

# Monazite U–Th–Pb EPMA and zircon U–Pb SIMS chronological constraints on the tectonic, metamorphic, and thermal events in the inner part of the Variscan orogen, example from the Sioule series, French Massif Central

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2	the tectonic, metamorphic and thermal events in the inner part of the Variscan
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4	
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#### 15 Abstract

- 16 In the northern Variscan French Massif Central, the Sioule metamorphic series exposes from
- 17 top to bottom the tectonic superposition of the Upper Gneiss Unit (UGU), Lower Gneiss Unit
- 18 (LGU), and Para-autochthonous Unit (PAU). The nappe stacking developed throughout two
- 19 prograde syn-metamorphic events:  $D_1$  is a top-to-the SW shearing coeval with a probable
- 20 Devonian migmatization, and  $D_2$  is a top-to-the NW shearing event. Both events were
- 21 completed before the unconformable deposition of the undeformed and unmetamorphozed
- 22 "Tufs anthracifères" formation, dated at ca 330 Ma (Late Visean). Furthermore, the UGU
- 23 experienced a high pressure metamorphism ascribed to a  $D_0$  event during which eclogite or
- 24 granulite crystallized in several parts of the UGU. Monazite U-Th-Pb, and zircon U-Pb SIMS

25	datings were carried out in order to constrain the ages of these $D_0$ , $D_1$ , and $D_2$ tectonic-
26	metamorphic events. These new geochronological results are placed in a P-T-t diagram
27	constructed for the UGU, LGU, and PAU. Monazite sampled in UGU, LGU, PAU rocks
28	yields similar 365-350 Ma ages consistent with the $D_2$ event dated in other places of the
29	French Massif Central. A zoned monazite grain from a granulitic paragneiss yields 416±15
30	Ma and 362 $\pm$ 14 Ma ages interpreted as those of the D <sub>0</sub> and D <sub>2</sub> events, respectively. Zircon
31	from the same granulitic paragneiss yields SIMS ages at 343±2 Ma, and 328±2 Ma that are
32	interpreted as recrystallization processes associated with post-thickening thermal events,
33	possibly recording the onset of orogenic collapse of the Northern Massif Central. It is worth
34	to note that neither monazite nor zircon recorded the $D_1$ event.
35	
36	Key words Variscan orogeny, monazite chemistry, monazite U-Th-Pb chemical dating,
37	zircon SIMS geochronology, granulite facies, monazite chemistry, French Massif Central
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#### 41 Introduction

42 Dating the syn-metamorphic rocks and their related ductile fabrics remains a challenge.

43 Indeed, many of the common metamorphic phases such as biotite, muscovite, feldspar, and amphibole can be dated by the  ${}^{40}$ Ar/ ${}^{39}$ Ar method, however it is well acknowledged that this 44 45 chronometer is highly sensitive to any late thermal perturbation, thus the radiometric ages 46 obtained by this method may represent cooling ages or "re-opening" ages rather than genuine 47 crystallization age of the dated minerals (see Jourdan et al 2014 for a recent review). Zircon is 48 also a common accessory phase used for U-Pb geochronology. Due to analytical 49 improvements, punctual measurements using SIMS, SHRIMP or LA-ICP-MS technique, zircon yields precise age, however as for the <sup>40</sup>Ar/<sup>39</sup>Ar method, the significance of zircon U-50 51 Pb geochronology must be carefully considered for dating metamorphic rocks since zircon 52 may crystallize or recrystallize at various stages of the rock history, including initial 53 magmatism formation stage for igneous protoliths. Monazite that may crystallize either from 54 magma or during metamorphic reactions appears as one of the most useful phase to assess the 55 timing of tectono-metamorphic events. Although separated monazite grains can be dated by 56 SIMS or LA-ICP-MS, the *in-situ* approach that preserves the textural relationships between minerals allows the deciphering of superimposed magmatic and/or metamorphic high-57 58 temperature stages. Furthermore, the size of the analytical spot (ca 20 µm) for LA-ICP MS 59 measurements generally prevents this detailed investigation as metamorphic monazite is often 60 small-sized, chemically zoned or partly dissolved. Nevertheless, though involving a non-61 isotopic technique, with a ca 5 to 15 Ma uncertainty, larger than the isotopic methods, the 62 monazite U-Th-Pb EPMA method can provide one of the best estimate of the metamorphic 63 mineral crystallization ages in polymetamorphic terranes. 64 Due to the abundance of metapelitic and felsic magmatic protoliths, the Variscan belt

appears as a good example to determine the ages of successive tectono-metamorphic events,

in polymetamorphosed and polydeformed domains, particularly when thermo-barometric
conditions preclude the use of temperature-sensitive geochronological methods such as
<sup>40</sup>Ar/<sup>39</sup>Ar. This paper aims to apply the monazite U-Th-Pb EPMA chemical, and zircon U-Pb
SIMS dating methods to the Sioule metamorphic series, in the Northern part of the Variscan
French Massif Central since in this area, the tectonic-metamorphic succession is already well
established, but radiometric constraints still rare, as presented in the following sections (e.g.
Faure et al. 1993, 2002; Schulz et al. 2001; Schulz 2009).

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- 74

#### 75 Geological outline of the Variscan French Massif Central

76 The stack of nappes

77 The French Massif Central (FMC thereafter) is an important segment of the Variscan Belt that

results of a complex interplay between two main continents, namely Gondwana, and

79 Laurussia, and several intervening microcontinents such as Avalonia, Mid-German

80 Crystalline Rise, and Armorica (e.g. Matte 2001; Cocks 2000; Faure et al. 2005; Murphy et al.

81 2011). It is now well acknowledged that the bulk architecture of the French Massif Central,

82 alike the southern part of the Massif Armoricain or the Vosges-Black Forest massif, is a stack

83 of metamorphic nappes developed diachronously from Late Silurian to Early Carboniferous at

the expense of the north Gondwana margin (Matte 1986; Ledru et al. 1989; Faure et al. 2005,

85 2009; Lardeaux et al. 2014; Fig. 1).

From bottom to top, and South to North, the following units are recognized (Figs. 1, 2). 1)

87 *The foreland basin* develops in the southernmost part of the Massif Central. It is an Early

- 88 Carboniferous (Visean-Serpukhovian) turbiditic basin that extends southward below the
- 89 coastal plain of the Mediterranean Sea. 2) The Paleozoic Fold-and-Thrust Belt consists of
- 90 Early Cambrian to Visean unmetamorphosed sedimentary rocks displaced to the South as

91 thrust sheets or kilometre-scale recumbent folds well. There, the Early Devonian (i. e. 92 Lochkovian, 416-411 Ma) unconformity argues for an Early Paleozoic tectonic event (at ca 93 420-400 Ma), which is also recorded to the North of the Massif Central in the Lower and 94 Upper Gneiss metamorphic units (cf. below). 3) The Para-autochthonous Unit that 95 overthrusts the Paleozoic Fold-and-Thrust Belt is formed by greenschist to epidote-96 amphibolite facies metapelites, metagreywackes, quartzites, and a small amount of marbles 97 and metabasites. In the northern Massif Central, the Para-autochthonous Unit crops out in 98 tectonic windows surrounded by the Lower Gneiss Unit. In the Cévennes area, some granitic 99 plutons, now transformed into orthogneiss, intruding the series, yield Cambrian to Early 100 Ordovician zircons (Bé Mézème et al. 2006). 4) The Lower Gneiss Unit (LGU) is composed 101 of metagreywackes, metapelites, and metarhyolites intruded by Cambrian to Early Ordovician 102 porphyritic alkaline granitoids transformed into augen orthogneiss during the tectonic-103 metamorphic events. The LGU underwent a Middle Devonian metamorphism leading to 104 crustal melting coeval with a ductile shearing dated in the Limousin at 375-370 Ma (Faure et 105 al. 2008) and attributed to the D<sub>1</sub> event (cf below). Moreover, the LGU also experienced a 106 medium pressure/medium temperature metamorphism characterized by a biotite-garnet-107 staurolite assemblage, dated at ca. 360-350 Ma (Costa 1989; Melleton et al. 2009), and attributed to the  $D_2$  event. 5) The Upper Gneiss Unit (UGU) forms the overlying nappe whose 108 109 the main protoliths are metapelites and metagrauwackes. This unit contains high-pressure 110 rocks, such as eclogites or high pressure (HP) granulitic orthogneiss that may locally reach the 111 coesite-eclogite facies (Santallier 1981; Pin and Vielzeuf 1988; Lardeaux et al. 2001; Berger 112 et al., 2010). The upper part of the UGU consists of Devonian migmatites, yielding zircon U-113 Pb ages at *ca* 385-380 Ma, attributed to the  $D_1$  event, and formed by the partial melting of 114 pelitic and quartzo-feldspathic rocks (Duthou et al. 1994; Faure et al. 2008). These rocks 115 contain amphibolite blocks derived from retrogressed eclogites that did not experience partial

116 melting. The HP metamorphism is dated at ca 420-400 Ma by U-Pb and Sm-Nd methods in 117 several locations (Pin and Peucat 1986; Paquette et al. 1995; Faure et al. 2005). 118 The five units described above represent the classical nappe pile of the FMC in which the 119 metamorphism increases from bottom to top. Furthermore, in the northern part of the French 120 Massif Central, the undeformed and unmetamorphozed "Tuffs anthracifères" series, which 121 consists of terrigenous rocks (sandstone, shales, conglomerates), coal measures, and felsic 122 magmatic rocks (dacites and rhyolites), is dated as Late Visean (ca 330 Ma, Bruguier et al. 123 1998). These rocks unconformably cover Late Devonian-Early Carboniferous plutonic or 124 metamorphic rocks of the UGU and LGU. The "Tuffs anthracifères" series provides a major 125 time constraint for the evolution of the French Massif Central. As the "Tuffs anthracifères" 126 series is coeval with the development of the Fold-and-Thrust Belt, it supports the north-south 127 diachronism in the deformation at the scale of the entire massif.

128

129 The polyphase tectono-metamorphic evolution

130 The Variscan geodynamic evolution of the French Massif Central remains disputed. Some 131 authors (e.g. Matte 1986; Ledru et al. 1989) consider that the formation of the FMC results of 132 a continuous north-directed oceanic then continental subduction from early Silurian to early 133 Carboniferous. In contrast, other authors (e.g. Pin 1990; Faure et al. 1997, 2008, 2009) argue 134 for a polycyclic evolution. The Early Silurian to Early Devonian events correspond to the Eo-135 Variscan cycle due the collision of the Armorica microcontinent with the north Gondwana 136 margin. From Middle Devonian to Early Carboniferous, the Variscan belt develops due to the 137 southward subduction of the Rheic Ocean below Gondwana followed by the collision of 138 Gondwana with Laurussia. In spite of differences between the two geodynamic scenarii, most 139 of authors agree on the main tectonic, metamorphic, and magmatic events. Six tectono-140 metamorphic events are recognized (Faure et al. 2009).

141 The earliest event, D<sub>0</sub>, corresponds to the high to ultra high-pressure metamorphism 142 recorded in the eclogite facies rocks of the UGU. Most of the HP rocks derive from mafic 143 protoliths, but also HP relics are also found in orthogneiss are locally found. In the Lyonnais 144 area, the UGU eclogites record pressure and temperature conditions of 1.8-2 GPa and 650-145 750°C, respectively (Lardeaux et al. 2001). On the basis of the HP metamorphism, the D<sub>0</sub> 146 event took place around 415 Ma (Pin and Peucat 1986).

147 The Early to Middle Devonian, tectonic-metamorphic event  $D_1$ , is coeval with the 148 crustal melting of the metapelites and orthogneiss of the UGU and LGU, whereas mafic rocks 149 are preserved as amphibolite restitic blocks in migmatites. The migmatisation is dated as 150 Middle Devonian (385-375 Ma) in the Limousin or Lyonnais areas (Duthou et al. 1994, Faure 151 et al. 2008 and enclosed references). P-T estimates from garnet-plagioclase and garnet-biotite 152 pairs in migmatites, indicate metamorphic conditions of  $0.7\pm0.05$  GPa and  $700\pm50^{\circ}C$ respectively (Mercier et al. 1991; Roig and Faure 2000). The flat-lying foliation exhibits a 153 154 NE-SW striking (N30 to N60E) stretching lineation marked by fibrolitic sillimanite or biotite in migmatites, and biotite or amphibole in amphibolites. Shear criteria indicate a top-to-the-155 156 SW displacement (Ledru et al. 1989; Faure et al. 1997; Roig and Faure 2000). Amphibolite in the northern Massif Central yields biotite and hornblende <sup>40</sup>Ar/<sup>39</sup>Ar cooling ages ranging from 157 158 390 Ma to 381Ma (Costa and Maluski 1988; Boutin and Montigny 1993). In northeastern 159 Massif Central, or Morvan area, Middle Devonian unmetamorphosed and undeformed 160 sedimentary rocks unconformably overlying migmatites with eclogite relics suggest that, at 161 least in the northern part of the Massif Central, the HP rocks were already exhumed before the Middle Devonian (Faure et al. 1997). 162

In the LGU, UGU, the dominant fabric is a NW-SE trending stretching lineation
developed either on flat-lying or steeply dipping foliations, which is attributed to the D<sub>2</sub> event
(Faure et al. 2005, 2008). Thermo-barometric constraints are well established in the Limousin

166 and Rouergue areas (Duguet et al. 2006; Bellot and Roig 2007). In the UGU, the syn-D<sub>2</sub> 167 minerals, crystallized along the NW-SE lineation, indicate 0.7-1 GPa and 600-700°C 168 metamorphic conditions. Paragneiss belonging to the LGU yield nearly similar conditions of 169 0.8-1 GPa and 550-600°C. In the LGU, amphibolites that never experienced the high-pressure 170 event indicate 0.8-1 GPa and 700-800°C metamorphic conditions (Santallier 1981). The D<sub>2</sub>, 171 event, characterised by a top-to-the-NW ductile shearing, is widely documented from the 172 southeast Massif Central up to the Southern part of the Massif Armoricain. This deformation 173 is stratigraphically dated as pre-Early Visean (i.e. older than 345 Ma) by the unconformity 174 recognized in the Brévenne area (e.g. Leloix et al., 1999 and enclosed references, Fig. 1). The age of the syn-D<sub>2</sub> MP/MT metamorphism is constrained by  ${}^{40}$ Ar/ ${}^{39}$ Ar method on biotite, 175 176 muscovite and amphibole, and by EPMA on monazite between 360 and 350 Ma (Costa 1989;

177 Leloix et al. 1999; Melleton et al. 2009).

178 The syn-metamorphic D<sub>3</sub> deformation is recognized only in the Para-autochthonous 179 Unit of the southern part of the Massif Central that experienced a first deformation at ca 340-180 335 Ma (Caron 1994; Faure et al. 2005). Further south, south-verging recumbent folds and 181 thrusts of the Paleozoic series dated of Late Visean-Early Namurian (ca 325 Ma) by the syn-182 orogenic sediments of the foreland basin, are also related to the  $D_3$  event. This progressive 183 southward younging of the nappe tectonics is coeval with the development of a flat-lying 184 foliation and a N-S to NE-SW striking stretching lineation. In contrast to the southern part of 185 the Massif Central, in its northern part, the Late Visean deformation develops under brittle 186 conditions. It is represented by a NW-SE stretching, related to the emplacement of NE-SW 187 striking dykes of basalt, dacite, and microgranite in the Tuff Anthracifères series. This 188 deformation is interpreted as the onset of the syn-orogenic extension (Faure 1995). 189 Ductile normal faults, such as Argentat or Nantiat faults, coeval with Serpukhovian-190 Bashkirian (or Namurian-Westphalian in W. European stratigraphy, *i.e.* 325-315 Ma)

191 synkinematic granite emplacement, argue for an extensional tectonic phase named the  $D_4$ 192 event (Faure 1995). As shown by petrofabric and AMS studies, most of the Late 193 Carboniferous leucogranite and porphyritic monzogranite plutons and their metamorphic 194 aureoles exhibit a well-defined NW-SE trending mineral or stretching lineation (Talbot et al. 195 2005; Joly et al. 2007). The tectonic-plutonic  $D_4$  event, widespread in the entire Massif 196 Central, is interpreted as a syn-orogenic extensional process since at this time, compression 197 was still active in the northern and southern outer zones of the Variscan belt, such as Ardenne 198 and Montagne Noire-Pyrénées, respectively.

199The last phase in the structural evolution of the French Massif Central is a Late200Carboniferous (Kasimovian-Ghzelian, or Stephanian)  $D_5$  late-orogenic extensional event that201is distinguished from the previous one by the NNE-SSW strike of the maximum stretching202direction. Intramontane coal basins develop either as half-grabens or left-lateral pull-aparts203depending on the strike and kinematics of the bounding fault that controls the basin opening204and sedimentary infill.

205

#### 206 **The Sioule metamorphic series**

207 The bulk architecture

208 Located in the northern part of the French Massif Central, the Sioule metamorphic series is 209 bound to the west by the Late Carboniferous Sillon Houiller sinistral wrench fault, and to the 210 east by the Oligocene Ebreuil and Limagne grabens (Figs. 1, 2). The Tréban Massif and the S<sup>te</sup>-Christine wrench fault are the northern and southern limits of the Sioule metamorphic 211 212 series, respectively (Fig. 2). The metamorphic series is intruded by two granitic plutons. The 213 Pouzol-Servant porphyritic microgranite is a biotite granite dated at *ca* 330 Ma (Pin, 1991). 214 The Echassières leucogranite is a small, differentiated, peraluminous pluton dated at ca 310 215 Ma (Duthou and Pin 1987; Cheilletz et al. 1992). The Sioule series is a classic example of an 216 inverted metamorphic sequence in the Massif Central (Grolier 1971). Three lithological and

217 structural units have been described (Richard 1938; Grolier 1971; Faure et al. 1993). From top 218 to bottom, they are: a) an upper unit composed of biotite-sillimanite gneiss, and biotite-219 sillimanite± cordierite migmatite developed at the expense of the underlying gneiss that 220 enclose a few meter-sized boudins of kyanite-garnet-plagioclase high-pressure granulitic 221 paragneiss partly retrogressed under amphibolite facies conditions (Ravier and Chenevoy 222 1979). This unit is correlated with the Upper Gneiss Unit (UGU) of the French Massif Central 223 (cf section 2); b) an intermediate unit formed by biotite-muscovite-sillimanite-K feldspar-224 garnet paragneiss corresponding to the Lower Gneiss Unit (LGU), and c) a biotite-staurolite-225 garnet micaschist that crops out around the Echassières pluton. This unit, which is structurally 226 below the two micas paragneiss of the LGU, lacks of orthogneiss and anatectic melts, it is 227 therefore correlated with the Para-autochthonous Unit (PAU).

228

229 Structural outline of the Sioule metamorphic series

230 In the study area, the observed structures, and the related kinematics account well for a multistage tectonic evolution. In agreement with previous works (Grolier 1971; Feybesse and 231 232 Teygey 1987; Faure et al. 1993; Schulz et al. 2001; Schulz 2009), the foliation is folded by 233 NNW-SSE trending km-scale upright synform and antiform (Fig.2). A WNW-ESE striking 234 stretching and mineral lineation is well developed in the foliation plane of the PAU, LGU, and 235 UGU. Microscope-scale shear criteria, such as sigmoidal micas, asymmetric pressure shadows 236 around feldspar or garnet, and S-shaped inclusion trails in garnet consistently indicate a top-237 to-the WNW shearing (Faure et al. 1993; Schulz 2009). These microstructural features are 238 relevant to the D<sub>2</sub> event of the Massif Central.

In the UGU, in addition to the main NW-SE D<sub>2</sub> deformation, a NNE-SSW striking
mineral and stretching lineation is also recognized (Grolier 1971; Feybesse and Teygey 1987;
Faure et al. 1993). This deformation, characterized by elongated biotite-sillimanite

242 aggregates, stretched K-feldspar, and axes of intrafolial folds parallel to the stretching 243 lineation, is associated with top-to-the SSW kinematic indicators. A Barrovian-type 244 metamorphism with biotite-sillimanite-K-feldspar assemblage is coeval with this deformation 245 event that appears less developed, and older, than the NW-SE D<sub>2</sub> one. In the upper part of the 246 UGU, the biotite-sillimanite±cordierite migmatite exhibits also the NNE-SSW mineral and 247 stretching lineation, with similar top-to-the-SW kinematics as those observed in the 248 underlying biotite-sillimanite gneiss. By correlation with the general tectonic-metamorphic 249 pattern of the FMC, this early phase is correlated with the D<sub>1</sub> event, thus a Middle Devonian 250 age, dated from 385 to 375 Ma is suggested (Faure et al. 2009) but not documented yet. 251 Furthermore, top-to-the-WNW and top-to-the-ESE shearing, characterized by shear 252 bands cutting through the regional foliation, sericite pressure shadows upon cataclazed 253 staurolite or garnet porphyroclasts, develop around the Echassières pluton. These kinematic 254 indicators, coeval with greenschist facies metamorphic conditions, are related to the 255 emplacement of the Echassière leucogranite pluton in an extensional setting. Thus this 256 deformation phase corresponds to the D<sub>4</sub> event (Faure et al. 2009). 257 In contrast with other areas of the French Massif Central, mafic eclogites formed 258 during the earliest D<sub>0</sub> event are not recognized in the Sioule area. Nevertheless, kyanite-259 garnet-plagioclase-rutile high-pressure granulitic gneiss, garnet-pyroxenite, and rare 260 serpentinite boudins are mapped in the UGU (Grolier 1971; Ravier and Chenevoy 1979). 261 These rocks argue for an early HP event probably equivalent to the  $D_0$  one. Moreover, in 262 paragneiss, as shown by the symplectitic texture, these HP garnet and kyanite grains are 263 surrounded by a biotite-sillimanite or muscovite assemblage representative of an amphibolite 264 facies retrogression developed during the  $D_1$  or  $D_2$  events (Fig. 3). As in most areas of the 265 Massif Central, the planar and linear fabrics related to the  $D_0$  event have been erased by 266 subsequent metamorphic recrystallization or deformation events. But since syn-D<sub>0</sub> minerals,

such as kyanite, core of garnet, and possibly monazite are preserved, the rocks may still besuitable to yield the age of the HP event.

269 Available time constraints in the Sioule metamorphic series are currently limited 270 (Faure et al., 2002; Schulz, 2009). The Late Visean Pouzol-Servant microgranite and the 271 Bashkirian-Moscovian Echassières leucogranite intrude the already metamorphosed and 272 ductilely deformed series, and thus provide the upper time limit for the  $D_0$ ,  $D_1$ , and  $D_2$ tectono-metamorphic events. An  ${}^{40}$ Ar/ ${}^{39}$ Ar survey has been carried out in the metamorphic 273 274 units of the Sioule series (Faure et al. 2002). All the minerals, whatever the rock type or the 275 tectonic unit, yielded similar ca 335-330 Ma ages that were interpreted as the Late Visean thermal overprint since these ages are similar, within the errors, to the age of the "Tuffs 276 277 Anthracifères" event. Thus, the ages of the different pre-Late Visean tectono-metamorphic 278 events in the Sioule series remain presently unknown.

279

#### 280 Sampling and petrological description

281 Twenty-four samples representative of the different metamorphic assemblages cropping out in 282 the Sioule series have been petrologically studied in order to bring the time constraints on the 283 metamorphic evolution of this series. After detailed microscopic observations, five samples 284 were selected due to their suitable parageneses and textures to ensure the presence of 285 accessory minerals such as monazite and zircon developed during the D<sub>1</sub> and D<sub>2</sub> tectono-286 metamorphic events. The sample location in the Sioule nappe pile, and the GPS position, are 287 provided in Figure 2 and Table 1, respectively. The PAU is represented by a staurolite-garnet 288 micaschist (SO3) located to the East of the Echassières pluton. The LGU is represented by a 289 two-mica paragneiss (SO17) to the SE of the same pluton. The UGU is represented by a 290 biotite-sillimanite gneiss (SO7), a migmatitic gneiss (SO11), and a biotite-kyanite-garnet 291 paragneiss (SO15), located on both limbs of the antiform. The microstructural descriptions

herein refer to thin sections cut parallel to the stretching lineation (X) and perpendicular to thefoliation plane (Z), i.e. in the XZ plane of the finite strain ellipsoid.

294 Sample SO3, belonging to the Para-autochthonous unit, consists of quartz, feldspar, 295 biotite, muscovite, garnet, staurolite, sericite, and chlorite. In the field, structural observations 296 show a low-angle ESE plunging foliation, and a N105E to N115E striking mineral lineation 297 (Fig. 3a2). The foliation cut by shear bands, and sigmoidal quartz veins indicate a top-to-the-298 SE sense of shear (Fig. 3a1). Under the microscope, cataclazed garnet and staurolite 299 surrounded by an asymmetric mantle of sericite developed during retrogression of the main 300 assemblage indicate the same sense of shear (Fig 4a). Furthermore, garnet exhibits sigmoidal 301 inclusions of quartz and opaque minerals indicating an earlier foliation. The asymmetry of 302 these inclusions indicates a synkinematic crystallisation of these porphyroblasts during a 303 noncoaxial deformation that is not in agreement with the top-to-the-SE sense of shear, but 304 instead argues for a top-to-WNW sense of shear developed during the D<sub>2</sub> event. According to 305 Schulz et al (2001), the thermobarometry from the PAU micaschist indicates a prograde P-T 306 evolution with increasing temperatures from 450°C to 600°C under slightly increasing 307 pressure from 0.7 to 0.8 GPa, followed by a P and T decrease indicated by the formation of 308 retrogressive sericite during the decompression. Scan Electron Microscope (SEM) 309 backscattered electron (BSE) observation of sample SO3 reveals the presence of 50 to 100 µm 310 sized, homogeneous, monazite grains included into biotite or crystallized along grain 311 boundaries.

Sample SO17 is a two-mica paragneiss presenting traces of incipient migmatization belonging to the LGU with a segregation of thick quartz-feldspar and thin biotite and sillimanite layers. The flat lying foliation contains a NW-SE striking mineral and stretching lineation related to the D<sub>2</sub> event. The mineral assemblage consists of quartz, feldspar, biotite, muscovite, chlorite, and accessory minerals including sillimanite, Fe-Ti-oxides, and monazite. Muscovite often presents a clear secondary character, growing without preferred orientation while biotite is partially altered into chlorite in line with a late thermal overprint (Faure et al. 2002). Biotite contains large subeuhedral monazite grains of *ca* 80-120  $\mu$ m size. Monazite also occurs as *ca* 80-100 $\mu$ m euhedral inclusions in biotite or quartz. Despite the existence of these two occurrences, in SEM BSE imaging, the monazite grains exhibit homogeneous internal textures without zoning.

323 Sample SO7 is a biotite-sillimanite migmatitic paragneiss belonging to the UGU 324 collected near Moulin du Bouchet (Fig. 2). This rock is composed of quartz, feldspar, garnet, 325 biotite, muscovite, rare staurolite and fibrolitic sillimanite. Field observations of coarse-326 grained quartz-feldspar leucosome parallel to the foliation indicate that this rock underwent a 327 limited partial melting. The biotite-sillimanite assemblage defines a pervasive foliation and a 328 N50E striking mineral and stretching lineation that can be assigned to the regional  $D_1$ 329 deformation. However, as already pointed out by Schulz (2009), the subeuhedral shape of the 330 garnet argues for a static growth after the development of the D1 structures. According to Schulz et al. (2001), the biotite-sillimanite gneiss of Moulin de Bouchet, which is located 331 332 close to our sample SO7, underwent a prograde metamorphic evolution at low pressure (0.4-333 0.6 GPa) and high temperature (650°C-700°C). Thus, this biotite-sillimanite paragneiss might 334 have also experienced the  $D_2$  event, although typical  $D_2$  NW-SE stretching lineation is not 335 observed. In this rock, all the analyzed monazite grains, observed in SEM BSE mode, are 336 homogeneous inclusions in biotite (Fig. 4b).

*Sample SO11*, collected at Pont de Menat (Fig. 2) is a migmatitic paragneiss belonging
to the upper part of the UGU. This rock is composed of alternations of coarse-grained quartzfeldspar leucosome and fine-grained biotite-fibrolitic sillimanite-garnet melanosome. Field
observation shows the occurrence of a pervasive deformation characterized by a flat-lying
foliation and a N120E trending mineral and stretching lineation assigned to the D<sub>2</sub> event (Fig.

3 c). All the analyzed monazite grains observed in SEM BSE mode are homogeneous
inclusions in biotite (Fig. 4c).

*Sample SO15* is a kyanite-garnet ± rutile paragneiss coming from Saugères (Fig. 2).
This rock is composed of biotite, feldspar, quartz, garnet, and kyanite. Garnet and kyanite are
1-5 mm sized clasts included in a well-foliated granoblastic matrix composed of quartz,
feldspar, and small biotite flakes (Fig. 3d). The kyanite-garnet assemblage that formed in
high-pressure granulite facies underwent an amphibolite facies retrogression as shown by
biotite overgrowths around garnet porphyroclasts (Ravier and Chenevoy 1979; Fig. 4d).
Monazite inclusions in garnet and in biotite exhibit a core-rim chemical zoning.

#### 352 Monazite EPMA chemical dating

353 Analytical method and strategy

354 Due to its high U and Th contents, and negligible initial Pb content (Parrish 1990), it is 355 now well acknowledged that a crystallization (or recrystallization) age can be determined 356 from monazite on the basis of its chemical composition in U, Th, and Pb determined by 357 electron probe microanalysis (EPMA). Such a simple and fast technique has been successfully 358 applied to several plutonic or metamorphic rocks (e.g. Suzuki and Adachi, 1991; Montel et 359 al., 1996; Cocherie et al., 1998, 2005; Cocherie and Albarède, 2001; Schulz et al., 2009; Faure 360 et al., 2010, and enclosed references). Monazite is a common accessory mineral in 361 metamorphic rocks, especially in metapelites. It crystallizes in a wide range of P-T conditions, 362 from greenschist facies (Cabella et al. 2001; Rasmussen et al. 2001) to amphibolite (Smith 363 and Barreiro 1990; Kohn and Malloy 2004) and granulite facies (Bé Mézème et al. 2006; Mc 364 Farlane and Frost 2009) in rocks metamorphosed under Barrovian P-T gradient during 365 prograde or retrograde paths. Previous studies have shown also that monazite can crystallize 366 in blueschist (Janots et al. 2006) or even eclogite facies (Nagy et al. 2002). Consequently,

analyzed monazite grains must be linked with petrological studies to delimit their P-T
crystallization domain. The chemical composition of monazite is an important parameter in
order to discuss the radiometric ages.

370 In situ chemical U-Th-Pb dating of monazite has been developed over the past fifteen 371 years allowed by technological progress performed on electron microprobe analyses (EPMA; 372 Suzuki and Adachi 1991; Montel et al. 1996; Cocherie et al. 1998, 2005). The method uses 373 the radiochronometer between U and Th parent and Pb radiogenic elements, considering the 374 restricted lead diffusion in monazite lattice below 900°C (Cocherie et al. 1998). The 375 determination of U, Th and Pb contents can be performed directly by EPMA in situ analyses 376 on polished thin section after textural optical microscope observation of crystallisation-377 deformation relationships between the constitutive minerals, and monazite internal structure 378 determination using a SEM in BSE mode. This approach provides petrological constraints on 379 the analyzed grains and thus allows us to discuss the significance of the calculated ages in the 380 crystallisation-deformation history of the rock.

381 SEM BSE was used to define heterogeneous chemical compositional domains of 382 monazite. In metamorphic rocks, contrasted chemical domains can be explained by the 383 superimposition of metamorphic events that record a polyphase evolution during orogenic 384 processes. Theoretical procedure to reduce the data and to calculate the average age from 385 individual spot analyses is given by Cocherie and Albarède (2001), Cocherie et al. (2005), and 386 Cocherie and Legendre (2007). The analysis of monazite grains directly from polished thin 387 sections was performed by a Cameca SX 50 EPMA co-operated by BRGM-CNRS-Orléans 388 University. An accelerating voltage of 20 kV, and a beam current of 100 nA were chosen as 389 operating conditions. Synthetic phosphates, glasses and natural minerals were used as 390 standards. The counting times (2/3 on peak and 1/3 on background) varies from 20s for Si, P, 391 Ca, Nd, Gd, Y, Th, Ce and La, to 100s for U and 120s for Pb. Considering such parameters,

392 the detection limit for Th, U and Pb is 150 ppm (Cocherie and Albarede, 2001). X-ray 393 element maps were also recorded with an accelerating voltage of 20 kV and beam current of 394 100 nA, using the wavelength-dispersive spectrometers. Details on the analytical procedure are the same as those used in Cocherie et al. (2005) and Faure et al. (2008, 2010). Age 395 396 calculations were done using the Isoplot/Ex (2.49) program of Ludwig (2001) and a Microsoft 397 Excel add-in program for determining U-Th-Pb ages from EPMA measurements (Pommier et 398 al. 2002). The  $2\sigma$  errors given on individual ages depend on U, Th, and Pb contents, and are 399 calculated by propagating the uncertainties of these elements (at 95% confidence level). 400 Because the mean age of an homogeneous age population is directly drived from the isochron 401 plot Th/Pb vs U/Pb, only the uncertainty on these two parameters (Th/Pb and U/Pb) are 402 required to calculate the uncertainty on the mean age.

The strategy followed for this study was to derive time constraints for the monazite included into the foliation forming biotite or in contact with it, in order to settle the crystallisation time of the main foliation. Consequently, we concentrated on subeuhedral to euhedral monazite grains. We focused on the dating of well-defined chemical domains so as not to shuffle different heterogeneous domains that would yield different crystallization periods.

409

410 Results

*Chemical composition of monazite grains*. SEM BSE images reveal chemically
homogeneous monazite grains for the four samples SO3, SO7, SO11, and SO17. Conversely,
the SO15 kyanite-garnet granulitic paragneiss sample presents zoned monazite grains. This
zonation corresponds to a core/rim zonation as described by Bé Mézème et al. (2006), mostly
due to the spatial distribution of U, Th and Pb. The EPMA analysis allows us to quantitatively
characterize the chemical composition of the dated grains. The average U/Pb and Th/Pb ratio

417 values, details concerning the number of spot analyses, and analyzed grains are provided in 418 Table 1. Representative monazite chemical compositions with uncertainties for samples SO3, 419 SO7, SO11, SO15, and SO17 are given in Table 2. For the granulitic gneiss (sample SO15), 420 core and rim chemical data have been separated. Monazite chemical data for each samples are 421 plotted in the Monazite  $(2REEPO_4)$  - Cheralite  $(CaTh(PO_4)_2)$  - Huttonite  $(2ThSiO_4)$  diagram 422 (Linthout 2007; Fig. 5a), in order to show the relative substitution between end-member 423 monazite phase and the cheralite or huttonite components (Fig. 5a). The cheralite substitution 424 is always low, as already observed for various metamorphic environments (e.g. Franz et al. 425 1996; Cabella et al. 2001), and huttonite substitution is absent or negligible. In samples SO3, 426 SO7, SO11 and SO17, monazite grains appear as homogeneous domains in BSE images, and 427 the analytical data scatter along the cheralite-monazite axis showing clearly a common 428 substitution. In sample SO15, the analyzed monazite grain is composed of two domains in the 429 BSE images, with a light (i.e. dense) core and a dark (i.e. light) rim. Data plotted in the 430 Monazite  $(2REEPO_4)$  - Cheralite  $(CaTh(PO_4)_2)$  - Huttonite  $(2ThSiO_4)$  diagram (Fig. 5a) show 431 a relatively high Ce-monazite component into the core of the grain (white diamond) and a 432 cheralite enriched rim (black diamond), corresponding to two different chemical domains. The (Th+U+Si+Pb) = f(REE+P) plot, modified from Franz et al. (1996), confirms the spread 433 434 of the analytical data for samples SO3, SO7, and SO11 along the CaThREE<sub>2</sub> axis 435 corresponding to the cheralite substitution (Fig. 5b). Monazite chemical data from the 436 staurolite micaschist SO3 (Fig. 5b) indicate a limited huttonite substitution pointed out by 437 small vertical variation in Th+U+Si+Pb. Chemical data of monazite from biotite-sillimanite 438 gneiss SO7 and SO11 show a more significant huttonite substitution than for SO3. The core 439 and the rim of dated monazite grain from sample SO15 are characterized by two distinct sets 440 of chemical compositions. The rim is richer in cheralite component than the core, even if 441 huttonite substitution in each domain seems to be similar (Fig. 5b). X-ray maps depicting the

semiquantitative distribution of Th, Ca, and U confirm the chemical differences between the core and the rim of SO15 grain (Fig. 5d). These maps show that the monazite rim of monazite is enriched in Th and Ca compared to the core. The U content is relatively equivalent in the core and rim, except for an enriched fringe in the rim. This domain was not analyzed because of holes on the polished surface. Despite the presence of several small-sized domains indicated by the Ca and Th contents, the entire core is homogeneous in Th and U composition.

449

*U-Th-Pb chemical dating.* In this section, the ages of the analyzed monazite grains
from samples SO3, SO 17, SO7, SO11, SO15 are presented following the tectonic order from
bottom to top (Fig. 2).

453 Staurolite micaschist SO3. Data points are highly scattered on the Th/Pb vs. U/Pb 454 diagram, leading to a small error envelope. The intercept ages are similar within errors, since 455 the U-Pb age (intercept with U/Pb axis), and the Th-Pb age (intercept with Th/Pb axis) are at 456 379 +54/-62 Ma, and 357+26/-23 Ma, respectively. The calculated MSWD of 1.3 indicates 457 that the population of 73 analyses is statistically homogeneous within the analytical errors 458 (Wendt and Carl 1991). As a consequence, it is possible to calculate the mean age at the 459 centroid where the precision is the best within the error envelope. Thus, the monazite grains 460 yield a mean age of  $363 \pm 8$  Ma (at  $2\sigma$  confidence level; Fig. 6a).

Two-mica gneiss (SO17). Data points are weakly scattered on the Th/Pb vs. U/Pb diagram, leading to a large error envelope. The intercept ages are similar within errors, since the intercept U-Pb age and the intercept Th-Pb age are at 399 +44/-53 Ma, and 328+31/-26 Ma, respectively. The calculated MSWD of 0.63 indicates that the population of 38 analyses is statistically homogeneous within the analytical errors (Wendt and Carl 1991). As a consequence, it is possible to calculate the mean age at the centroid where the precision is the 467 best within the error envelope. Thus, the monazite grains yield a mean age of  $351 \pm 5$  Ma (at 468  $2\sigma$  confidence level; Fig. 6b).

469 Biotite-sillimanite gneiss (SO7). Data points are relatively scattered on the Th/Pb vs. 470 U/Pb diagram, leading to a small error envelope. The intercept ages are similar within errors, 471 since the intercept U-Pb age and the intercept Th-Pb age are at 331+39/-46 Ma, and 371+33/-472 28 Ma, respectively. The calculated MSWD of 1.19 indicates that the population of 73 473 analyses is statistically homogeneous within the analytical errors (Wendt and Carl 1991) and 474 as a consequence, it is possible to calculate the mean age at the centroid. Thus, the monazite 475 grains yield a mean age of  $355 \pm 7$  Ma (at  $2\sigma$  confidence level; Fig. 6c). 476 Migmatitic gneiss (SO11) of Pont de Menat. Data points on the Th/Pb vs. U/Pb 477 diagram are less scattered than for previous sample, which may lead to a larger error envelop. 478 However, the large number of analyses (n = 174) decreases the envelop size. The intercept 479 ages are similar within errors, since the intercept U-Pb age and the intercept Th-Pb age are at 480 342+45/-51 Ma, and 373+24/-21 Ma, respectively. The calculated MSWD of 0.98 indicates 481 that the population of 174 analyses is statistically homogeneous within the analytical errors 482 (Wendt and Carl 1991). As a consequence, it is possible to calculate the mean age at the 483 centroid. Thus, the monazite grains yield a mean age of  $363 \pm 4$  Ma (at  $2\sigma$  confidence level; 484 Fig. 6d).

Kyanite-garnet granulitic paragneiss (SO15) of Saugères. Monazite in sample SO15 exhibits a core-rim zonation of the grain, and in consequence, EPMA data were processed separately for the different domains in order to calculate two isochron ages. 47 and 21 analyses were performed in the core and rim of a single grain, respectively. The 47 analyses from the core on the Th/Pb vs. U/Pb diagram are clustered, leading to a large error envelope. The intercept ages are similar within errors, since the intercept U-Pb age and the intercept Th-Pb age are at 517+281/-322 Ma, and 390+81/-57 Ma, respectively. The calculated MSWD of

492	1.15 indicates that the population of the 47 analyses is statistically homogeneous within the
493	analytical errors (Wendt and Carl 1991). As a consequence, it is possible to calculate a mean
494	age at the centroid. Thus, the core of this monazite grain yields a mean age of $416 \pm 15$ Ma (at
495	$2\sigma$ confidence level; Fig. 6e).
496	Moreover, the 21 analyses performed from the monazite rim are well-scattered,
497	leading to a small error envelope. The intercept ages are similar within errors, since the
498	intercept U-Pb age and the intercept Th-Pb age are at 423+74/-88 Ma, and 338+36/-30 Ma,
499	respectively. The calculated MSWD of 0.93 indicates that the population of 21 analyses is
500	statistically homogeneous within the analytical errors (Wendt and Carl 1991). As a
501	consequence, it is possible to calculate the mean age at the centroid where the precision is the
502	best within the error envelope. Thus, the monazite grains yield a mean age of $362 \pm 14$ Ma (at
503	$2\sigma$ confidence level; Fig. 6f).

504

#### 505 Zircon U-Pb SIMS dating

506 Analytical procedure

507 Zircon concentrates were separated from a ca 5 kg of the granulitic paragneiss sample SO15 508 using standard density and magnetic separation techniques. Zircon grains, together with 509 standard zircons TEMORA 2, 91500, and an in-house standard zircon Qinghu, were mounted 510 in an epoxy mount that was then ground and polished to section the crystals in half for 511 analysis. Zircons were imaged with transmitted and reflected light photomicrographs as well 512 as cathodoluminescence (CL) to reveal their internal structures, and the mount was vacuum-513 coated with high-purity gold prior to SIMS analyses. Measurements of U, Th and Pb isotopes 514 were conducted using a Cameca IMS 1280 SIMS at the Institute of Geology and Geophysics, 515 Chinese Academy of Sciences in Beijing. Analytical procedures are similar to those reported 516 by Li et al. (2009). The primary  $O_2^-$  ion beam spot is about  $20 \times 30 \ \mu m$  in size. Positive

secondary ions were extracted with a 10 kV potential. In the secondary ion beam optics, a 60 517 518 eV energy window was used, together with a mass resolution of *ca* 5400 (at 10% peak height), to separate Pb<sup>+</sup> peaks from isobaric interferences. A single electron multiplier was 519 520 used in ion-counting mode to measure secondary ion beam intensities by peak jumping mode. 521 Analyses of the standard zircon TEMORA 2 were interspersed with unknown grains. Each 522 measurement consists of 7 cycles. Pb/U calibration was performed relative to zircon standard TEMORA 2 ( $^{206}$ Pb/ $^{238}$ U age = 417 Ma, Black et al. 2004); U and Th concentrations were 523 524 calibrated against zircon standard 91500 (Th = 29 ppm, and U = 81 ppm, Wiedenbeck and 525 Allen 1995). Measured compositions were corrected for common Pb using non-radiogenic <sup>204</sup>Pb, and an average of crustal composition (Stacey and Kramers 1975) was used for the 526 527 common Pb. Data reduction was carried out using the Isoplot/Ex v. 2.49 program (Ludwig 528 2001). In order to monitor the external uncertainties of SIMS U-Pb zircon measurements, an 529 in-house standard zircon Qinghu was alternately analyzed as an unknown together with other 530 unknowns. Twenty measurements on Qinghu zircon (Table 3) yield a Concordia age of  $160 \pm$ 531 1 Ma (2SE, MSWD of concordance = 0.92), which is in good agreement within errors with 532 the recommended U-Pb age of  $159.5 \pm 0.2$  Ma (Li et al. 2009). Zircon U-Pb age data are 533 presented in Table 3.

534

535 Results

536 Fifty-five zircon grains from the SO 15 granulitic paragneiss have been analyzed (Table 3).

537 All but four zircons are concordant within analytical uncertainties (discordance degree <4%).

538 The dated zircons can be divided into two groups in terms of their morphological feature, ages

- and Th/U ratio. In group 1, zircons are mostly sub-rounded to multifaceted grains and yield
- ages between 324 and 349 Ma. They are characterized by low Th/U ratio (<0.1) and faint CL
- 541 luminescence (Table 3, Fig. 7), similar to metamorphic zircons (Corfu et al. 2003). Contrarily,

Group 2 zircons are dated between 368 Ma and 1.99 Ga (Table 3). They are mostly oval 542 543 shaped grains showing concentric zoning under CL (Fig. 7), and relatively high Th/U ratio 544 (mostly >0.1), similar to the magmatic zircons (Corfu et al. 2003). Among them, four zircons (spots @06, 26, 27 and 37) are highly discordant, with <sup>207</sup>Pb/<sup>206</sup>Pb ages between 1629 and 545 546 1994 Ma. On the probabilistic histogram of U-Pb ages for 51 concordant analyses (Fig 8), 547 Group 1 zircons form one major peak at 342 Ma (n = 24) and a subordinate peak at 327 Ma (n548 = 6), corresponding to Concordia age of  $343\pm2$  Ma and  $328\pm4$  Ma, respectively, on a U-Pb 549 Concordia plot (Fig. 9). Thus, Group 1 zircons are most likely metamorphic in origin. The 550 remaining 21 Group 2 zircons display a scattered age pattern ranging from 370 to 800 Ma, 551 which might be detritus zircons of magmatic origin with or without later metamorphic and 552 hydrothermal overprinting.

553

#### 554 **Discussion**

555 Many studies have pointed out the relationships between the age and the chemical 556 composition of monazite grains in polymetamorphic terranes (e. g. Martins et al., 2009; Kelly 557 et al., 2012). In samples SO3, SO7, SO11 and SO17, SEM BSE images of monazite grains 558 revealed homogeneous grains. The chemical compositions of the analysed monazite grains 559 cluster near the Monazite-(Ce) pole and scatter along the cheralite axis (Fig. 5a and b). The 560 weak data dispersal along the cheralite substitution line indicates that the analysed grains 561 represent a rather homogeneous population. Monazite grains from sample SO15 that exhibit a 562 light (i.e. dense) core and a dark (i.e. light) rim in BSE images, are scattered between the 563 cheralite (CaTh(PO<sub>4</sub>)<sub>2</sub>) and huttonite (2ThSiO<sub>4</sub>) substitutions (Fig. 5b). Compared with the 564 other analysed samples, such a difference is explained by a high REE (La, Ce, Pr, Nd) content 565 and absence of Y in the core of monazite grains from sample SO15 (Table 2). The analysed 566 spots in both the core and rim parts delimit two clusters with different chemical composition.

567 It is noteworthy that i) the chemistry of the SO15 monazite rim is close to the chemistry of the 568 other analysed monazite grains, and ii) the reaction leading to the rim crystallisation occurred 569 contemporaneously to the crystallisation of the other monazite grains in samples SO3, SO7, 570 SO11 and SO17. Datings carried out in this study have been made on grain populations with 571 homogeneous chemical composition that make us confident about the age interpretation. 572 The kyanite-garnet granulitic gneiss SO15 preserves a core domain within monazite 573 yielding a *ca* 415 Ma age. This Early Devonian age can be assigned to the  $D_0$  event 574 recognized in the UGU in several places in the Massif Central (Pin and Peucat 1986; Pin and 575 Vielzeuf 1988; Paquette et al. 1995). All the dated samples, whatever the tectonic unit to 576 which they belong, yielded similar within the error, monazite U-Th-Pb ages at ca 365-350 Ma 577 (Fig. 10). Since they share common structural features, including a similar NW-SE striking, 578 D<sub>2</sub>, stretching lineation, which is well developed in samples SO3, SO11, SO17, and SO15, the 579 363-351 Ma ages yielded by the monazite in these rocks can be attributed to the D<sub>2</sub> tectono-580 metamorphic event. Similar ages were already reported for the D<sub>2</sub> tectono-metamorphic event 581 in other areas of the French Massif Central, such as the Lot series (Costa 1989) or Limousin 582 (Melleton et al. 2009). Moreover, these ages are consistent, within the errors, with the 583 emplacement age of the Guéret and S<sup>t</sup>-Gervais d'Auvergne plutons (Figs 1, 2) that exhibit a 584 NW-SE striking magmatic lineation (Faure et al. 2005; Cartannaz et al. 2007). These plutons, 585 dated at 360-350 Ma, emplaced at the end of the D<sub>2</sub> event (Berthier et al. 1979; Cartannaz et 586 al. 2007; Faure et al., 2009). The SO11  $D_1$  migmatite sample, deformed during the  $D_2$  event, 587 yields also a monazite U-Th-Pb age at ca 363 Ma, which is consistent with the Late 588 Devonian-Early Carboniferous age of the D<sub>2</sub> event. 589 It is worth to note that although the  $D_1$  migmatization is preserved in samples SO11,

and SO7, the P-T conditions of 0.4-0.6 GPa and 550-600°C, calculated in the UGU from a

sample close to our sample SO7 (Schulz et al. 2001, Schulz 2009) are not in agreement with

592 the  $D_1$  event, as defined in other areas of the French Massif Central (e.g. Santallier 1981; 593 Mercier et al. 1991; Lardeaux et al. 2001; Bellot and Roig 2007), but instead represent a low 594 pressure-medium temperature event similar to the D<sub>2</sub> one as observed in the Limousin, Lot or 595 Rouergue areas (Duguet et al. 2006; Bellot and Roig 2007; Melleton et al 2009). Thus this 596 biotite-sillimanite paragneiss might have also experienced the D<sub>2</sub> event, even if the typical 597 NW-SE D<sub>2</sub> stretching lineation is not observed in this rock. Furthermore, the NE-SW striking 598  $D_1$  mineral lineation is clearly observed in sample SO17. In these three rocks, the analyzed 599 monazites do not record the Devonian migmatization dated between 385 Ma and 375 Ma in 600 Lyonnais (Duthou et al. 1994), Limousin (Faure et al. 2008) and S. Brittany (Cocherie et al. 601 2005). This result can be variously interpreted. Firstly, monazite was unable to crystallize 602 under the D<sub>1</sub> metamorphic conditions; secondly, monazite effectively crystallized, but it may 603 have been dissolved during the HT event, and then neocrystallized in biotite during the 604 prograde D<sub>2</sub> event; thirdly, due to the limited number of analyzed grains, our sampling missed 605 any monazite grain formed during the D<sub>1</sub> event. Though the first hypothesis appears unlikely, 606 since aluminous composition protoliths are suitable for monazite crystallization, the other two 607 explanations cannot be discarded. Thus, the 365-350 Ma monazite ages obtained from 608 samples SO3, SO17, SO7, SO11, and the monazite rim from sample SO15 are attributed to 609 the neocrystallization or the complete recrystallization of monazite during the  $D_2$  event, 610 leading to the total loss of any early monazite. In order to get a better understanding of the 611 thermo-barometric and chemical conditions that allow monazite crystallization or dissolution 612 with respect to other phases such as garnet and biotite, a detailed analysis of the chemical 613 elements exchanges during the succession of the crystallization-deformation events would be 614 necessary. On the contrary to Limousin or Maures areas (e. g. Duguet et al. 2006; Bellot and 615 Roig 2007; Oliot et al. 2015), such a study is not available yet for the Sioule metamorphic 616 series.

617 Besides, zircon from the granulitic paragneiss yields U-Pb SIMS ages younger than 618 the monazite ones at 343±2 Ma, and 328±2 Ma. The latest is close to the whole-rock Rb/Sr 619 age of the Pouzol-Servant microgranite at 330 Ma (Duthou and Pin 1987; Pin 1991), and also to the cooling ages yielded by the  ${}^{40}$ Ar/ ${}^{39}$ Ar analyses on biotite, muscovite and amphibole for 620 621 the entire nappe-stack of the Sioule metamorphic series, either in the Upper Gneiss, Lower 622 Gneiss, and Para-autochthonous Units (Faure et al. 2002; Fig. 11). As pointed out in the 623 "Geological Setting" section, the *ca* 330 Ma age is that of the "Tufs anthracifères" 624 magmatism widespread in the northern part of the French Massif Central (Fig. 11). The <sup>40</sup>Ar/<sup>39</sup>Ar ages of the Sioule metamorphic rocks, younger than the Late Visean unconformity, 625 626 have been interpreted as a thermal reset provided by the high heat flux during the onset on the 627 extensional tectonics in the northern part of the French Massif Central. Thus we suggest here 628 that the zircon age at  $328\pm4$  Ma Ma might be due to recrystallizations processes, possibly 629 assisted by hydrothermal fluids, also as a consequence of the Late Visean thermal reset. 630 Indeed, the LGU and PAU rocks exhibit several evidence of retrogression such as chlorite 631 developed at the expense of biotite, sericite surrounding staurolite, (Fig 4a) and albite 632 overprinting the foliation. These minerals argue for a late metamorphic stage associated with 633 fluid circulation. In the present state of knowledge, detailed petrological investigations to 634 characterize this hydrothermal event are not available. The 343±2 Ma age yielded by 24 635 zircon grains is more difficult to interpret in the geological framework. This age 636 corresponding to the majority of analyzed concordant grains is different, within the error, 637 from the 328±4 Ma age. Furthermore, such a Middle Visean age is rarely reported in the 638 northern part of the Massif Central. However, to the East of the study area, in the Lyonnais, 639 syn-kinematic granites emplaced along a NE-SW striking dextral strike-slip fault yield biotite and muscovite  ${}^{40}\text{Ar}/{}^{39}\text{Ar}$  ages at ca 346±3 Ma and 349±3 Ma, respectively (Costa et al. 1993). 640 641 Such a NE-SW strike-slip shearing is not identified in the Sioule area, but the NW-SE striking S<sup>te</sup>-Christine dextral strike slip fault develops in the southern boundary of the Sioule series
(Fig. 2). One possible interpretation would be to relate this *ca* 343 Ma (i. e. Middle Visean)
age to fluid circulations coeval with a transcurrent shearing event too. Importantly, the zircon
SIMS measurement did not recorded the 365-350 Ma D<sub>2</sub> ages.

646 Previous monazite U-Th/Pb ages (Schulz 2009) range between 343 Ma and 327 Ma, 647 i.e. much younger than those reported in this study. These dates were attributed to two distinct events, in agreement with the  ${}^{40}\text{Ar}/{}^{39}\text{Ar}$  ages (Faure et al. 2002), the monazite chemical ages 648 649 younger than 340 Ma were considered as due to the Late Visean hydrothermal event related to 650 the "Tufs anthracifères" magmatism. Conversely, ages older than 340 Ma were considered as 651 minimum ones associated to a top-to-the-SE tectonic event. However, structural 652 investigations demonstrated that the top-to-the-SE shearing was coeval with the emplacement 653 of the Echassières pluton at *ca* 310 Ma (Feybesse and Teygey 1987; Faure et al. 1993). The 654 discrepancy between our results and the previous ages by Schulz (2009) can also be explained 655 considering the data reduction method. The previous study uses frequency histograms to 656 process the analytical data associated with very large, routinely ca 15 Ma, errors. Moreover, 657 the frequency diagram method provides a mean age systematically younger than the one given 658 by the Th/Pb= f(U/Pb) isochron method (Cocherie and Albarède 2001). For example, the 659 weighted average age computed here for the monazite core and rim of sample SO15 (Fig. 5c) 660 also yields ages at  $392\pm16$  Ma, and  $359\pm18$  Ma younger than  $416\pm8$  Ma, and  $362\pm14$  Ma 661 yielded by the isochron method. In addition, the results provided by the isochron method are generally closer to the ICP-MS isotopic measurements than those provided by the histogram 662 663 method (Cocherie et al. 1998; Cocherie and Albarède 2001), and thus are preferred here. As mentionned in the "Sioule metamorphic series" section, in the study area, two pre-Late Visean 664 665 events, namely  $D_1$  and  $D_2$ , are recognized. However, the age of the  $D_1$  and  $D_2$  range between

385-375 Ma and 360-350 Ma, respectively. Therefore, the *ca* 340 Ma monazite ages cannot
be ascribed to a single tectono-metamorphic event.

668 Our new monazite chemical ages can be used to place time constraints in the pressure 669 (P)-temperature (T) plot in order to settle the P-T-t paths experienced by each unit. P-T 670 estimates from the Sioule area (Schulz et al. 2001) are consistent with P-T conditions from 671 other areas of the Massif Central (e.g. Santallier 1981; Mercier et al. 1991; Lardeaux et al. 672 2001; Bellot and Roig 2007; Faure et al. 2008). The UGU, LGU, and PAU underwent 673 different P-T paths depending on their location in the French Massif Central. In the Lyonnais 674 area, the coesite bearing eclogite argue for P conditions about 2.7-2.8 GPa (Lardeaux et al. 675 2001) whereas such ultra-high pressure conditions are not recorded (or not preserved) in the 676 Sioule area. On the basis of petrological studies in the Limousin and Artense areas (Santallier 677 1981, Mercier et al. 1991), peak-conditions of ca 2 GPa for ca 800°C are inferred for the 678 Sioule granulite (Fig. 12). Sample SO15 places a time constraint at *ca* 415 Ma on this HP 679 event. On the contrary, in the Limousin area, the LGU reached a maximum P value of 1.1-1.2 680 GPa for 500 °C (Bellot and Roig 2007; Faure et al. 2008). During their exhumation, the UGU 681 and LGU followed nearly isothermal decompression paths coeval with migmatization, and 682 subsequent cooling during the D<sub>1</sub> event at 385-375 Ma. The minimum P-T conditions 683 experienced by the UGU and LGU during this eo-Variscan cycle are not precisely settled. 684 Since andalusite replacing sillimanite or kyanite is never observed in the UGU or LGU, we 685 assume that the two units were not exhumed close to the surface but remained burried in the 686 upper crust, even if aluminosilicates might have remained stable during exhumation. 687 Conversely, the PAU never experienced the D<sub>1</sub> metamorphism nor migmatization. This unit 688 records maximum P and T values of 0.7-0.8 GPa, and 500-600°C, respectively. Our monazite 689 age of sample SO3 at 363±8 Ma constrains the age of the MP/MT metamorphism coeval with 690 the top-to-the-NW D<sub>2</sub> event. During the same late Devonian-Early Carboniferous period, the

691	UGU and LGU experienced a second metamorphic cycle leading to maximum P-T conditions
692	of 0.7-0.8 GPa, and 500-600°C similar to those of the PAU. Our monazite U-Th-Pb ages at
693	362Ma, 363 Ma, 355 Ma, and 351 Ma, for samples SO10, SO11, SO7, and SO17,
694	respectively represent the age of the $D_2$ event recorded in the UGU and LGU (Fig. 12).
695	After the completion of the two Variscan metamorphic cycles, the Sioule metamorphic
696	series, like most of the areas in the northern part of the FMC, experienced a huge late
697	orogenic magmatic episode, at ca 335-330 Ma, represented both by deep seated plutonism,
698	dyke emplacement, and aerial volcanism. As already proposed to account for the ${}^{40}\text{Ar}/{}^{39}\text{Ar}$
699	ages, the hydrothermalism was responsible for the complete resetting of the zircon U-Pb
700	isotopic systems during the Tufs Anthracifères event but the U-Th-Pb chronometer was not
701	affected by this late thermal event.
702	
703	Conclusion
704	This geochronological study in a key area of the Variscan French Massif Central, casts light
705	on the importance of structural and petrological constraints when dealing with age
706	determination of thermal, tectonic and metamorphic events in orogens. These new radiometric
707	ages yielded by monazite and zircon included in the macroscopic foliation allow us to place
708	time constraints on the succession of superimposed deformation and related metamorphic
709	events in the Sioule area. The early Variscan HP/HT $D_0$ event occurred at 416±15 Ma and the
710	MP/MT $D_2$ event, coeval with the top-to-the NW shearing took place between 365 Ma and
711	350 Ma. On the contrary, the $ca$ 380 Ma D <sub>1</sub> event has not been recorded by monazite. Our
712	study emphasizes that radiometric ages must be carefully discussed in the light of the regional
713	geological setting. Particularly, the knowledge of the thermo-barometric and hydrothermal
714	processes must be a pre-requisite for the understanding of the geochronological data.

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- 1024 Figure Captions
- 1025 Fig. 1. A: Location of the French Massif Central in the European Variscan framework. B:
- 1026 Structural map of the Massif Central (modified from Ledru et al., 1989, and Faure et al.,
- 1027 2009). D<sub>1</sub>, D<sub>2</sub>, D<sub>3</sub> refer to the stretching lineation and kinematics referred to the Early
- 1028 Devonian, Early Carboniferous, and Middle Carboniferous tectono-metamorphic events,
- 1029 respectively. SGA: S<sup>t</sup>-Gervais d'Auvergne pluton
- 1030
- Fig. 2. Structural map, and NW-SE cross-section of the Sioule series with sampling sites and
  microstructural data, after Grolier (1971), Feybesse and Teygey (1987), and Faure et al.
- 1033 (1993).
- 1034
- 1035 Fig. 3. Examples of representative macroscopic field structures of the Para-autochthonous (a),
- 1036 Lower Gneiss (b) and Upper Gneiss (c, d) Units. a1: outcrop of biotite-staurolite micaschist
- 1037 of sample SO3, with top-to-the-SE sigmoidal quart veins, a2: close-up of the mineral lineation
- 1038 marked by elongated biotite and staurolite porphyroblasts. This NW-SE lineation results of
- 1039 the superposition of  $D_2$  and  $D_4$  events. b) D1 NE-SW striking isoclinal folds at Chouvigny. c)

Biotite-sillimanite paragneiss with S1 flat-lying foliation and L1 mineral lineation, close to
sample SO7. d) hand sample of garnet-kyanite granulitic paragneiss at Saugères (sample SO
1042 15). Note the biotite rim around garnet developed during the retrogression of the granulite
facies metamorphic assemblage into ampohibolite facies ones.

1044

1045 Fig. 4. Polarized light micrographs of analyzed samples showing the textural relationships of 1046 the dated monazite grains with other minerals (see location in Fig. 2). a: PAU micaschist 1047 sample SO3 from the Para-autochthonous Unit showing D<sub>2</sub> staurolite mantled by a sericite 1048 rim and deformed during the D<sub>4</sub> event. b: biotite-garnet-sillimanite gneiss (sample SO7) from 1049 the Upper Gneiss Unit with monazite included in biotite. c: biotite-sillimanite migmatite 1050 (sample SO11) foliated and lineated during the D<sub>2</sub> event, the dated monazite is included in 1051 biotite. d: kyanite-garnet granulitic paragneiss (sample SO15) with garnet surrounded by a 1052 biotite rim developed during the  $D_2$  event.

1053

1054 Fig. 5. a: plot of the analyzed monazites in the 2REEPO<sub>4</sub>-CaTh(PO<sub>4</sub>)<sub>2</sub>-2ThSIO<sub>4</sub> system 1055 (Linthout, 2007) showing that the grains are close to the Ce-monazite pole. b: plot of the 1056 analyzed monazite in the Th+U+Si+Pb vs REE+P diagram (Franz et al., 1996). c: weighted 1057 average age of the core and rim of monazite contained in kyanite-garnet paragneiss of 1058 Saugères (sample SO15). d: X-ray compositional map for Th, Ca, U of the analyzed monazite 1059 grain in the granulitic paragneiss sample SO15, showing the chemical zoning with a Th, Ca, 1060 and U enriched rim. White and black squares represent EPMA analyse points for core and 1061 rim, respectively.

1062

Fig. 6. Th/Pb vs U/Pb plots with SEM BSE pictures of dated monazites, sample location
shown on Fig. 2, and GPS in Table 1. a, b, c, b: Upper Gneiss Unit. a) core of monazite grain

1065	from SO 15 granulite (Saugères); b) rim of monazite grain from SO15 granulite (Saugères); c)
1066	biotite-sillimanite migmatitic gneiss SO11 from Pont de Menat; d) biotite-sillimanite
1067	migmatitic paragneiss SO7 from Moulin de Bouchet; e) biotite-staurolite micaschist SO3
1068	from the Para-autochthonous Unit; f) two-mica paragneiss SO17 from the Lower Gneiss Unit.
1069	
1070	Fig. 7. Cathodoluminescence (CL) images for representative zircons sample SO 15. The white
1071	ellipses in the CL images represent the spots of SIMS U-Pb analysis. SIMS spots are 30 $\mu m$ in
1072	length for scale.
1073	
1074	Fig. 8. Probability histogram of U-Pb age for 51 concordant analyses showing the two groups
1075	of zircon ages. Group 1 zircons have a main peak at 342 Ma and a secondary one at 327 Ma.
1076	
1077	Fig. 9. Zircon U-Pb Concordia age of granulitic paragneiss sample S015.
1078	
1079	Fig. 10. Synthetic map summarizing the available radiometric ages obtained through different

1080 methods, whole rock Rb/Sr, biotite and muscovite  ${}^{40}$ Ar/ ${}^{39}$ Ar, monazite U-Th-Pb, and zircon

1081 U-Pb SIMS for the Sioule series.

1082

1083 Fig. 11. Synoptic chart of the radiometric ages from the Sioule series presented in this paper,

1084 compared with the ages of the  $D_1$ ,  $D_2$ ,  $D_3$  tectono-thermal events for the French Massif

1085 central, and the Guéret and S<sup>t</sup>-Gervais biotite granites, and Pouzol-Servant microgranite.

1086 (1): Average biotite, muscovite, and hornblende  ${}^{40}$ Ar/ ${}^{39}$ Ar ages from Faure et al. (2002); (2):

1087 zircon U-Pb age of the Pouzol-Servant microganite (Pin 1991); (3): zircon U-Pb SHRIMP and

1088 monazite U-Th-Pb chemical ages of the Guéret-St-Gervais plutons (Cartannaz et al. 2007).

1090	Fig. 12. P-T-t paths for the Upper Gneiss Unit (UGU), Lower Gneiss Unit (LGU), and Para-
1091	Autochthonous Unit (PAU) modified from Mercier et al. 1991, Lardeaux et al., 2001; Schulz
1092	et al., 2001; Bellot and Roig 2007; Faure et al. 2007; Schulz 2009) emphasizing the
1093	polycyclic evolution. During the eo-Variscan cycle, the UGU experienced the $D_0$ high to
1094	ultra-high pressure metamorphism, and Devonian migmatization during its $D_1$ exhumation.
1095	The LGU underwent burial under lower pressure conditions than the UGU, then Devonian
1096	exhumation and migmatization during $D_1$ . During the Late Devonian-Early Carboniferous $D_2$
1097	event, the PAU followed its first burial and exhumation cycle, whereas the LGU and UGU
1098	experienced a second metamorphic cycle before its Late Visean exhumation.
1099	
1100	Table 1. Dated sample locations and analytical data
1101	
1102	Table 2. Electron microprobe analyses of dated monazites
1103	

1104 Table 3. Zircon U-Pb zircon analyses of granulitic paragneiss sample SO15

Figure 1 Click here to download high resolution image









Figure 5 Click here to download high resolution image



Fig. 5



 $\partial g \, d$ 











Fig. 11

Figure 12 Click here to download high resolution image



Sample	LAT	LON	Nbr of grains	Pb (ppm)	U (ppm)	Th (ppm)	U/Pb	Th/Pb	lsochron age ±2σ	n
SO3	46°10'19.25"	3°01'30.80"	4	839	4885	37366	6.2	44.2	363	73
				±	±	±	±	±	±	
				253	1596	14335	2.3	8	8	
SO7	46°06'09.60"	3°02'28.20"	4	1013	8039	39089	7.9	39.1	355	73
				±	±	±	±	±	±	
				147	2514	5186	2	6.3	7	
SO11	46°06'01.19"	2°55'15.60"	4	914	5365	39639	5.9	43.8	363	17
				±	±	±	±	±	±	4
				138	1296	5835	1.2	5.4	4	
SO15	46°07'46.99"	2°54'05.36"	1	586	1960	26843	3.4	46.8	361	47
Rim				±	±	±	±	±	±	
				103	614	3470	1	7.9	14	
SO15			1	808	3922	38519	4.5	49.5	416	21
Core				±	±	±	±	±	±	
				160	2648	3886	2.2	11.1	15	
SO17	46°10'31.26"	2°54'48.07"	6	1179	8347	49787	7.2	41.8	351	38
				±	±	±	±	±	±	
				235	1141	15110	1.2	4.8	5	

### Table 1. Dated sample locations and analytical data

Sample	UO <sub>2</sub>	PbO	$Nd_2O_3$	CaO	P <sub>2</sub> O <sub>5</sub>	ThO <sub>2</sub>	$Gd_2O_3$	Ce <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	$Sm_2O_3$	$La_2O_3$	Y <sub>2</sub> O <sub>3</sub>	Pr <sub>2</sub> O <sub>3</sub>
	0.55	0.10	12.27	0.88	29.27	4.25	1.34	29.27	0.67	2.21	14.08	0.99	3.51
SO3	±	±	±	±	±	±	±	±	±	±	±	±	±
	0.18	0.03	0.54	0.27	0.5	1.63	0.28	0.94	0.99	0.16	0.78	0.26	0.15
	0.91	0.13	11.16	1.04	28.82	4.45	1.57	28.33	0.28	1.98	13.9	2.11	3.29
SO7	±	±	±	±	±	±	±	±	±	±	±	±	±
	0.29	0.02	0.45	0.11	0.58	0.59	0.15	0.51	0.13	0.16	0.31	0.64	0.1
	0.61	0.1	11.9	0.99	29.39	4.48	1.32	28.85	0.25	2.03	14.92	0.74	3.44
SO11	±	±	±	±	±	±	±	±	±	±	±	±	±
	0.15	0.01	0.32	0.11	0.92	0.68	0.29	0.94	0.11	0.14	0.6	0.86	0.11
\$015	0.22	0.06	12.34	0.65	29.33	3.05	0.51	32.19	0.15	1.62	16.55		3.65
Rim	±	±	±	±	±	±	±	±	±	±	±	0	±
NIIII	0.07	0.01	0.34	0.49	0.49	0.39	0.08	0.37	0.17	0.12	0.52	0	0.09
\$015	0.44	0.09	12.44	0.81	29.1	4.39	0.76	30.78	0.32	1.93	15.86		3.57
Core	±	±	±	±	±	±	±	±	±	±	±	0	±
core	0.3	0.02	0.23	0.14	0.48	0.44	0.14	0.59	0.15	0.15	0.32	0	0.08
	0.95	0.15	10.98	1.35	28.04	5.67	1.55	27.26	1.11	1.88	13.36	2.56	3.23
SO17	±	±	±	±	±	±	±	±	±	±	±	±	±
	0.13	0.03	0.55	0.24	1.03	1.72	0.13	0.74	2.79	0.17	0.39	0.28	0.13

## Table 2. Electron microprobe analyses of dated monazites

Table 2

Table 3 SIMS U-Pb zircon data					Radiogenic isotopic ratios						Age (Ma)					
Sample/	[U]	[Th]	Th/U	<b>f</b> 206	<sup>207</sup> Pb	±σ	<sup>207</sup> Pb	±σ	<sup>206</sup> Pb	±σ	<sup>207</sup> <u>Pb</u>	±σ	<sup>207</sup> Pb	±σ	<sup>206</sup> Pb	±σ
spot #	ppm	ppm		%	<sup>206</sup> Pb	%	<sup>235</sup> U	%	<sup>238</sup> U	%	<sup>206</sup> Pb		<sup>235</sup> U		<sup>238</sup> U	
SO-15A@01	904	28	0,03	0,00	0,0534	0,77	0,401	1,69	0,0544	1,50	346,5	17,3	342,2	4,9	341,6	5,0
SO-15A@02	231	94	0,41	0,01	0,0580	1,33	0,675	2,05	0,0844	1,56	530,5	28,9	523,8	8,4	522,3	7,8
SO-15A@03	743	34	0,05	0,00	0,0538	0,87	0,408	1,74	0,0549	1,51	364,6	19,4	347,3	5,1	344,8	5,1
SO-15A@04	673	21	0,03	0,00	0,0528	1,06	0,397	1,88	0,0545	1,55	320,0	23,8	339,4	5,4	342,2	5,2
SO-15A@05	819	25	0,03	0,01	0,0529	0,89	0,402	1,74	0,0550	1,50	326,6	20,0	342,8	5,1	345,2	5,0
SO-15A@06	289	83	0,29	0,03	0,1226	1,44	3,497	3,62	0,2069	3,32	1994,2	25,4	1526,5	29,0	1212,2	36,8
SO-15A@07	250	106	0,43	0,39	0,0534	1,75	0,541	2,31	0,0734	1,51	346,5	39,1	438,8	8,3	456,6	6,6
SO-15A@08	632	85	0,13	0,02	0,0703	1,81	1,295	2,48	0,1335	1,70	937,8	36,6	843,5	14,3	808,1	12,9
SO-15A@09	377	159	0,42	0,10	0,0565	1,07	0,527	2,19	0,0677	1,91	470,5	23,6	430,0	7,7	422,4	7,8
SO-15A@10	953	22	0,02	0,07	0,0532	0,82	0,387	1,72	0,0528	1,51	338,0	18,5	332,4	4,9	331,6	4,9
SO-15A@11	843	107	0,13	0,02	0,0545	0,92	0,517	1,77	0,0687	1,50	393,2	20,6	423,0	6,1	428,5	6,2
SO-15A@12	661	37	0,06	0,02	0,0553	0,87	0,416	1,74	0,0546	1,50	423,5	19,3	353,3	5,2	342,7	5,0
SO-15A@13	444	15	0,03	0,00	0,0538	1,08	0,404	1,85	0,0545	1,50	361,7	24,2	344,7	5,4	342,2	5,0
SO-15A@14	817	115	0,14	0,00	0,0569	0,69	0,534	1,72	0,0681	1,57	487,0	15,2	434,7	6,1	424,9	6,5
SO-15A@15	389	324	0,83	0,01	0,0583	0,91	0,668	1,75	0,0831	1,50	542,5	19,7	519,7	7,2	514,5	7,4
SO-15A@16	1693	280	0,17	0,09	0,0501	0,55	0,531	1,64	0,0687	1,54	454,6	12,1	432,4	5,8	428,3	0,4
50-15A@17	262	213	0,37	0,00	0,0591	1,05	0,707	2,33	0,0808	2,08	229,0	22,7	542,9 242.4	9,8	242.0	10,7
SO-15A@10	757	20	0,05	0,02	0,0532	0,94	0,401	1,70	0,0547	1,52	267.2	21,1	342,4 350.6	5,2	242,0	5,1
SO-15A@19	206	20	0,04	0,02	0,0539	0,99	0,412	1,00	0,0555	1,50	307,Z	22,2	330,0 491 7	5,5	340, I 170.0	5,1
SO-15A@20	616	27	0,30	0,00	0,0576	0,93	0,007	1,70	0,0773	1,52	307 7	20,5	335.6	5.0	326.7	1,0
SO-154@22	1050	42	0.04	0.07	0 0537	1 26	0.408	1 06	0.0550	1,50	359.8	28.2	347 2	5.8	345 4	5 1
SO-15A@23	758	36	0.05	0.02	0.0537	0.86	0,391	1,30	0.0528	1,50	357.3	19.4	335 1	5.0	331 9	49
SO-15A@24	536	837	1.56	0.05	0.0547	0.98	0,458	1.80	0.0607	1.51	400 2	21.8	382.9	57	380 1	5.6
SO-15A@25	954	203	0.21	0.18	0.0543	1.03	0.417	1,82	0.0557	1,50	381.5	23.0	353.6	5.5	349.4	5,1
SO-15A@26	734	325	0.44	0.23	0.1111	0.63	3.048	2.00	0,1989	1.89	1818.0	11.5	1419.8	15.4	1169.6	20.3
SO-15A@27	735	413	0.56	0.02	0.1087	0.71	3.219	1.69	0.2147	1.54	1778.2	12.8	1461.7	13.2	1253.9	17.5
SO-15A@28	845	27	0.03	0.09	0.0527	1.09	0.377	1.85	0.0520	1.50	315.0	24.5	325.1	5.2	326.5	4.8
SO-15A@29	586	447	0,76	0.05	0,0696	0,75	1,251	1,77	0,1304	1,60	916,0	15,3	823,9	10,0	790,2	11,9
SO-15A@30	910	365	0,40	0,09	0,0568	0,78	0,575	1,69	0,0733	1,50	485,2	17,1	461,0	6,3	456,2	6,6
SO-15A@31	693	187	0,27	0,56	0,0555	1,57	0,476	2,18	0,0622	1,50	430,5	34,7	395,2	7,2	389,2	5,7
SO-15A@32	434	162	0,37	0,24	0,0557	1,21	0,479	1,98	0,0623	1,56	441,3	26,7	397,2	6,5	389,7	5,9
SO-15A@33	388	67	0,17	0,00	0,0541	1,09	0,451	1,86	0,0605	1,51	374,0	24,4	378,1	5,9	378,8	5,5
SO-15A@34	396	73	0,18	0,04	0,0550	1,10	0,495	1,89	0,0653	1,54	410,6	24,3	408,3	6,4	407,9	6,1
SO-15A@35	958	29	0,03	0,02	0,0529	0,77	0,398	1,69	0,0546	1,50	324,9	17,4	340,4	4,9	342,6	5,0
SO-15A@36	579	129	0,22	0,03	0,0540	0,93	0,437	1,78	0,0587	1,52	370,1	20,7	368,1	5,5	367,8	5,5
SO-15A@37	244	60	0,25	0,11	0,1003	2,17	1,531	2,84	0,1107	1,83	1629,2	39,8	942,8	17,6	676,9	11,8
SO-15A@39	1787	32	0,02	0,00	0,0529	0,61	0,397	1,63	0,0544	1,51	324,0	13,9	339,4	4,7	341,6	5,0
SO-15A@40	1767	22	0,01	0,05	0,0534	0,62	0,400	1,64	0,0543	1,52	347,3	14,0	341,8	4,8	340,9	5,0
SO-15A@41	1067	79	0,07	0,01	0,0533	0,70	0,398	1,66	0,0541	1,50	343,2	15,8	340,0	4,8	339,5	5,0
SO-15A@42	648	161	0,25	0,04	0,0541	1,14	0,491	1,97	0,0659	1,61	373,2	25,4	405,5	6,6	411,2	6,4
SO-15A@43	604	28	0,05	0,01	0,0538	0,92	0,404	1,77	0,0545	1,51	362,3	20,7	344,6	5,2	342,0	5,0
SO-15A@55	606	22	0,04	0,05	0,0541	1,39	0,407	2,05	0,0545	1,51	377,3	30,9	346,6	6,0	342,0	5,0
SO-15A@57	636	35	0,06	0.03	0,0531	1,36	0,391	2,03	0,0535	1,50	331,7	30,5	335,2	5,8	335,7	4,9
SO-15A@58	649	52	0.08	0.00	0.0608	0.94	0.818	1.84	0.0976	1.58	631.1	20.2	606.9	8.5	600.4	9.1
SO_15A@50	1077	1/32	0.72	0,00	0.0503	0.64	0 772	1.63	0.0044	1 50	576.8	13.0	580.7	7.2	581 7	83
	301	1452	0,72	0,06	0,0000	4.07	0,112	1,00	0,0544	1,00	004.0	10,0	201,7	, z	001,7	0,0
SU-15A@60	781	22	0,03	0,07	0,0528	1,37	0,376	2,04	0,0516	1,50	321,6	30,9	324,0	5,7	324,3	4,8
SO-15A@61	477	31	0,07	0,00	0,0539	1,57	0,405	2,17	0,0544	1,50	368,0	34,9	345,0	6,4	341,6	5,0
SO-15A@62	725	25	0,03	0.00	0,0530	1,28	0,395	1,97	0,0541	1,50	327,4	28,8	338,0	5,7	339,5	5,0
SO-15A@63	810	31	0.04	0.02	0.0533	1.21	0.399	1,93	0.0543	1.50	343.0	27.2	341.0	5.6	340 7	5.0
SO_15A@64	606	20	0.04	0,02	0.0524	1.97	0.202	2 / 1	0.0510	1 50	344 5	11 7	300 0	6.0	325.0	10
	090	20	0,04	0,09	0,0004	1,07	0,002	2,41	0,0019	1,02	000.0	+1,7	020,Z	0,0	323,9	4,0
SU-15A@65	573	26	0,05	0,00	0,0528	1,50	0,393	2,12	0,0539	1,50	320,8	33,7	336,2	6,1	338,5	4,9
SO-15A@66	2192	98	0,04	0,11	0,0524	1,03	0,403	1,82	0,0557	1,50	304,3	23,3	343,7	5,3	349,5	5,1
SO-15A@67	577	227	0,39	0.15	0,0517	1,76	0,384	2,31	0,0539	1,50	272,0	39,8	330,0	6,5	338,3	4,9
SO-15A@68	609	16	0,03	0,00	0,0517	1,48	0,394	2,12	0,0553	1,52	274,2	33,5	337,6	6,1	346,9	5,1
Qinghu@1	1552	930	0,60	0.04	0,0496	0,92	0,173	1,80	0,0253	1,55	176,0	21,2	162.3	2,7	161,3	2,5
Qinghu@2	1209	554	0.46	0.00	0 0491	1.00	0 170	1.80	0 0252	1.50	150.4	23.3	159.6	27	160.2	24
Qinghu@2	203	554	0,40	0,00	0,0500	0.05	0.174	1,00	0,0254	1,00	204.0	20,0	160.0	2,1	150,2	2,7
ungnu@3	2014	557	0,28	0,01	0,0502	0,95	0,174	1,78	0,0251	1,50	204,0	22,0	162,8	2,7	159,9	2,4
Qinghu@4	945	409	0,43	0,00	0,0500	1,94	0,172	2,45	0,0249	1,50	195,2	44,5	160,9	3,7	158,6	2,4
Qinghu@5	633	227	0,36	0,00	0,0490	1,40	0,170	2,06	0,0251	1,51	146,2	32,5	159,0	3,0	159,9	2,4
Qinghu@6	1362	797	0.59	0 11	0.0499	1.21	0.174	1.93	0.0253	1.51	188.3	27.9	162 6	2.9	160.8	24
Oinghu@7	2550	1044	0.44	0,11	0.0404	0.70	0.174	1.66	0.0256	1 50	152.0	16 4	162.6	2,0	162.0	<u> </u>
angnu@/	2009	1041	0,41	0,01	0,0491	0,70	0,174	1,00	0,0250	1,50	153,9	10,4	102,0	2,5	103,2	2,4
Qinghu@8	1301	551	0,42	0,00	0,0496	0,96	0,171	1,78	0,0250	1,50	175,2	22,4	160,5	2,7	159,5	2,4
Qinghu@9	703	300	0,43	0,07	0,0482	1,46	0,165	2,09	0,0249	1,50	107,3	34,1	155,4	3,0	158,6	2,4
	980	440	0.45	0.10	0,0498	1,23	0,174	1,94	0,0253	1,50	184.4	28.3	162.7	2.9	161.2	2.4
Qinghu@10	500					, = =		,	= = = =	,	, .	-,-	, .	_,_	,	-, -
Qinghu@10 Qinghu@11	980	440	0.45	0.10	0 0408	1 23	0 174	1 9/	0 0253	1 50	184 4	28.3	162 7	29	161 2	24
Qinghu@10 Qinghu@11	980	440	0,45	0,10	0,0498	1,23	0,174	1,94	0,0253	1,50	184,4	28,3	162,7	2,9	161,2	2,4