer Suordakh basalts and with Ba contents much 535 higher than any Suordakh basalts. This indicates that the K, Rb and Ba concentrations in the pristine Arrikine gabbro were probably lower than that currently observed. By opposition, the 536

other elements (HFSE, Th) can be considered as pristine, being not mobilized by an aplitic 537 fluid. 538

If the Arrikine gabbro composition is similar to the intraplate alkaline continental 539 540 high-Ti alkali basalt or gabbro provinces, it is distinct from the intraplate high-Ti potassic 541 basalts from the Virunga (Rogers et al., 1998), except for Ba. By opposition to the latter, the sources of the formers are considered to be a more or less enriched asthenospheric source with 542 very low lithospheric participation, similarly to Hawaii volcanoes but in a continental setting. 543 The similarity of the geochemistry of the Arrikine sill with the lower Devonian Aïr 544 545 magmatism concurs to support the lower Devonian age determined in the paleomagnetic 546 section.

Based on the silica content, a simple calculation leads to an estimated liquidus 547 548 temperature of 1280°C. When considering the experimental work of Botcharnikov et al. (2008) determining a liquid line of descent for ferro-basalts, the Arrikine concentrations in 549  $550$  SiO<sub>2</sub>, MgO, TiO<sub>2</sub>, K<sub>2</sub>O, Na<sub>2</sub>O, Al<sub>2</sub>O<sub>3</sub>, FeO, CaO point all to a temperature of c. 1080<sup>o</sup>C, 551 which is the temperature where the maximum concentration in FeO and  $TiO<sub>2</sub>$  is attained. This 552 is true when considering nearly anhydrous magmas, which is the case of Arrikine  $(0.2\% \text{ H}_2\text{O},$ 553 Table 5). Considering the temperature of the mantle partial melting at  $1280^{\circ}$ C and the 554 crystallization of the Arrikine sill at 1080°C seems reasonable. This is indeed the temperature 555 range observed in experimentation concerning Fe-rich basalts (Chevrel et al., 2014). The 556 calculated viscosity at liquidus temperature is very low  $(0.17 \text{ Pas})$ , as it is the case for Fe-rich 557 basalts whose viscosity is less than 1 Pas (Chevrel et al., 2014). In a 3 m-width dyke, a magma rise speed higher than 10 m/s can be proposed, a speed that limits fractional 558 559 crystallization and interaction with country-rocks during ascent (Chevrel et al., 2014). This implies that the differentiation that affected the Arrikine magma (low Cr contents) occurred at 560 561 depth.

Most of these provinces are considered to be or having been fed by a mantle plume. 562 563 Notwithstanding the fact that the existence of mantle plumes is currently challenged (e.g. Foulger and Hamilton, 2014), there is no one argument for a plume origin in the case of the 564 565 Arrikine sill: very small magmatic volume, no swell, no rift, no magmatic track, even if a similar asthenospheric mantle source can be inferred for the Arrikine sill. The proposed 566 alternative will be discussed below in the geodynamic section. 567

568

- 569 **7. Emplacement and reactivation of the Arrikine sill: why there and why at these times?**  570
- 571 *7.1 Regional considerations*
- 572 *7.1.1. The time of emplacement, the Lower Devonian*

During the Pan-African orogeny and the Phanerozoic, the Murzuq craton played an 573 important role as a rheologically rigid body. Magmatism and compressive (Pan-African) or 574 575 extensional (Phanerozoic) tectonics occurred around it, and the Murzuq basin developed as an intracontinental sag basin in a similar way as Al Kufra and Chad basins (Heine et al., 2008). 576 Formation of the Murzuq basin did not require accommodation of tectonic subsidence through 577 faulting and rifting but resulted from the negative buoyancy caused by thick cratonic 578 579 lithosphere (Ritzmann and Faleide, 2009). As a consequence, the Murzuq craton is devoid of 580 magmatism except along its metacratonic boundaries where Cenozoic volcanic fields are 581 particularly present all around it (Fezaa et al., 2010; Liégeois et al., 2013). Indeed, the West African Cenozoic volcanism is attributed to tectonic reactivations induced by the Europe-582 583 Africa convergence (Liégeois et al., 2005; Beccaluva et al., 2007; Lustrino et al., 2012; 584 Radivojević et al., 2015). These reactivations, even induced by the rather low stress propagating in remote intracontinental regions, must affect the whole lithospheric thickness 585 586 allowing for small mantle melt volumes to reach the surface. This is the case of the megashear zones within metacratonic areas that are rigid but fractured (Liégeois et al., 2003, 2005, 587 2013; Bardintzeff et al., 2011) through vertical planar delamination as recently evidenced by 588 589 magnetotelluric studies in Hoggar (Bouzid et al., 2015).

The Arrikine sill is lower Devonian in age and intruded Silurian sediments. It can be 590 correlated with Paleozoic dolerites in Niger (Fig. 1B), which are also intrusive within Silurian 591 sediments, suggesting a regional event along the southwestern border of the Murzuq basin, 592 593 even if limited in volume.

594 By opposition, no direct evidence of magmatism, even as detrital pebbles, were described within the sedimentary Devonian formations of the Murzuq basin (Bellini and 595 596 Massa, 1980). This fact is tentatively related to the so-called Caledonian event (base of the 597 Devonian) that prevented the deposition of lower Devonian sediments (Ghienne et al., 2013). This "Caledonian" event" was marked in the Murzuq basin by large scale uplifts and 598 secondary highs roughly oriented SSE–NNW in SW Libya, i.e. parallel to the Pan-African 599 600 major shear zones in the Djanet and Edembo terranes (Davidson et al., 2000; Ghienne et al., 2013) constituting the western metacratonic boundary of the Murzuq craton (Fezaa et al., 601 2010). 602

This means that the Arrikine sill intruded during a period of reactivations of the mega-603 shear zones of the region, known to be former terrane boundaries and as such of lithospheric 604 scale (Fezaa et al., 2010). The Aïr ring complex province also resulted from the reactivation 605 of a major shear zone, the Raghane shear zone (Moreau et al., 1994), which corresponds to 606 607 the western boundary of the Saharan metacraton (Liégeois et al., 1994; Abdelsalam et al., 2002). The shift from the rapid vertical ascent along the mega-shear zone of the low-viscosity 608 Arrikine magma to the widespread horizontal sill development can be tentatively attributed to 609 610 the characteristics of the Silurian Acacus formation. The latter is a massive level (about 100 m 611 thick) overlying lagunal deposits that include gypsum-rich beds (Freulon, 1964). We thus suggest that the Arrikine magma intruded within evaporitic-rich facies, which, at the contact 612 with the high temperature magma, became much more easily deformable and moreover 613 released water due to gypsum transformation, largely blocking the magma ascent. However, 614 looking at the age of the magmatism and at the stratigraphic series, the Arrikine sill should 615 616 have intruded the cover at shallow depth (of the order of less than  $300 \text{ m}$ ), in agreement with the moderate size (PSD) of the magnetite grains. These renders the presence of Arrikine aerial 617 products likely but in that case it is likely that they were eroded and transported far away by 618 619 the large fluvial system that developed at this period in the Murzuq basin (Ghienne et al., 2013) as it was the case for most of the Lower Devonian deposits in the Murzuq basin 620 (Davidson et al., 2000). 621

This kind of emplacement would be favorable to suggest a link between the Arrikine 622 magmatism and the magmatic rocks proposed to be at the origin of sand injections in the 623 Murzuq area (Fig. 1A; Moreau et al., 2012). Indeed, these peculiar structures occur in the 624 625 Silurian sediments around the Murzuq basin in a similar manner as the Arrikine sill and are considered as Devonian in age. These sand injections would be the result of the combined 626 effects of tectonic uplift, igneous intrusions in the sand-rich Cambrian-Ordovician sediments 627 and overpressures generated by the paleo-relief existing below the Silurian strata (Moreau et 628 al., 2012). The Arrikine sill would correspond to the magma that intruded the Silurian 629 630 sediments, lower Devonian sediments having not deposited in the area due to uplift. Let us remark that the three known occurrences of sand injections (Mt Telout, In-Ezzane to the west 631 and El Meherschema to the east; Fig. 1A) are also located around the Murzuq craton, within 632 its metacratonic margin, the Tibesti area being the equivalent of the Djanet and Edembo 633 634 terranes from Eastern Hoggar (Fezaa et al., 2010).

635 *7.1.2. The time of rejuvenation, the Mid-Carboniferous* 

636 The K-Ar method has given an age of  $325.6 \pm 7.7$  Ma that we consider as close to that of a rejuvenation event during the Serpukhovian, i.e. from uppermost Visean to upper 637 638 Bashkirian when taking into account the error bracket-attributed to aplitic fluid percolation. Such an age is also that of the erasure of the primary magnetization in the older sedimentary 639 rocks of the Saharan platform (Henry et al., 2004). It corresponds thus to the second main 640 tectonic event in the area. This Mid-Carboniferous compression event led to the major so-641 called Hercynian unconformity that affected the upper part of the Paleozoic sequence in the 642 Murzuq Basin but whose initial phases started during middle Carboniferous (Bellini and 643 Massa, 1980), or at the Visean-Serpukhovian boundary (Fröhlich et al., 2010 and references 644 therein). This event was responsible for the uplift of the Tiririne High (NW Murzuq basin) at 645 the end of the Visean, removing the Assedjefar-Marar (Lower Carboniferous) sequence 646 647 (Echikh and Sola, 2000). It began with transpressional movements along a series of NNE-648 SSW trending wrench faults (parallel to the Murzuq craton boundary), producing a NE-SW en 649 echelon pattern of folds on the Tiririne High (Echikh and Sola, 2000). At the scale of the Murzuq basin, the last marine deposits are of Moscovian age (e.g. Massa et al., 1988; Amenna 650 51 et al., 2014) and the upper Carboniferous is nearly entirely lacking (due to uplift or to erosion) 6 and only some red lacustrine mudstone (e.g. upper Carboniferous Tiguentourine Formation) 652 653 can be rarely found (Davidson et al., 2000).

Such a major compressive event, with transpressional movements and various uplifts 654 at the regional scale is favorable for fluid movements, able to reset the K-Ar geochronometer 655 that in turn is able to date it. A Serpukhovian reactivation ("Hercynian") occurred at the same 656 place as the early Devonian ("Caledonian") reactivation. This indicates that emplacement of 657 8 the Arrikine sill was related to the lithospheric character of the mega-shear zones of the region 65 (Fezaa et al., 2010). This implies that these intraplate reactivations are quite important and 659 must be considered. 660

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#### 662 *7.2 Global considerations*

For a long time, the use of the "Caledonian" and "Hercynian" expressions was 663 suggested for the intraplate intense tectonic reactivations that occurred around the Murzuq 664 craton. Causes must be found in events that occurred at the plate margins at these periods. In 665 addition to tectonic reactivations, volcanism may be generated by adiabatic pressure release of 666 7 uprising asthenosphere in response to stress resulting from far field collisions such as, for the 66 West African Cenozoic volcanism, the Africa-Europe collision (Liégeois et al., 2005). Indeed, 668

669 these "Caledonian" and "Hercynian" effects are known all over the Saharan platform, to the north of Hoggar (Haddoum et al., 2001; Galeazzi et al., 2010), around the West African 670 671 craton (Michard et al., 2008) or within the Algerian petroleum basins (Galeazzi et al., 2010) and in the whole Northern Africa (Guiraud et al., 2005). The Alpine reactivation is generally 672 673 superimposed on the Hercynian reactivated structures (Smith et al., 2006, Derder et al., 2009). These "Caledonian" and "Hercynian" tectonic events are also recorded at the regional scale by 674 675 detrital zircons in the Murzuq basin (Morton et al., 2011).

The Arrikine sill emplacement, as well as that of the Aïr ring-complex province 676 (Moreau et al., 1994) at c. 410 Ma, occurred during a continental breakup, when the Hun 677 terranes separated from the northern Gondwana margin (Stampfli and Borel, 2002, Blackey, 678 2008 and references therein). Such a geodynamical environment is favorable to generate 679 intraplate tectonics and magmatism as it was the case, for West Africa, during the continental 680 breakup. This led to the Atlantic ocean opening that generated the Central Atlantic Magmatic 681 Province (CAMP; Marzoli et al., 1999), which extends in West Africa (e.g. Chabou et al., 682 683 2007, 2010), SW Europe (Callegaro et al., 2013) and even Sicily (Cirrincione et al., 2014), and East America, including Bolivia (Bertrand et al., 2014). This province, even tholeiitic, 684 comprises abundant high-Ti basalts related to an asthenospheric mantle source (Deckart et al., 685 2005). We thus suggest that the Arrikine sill, and associated magmatism, emplaced due to this 686 continental breakup that also generated the Caledonian unconformity. 687

688 The mid-Carboniferous reactivation with an age at  $325.6 \pm 7.7$  Ma as determined by 689 the K-Ar method is easily related to the late Variscan episode that resulted from the collision between Gondwana, especially NW Africa, and southern Europe from 330 Ma to early 690 Permian (Schulmann et al., 2014). The fact that this important event generated no magmas 691 (except the aplitic metasomatism) can be related to the transpressive character of this 692 reactivation, unable to allow magmas to rise if no major horizontal movements occurred, as it 693 is the case in an intraplate setting. 694

To sum up, we propose that the Arrikine sill was emplaced along a major lithospheric 695 weakness zone, the margin of the Murzuq craton during the Gondwana-Hun continental 696 697 breakup. Similarly, the reactivation of the major Raghane shear zone bordering the Saharan metacraton that allowed the emplacement of the Aïr ring complex province (Moreau et al., 698 1994), can be attributed to this continental break-up. This lithosphere weakness zone was 699 700 reactivated during the late Variscan orogeny that involved Gondwana. These two events correspond to the two major unconformities with important sedimentary hiatus known in the 701 702 region, the so-called Caledonian and Hercynian unconformities.

#### 703

#### 704 **8. Conclusions**

705

The newly discovered Arrikine sill is a large magmatic object (pyroxene-rich gabbro) 706 of Lower Devonian age emplaced in Silurian sediments, rather unexpected in far-east Hoggar. 707 Although emplaced in an intraplate environment, its datation was not straightforward: all 708 zircons are older than the country rocks and are thus inherited (most zircons are at c. 2030 Ma 709 710 with additional ages at c. 2700 Ma and in the 766-598 Ma age range) and its rejuvenated K-Ar 711 age of  $325.6 \pm 7.7$  Ma relates to a remobilization event due to a K-rich aplitic fluid that reset the isotopic system and that is recorded by secondary minerals (K-feldspar, acid plagioclase, 712 quartz). By contrast, the paleomagnetic pole is reliable and allows dating of the Arrikine sill 713 emplacement in the 400-415 Ma age interval (mid-Lochkhovian-mid-Emsian). 714

Geochemically, the Arrikine gabbro has the composition of a high-Ti alkali basalt, 715 716 actually a high-Ti hawaiite (sodic trachybasalt), with  $46\%$  SiO<sub>2</sub>,  $4\%$  MgO,  $\%$ Na<sub>2</sub>O-2> $\%$ K<sub>2</sub>O, 717 absence of chromite and 5%  $TiO<sub>2</sub>$ . As a whole, this chemistry is close to that of the Mauna Loa volcano in Hawaii (Wanless et al., 2006), in agreement with an enriched mantle origin 718 needed for such a high-Ti/low-Si composition. This peculiar chemistry is similar to several 719 intraplate continental provinces such as the Trias rift-related from SW China, the high-Ti 720 basalts from the Emeishan LIP (Zhang et al., 2013), the Ordovician Suordakh and Cambrian 721 722 Kharaulakh mafic rocks from Siberia (Khudoley et al., 2013) or the 1.5 Ga Yangtse mafic province (Fan et al., 2013). It is also similar to some lithologies belonging to the lower 723 Devonian Aïr ring complexes province in Niger, such as the Abontorok ring complex (Brown 724 et al., 1989). The peculiarity of these compositions supports so a lower Devonian age for the 725 Arrikine sill, as is proposed in the paleomagnetic section. The Arrikine gabbro composition 726 727 points to a liquidus temperature of 1280°C and a temperature of 1080°C for the magma when 728 it crystallized close to the surface.

Geodynamically, the Arrikine sill emplaced in Silurian sediments belonging to the 729 western boundary of the Murzuq craton, actually its metacratonic margin (Fezaa et al., 2010; 730 Liégeois et al., 2013). Its emplacement is related to the reactivation of this metacratonic 731 margin due to a low stress that propagated in this remote intracontinental region. This was a 732 consequence of "Caledonian" events that also generated in the area the so-called Caledonian 733 unconformity, witness of the absence of lower Devonian sediment deposition (e.g. Ghienne et 734 al., 2013). A link with the magmatic rocks at the origin of the sand injections at the Murzuq 735 736 craton margins (Moreau et al., 2012) is suggested. The Aïr ring complexes were emplaced at

the same period, also along a major lithospheric discontinuity, the Raghane shear zone 737 (Moreau et al., 1994), which is the western boundary of the Saharan metacraton (Abdelsalam 738 739 et al., 2002).

The Mid-Carboniferous rejuvenation dated by the K-Ar age  $(325.6 \pm 7.7 \text{ Ma})$  and marked by a aplitic fluid phase can be correlated with the erasure of the primary 741 742 magnetization in the older sedimentary rocks of the Saharan platform (Henry et al., 2004). It corresponds to the second main Phanerozoic tectonic event in the area marked by the so-743 called Hercynian unconformity, responsible for important transpressive vertical movements 744 745 such as the uplift of the Tiririne High (NW Murzuq basin).

Emplacement (c. 410 Ma) and rejuvenation (c. 326 Ma) of the Arrikine sill are thus 746 attributed to intense tectonic reactivations that occurred around the Murzuq craton, able to 747 generate magmas of mantle origin through adiabatic pressure release of uprising 748 asthenosphere along mega-shear zones. The effects of these two events being known all over 749 the Saharan platform, it is concluded that they resulted from major events that occurred at 750 plate boundaries. We suggest that the extensional event in the Arrikine area at 410 Ma is 751 linked to the continental breakup when the Hun terranes separated from the northern 752 Gondwana margin (Stampfli and Borel, 2002, Blackey, 2008 and references therein) and, that 753 the compressional event at c. 325 Ma reflects the far-field effects of the collision between 754 Africa (belonging to Gondwana) and southern Europe (Schulmann et al., 2014). 755

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- 1079 **Figure caption**

1078

- Figure 1: Geological map of the southwestern Murzuq basin margin: A) Location in the North 1080 African context based on Lelubre et al., 1962; Liégeois et al., 2003, 2005; Fezaa et al., 1081 2010. White rectangles indicate the circular structures discussed in the text: Mt T.= 1083 Mount Telout; I.E.= In-Ezzane; El Meh.= El Meherschema. B) Geological map of the 1084 studied area (modified after Le Caignec et al, 1957, Fezaa et al., 2010 and Geological 1085 Map of Libya, 1985, SW sheet).
- Figure 2: Detailed map of the Arrikine sill, from image analysis through Landsat 8 satellite 1086 images and from field observations and measurements: a) Names on the map concern 1087 geographical location of the two main "oueds" in this area. A color composite 1088 analysis, using TM6, TM3 and TM1 bands with contrast enhancement, highlights a 1089 NW-SE dark band corresponding to magmatism observed and sampled on the field. 1090 Contrasting spectral responses allow differentiating the Silurian clays in purple and the 1091 Devonian sandstones in brown. b) Names on the map concern geological formations. 1092 The relative homogeneity of responses within these different areas, leads us to try to 1094 specify the extension and edges of the sill. Four classes have been defined (green clays, marls and limestones; red dark rocks; brown sandstones; white alluvial 1095 1096 material). The result is a classified image by maximum likelihood method. The Cambro-Ordovician levels are not differentiated by this classification and the 1097 1098 magmatism in red is well underlined with its NW-SE orientation. However, the northern part of the red area shows a surprising lateral eastward extension. 1099 Comparison with the figure 2a rather suggests drainage of dark material by the Oued 1100 Arrikine. The paleomagnetic sampling sites are indicated with numbers by yellow 1101 squares (gabbro) and black dots (sedimentary formations). Cambro-Ordovician (ks), 1102 Silurian (s) with its two formations (Oued In Djerane and Acacus) and Devonian (d). 1103 Yellow circles correspond to injectites. 1104

1105 Figure 3: U-Pb concordia diagram.

- Figure 4: Thermal demagnetization curves for representative samples of the dark facies. 1106
- 1107 Figure 5: Typical thermomagnetic curves (normalized low field susceptibility  $K/K_0$  as a 1108 function of the temperature  $T - in °C$  for representative samples of the gray (IZ716) 1109 and dark (IZ696) facies of the gabbro.
- Figure 6: a, b): Hysteresis loops (a: sample IZ725 and b: sample IZ678, both for dark facies); 1110 Her is the remanent coercive force, Field H in Tesla. c) Day plot (Day et al. 1977) of the hysteresis ratios; PSD (pseudo-single domain) and MD (multidomain) areas for 1112 1113 magnetite.
- Figure 7: Orthogonal vector plots (filled circles: horizontal plane, crosses: vertical plane), in 1114 stratigraphic coordinates for AF (a, c and e), thermal (b, d, f) and mixed (g) treatments 1115 for samples IZ659A (a), IZ664A (b), IZ709A (c), IZ739A (d), IZ711A and B (e, f) and 1116 1117  $IZ660A(g)$ .
- Figure 8: Equal-area plot after dip correction (a) of ChRMs C and (b) of the mean-site 1118 paleomagnetic directions with associated 95% confidence zone (open circles: negative 1119 1120 inclinations).
- Figure 9: In North-West Africa coordinates, comparison of the Arrikine pole with Lower 1121 Devonian poles (see text), with the 560-210 Ma Gondwana APWP (in red 560-250 Ma 1122 1123 - Amenna et al., 2014, Henry 2015 – in orange 240-210 Ma - Domeier et al., 2012), the Africa APWP (in green 200-0 Ma - Besse and Courtillot, 2002) and in blue the 1124 430-380 Ma Gondwana APWP of Torsvick et al. (2012). Paleomagnetic poles with 1125 associated confidence zone of Mt. Leyshon Devonian dykes (Mt. LDD) in Australia 1126 (Clarck, 1996), Snowy River Volcanics (SRV) in Australia (Schmidt et al., 1987), 1127 1128 Herrada Member, Sierra Grande (HM) in Argentina (Rapalini and Vilas, 1991), Aïr intrusives (Aïr) in Niger (Hargraves et al., 1987), Tin Serririne (TS – Derder et al., 1129 2006) and Arrikine (ARK) in Algeria (this study). Confidence zones are also presented 1130 for the 250 or 500 Ma poles of the APWPs. 1131
- Figure 10: Arrikine sill representative sample AF-ALG/Dj-Site58 compared with rocks 1132 having also a high-Ti basaltic composition (Aïr, Niger (Abontorok ring-complex): 1133 Brown et al., 1989; Siberia: Khudoley et al., 2013, Virunga: Rogers et al., 1998, 1134 Hawaii: Wanless et al., 2006, Yangtse: Fan et al., 2013, SW China: Zhang et al., 1135 2013). (A) REE normalized to chondrites (Taylor and McLennan, 1985), (B) Di 58 spectrum for incompatible elements normalized to MORB (Sun, 1980 and Pearce, 1137

- 1982) compared with high-Ti basaltic composition of rocks of Siberia, Virunga, 1138 Hawaii, Yangtse and SW China (see references above) 1139
- Figure 11: Arrikine gabbro AF-ALG/Dj-Site58 spectrum for incompatible elements 1140 normalized to the Primitive Mantle (Sun and McDonough, 1989) compared with 1141 1142 Siberian high-Ti basalts (Suordakh event, Eastern margin of the Siberian craton; 1143 Khudoley et al., 2013).
- 1144

1145 Table 1: Microprobe analysis

- 1146 Table 2: K-Ar dating of the Arrikine sill.
- Table 3: U-Th-Pb laser ablation ICP-MS analyses for zircons extracted from the Arrikine 1147 1148 gabbro.
- 1149 Table 4: Mean direction: Site, number of ChRM N, Declination D and Inclination I before 1150 and after dip correction, Fisher (1953) parameters k and  $\alpha_{95}$  and VGP or paleomagnetic pole: Lat. (°S), Long (°E), with for the latter corresponding Fisher 1152 parameters K and A<sub>95</sub>.
- Table 5: Major and trace elements for the Arrikine sill measured on a Horiba-Jobin-Yvon 1153 Ultima 2 ICP-AES at the IUEM, Pôle de Spectrométrie Océan, Brest, France, by 1154 Céline Liorzou. CIPW norm and other parameters have been calculated using Norm4 1155 Excel spreadsheet from Kurt Hollocher: 1156
- (http://minerva.union.edu/hollochk/c\_petrology/norms.htm). 1157







Table 3: U-Th-Pb laser ablation ICP-MS analyses for zircons extracted from the Arrikine gabbro









24°10' N

24°05' N

24°00'N

23°55'N



ACCEPTED MANUSCRIPT

24°10' N

24°05' N

N.00<sub>°</sub>

23°55'N







































A large mafic sill has been discovered in western border of the Murzuq basin Emplacement age around 410Ma has been obtained by paleomagnetic dating Emplacement is related to reactivation of mega-shear zones at cratons boundaries Obtained K/Ar age is that of a rejuvenation attributed to aplitic fluid percolation These ages correspond to that of the Caledonian and Hercynian unconformities