1 Revision 2

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Granitoid magmas preserved as melt inclusions in high-grade metamorphic rocks

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17 **Abstract**

18 This review presents a compositional database of primary anatectic granitoid magmas, entirely based

19 on melt inclusions (MI) in high-grade metamorphic rocks. Although MI are well known to igneous

20 petrologists and have been extensively studied in intrusive and extrusive rocks, MI in crustal rocks that

21 have undergone anatexis (migmatites and granulites) are a novel subject of research. They are generally

22 trapped along the heating path by peritectic phases produced by incongruent melting reactions. Primary

23 MI in high-grade metamorphic rocks are small, commonly 5-10 μm in diameter, and their most

24 common mineral host is peritectic garnet. In most cases inclusions have crystallized into a

25 cryptocrystalline aggregate and contain a granitoid phase assemblage (nanogranitoid inclusions) with

26 quartz, K-feldspar, plagioclase and one or two mica depending on the particular circumstances. After

27 their experimental remelting under high confining pressure, nanogranitoid MI can be analyzed

28 combining several techniques (EMP, LA-ICP-MS, NanoSIMS, Raman). The trapped melt is granitic

29 and metaluminous to peraluminous, and sometimes granodioritic, tonalitic and trondhjemitic in

30 composition, in agreement with the different P-T-a_{H2O} conditions of melting and protolith composition,

31 and overlap the composition of experimental glasses produced at similar conditions. Being trapped

32 along the up-temperature trajectory –as opposed to classic MI in igneous rocks formed during down-

33 temperature magma crystallization– the fundamental information provided by nanogranitoid MI is the

34 pristine composition of the natural primary anatectic melt for the specific rock under investigation. So

35 far ≈600 nanogranitoid MI, coming from several occurrences from different geologic and geodynamic

36 settings and ages, have been characterized. Although the compiled MI database should be expanded to

37 other potential sources of crustal magmas, MI data collected so far can be already used as natural

38 "starting-point" compositions to track the processes involved in formation and evolution of granitoid

39 magmas.

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Introduction

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43 Granitoid rocks and their extrusive counterparts represent the largest products of magmatic activity on

44 continental crust. In zones of crustal thickening associated with collisional orogens, there are several

45 examples of crystalline basements which have experienced repeated episodes of high-temperature

46 metamorphism, partial melting and extraction of granitoid melts (Sawyer et al., 2011; Brown, 2013).

47 On the other hand, in continental margin arc systems there is much field and geochemical evidence of

48 melting of crustal rocks and hybridization between crustal- and mantle-derived magmas, resulting in

49 the formation of batholiths (Barnes et al., 2002; Annen et al., 2006; Kemp et al., 2007; Gray and

50 Kemp, 2009). Therefore, anatexis of crustal protoliths and formation of anatectic granitoid magmas are

51 key processes for the formation of new crust and its geochemical differentiation (Brown and Rushmer,

52 2006; Hacker et al., 2011; Sawyer et al., 2011). Field geology, experimental petrology, thermodynamic

53 calculations, numerical modeling, whole-rock geochemistry and Hf-O isotopes in zircon represent the

54 major tools for understanding the origin and evolution of anatectic granitoid magmas (see Annen et al.,

55 2006; Brown, 2013; Clemens, 2006; Kemp et al., 2007; Sawyer et al., 2011; White et al., 2011).

56 Despite the vigorous debate on the petrogenesis of granitoid magmas which began more than 60 yr ago

57 (Read, 1948), conflicting views still exist, in particular concerning the melting processes at the source

58 and the differentiation processes which modify granitoid chemistry (Clemens and Watkins, 2001;

59 Clemens and Stevens, 2012, 2015; Brown, 2013; Weinberg and Hasalová, 2015a, b; and discussion

60 therein).

61 Here we present a novel and alternative method to investigate crustal magmas, providing a

62 review of the current research on melt inclusions (MI) in high-grade metamorphic rocks, the source

63 where crustal magmas originate. After discussing the origin of MI by incongruent melting in

64 migmatites, granulites and anatectic enclaves and presenting the data collected mainly by the research

65 group of the authors, along with the adopted experimental and analytical strategies, we highlight how

66 such tiny inclusions of melt can constrain the origin and evolution of granitoid magmas produced by

67 anatexis of crustal rocks.

68

Changing the viewpoint: entrapment of melt inclusions upon heating

70 MI are small volumes of silicate melt enclosed in minerals (Roedder, 1984). First recognized in the 19th

71 century by Sorby (1858), MI hosted in minerals of igneous rocks have been long studied and widely

72 used in the last forty years (see dataset reported in Kesler et al., 2013), becoming a worldwide-accepted

73 technique to investigate melt evolution in magmatic systems (Clocchiatti, 1975; Roedder, 1979;

74 Sobolev, 1996; Frezzotti, 2001; Schiano, 2003; Bodnar and Student, 2006; Thomas and Davidson,

75 2012; Audétat and Lowenstern, 2014; and references therein). These MI (hereafter "classic MI") are

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76   generally trapped during crystallization of magmas, i.e. along the cooling path where the host crystal is

77   crystallizing *from* the melt which is being entrapped (Fig. 1). MI-bearing phenocrysts in lavas are a

78   typical example of this mode of occurrence. Being trapped along the down-temperature trajectory (Fig.

79   1), such classic MI represent therefore snapshots on the liquid line of descent of silicate liquids (i.e.,

80   they represent variably evolved melts) and, particularly in the case of felsic systems, they have

81   represented a key tool to investigate and understand the genesis of a variety of ore deposits (Audétat

82   and Lowenstern, 2014 and references therein). Even in the case of entrapment by the first liquidus

83   phase (for example olivine in some mantle-derived magmas), classic MI often approximate the

84   composition of the parental melt (i.e., the most primitive melt found in an area) and not that of the

85   primary one (i.e., the melt derived directly by partial melting of the source; see Fig. 1 in Bartoli et al.,

86   2014).

87   In order to represent primary melt compositions, MI should be entrapped in the source region,

88   before magmas undergo segregation and differentiation processes such as mixing and/or mingling with

89   other magma batches, double-diffusive convection, Soret diffusion, cumulus phenomena, fractional

90   crystallization, volatile exsolution and assimilation of residual or exotic material (e.g., Burnham, 1967;

91   DePaolo, 1981; Lesher et al., 1982; Barbarin 1988; Candela, 1997; Weinberg et al., 2001; Stevens et

92   al., 2007; Brown, 2013). Indeed, all these processes produce final products –i.e. most of the extrusive

93   and intrusive rocks we collect in the field– which are markedly different from the original melt

94   produced at the source.

95   The continental crust (i.e. the source region of the anatectic crustal magmas) mostly melts

96   through incongruent melting reactions (Weinberg and Hasalová, 2015a and references therein). Along

97   with magma crystallization, such reactions fulfill the fundamental requirement to form MI, i.e. the

98   growth of a mineral in the presence of a melt phase. Indeed during incongruent melting, both a silicate

99   liquid and a solid phase –the peritectic mineral– are produced contemporaneously along the up-

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100   temperature path (Fig. 1). In such a scenario, the peritectic phase can trap droplets of the melt with

101   which it is growing. Shielded by the peritectic host, these MI can provide us with the pristine

102   geochemical signatures ("the starting point" as stated by Sawyer et al., 2011) of magmas formed by

103   crustal anatexis. Although the possibility of trapping droplets of melt during incongruent melting may

104   appear intuitive, MI in high-grade metamorphic rocks have received only little consideration in the past

105   decades. After the first detailed microstructural and microchemical description of MI in regionally

106   metamorphosed granulites of southern India (Cesare et al., 2009), many other occurrences have been

107   reported worldwide in the last six years (e.g. Cesare et al., 2011, 2015; Ferrero et al., 2012, 2014, 2015;

108   Groppo et al., 2012; Mosca et al., 2012; Bartoli et al., 2013a, 2014; Darling, 2103; Kawakami et al.,

109   2013; Barich et al., 2014; Carosi et al., 2015; Massonne, 2014), demonstrating that MI in high-grade

110   metamorphic rocks are more common than expected. More rarely, MI have also been described in

111   enclaves of partially-melted continental crust brought to the Earth's surface in lavas. This is the case of

112   the anatectic metasedimentary enclaves from the Neogene Volcanic Province (NVP) of southern Spain

113   (Cesare et al., 1997) – also known as "erupted migmatites" (Zeck, 1970). From all the above

114   considerations it is clear that petrologists can utilize a novel tool, i.e. MI trapped by peritectic minerals,

115   to gain previously unavailable information, and to better understand the origins and evolution of crustal

116   magmas.

117

Microstructural characterization of melt inclusions

119   In most migmatites, granulites and anatectic enclaves investigated so far, MI have been mostly found in

120   garnets of the melt-depleted melanosome portions of the rock (Tab. 1), because this mineral is the most

121   common peritectic phase associated with crustal melting reactions in a wide P–T–X range (see Cesare

122   et al., 2011; Ferrero et al., 2012 and references therein). Other MI-bearing phases are plagioclase and

123   zircon, and less commonly cordierite, andalusite, ilmenite, hercynite and monazite (Tab. 1; Cesare et

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124 al., 2015). Crustal enclaves contain abundant MI both in reactants and peritectic phases, e.g.,

125 plagioclase and garnet (Cesare and Maineri, 1999; Cesare et al., 2003; Acosta-Vigil et al., 2007, 2010).

126 Indeed, during melting with some degree of reaction overstepping, some reactant phases in the rock

127 matrix may recrystallize, equilibrate with the melt and trap MI (Cesare et al., 2015).

128   From a microstructural point of view, MI may occur isolated, distributed in zonal arrangements,

129 or in clusters with random distribution within the host mineral (Fig. 2; Ferrero et al., 2012; Barich et

130 al., 2014). These modes of occurrence unequivocally demonstrate the primary nature of MI (Roedder,

131 1979). MI are generally small (<20 μm) and often show negative crystal shape (Fig. 3a-d). Locally,

132 larger MI have been described (up to 150-200 μm, Barich et al., 2014; Ferrero et al., 2015). Variable

133 degrees of crystallization have been observed and MI range from glassy to fully crystallized (Fig. 2e,

134 3a-e). The most common daughter minerals found within crystallized MI are quartz, alkali feldspar,

135 plagioclase, biotite and muscovite (Fig. 3a-c). In MI from near ultrahigh pressure conditions,

136 metastable polymorphs of albite, K-feldspar and quartz have also been identified (Fig. 3b; see also

137 Ferrero et al. 2016). A large number of minerals trapped during inclusion formation has been found

138 within MI, the most common being graphite, zircon, rutile and kyanite. Owing to the very fine grain-

139 size (often <1 μm) and the granitic phase assemblage observed in totally crystallized MI, they have

140 been referred to as "nanogranites" (Cesare et al., 2009). Because recent microchemical investigations

141 have demonstrated the existence of granodiorite, trondhjemite and tonalite compositions (Carosi et al,

142 2015; this study), the totally crystallized polycrystalline MI will be referred, hereafter, to as

143 "nanogranitoid inclusions". Nanogranitoids can display a diffuse micro- to nano-porosity, which

144 contains liquid H_2O, as evidenced by micro-Raman mapping (Bartoli et al. 2013a). Nanogranitoids may

145 also coexist with primary C-bearing fluid inclusions (Fig. 3f). As both inclusion types are primary, they

146 indicate conditions of fluid-melt immiscibility, thus suggesting COH fluid- present anatexis (Cesare et

147 al., 2007; Ferrero et al., 2011). Preserved glassy MI are abundant in anatectic enclaves owing to the fast

148   cooling (quench) related to the magma uprise and extrusion (Fig. 3d). On the other hand,

149   nanogranitoids and partially crystallized MI are typically found in slowly cooled, regionally

150   metamorphosed migmatite and granulite terranes (Fig. 3a-c). Surprisingly, some preserved glassy MI

151   coexisting with nanogranitoids have been recognized also in migmatitic terranes (Fig. 3e; see also

152   Cesare et al., 2009; Bartoli et al., 2013a,b; Barich et al., 2014). This geological oddity (i.e., the glass

153   preservation in slowly cooled rocks) has been ascribed to either pore size effect (Cesare et al., 2009), or

154   the heterogeneous distribution of nucleation sites (Ferrero et al., 2012), or the high viscosity of the

155   trapped melt (Bartoli et al., 2015).

156

Chemical characterization of melt inclusions

158   **Re-melting nanogranitoid inclusions**

159   The experimental re-melting of nanogranitoid inclusions is a prerequisite in order to retrieve the

160   original composition (in terms of major and volatile contents) of the trapped melt. Because anatexis

161   takes place in rocks of the middle to lower continental crust, at the bottom of thickened continental

162   crust, and even in continental materials subducted to mantle depths, re-melting experiments of

163   nanogranitoids must be performed under high confining pressure, using a piston-cylinder apparatus, to

164   prevent the inclusion from decrepitating, volatile loss and melt contamination. Indeed, as a

165   consequence of the H_2O loss, crystallized inclusions can be completely re-melted only at temperatures

166   higher than the trapping temperatures –i.e., H_2O loss results in an increased solidus temperature–

167   leading to an inevitable melt-host interaction (see details in Bartoli et al., 2013c). The first attempts to

168   remelt nanogranites using the routine technique in igneous petrology, namely the high-temperature

169   heating stage at ambient pressure (Esposito et al., 2012), produced the decrepitation of the

170   nanogranitoid inclusions, making them poorly suited for a geochemical study (Cesare et al., 2009).

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171   The P-T conditions to be used during piston cylinder remelting should be initially established

172   based on phase equilibria modelling or classic thermobarometry, that can provide a fair estimate of the

173   (presumed) P-T conditions of MI entrapment (Bartoli et al., 2013a, c; Ferrero et al., 2015). Conditions

174   of further experiments are then fine-tuned based on the results of the first runs. Despite the time

175   consuming pre- and post-run sample preparation (compared to the heating stage method), and the trial-

176   and-error nature of the experimental approach, this method can successfully re-melt the nanogranitoids

177   under high confining pressure (see Bartoli et al.,2013c; Ferrero et al., 2015).

178

179   **Building up the compositional database**

180   Working with MI in partially melted rocks (i.e. their identification, re-homogenization and chemical

181   characterization) is challenging due to their small diameter, often <20 μm (Fig. 2, 3). Despite these

182   difficulties, originally glassy and successfully re-melted MI can be analyzed for the concentrations of

183   most elements in the periodic table. Major element contents are routinely measured by electron

184   microprobe (EMP), analyzing secondary standards in the same analytical session to monitor the alkali

185   migration process and eventually calculate correction factors (Ferrero et al., 2012). Indeed, the small

186   size of MI often demands the use of a beam diameter of ≈1 μm (Bartoli et al., 2013a), resulting

187   inevitably in Na loss during EMP analysis of hydrous felsic glasses (Morgan and London, 1996,

188   2005a). The Laser Ablation Inductively Coupled-Plasma Mass-Spectrometry (LA-ICP-MS) has been

189   used to measure trace element contents of MI, drilling the glassy MI exposed on the host surface or the

190   nanogranitoid inclusions entirely enclosed in the host. In the latter case, one of the major elements

191   measured by EMP in coexisting glassy or re-melted MI can be used as internal standard for the

192   deconvolution of the mixed signal (Halter et al., 2002). H_2O, which is the most important volatile

193   dissolved in crustal magmas (Burnham, 1975), can be quantified by different methods: deficiency from

194   100% in the EMP analysis (Acosta-Vigil et al., 2007), Raman spectroscopy (Bartoli et al., 2013a;

195 Ferrero et al., 2015) and secondary ion mass spectrometry (SIMS/NanoSIMS: Bartoli et al., 2013c,

196 2014). Using NanoSIMS and Raman spectroscopy, the H_2O concentration of glass can be determined

197 with a high spatial resolution of 1-2 μm. Bartoli et al. (2014) applied different methods to measure H_2O

198 in MI from amphibolite-facies migmatites and observed that, compared to NanoSIMS concentrations,

199 the difference of EMP totals from 100% generally yields slightly higher H_2O contents (up to

200 approximately 15% relative), whereas Raman spectroscopy underestimates H_2O (up to approximately

201 20% relative) when c[1]ompared to NanoSIMS values. Ferrero et al. (2015) reported lower differences

202 between H_2O values determined by Raman and EMP difference method (approximately 9% relative on

203 the average value).

204 By applying all these techniques, ≈600 MI –including re-melted nanogranitoids in migmatites

205 and granulites and glassy MI in anatectic enclaves– have been characterized to date (Fig. 4), leading to

206 the construction of an extensive geochemical database of MI in high-grade metamorphic rocks. In

207 detail, the compositional database consists of ≈600 and ≈200 major and trace elements analyses,

208 respectively, and ≈140 measurements of H_2O by either SIMS, NanoSIMS or Raman. The interested

209 reader may refer to the Supporting Information (Supplementary Tables 1[1] and 2[1]) and to Cesare et al.

210 (2015) for tables where all the presented and discussed analyses are reported.

211 The analysed MI come from three continents (Europe, Asia and Africa) and are related to

212 different geologic and geodynamic settings (Tab. 1) such as, i) the late orogenic, low-to-medium P

213 extensional setting represented by the Neogene Volcanic Province of southern Spain, ii) the low-P

214 dynamothermal aureole of Ojén (Betic Cordillera, S Spain), iii) the low- and medium-P collision-

215 related magmatism of the Maghrebian (La Galite, Tunisia) and Himalayan (Kali Gandaki, Nepal)

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[1] Deposit item AM-XXX, Supplemental Material. Deposit items are free to all readers and found on the MSA web site, via the specific issue's Table of Contents (go to http://www.minsocam.org/MSA/AmMin/TOC/).

216   mountain belts, respectively, iv) the low-to-medium P and ultrahigh-T regional metamorphism of the

217   Kerala Kondalite Belt (India) and v) the near ultrahigh-P metamorphism of the Bohemian Massif

218   (Orlica-Śniez nik Dome, central Europe). MI have been found in peraluminous metasedimentary

219   rocks and metaluminous metaigneous rocks, and are inferred to have formed at conditions varying from

220   670 to 950 °C and 4 to 27 kbar, from Proterozoic to Quaternary/Pleistocene (Tab. 1). A complete list of

221   published occurrences of MI in migmatites and granulites, along with a description of the host rock and

222   conditions of melting, is reported in Cesare et al. (2015). For the sake of completeness, it should be

223   noted that the research group of the writers has recently found MI also in Proterozoic and Archean

224   rocks from the Americas, as well as from Antarctica and Africa, but their microstructural and

225   microchemical characterization is still in progress.

226

227   **Reliability of the MI as records of melt compositions**

228   During and after entrapment of melt its composition may be modified by several processes, making the

229   MI unrepresentative of the bulk melt in the rock (Roedder, 1984). Many processes such as the

230   crystallization of the host on MI walls, the diffusional exchange of cations between the crystalline

231   phases within nanogranitoids and the host mineral, the nucleation of bubbles by volume contraction

232   and the retrograde fluid infiltration have been widely discussed by Acosta-Vigil et al. (2010, 2012),

233   Bartoli et al. (2013c, 2014) and Cesare et al. (2015). Here, we consider two processes, with which

234   igneous petrologists working with classic MI are more familiar: the boundary layer effect and diffusive

235   H_2O re-equilibration.

236   Boundary layers may develop at the crystal-melt boundary, when the rate of crystal growth is

237   faster than the rate of cation diffusion (Bacon, 1989). This results in a layer immediately adjacent to the

238   growing crystals that is generally depleted in compatible elements and enriched in incompatible

239   elements (Kent, 2008). If these boundary layer melts are trapped as MI, their chemical compositions

10

240   are obviously not representative of the bulk melt. It should be noted, however, that i) examples of

241   boundary layers have been mainly observed in experimental runs, whereas many studies on natural

242   classic MI do not report compositions affected by boundary layer phenomena –see review papers of

243   Kent (2008) and Audétat and Lowenstern (2014), and ii) experimental boundary layers commonly

244   formed at high degrees of undercooling (≈100-200 °C) around skeletal and dendritic crystals (e.g.

245   Faure and Schiano, 2005; Morgan and London, 2005b). In particular, Faure and Schiano (2005) have

246   demonstrated that the melt trapped in polyhedral crystals is representative of the parental melt, whereas

247   the composition of MI hosted in skeletal or dendritic forsterite crystals lies away from the predicted

248   liquid line of descent. It is important to stress again that MI in high-grade metamorphic rocks are

249   trapped along the heating path in polyhedral peritectic crystals (as opposed to the MI formation during

250   cooling/undercooling in crystallizing magmas). Results from a thorough study of primary MI hosted in

251   garnets of migmatites from the Ojén unit (Betic Cordillera, S Spain) and formed at increasing

252   temperatures (from ≈700 to ≈820 °C), show that MI compositions are mostly controlled by melting

253   conditions, and not by boundary layer systematics. Thus, Fe (compatible with respect to the host

254   garnet) and K (incompatible) are enriched in MI formed at higher temperatures, whereas low-

255   temperature MI have higher H and Na (both incompatible) (Bartoli et al., 2014, 2015). Therefore, the

256   boundary layer effect does not seem to have played an important role in shaping the geochemistry of

257   these trapped melts. Likewise, mass balances show that boundary layer phenomena do not control the

258   differences in major elements compositions among MI hosted in different minerals of anatectic

259   enclaves of SE Spain (Cesare et al., 2015). Regarding the trace element concentrations, MI in several

260   plagioclase and garnet crystals show remarkably similar concentrations in incompatible elements (e.g.

261   Li, Rb, Cs, B, Be, Zn, As, Zr, Th, U), indicating that boundary layer phenomena are negligible for these

262   elements. Conversely, the trace elements compatible or very slightly incompatible in the host (Sr, Ba

263   and Eu for plagioclase, and Y and HREE for garnet) are depleted in the MI due to interaction with the

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264   host phase (see Acosta-Vigil et al., 2012). In conclusion, the available data seem to indicate that MI are

265   representative of the bulk melt for the major and incompatible trace elements.

266   Recently, the reliability of olivine-hosted classic MI in preserving the pre-eruptive H_2O

267   contents of degassed lavas has been challenged by experimental studies, which have induced huge H_2O

268   variations in MI within olivine (e.g., from ≈3.8 wt% H_2O to ≈800 ppm; Hauri, 2002; Massare et al.,

269   2002; Portnyagin et al., 2008; Gaetani et al. 2012). On the other hand, Audétat and Lowenstern (2014)

270   have noted that most of the above experiments were done under unrealistic conditions and that many

271   studies on natural MI in olivine demonstrate that the original volatile content of the trapped melt can be

272   preserved after entrapment. Without entering in this "magmatic" dispute, we note that the two

273   contrasting mechanisms of primary MI entrapment (i.e. on the liquidus during crystallization of

274   magmas, and on or close to the solidus during incongruent melting of crustal rocks) along with the

275   contrasting P-T histories may result in different processes affecting the H_2O content of the trapped

276   melts. Compared to crystalline rocks which have experienced prograde anatexis, olivine-hosted MI

277   within magma batches represent the most suitable system for diffusive H_2O re-equilibration. Indeed,

278   during ascent of magmas, important gradients of H_2O concentration and pressure can be continuously

279   formed between the MI and the external magma. On the contrary, in the partially melted crust MI-

280   bearing peritectic phases coexist with discrete fractions of melt scattered in a mostly solid matrix. As

281   discussed by Bartoli et al. (2014), the large differences in physical parameters (e.g. temperature and, in

282   turn, hydrogen diffusivities, pressure and size of MI-bearing crystals) can result in times for the

283   diffusive H_2O re-equilibration which are >10 orders of magnitude greater in migmatitic and granulitic

284   terranes than in the plumbing of magmatic systems.

285   It might be argued that if crustal rocks undergo a long thermal history, reaching temperatures

286   much higher than the trapping temperatures and producing higher amounts of melt, H_2O contents in MI

287   could re-equilibrate with the external drier melt. In this case, three important observations must be

288   taken into account. Firstly, there is much field, microstructural and geochemical evidence indicating

289   that most of the crustal melts generated in the source area have been extracted (Brown, 2013).

290   Secondly, melt accumulation and extraction from anatectic rocks is expected to occur very early after

291   crossing the solidus and to continue during the prograde path, at very low melt fractions (<0.07;

292   Rosenberg and Handy, 2005; Jamieson et al., 2011), leaving residual granulites enriched in peritectic

293   minerals (Brown, 2013). Thirdly, very rapid rates of melt extraction have been reported in the

294   literature. For example, Harris et al. (2000) and Villaros et al. (2009) calculated residence times of S-

295   type granites at the source as short as 50 and 500 years, respectively. Therefore, high temperature

296   conditions do not implicitly mean that MI in residual migmatites and granulites experienced diffusive

297   H_2O re-equilibration. The successful re-homogenization of nanogranitoids at temperatures similar to

298   those inferred by phase equilibria calculations or thermobarometry, as reported by Bartoli et al. (2013a,

299   c) and Ferrero et al. (2015), strongly supports the idea that MI can behave as a closed system. Rather,

300   MI compositions could be compromised by fluid infiltration, hydration and retrogression occurring

301   during the subsolidus low-temperature evolution in crystalline basements. However, these processes

302   leave clear microstructural evidence such as the presence of low-temperature minerals within the

303   nanogranitoid inclusions like chlorite (replacing biotite) and pyrite (see Fig. 19 in Cesare et al., 2015).

304   All the above observations strongly suggest that the MI database corresponds to that of primary

305   anatectic melts.

306

Major and trace elements, and H_2O contents

307

308   In general, the compiled compositional database for MI in high-grade metamorphic rocks reports

309   compositions rich in SiO_2 (≈64-79 wt%; on a hydrous basis), with variable Al_2O_3 (≈10-18 wt%), Na_2O

310   (≈1-6 wt%) and K_2O contents (≈0-8 wt%) (Fig. 4). Most MI have very low to low-moderate

311    $FeO_t+MgO+TiO_2$ concentrations (≈0.25-2.5. wt%), whereas a small fraction of the dataset (≈10%)

<div align="center">13</div>

312   show values up to ≈4.0-5.0 wt%. CaO is low (<1.5 wt%), except for MI from Kali Gandaki where CaO

313   content can reach values as high as 4 wt% (Fig. 4). H_2O shows extremely variable concentrations (0-12

314   wt%) which are related to different P-T conditions and fluid regimes during crustal melting.

315   Regarding the trace elements, to date the dataset is limited to two case studies from S Spain, i.e.

316   crustal enclaves from El Hoyazo and migmatites from Ojén. Despite the different tectonic settings,

317   anatectic history and host rocks and crystals, the trace elements patterns show remarkably similar

318   characteristics when compared with the composition of the upper continental crust: MI are generally

319   enriched in elements (Cs, Rb, Li, Be and B) partially to totally controlled by muscovite and biotite, and

320   depleted in elements (Ba, Th, REE, Sr, Zr, Hf) hosted by feldspars and accessory phases (zircon and

321   monazite) (Fig. 5; see also Fig. 13 of Acosta-Vigil et al., 2012). Overall, MI from Ojen migmatites are

322   characterized by higher contents of Cs, Rb, B, U, Nb, Zr, Sm, V, Zn, lower contents of Ba and Pb, and

323   variable concentrations of Th, La and Ce with respect to MI in NVP enclaves (Fig. 5). This may reflect

324   differences in bulk host rock compositions and/or mechanisms of anatexis (i.e. different melting

325   reactions, different extent of equilibration between melt and residuum; see Acosta-Vigil et al., 2010,

326   2012).

327

328   One composition, different types of granitoids

329   Approximately 20 different classification schemes for granitoid rocks have been proposed in the last 40

330   years (Barbarin, 1999; Frost et al., 2001, and references therein). To describe the composition of crustal

331   magmas trapped in peritectic phases of high-grade metamorphic rocks, we have followed some of the

332   most commonly used classification schemes. Based on their normative compositions and considering

333   the compositional fields of O'Connor (1965), the majority of MI analyzed up to date correspond to

334   granites, whereas nanogranitoids from Kali Gandaki (Nepal) plot in the granodiorite, trondhjemite and

335   tonalite fields (Fig. 6). MI are generally slightly (ASI=1.0-1.1; ASI = mol. $Al_2O_3/(CaO+Na_2O+K_2O)$)

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336   to strongly peraluminous, with ASI up to 1.5 (Fig. 7). However, some proportion of the MI enclosed in

337   orthogneisses of the Bohemian Massif and tonalites of La Galite, are metaluminous or located at the

338   boundary between the metaluminous and peralkaline fields (ASI=0.8-1.0, alkalinity index up to -0.02)

339   (Fig. 7). Chappell and White (1974) proposed the first, modern geochemical scheme for the

340   classification of granitic (s.l.) rocks, and recognized two distinct granitoid types: the S-type

341   (supracrustal type) derived from metasedimentary rocks and the I-type (infracrustal type) inferred to

342   have formed from metaigneous sources (see also Chappell, 1984, 1999; Chappell and White, 1992,

343   2001). Considering their ASI, most analyzed MI correspond to S-type granitoids, with only 8% of the

344   MI plotting in the I-type field (Fig. 7). Remarkably, almost the totality of MI produced from anatexis of

345   metaigneous rocks correspond to I-type granitoid compositions (Fig. 7).

346   Frost et al. (2001) introduced a geochemical classification for granitoid rocks which has

347   achieved wide use (see also Frost and Frost, 2008). Using the compositional parameters suggested by

348   these authors, MI are mostly ferroan granitoids, from alkali-calcic to calc-alkalic, even though some

349   spread can be observed (Fig. 8). MI from Kali Gandaki rocks, instead, depart markedly from the other

350   crustal melts and plot in the calcic field (Fig. 8). As stated by Frost and Frost (2008), calcic

351   compositions are typical of melts produced by fluid-present melting.

352

Insights on the origins and evolution of granitoid magmas

Melt inclusions and the model (haplo)granite system

355   The origin of granitoid rocks has been strongly debated in the middle half of the past century by N.L.

356   Bowen and H.H. Read (see Bowen, 1948; Gilluly, 1948; Read 1948). This controversy ended a decade

357   later, when the magmatic origin of granite (s.l.) was definitely demonstrated via experimental petrology

358   by Tuttle and Bowen (1958). These authors showed that melting of a ternary mixture of quartz, albite

359   and orthoclase –i.e. a simple granitic system called haplogranite (Qtz-Ab-Or), starts at relatively low

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360   temperatures, typical of the continental crust, provided that the system is H_2O-saturated. After the

361   pioneering work of Tuttle and Bowen (1958), an impressive number of studies have been performed to

362   understand the effect of pressure and H_2O, and to a lesser extent of the other minor components, on the

363   phase relations of the haplogranitic system (Johannes and Holtz, 1996; see below). Also, whole-rock

364   data have been often compared to the eutectic/cotectic compositions of haplogranite system to identify

365   primary compositions of crustal melts (e.g. Olsen and Grant, 1991; Symmes and Ferry, 1995; Slagstad

366   et al., 2005) or to infer the depth of magma-storage (e.g. Blundy and Cashman, 2001; Almev et al.,

367   2012; Gualda and Ghiorso, 2013).

368   Because the haplogranitic system is a relevant reference frame to discuss granite petrogenesis,

369   and because MI in high-grade metamorphic terranes seem to provide the compositions of the primary

370   melts produced by melting of natural crustal rocks, it is important to see how MI data plot in the

371   pseudoternary normative Qtz-Ab-Or diagram. Here, granitoid MI from each locality show some spread

372   and a distinctive composition with respect to each other (Fig. 9a). They are commonly located towards

373   the center of the diagram, often in the Qtz field and above the corresponding cotectic lines at the

374   pressure of melting, between the H_2O-saturated eutectic point and the Qtz–Or join (Fig. 9a).

375   Considering the relatively wide compositional spectrum of the host rocks, it is clear from Figure 9a that

376   the concept of haplogranitic "minimum melt" composition finds little applicability in natural multi-

377   component systems, as even low temperature melts may significantly depart from eutectic

378   compositions. Indeed, natural rocks are more complex than a model system such as the haplogranite,

379   and additional components such as Ca, Ti, Fe and Mg play an important role in modifying the liquidus

380   and solidus phase relationships. For example, Wilke et al. (2015) noted that the effect of An on the

381   phase relationships of the haplogranite system is much more pronounced if small amounts of Fe and Ti

382   ($\approx$1 and $\approx$0.2 wt%, respectively) are present.

383   In Figure 9b we have summarized the effects of P, H_2O and other components (e.g. Al and Ca)

384   on the position of the minimum and eutectic points and of the cotectic lines in the ternary system Qtz-

385   Ab-Or, as reported in the literature. MI often cluster above the corresponding cotectic lines, which, in

386   accordance with experimental studies, suggests that excess Al and Ca play an important role in moving

387   the eutectic compositions and the position of cotectic lines towards the Qtz apex (Fig. 9a, b). Most MI

388   show some spread in the Qtz/feldspar ratio compared to their small variation in Ab/Or, forming trends

389   at relatively high angle with the cotectic line (Fig. 9a). This spread of MI data parallel to the Qtz-

390   feldspar direction suggests a control by diffusion in the melt for the major element compositions at the

391   time of MI entrapment (Fig. 9b). Indeed, it has been experimentally shown that the sluggish diffusion

392   of Si and Al versus rapid diffusion of alkalis in granite melts produced by partial melting of quartzo-

393   feldspathic protoliths result in linear trends in the Qtz-Ab-Or diagram (see Fig. 10 in Acosta-Vigil et

394   al., 2006).

395   The decrease of a_{H2O} moves the eutectic point towards the Qtz–Or join without affecting the Qtz

396   content (Fig. 9b). The majority of MI are located between the composition of the H_2O-saturated

397   eutectic and the Qtz–Or side. Although this shift towards higher Or/Ab ratios can be partly related to

398   the presence of Ca in natural systems (Fig. 9b), the collected data support the widespread idea that

399   anatexis and formation of crustal melts generally occur under H_2O-undersaturated conditions (e.g.

400   Thompson, 1990; Clemens and Watkins, 2001). It is important to note, however, that the absence or

401   low proportion of H_2O-saturated melts does not represent a proof for fluid-absent melting, as even

402   H_2O-fluxed melting is generally expected to produce final melts undersaturated in H_2O (Weinberg and

403   Hasalová, 2015b).

404   The reduction of the a_{H2O} during anatexis can be related to two different scenarios: i) melting

405   along a prograde path, in which all the free H_2O present in the rock below the H_2O-saturated solidus is

406   dissolved in the melt at the solidus and melting then continues through fluid-absent reactions; and ii)

17

407   presence of other components in excess fluids, like the carbonic species CO_2 and CH_4, that reduce the

408   activity of H_2O.. A clear example of scenario i) is represented by the Ojén MI. The peraluminous

409   metagreywacke of Ojén underwent prograde anatexis and MI in metatexites represent the first melt

410   produced immediately beyond the fluid-saturated solidus at ≈660-700 °C, ≈4.5-5 kbar (Bartoli et al.,

411   2013c), whereas MI in diatexites reflect the composition of melt formed at higher temperature, under

412   H_2O-undersaturated conditions (≈820 °C; Bartoli et al., 2015). In agreement with this evolution and

413   with the bulk rock composition, MI in diatexites show higher Or/Ab compared to MI in metatexites

414   (Fig. 9a; see also Bartoli, 2012), and the H_2O content of MI decreases from ≈7 to ≈3 wt % with

415   increasing temperature (Bartoli et al., 2015).

416   In scenario ii), the presence in the fluid of carbonic species characterized by low solubility in

417   granitoid melts can result in the phenomenon of immiscibility. As a matter of fact, cordierite, garnet

418   and plagioclase crystals from enclaves of the NVP (SE Spain) preserve spectacular microstructural

419   evidence of immiscible trapping (Fig. 3f), where granitic MI coexist with primary fluid inclusions

420   which are CO_2-dominated (>85 mol%), with minor amounts of N_2 and CH_4, and traces of CO and H_2

421   (see Cesare et al., 2007; Ferrero et al., 2011). Fluid-melt immiscibility is also documented in

422   metaluminous granitoids at La Galite, Tunisia (Ferrero et al., 2014). The origin of CO_2-rich fluids in

423   the deep crust is strongly debated (Santosh and Omori, 2008; Huizenga and Touret, 2012). In the

424   graphite-bearing enclaves from NVP the occurrence of CO_2 has been interpreted as internally generated

425   by Fe^{3+} reduction and graphite oxidation during incongruent melting of biotite (Cesare et al., 2005).

426   Conversely, infiltration of CO_2 from the mantle or from other crustal sources has been postulated for

427   rocks from La Galite (Ferrero et al., 2014).

428   MI from Kerala Khondalite Belt (India) plot very far from haplogranitic minimum melts, close

429   to the Qtz–Or sideline and point to melting temperatures well above minimum or eutectic temperatures,

430   in agreement with the ultra-high temperature (>900 °C) origin of the rocks and the very low H_2O

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431   content (≈0.7 wt%; Cesare et al., 2009) of the MI. Similar compositions have been obtained by

432   experimental melting of pelitic protoliths at 5-7 kbar, 900-950 °C (Fig. 9a). On the other hand,

433   nanogranitoids from Kali Gandaki (Nepal) are clearly displaced towards the Qtz–Ab sideline and

434   reflect the compositions of crustal melts produced by H_2O-fluxed melting (Carosi et al., 2015). The

435   latter inclusions show the highest H_2O contents (up to 12 wt% by EMP differences) and CaO, and the

436   lowest K_2O (Fig. 4), partly overlapping the composition of experimental glasses produced in the

437   presence of excess H_2O at variable P-T conditions (6-14 kbar, 675-775 °C; Fig. 9a). Indeed, the

438   presence of free H_2O decreases the plagioclase + quartz solidus more strongly than it depresses the

439   stability of micas, producing tonalite/granodiorite/trondhjemite melts (Weinberg and Hasalová, 2015a

440   and references therein). Melt inclusions from the orthogneiss of the Bohemian Massif, re-homogenized

441   at 875 °C and 27 kbar, show the highest content of normative Ab (Fig. 9a) explained by melting at near

442   ultrahigh-P conditions (increasing pressure moves haplogranitic eutectics closer the Ab apex; Fig. 9b).

443   Experimental melts produced at 24-30 kbar, 870-950 °C by melting of different crustal protoliths plot

444   in the same area (Fig. 9a; Ferrero et al., 2015).

445

446   **Characterizing the melting processes at the source**

447   Granitoid MI in high-grade metamorphic rocks are providing more and more robust and consistent

448   information on the chemistry of natural crustal melts (see above). In addition, they represent a new tool

449   to constrain mechanisms (i.e. melting reactions, melting mode, extent of equilibration with the solid

450   residue) and conditions (P-T-a_{H_2O}) of formation of crustal magmas, and hence open a new window into

451   the anatectic history of the partially melted continental crust (Fig. 1; Acosta-Vigil et al., 2010).

452   An example of this novel approach is the study of enclaves from El Hoyazo (Neogene Volcanic

453   Province, SE Spain; Acosta-Vigil et al., 2007, 2010, 2012). These rocks represent fragments of

454   continental crust which underwent partial melting and extraction of about 30-60 wt% of granitic melt

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455   (Cesare et al., 1997; Cesare, 2008). Here, melt (undevitrified glass owing to rapid cooling of the host

456   dacitic lava) has been chemically investigated both within plagioclase and garnet (see compilation of

457   Fig. 5) and in the rock matrix where it often occurs along foliation planes (i.e. the equivalent of

458   leucosomes in regional migmatites and granulites). Melt inclusions show the highest concentrations of

459   Li, Cs and B, whereas the matrix glass is characterized by higher contents of FRTE (first row transition

460   elements), Y, Zr, Th, REE and higher Rb/Cs and Rb/Li (Fig. 10a; see Acosta-Vigil et al., 2010). The

461   concentrations of Zr and LREE were used to calculate the zircon and monazite saturation temperatures

462   (Watson and Harrison, 1984; Montel, 1993), in order to link MI and potential melt-producing reactions.

463   This approach represents an important advance in granitoid petrology, as the initial temperature of

464   magmas at the source has been long considered to be an inaccessible parameter for allochthonous

465   granites (see Miller et al., 2003). Melt inclusions in plagioclase yield the lowest temperatures (≈665-

466   715 °C), whereas MI in garnet (≈685-750 °C) and matrix glass (≈695-800 °C) show higher

467   temperatures (Fig. 10b). Because the analyzed melts seem to be undersaturated to some extent in the

468   accessory phases (Acosta-Vigil et al., 2012), the obtained temperatures have to be considered as

469   minimum estimates (Miller et al., 2003; Acosta-Vigil et al., 2010). In summary, based on the

470   composition of MI within several hosts and matrix glasses, calculated accessory mineral saturation

471   temperatures, and microstructural relationships, it was concluded that the deep crust beneath the

472   Neogene Volcanic Province of SE Spain underwent rapid heating and melting: MI in plagioclase reflect

473   the earliest granitic melts produced by fluid-present to fluid-absent muscovite melting, whereas MI in

474   garnet were produced simultaneously to slightly later via fluid-absent melting of muscovite (Fig. 10c).

475   In contrast, the generation of granitic melts characterized by higher contents of FRTE and REE (the

476   matrix melt) occurred at higher temperatures by biotite dehydration-melting reactions (Fig. 10c). In all

477   cases melt and residual minerals were at, or close to, equilibrium with respect to most trace elements,

478   except for garnet, and some undersaturation in accessory minerals (Acosta-Vigil et al., 2012).

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479

The "starting-point" composition

In order to understand which granitoid rocks reflect primary crustal melts and, subsequently, what

changes crustal magmas have undergone either near the source region or during their journey towards

Earth's surface, "starting-point" compositions are required (Sawyer et al., 2011). Because leucosomes

commonly do not represent primary melts (Sawyer, 2008 and discussion therein), a widespread

approach among crustal petrologists is to assume the composition of experimental glasses as

representative of crustal melt composition at the source, and to use such composition in mass balance

calculations to track the processes involved in magma evolution and crustal differentiation (e.g.,

Milord et al., 2001; Solar and Brown, 2001; Barnes et al., 2002; Guernina and Sawyer, 2003; Stevens

et al., 2007; Sawyer, 2008; Hacker et al., 2011).

The fundamental advance provided by the study of nanogranitoid MI in high-grade

metamorphic rocks is that they make accessible the composition of the natural primary anatectic melt

for the specific rock under investigation at its specific P-T-X_{H2O} conditions (Bartoli et al., 2013a;

Ferrero et al., 2015; this work). Despite the number of MI analyses is already significant (see above),

the compiled MI database should be expanded to cover other potential sources of crustal magmas (e.g.

intermediate to mafic protoliths) and a wider spectrum of P-T-a_{H2O} conditions under which continental

crust may melt (i.e. to date the number of MI case studies is still quite low compared to the number of

experimental studies). However, the already significant MI dataset collected so far can be already

useful to start discussing some of the previously proposed inferences and models regarding crustal

melting and the formation and evolution of granitoid magmas. Clearly, in order to make inferences on

petrogenetic processes affecting anatectic magmas, and in the absence of specific primary melt

compositions from MI for their particular study area, authors should consider using the MI

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502   compositions in the published dataset for the conditions (P-T-X_{H2O}-bulk rock composition) closest to

503   the particular rocks that they are investigating.

504   Figure 11a-d provides the compositional variations of MI in terms of mol% of Fe_{Tot}+Mg+Ti,

505   Na+Ca, K and Si+Al. The major crystalline phases are reported along with MI data. These types of

506   diagrams have been previously used to model crustal reworking by anatexis (see Solar and Brown,

507   2001; Barnes et al., 2002; Korhonen et al. 2010; Farina et al., 2015; Yakymchuk et al., 2015). Host

508   rock compositions form different clusters according to their nature (for example, metapelites show the

509   highest Fe_{Tot}+Mg+Ti values whereas metagreywackes are enriched in Si and K); MI plot away from

510   the Fe_{Tot}+Mg+Ti apex, into the bottom half of the diagrams at variable (Na+Ca)/K and

511   (Na+Ca)/(Si+Al) ratios, providing a slightly variable though distinctive melt composition (Fig. 11a-d).

512   This is in agreement with the fact that the composition of the source and conditions of anatexis play a

513   major control on melt chemistry.

514   The Himalayan Leucogranites (HL) are commonly considered to represent nearly pure melts

515   produced by low-temperature (≈700 °C) melting of metapelites and metagreywackes (Le Fort et al.,

516   1987). As a matter of fact, most HL overlap with the low-temperature MI (i.e those hosted in garnet

517   from the Ronda metatexites and in garnet and plagioclase of NVP enclaves), supporting their primary

518   nature (Fig. 12e, f). Some HL compositions, however, do not resemble MI compositions and plot in the

519   proximity of the lower portion of the field occupied by most MI data (Fig. 11e, f). It is important to

520   note that the difference in mafic components (Fe, Mg and Ti) between HL and MI cannot be related to

521   contamination of MI by the host phase during analysis, as MI hosted in Fe-, Mg- and Ti-free hosts such

522   as plagioclase show similar and sometimes higher Fe+Mg+Ti contents with respect to MI hosted in

523   garnet (Fig. 11b, d). On one hand, the observed discrepancy can be related to an incomplete MI dataset

524   (i.e. incomplete sampling of MI in high-grade metamorphic rocks). On the other hand, it could indicate

525   a non-primary nature of some HL. For instance, Scailett et al. (1990) stated that some geochemical

526 features of Gangotri leucogranites (for which compositions sometimes depart from those of MI; Fig.

527 11e) can be explained by fractionation of the early crystallizing phases. Their compositions often

528 depart from the compositions of primary HL in the opposite direction of the Bt and Ms fields (Fig. 11e,

529 f). As a matter of fact, some Gangotri leucogranites have lower Co, V and B contents than MI in Ronda

530 metatexites and lower Li content than MI in garnet of NVP, in agreement with a mica fractionation

531 process. It should be noted that petrographic and experimental evidence indicate that biotite was likely

532 the first mineral to crystallize in many HL (Scaillet et al., 1995). Therefore, the available MI data seem

533 to support a primary nature for the majority of HL, even though fractional crystallization processes

534 might have occurred locally, in agreement with results from previous studies (i.e. Le Fort et al., 1987;

535 Scaillet et al., 1990). It is important to note that MI from Kali Gandaki (Nepal) represent Ca-rich melts

536 produced during Eocene prograde metamorphism (Carosi et al., 2015; Iaccarino et al., 2015) and,

537 therefore, they are not related to the Miocene HL formed during exhumation stage (Harris and Massey,

538 1994).

539   The Layos Granite (Toledo Complex, central Spain) represents a S-type, strongly peraluminous

540 Crd-bearing granitoid suite, varying from quartz-rich tonalite to melamonzogranites and formed at

541 about 800-850 °C by biotite melting of metapelites (Barbero and Villaseca, 1992). Most of these rocks

542 plot away from MI datasets, towards the $Fe_{Tot}+Mg+Ti$ apex (Fig. 11e, f). The authors explained the

543 observed compositional variability of the suite by a "restite unmixing" model (Barbero and Villaseca,

544 1992). This model was proposed by Chappell et al. (1987) to explain the geochemical variability of

545 granitoid rocks and states that the majority of granites represent mixtures between a low-T melt and the

546 restite, i.e. solid materials that were residual from the source. Therefore, granites were thought to image

547 their source in a simple way: compositional variations observed within granitoid suites reflect varying

548 degrees of restite unmixing with a minimum or near-minimum temperature (haplogranitic) melt

549 (Chappell et al., 1987). However, this model has received several criticisms from the geochemical,

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550 petrographic and physical point of view (see Wall et al., 1987; Clemens and Mawer, 1992; Clemens,

551 2003; Vernon, 2010; Clemens and Stevens, 2012 for additional details). The fact that the composition

552 of the primary melt produced in the source may differ markedly from that of minimum melt, as clearly

553 demonstrated by MI study (see discussion above), demonstrates that the assumption that the melt

554 component has an eutectic composition is not applicable in natural systems. Recently, Stevens et al.

555 (2007) and Clemens and Stevens (2012) proposed a revised version of the restite unmixing model,

556 called "peritectic assemblage entrainment" or "selective peritectic phase entrainment". Following this

557 model, the more mafic granitoid rocks result from the entrainment of the solid peritectic products of the

558 melting reaction to the melt (although how this would occur is not fully explained), rather than from the

559 entrainment of a proportion of the total solid mineral assemblage remaining after the melting reaction.

560 Regarding the Layos Granite, only one data point plots in the MI field (overlapping the composition of

561 MI trapped in Crd from metapelites and formed at ≈850°C; Fig. 11e, f) and, in turn, seems to reflect a

562 primary origin. The rest of data plots between this primary melt composition and the Fe_{Tot}+Mg+Ti apex

563 and does not form a trend towards the (Si+Al)/10 apex. Similarly, in the (Fe_{Tot}+Mg+Ti)–(Si)–(Al)

564 diagram (not shown), Layos granites do not show compositional trends towards the Si and Al apexes.

565 According to the model proposed by Stevens et al. (2007), this chemical signature can be explained by

566 differential entrainment of peritectic cordierite (up to 30% of Crd has been documented in these rocks),

567 without the involvement of residual Qtz and Als, i.e. the restite unmixing model does not seem to be

568 applicable in this specific case.

569 The Malani rhyolites (western India) are considered to be the product of high-T crustal anatexis

570 (Maheshwari et al., 1996), and the fact that the majority of lava compositions match those of MI

571 supports their primary origin (Fig. 11c). Strongly peraluminous, K-rich Malani rhyolites resemble the

572 glassy MI from Kherala Khondalite Belt, in agreement with the inference that the rhyolites were likely

573 produced by melting of metapelites at temperatures ≥ 850 °C (Maheshwari et al., 1996). At the same

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574   time, the comparison with MI would suggest a more complex scenario for the genesis of these anatectic

575   lavas. Malani rhyolites have variable (Na+Ca)/K ratio and some lava compositions overlap the

576   compositions of MI found in NVP metapelitic enclaves, Ronda metatexites and La Galite granodiorite

577   and formed at variable temperatures (≈700-820 °C) (Fig. 11c). These lavas range from peraluminous,

578   through metaluminous to peralkaline. Notably, almost the totality of metaluminous/peralkaline lavas

579   approach the metaluminous/peralkaline compositions of MI from La Galite granodiorite. The observed

580   compositional variability can reflect the presence of distinct magma batches in the Malani rhyolite

581   complex, formed and extracted during prograde heating of a compositionally heterogeneous crustal

582   source. In this sense, other work has verified the occurrence of a heterogeneous crystalline basement

583   (composed of both granodiorites, orthogneisses and metasedimentary rocks) beneath Malani rhyolites

584   (Pandit et al., 1999; Sharma, 2004). Instead a single composition seems to reflect the entrainment of

585   mafic minerals (Fig. 11c).

586   In Figure 12 we have compared the composition of MI with a dataset of ≈520 experimental glasses

587   reported in literature and produced at variable P-T-a_{H2O} conditions and using different starting

588   materials, i.e., metasedimentary and felsic (meta)granitoid protoliths. Remarkably, MI and glasses from

589   experiments seem to be characterized by similar compositions and compositional ranges, even though

590   experimental melts may show higher Fe_{Tot}+Mg+Ti values than MI (Fig. 12). Koester et al. (2002)

591   reported glasses, produced at 700°C and 5-15 kbar, characterized by anomalously high FeO, TiO_2 and

592   MgO contents (e.g., FeO≈9.5 wt%; see red dots in Fig. 12), in contrast with their low-temperature

593   origin. The authors argued that the lack of chemical equilibrium was responsible for the heterogeneous

594   chemical compositions of melt observed in near-solidus experiments (Koester et al., 2002; see also

595   Gardien et al., 1995). Indeed, as any other petrologic tool, partial melting experiments may suffer from

596   potential pitfalls, in particular: impossibility of bulk compositional changes (in experiments there is no

597   H_2O and melt loss), slow kinetics of reactions, loss of iron from silicate phases to experimental

598 capsules, lack of equilibration, overstepping of melting reactions and difficulty of obtaining reliable

599 glass analyses in near-solidus experiments (Ratajeski and Sisson, 1999; Gardien et al., 2000; White et

600 al., 2011; Webb et al., 2015). Nonetheless, when the low-temperature ($\leq$ 720 °C) experiments are

601 considered, with the exception of the analyses reported in Koester et al. (2002), the produced melts

602 show low and variable Fe_{Tot}+Mg+Ti contents, overlapping the compositions of low-temperature MI

603 (Fig. 12).

604   From all the above considerations, it is clear that MI are an useful tool with which to discuss the

605 petrogenesis of granitoid rocks, and can complement experimental glasses to constrain differentiation

606 processes occurring in anatectic magmas. Researchers working on petrogenesis of crustal granitoids

607 can now look for and study MI in migmatites and granulites considered to be the source region of the

608 studied magmas, or in peritectic garnet entrained in the granitoid rocks in order to fix the real primary

609 composition for their specific case study. In particular, as the number of experimental studies presently

610 reporting trace element composition of melt is limited, MI are an important contribution that provides

611 the trace element compositions of natural anatectic melts.

612

Implications

613

614 Nanogranitoid melt inclusions in high-grade metamorphic rocks are a novel subject of research and are

615 becoming a matter of considerable scientific interest, as they allow the analysis *in situ* of the natural

616 primary melts produced by crustal melting. The size of nanogranitoid MI (commonly 10 μm in

617 diameter) and of daughter minerals crystallized in them (often ≤1 μm) pose analytical challenges, and

618 the accurate re-homogenization and compositional characterization of these small data repositories is

619 far from being routine. However, we have shown here that, albeit slowed down by many experimental

620 and analytical difficulties, the study of these MI opens new horizons for metamorphic and igneous

621 petrology. Technological improvement in the coming years will make the study of MI easier, and we

622 believe that their chemical characterization, in particular of volatiles, trace elements and radiogenic

623 isotopes, will provide exciting new results in many topics, such as granitoid petrogenesis, UHT and

624 UHP metamorphism, secular variations of felsic magmatism, volatile recycling, crust formation and

625 silicate Earth differentiation.

626 **Acknowledgements**

637

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1062

1063 **Figure captions**

1064 FIGURE 1. Schematic T-t diagram showing the main processes occurring during melt production

1065 (heating or prograde path) and melt consumption (cooling or retrograde path) in the partially melted

1066 continental crust. MI can be trapped during incongruent melting (arrow 1) and igneous crystallization

1067 (arrow 5). Mineral abbreviations after Kretz (1983). Ideal incongruent melting reaction from Vielzeuf

1068 and Holloway (1988).

1069

1070 FIGURE 2. Photomicrographs of primary nanogranitoid inclusions. (**a**) Garnet porphyroblasts from a

1071 partially melted pelitic enclave from El Hoyazo, SE Spain (additional information in Cesare et al.,

1072 1997). MI are distributed in zonal arrangements (red arrows). (**b**) Peritectic garnet in porphyroblastic

1073 gneisses from the anatectic sequence of Jubrique, Betic Cordillera, S Spain (see also Barich et al.,

1074 2014). Square: area enlarged in (c). (**c**) Close-up of inner part of the garnet crystal in (b) where clusters

1075 of micrometric inclusions are present. The presence of MI clusters with random distribution

45

1076   unequivocally demonstrates their primary nature. (**d**, **e**) Plane-polarized and crossed polars images

1077   respectively of the crystallized inclusion marked by a red arrow in (c). Multiple birefringent phases are

1078   recognizable within the inclusion.

1079

1080   **FIGURE 3.** SEM backscattered images (**a-e**) and plane-polarized light photomicrograph (**f**) of the

1081   microstructures of melt inclusions. (**a**) Fully crystallized nanogranite in khondalites (India). (**b**) Fully

1082   crystallized MI from Bohemian Massif. In this case, polymorphs have been identified by Raman

1083   spectroscopy (Ferrero et al., 2016). (**c**) Partially crystallized inclusion with internal porosity (red

1084   arrows, respectively) from Ronda metatexites. (**d**) Glassy MI in anatectic enclaves from the Neogene

1085   Volcanic Province, SE Spain. (**e**) Coexistence of glassy and crystallized MI in Ronda diatexites. (**f**)

1086   Cluster of associated fluid and melt inclusions (red and yellow arrows), indicative of fluid

1087   immiscibility in a graphite-bearing crustal enclaves.

1088

1089   **FIGURE 4.** Harker diagrams showing the major element concentrations (wt%) of melt inclusions in

1090   crustal enclaves and in migmatites and granulites (data from Supplementary Tables 1^1 and 2^1 of

1091   Supporting Information). (**a**) SiO_2 *vs.* Al_2O_3. (**b**) SiO_2 *vs.* Na_2O. (**c**) SiO_2 *vs.* K_2O. (**d**) SiO_2 *vs.*

1092 $FeO_t+MgO+TiO_2$. (**e**) SiO_2 *vs.* CaO. Numbers in parentheses indicate the number of analyses for each

1093   case study.

1094

1095   **FIGURE 5.** Chondrite-normalized trace element patterns (normalizing values from Sun and

1096   McDonough, 1989). (**a**) Normalized trace element patterns for melt inclusions in crustal enclaves of

1097   NVP, SE Spain (from Acosta-Vigil et al., 2010). (**b**) Normalized trace element patterns for melt

1098   inclusions in migmatites from Ojen, S Spain (Bartoli, unpublished data). The average upper continental

1099   crust composition is reported for comparison (data from Rudnick and Gao, 2014).

46

1100

1101   **FIGURE 6.** An-Or-Ab diagram with fields for granite, quartz-monzonite, granodiorite, trandhjemite and

1102   tonalite after O'Connor (1965). Symbols as in Figure 4.

1103

1104   **FIGURE 7.** Plot of ASI [aluminum saturation index = molar $Al_2O_3/(CaO+Na_2O+K_2O)$] *vs*. AI [alkalinity

1105   index = molar $Al_2O_3-(Na_2O+K_2O)$; see Frost and Frost, 2008]. Dotted line separates the compositional

1106   fields of I- and S-type granites s.l. (from Chappell and White, 1974; Chappell, 1999). Symbols as in

1107   Figure 4.

1108

1109   **FIGURE 8.** Geochemical classification of crustal melts following Frost et al. (2001) and Frost and Frost

1110   (2008). (**a**) Fe-index [$FeO_T/(FeO_T+MgO)$] *vs*. SiO_2 (wt%). (**b**) MALI (modified alkali-lime index =

1111 $Na_2O + K_2O – CaO$) *vs*. SiO_2 (wt%). Data on a volatile free basis. Symbols as in Figure 4.

1112

1113   **FIGURE 9.** (**a**) CIPW Qtz-Ab-Or diagram showing the normative compositions of all analyzed

1114   nanogranitoid melt inclusions. Symbols as in Figure 4. Black dots and lines show the eutectic points

1115   and cotectic lines for the subaluminous haplogranite system at $a_{H_2O} = 1$ and different pressures (Holtz

1116   et al., 1992; Luth et al., 1964; Huang and Wyllie, 1975). References to experimental glasses: PDJ91,

1117   950 °C, 7 kbar (Patiño Douce and Johnston, 1991); D03, 900 °C, 5 kbar (Droop et al., 2003); GC03,

1118   675-775 °C, 6-14 kbar, H_2O-present (Garcia-Casco et al., 2003); PDH98, 700-775 °C, 6-10 kbar, H_2O-

1119   present (Patiño Douce and Harris, 1998); SF15: 870-950 °C, 16-30 kbar (see Ferrero et al., 2015). (**b**)

1120   Normative Qtz-Ab-Or diagram showing the displacement of eutectic points and cotectic lines as a

1121   function of P, a_{H_2O}, An and Al_2O_3 contents. Black dots and lines as in (a). Light blue arrow: effect of

1122   increasing pressure (Johannes and Holtz, 1996 and references therein). Red arrows: effect of decreasing

47

1123 a_{H_2O} at 2, 5 and 10 kbar (white dots are eutectic points at different a_{H_2O}; Ebadi and Johannes, 1991;

1124   Becker et al., 1998). Violet arrow: effect of increasing Al content at 2 kbar and a_{H_2O}=0.5, i.e. from a

1125   subaluminous to peraluminous system (Holtz et al., 1992). Green arrow: effect of increasing An

1126   content at 2 kbar and a_{H_2O}=0.5. With the addition of An the phase diagram changes to a eutectic system

1127   with respect to the haplogranite system with a minimum (see green lines; Wilke et al., 2015). Double

1128   grey arrow: effect of diffusive transport properties of the melt on the composition of experimental

1129   glasses (Acosta-Vigil et al., 2006). It should be noted that the addition of significant amounts of B and

1130   F to the system (>1wt%) moves eutectic points and cotectic lines towards Ab-rich compositions

1131   (Johannes and Holtz, 1996). However, the effects of B and F are not reported here because these

1132   elements are present in very low amounts in the investigated MI (< 0.1 wt%; Acosta-Vigil et al., 2007).

1133   See text for details.

1134

1135   **FIGURE 10. (a)** Variation diagram showing the trace element contents for glassy MI and matrix glass in

1136   the crustal enclaves hosted in El Hoyazo dacite (NVP, SE Spain). **(b)** Zircon and monazite saturation

1137   temperatures calculated for all analyzed glasses of enclaves. Symbols as in (a). Diagonal line marks the

1138   geometrical location of points where both temperatures coincide. **(c)** Prograde history of melt formation

1139   in the continental crust beneath the Neogene Volcanic Province of SE Spain, estimated by accessory

1140   mineral saturation temperatures (redrawn after Acosta-Vigil et al., 2010). The relevant experimentally-

1141   determined melting reactions in the granitic and metapelitic systems are reported. HW81 refers to

1142   Huang and Wyllie (1981), P76 to Peto (1976), PDH98 to Patiño Douce and Harris (1998), PDJ91 to

1143   Patiño Douce and Johnston (1991) and VH88 to Vielzeuf and Holloway (1988).

1144

1145   **FIGURE 11.** Compositions (in mol%) of analyzed nanogranitoid melt inclusions in terms of

48

1146 $(Fe_{Tot}+Mg+Ti)$, $(Na+Ca)$, $(Si+Al)/10$ and K (after Solar and Brown, 2001; Barnes et al., 2002). The

1147   compositional fields of major crystalline phases and host rocks are reported. Bulk compositions of

1148   crystalline rocks are from Bartoli (2012), Groppo et al. (2012), Ferrero et al. (2014, 2015) and

1149   Iaccarino et al. (2015). Data for KKB rocks are not available. In the case of El Hoyazo samples, both

1150   chemical compositions of i) Grt-Bt and Crd-Sp enclaves and ii) low-grade metapelites from the

1151   Alpujarride basement (considered to approximate the source composition before melt extraction) are

1152   plotted (data from Cesare et al., 1997). (**a, b**) $(Fe_{Tot}+Mg+Ti)$–$(Na+Ca)$–K diagrams for MI from

1153   crystalline rocks (a) and from crustal enclaves (b). (**c, d**) $(Fe_{Tot}+Mg+Ti)$–$(Na+Ca)$–$(Si+Al)/10$ diagrams

1154   for MI from crystalline rocks (c) and from crustal enclaves (d). (**e, f**) Comparison between MI and

1155   crustal magmas. Manaslu leucogranites: data from Le Fort (1981, 1987) and Guillot and Le Fort

1156   (1995). Gangotri leucogranites: data from Scaillet et al. (1990). Everest/Masang Kang leucogranites:

1157   data from Visonà et al. (2012). Layos granite: data from Barbero and Villaseca (1992). Manali

1158   rhyolites: data from Maheshwari et al. (1996) and Sharma (2004). Yellow stars containing black dots

1159   indicate metaluminous or peralkaline Malani rhyolites. See text for details.

1160

1161   **FIGURE 12.** Comparison among the $Fe_{Tot}+Mg+Ti$, $(Na+Ca)$, $(Si+Al)/10$ and K concentrations (in

1162   mol%) of melt inclusions and glasses from partial melting experiments. Experimental glasses were

1163   produced at T=650-950 °C, P=1-50 kbar, variable a_{H2O} and from metasedimentary and felsic ($SiO_2 \geq 62$

1164   wt%) (meta)granitoid protoliths. Data from: Conrad et al. (1988); Le Breton and Thompson (1988);

1165   Vielzeuf and Holloway (1988); Brearley and Rubie (1990); Patiño Douce and Johnston (1991); Holtz

1166   and Johannes (1991); Skjerlie and Johnston (1993); Icenhower and London (1995, 1996); Gardien et al.

1167   (1995, 2000); Patiño Douce and Beard (1995, 1996); Singh and Johannes (1996); Montel and Vielzeuf

1168   (1997); Patiño Douce and Harris (1998); Pickering and Johnston (1998); Castro et al. (1999);

1169   Litvinovsky et al. (2000); Koester et al. (2002); Nair and Chacko (2002); García-Casco et al. (2003);

1170   Droop et al. (2003); Grant (2004, 2009); Schmidt et al. (2004); Spicer et al. (2004); Lopez et al. (2005);

1171   Tropper et al. (2005); Acosta-Vigil et al. (2006); Auzanneau et al. (2006); Watkins et al. (2007);

1172   Hermann and Spandler (2008); Ward et al. (2008); Ferri et al. (2009). Compositions of melt inclusions

1173   and major crystalline phases as in Fig. 11. Red dots: experimental glasses produced at 700°C and 5-15

1174   kbar (from Koester et al., 2002). Dashed black lines reflect the maximum Fe_{Tot}+Mg+Ti contents

1175   reported for low-temperature (650-720 °C) experimental glasses, with the exception of analyses

1176   reported in Koester et al. (2002). See text for details.

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1178

1179

1180   **TABLE 1.** Occurrences of nanogranitoid melt inclusions discussed in the text.

Locality	Host rocks	Host minerals	T, P conditions	Reference
Neogene Volcanic Province (NVP), SE Spain	Metapelitic enclave	Grt, Pl, And, Crd, Ilm	≈615-850, ≈2-7 kbar	1, 2, 3, 4, 5, 6
Ojén, Ronda, S Spain	Quartzo-feldspathic metatexite	Grt	≈660-700, ≈4.5-5 kbar	7, 8
Ojén, Ronda, S Spain	Quartzo-feldspathic diatexite	Grt	≈820, ≈6-6.5 kbar	9
La Galite, Tunisia	Tonalitic and garnetitic enclave	Grt	≈770-820, ≈5 kbar	10
Barun, Nepal Himalaya	Gneiss	Grt	≈800-860, ≈8 kbar	11
Kerala Khondalite Belt (KKB), S India	Khondalites	Grt	≈900, ≈6-8 kbar	11, 12
Orlica-Śnieżnik Dome, Bohemian Massif	Felsic granulite	Grt	≈875, ≈27 kbar	13

1181

1182   1: Acosta-Vigil et al. (2007); 2: Acosta-Vigil et al. (2010); 3: Acosta-Vigil et al. (2012); 4: Cesare et al. (2003); 5: Cesare et al. (2007);
1183   6: Ferrero et al. (2011); 7: Bartoli et al. (2013a); 8: Bartoli et al. (2013b); 9: Bartoli et al. (2015); 10: Ferrero et al. (2014); 11: Ferrero et
1184   al. (2012); 12 Cesare et al. (2009); 13: Ferrero et al. (2015).

T

Solidus

Metasedimentary continental crust

t
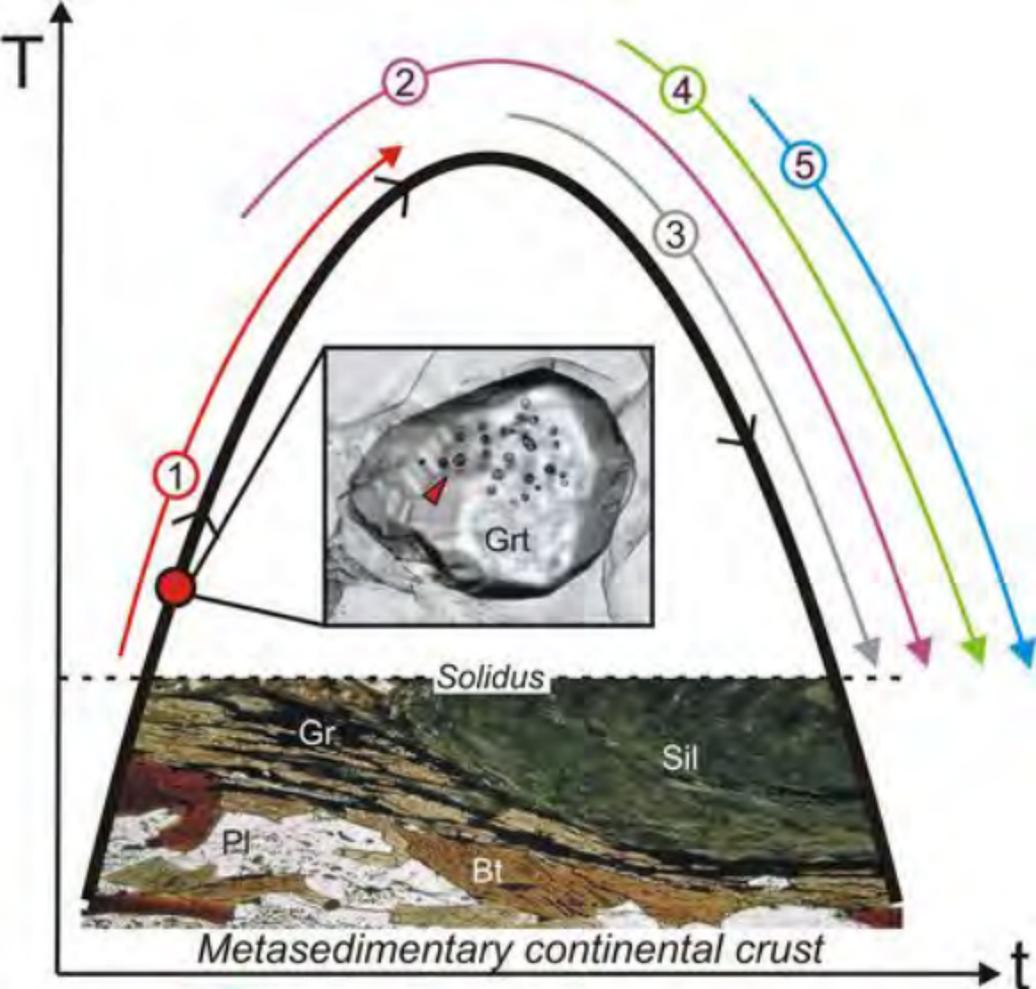
① Melt formation and MI entrapment
Bt + Sil + Pl + Qtz = Grt + melt (± Kfs)

② Melt segregation

③ Magma crystallization and differentation

④ Volatile degassing

⑤ Entrapment of "classic" MI

(a) Bt, Grt, Pl, Kfs, Kfs, Qtz — 1μm

(b) Bt, Kfs/Kok, Grt, Qtz/Crs, Kml — 5μm

(c) Ms, Bt, Grt, Qtz, glass, Kfs, glass — 1μm

(d) Grt — 5μm

(e) crystallized MI, glass — 5μm

(f) Crd — 10μm

Migmatites/granulites

MI in garnet

- ◯ Ojen met. *(31)*
- ● Ojen diat. *(38)*
- ◯ Kerala Kondalite Belt *(14)*
- ● La Galite *(23)*
- ☐ Barun *(4)*
- ■ Kali Gandaki *(18)*
- ◆ Bohemian Massif *(18)*

Crustal enclaves

- MI in Pl *(247)*
- MI in Grt *(78)*
- MI in Crd *(61)*
- MI in And *(31)*
- MI in Ilm *(18)*

(a)

Melt inclusion/chondrite

MI in Pl (20-24)
MI in Grt (10-32)
Average upper continental crust

Cs Rb Ba Pb Li Be B Th U Nb Ta La Ce Sr Nd Sm Zr Hf Eu Ti Gd Dy Y Er Yb Lu V Zn Cu Cr Sc Co Ni

(b)

Melt inclusion/chondrite

MI in Grt, Ojen metatex. (3-81)
MI in Grt, Ojen diatex. (4-64)
Average upper continental crust

Cs Rb Ba Pb Li Be B Th U Nb Ta La Ce Sr Nd Sm Zr Hf Eu Ti Gd Dy Y Er Yb Lu V Zn Cu Cr Sc Co Ni

An

Ab   Or

10   30

50   50

granodiorite

tonalite   70

qtz-monzonite

30

granite

trondhjemite   90

90   70   50   30   10

(a) Ternary diagram with Qtz–Ab–Or apices. Labels include GC03, PDJ91, PDH98, SF15, D03, and pressure isobars at 5 kbar, 10 kbar, 20 kbar, 30 kbar.

(b) Ternary diagram with Qtz–Ab–Or apices. Labels include "filtrate-controlled composition", "increasing norm. An", "ASI>1", "decreasing a_{H_2O}", "increasing P", with pressure isobars at 2 kbar, 5 kbar, 10 kbar, 20 kbar, 30 kbar.

Figure (c) P–T diagram with insets (a) Rb/Cs (ppm) vs Th (ppm) and (b) T_{Zrn} (°C) vs T_{Mnz} (°C).

Labels in diagram:
- P(kbar) — vertical axis
- T(°C) — horizontal axis
- Ky
- Sil
- HW81 — Qtz+Pl+Kfs+Ms+H$_2$O melt
- P76 — Ms+Pl+Qtz+H$_2$O / Als+melt
- PDH98 — Ms+Pl+Qtz / Kfs+Sil+Bt+melt
- PDJ91 — Bt+Pl+Sil+Qtz / Grt+melt
- VH88 — Bt+Pl+Sil+Qtz / Grt+melt+(Kfs)
- MI in Pl
- MI in Grt
- matrix glass
- host dacite

Inset (a): Rb/Cs (ppm), Th (ppm)
- MI in Pl
- MI in Grt
- Matrix glass

Inset (b): T_{Zrn} (°C), T_{Mnz} (°C)

Starting material:
● metasedimentary
○ (meta)granitoid

(a) Fe$_{Tot}$+Mg+Ti

700 °C, 5-15 kbar (Ko02)

Na+Ca — K

(b) Fe$_{Tot}$+Mg+Ti

700 °C, 5-15 kbar (Ko02)

Na+Ca — (Si+Al)/10