

# Aquamarine from Massangana batholith, Rondônia State: mineral chemistry and fluid inclusion data

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## Abstract

Beryl is usually found in granite-pegmatite systems. The addition of chromophore elements (V, Cr, Mn, Fe) into the crystalline structure favors color changes in beryl and thus generates some of the world's expensive gems such as emerald,morganite, heliodor and aquamarine. The Massangana polyphasic batholith is a well-known cassiterite, wolframite and gems deposit in the Rondônia state. These metals and blue-gems (topaz and aquamarine) are located in feldspar-rich pegmatite granite bodies. The aquamarine crystals show color ranging from light- to medium-blue and display concentric growth zones. Electron-probe microanalyses revealed that the Fe is the main chromophore element, occupying the octahedral Al-site, while Na had an important role in the charge balance, inserted in the channel sites together with H<sub>2</sub>O. The irregular supply of Fe and Na during the nucleation and growth of aquamarine was the main cause for the color change. A fluid inclusion study indicated that the aquamarine growth under an aqueous fluid system (H<sub>2</sub>O-NaCl), with low salinity, low density and total homogenization temperature between 243°-315° C, is compatible with final temperatures in the pegmatite pocket zone.

**Keywords:** aquamarine, Massangana batholith, Rondônia, electron probe micro-analysis (EPMA), mineral chemistry, microthermometry, fluid inclusion.

## 1. Introduction

Gem-quality beryl is usually found in fractionated-granitic or granite-pegmatite systems. Beryl is a cyclosilicate with its simplest chemical formula  $\text{Be}_3\text{Al}_2[\text{Si}_6\text{O}_{18}]$ , organized in hexagonal rings of Si-tetrahedra structure parallel to (0001), cross-linked together both by Be-tetrahedra and Al-octahedra sites, forming a three-dimensional framework. This crystalline structure has channels parallel to the c-axis that may host alkali,  $\text{H}_2\text{O}$  and  $\text{CO}_2$  molecules, favoring complex replacement by other cations, such as  $\text{Fe}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Mg}^{2+}$  and  $\text{Li}^+$  (Morosin, 1972; Aurisicchio *et al.*, 1988; Sherriff *et al.*, 1991; Artioli *et al.* 1993). The addition of the chromophore elements (Cr, V, Mn, Fe) into

the Al-structure favors color changes in beryl (Hawthorne and Huminicki, 2002), and thus generating some of the world's expensive gems such as emerald (Cr, V),morganite (Mn), heliodor (Fe, Mn) and aquamarine (Fe).

Brazil is a world's producer of aquamarine, whose main deposits are in Minas Gerais, Espírito Santo, Bahia, Paraíba, Rio Grande do Norte and Rondônia states (CPRM 2007, Barreto and Bittar 2010). In the Rondônia state, the Massangana batholith is a well-known cassiterite and wolframite deposit, as well as producer of some gems (mainly topaz and beryl), which have been exploited applying an artisanal

mining-type known as “garimpo”. In this deposit, the metals and gems occurs within pegmatites and hydrothermal veins, but also occur in paleo-alluvial deposits. Despite these gems being known and traded over time, information about the gemological quality and mineralogical properties are still insufficient or inexistent (e.g. Souza *et al.*, 2003; Debowski *et al.*, 2013). Herein, we report on chemistry and fluid inclusion compositions of aquamarine, a blue variety of beryl from Massangana batholith, thus adding information about the mineralogical characteristics and the physical-chemical nature of the paleofluids trapped in this gem.

## 2. Analytical methods

All analytical procedures were performed at the laboratories at the Geoscience Institute of the Brasília University (IG-UnB). The studied samples were cut in tablets, with orientations parallel and perpendicular (basal section) to the c-axis. A thin section cut was made according to the basal plane of the sample for petrographic and electron probe microanalysis (EPMA). In addition, two double-polished thin sections (around 1 mm thickness), cut according to the basal plane and to the c-axis, were prepared for the study of fluid inclusions.

The chemical composition of aquamarine was obtained through

electron probe microanalysis techniques (EPMA). A JEOL JXA-8230 micro-analyzer with five coupled wavelength dispersive spectrometers (WDS) was used. The analytical conditions applied were: accelerating voltage of 20 kV, beam current of 40 nA, beam diameter of 1–2  $\mu\text{m}$ , and counting times of 15 and 10 s for peak and background positions, respectively. The data reduction was performed with the ZAF program and the following standards were used: microcline (K, Al, Si), albite (Na), andradite (Ca, Fe), forsterite (Mg), pyrophanite (Mn, Ti), chromite (Cr) and pollucite (Cs). The results are reported as wt.% oxide, and the number of ions

in mineral formula were calculated on the basis of 3 Be and 18 O atoms per formula unit (apfu). For  $\text{H}_2\text{O}$  content calculation, the equation proposed by Marshall *et al.*, (2016) was applied.

The microthermometric measurements were carried out using a LINKAM THMS-600 heating-freezing system coupled to an Olympus BX-51 petrographic microscope with 10x and 50x long distance objectives. The calibration-stage was performed using synthetic fluid inclusion standards, applying speed rates from 10° to 5° C/min, with an estimated accuracy of  $\pm 0.3^\circ\text{C}$  for the freezing (+25° to -100°C) and  $\pm 5^\circ\text{C}$  for the heating (up to 400°C).

## 3. Geological setting

In the State of Rondônia occurs several Meso- to Neoproterozoic rapakivi granitic associations (Fig. 1a), emplaced during successive magmatic episodes between 1606 and 974 Ma, which make up the so-called Rondônia Tin Province (Kloosterman, 1968; Priem *et al.*, 1966 and 1971; Leal *et al.*, 1978; Isotta *et al.*, 1978; Bettencourt *et al.*, 1999; CPRM, 2007). However, the most significant metal concentrations (Sn, W, Nb, Ta) and gems are associated to A-type highly-fractionated magmatic episodes, occurred between 1314 and 974 Ma, and are represented by São Lourenço-Caripunas (1314-1309 Ma), Santa Clara (1082-1074 Ma) and Rondônia (995-974 Ma) intrusive suites (Bettencourt *et al.*, 1999).

The Massangana batholith belongs

to the Rondônia Suite, and intrudes the Paleoproterozoic basement rocks of the Jamari Complex (Isotta *et al.*, 1978; CPRM, 2007). It is a polyphasic granitic system with outstanding contrast through remote sensor and aerogeophysical products, marked by its ESE-WNW elliptical shape with ring-fault structure (Fig. 1b), and related to the successive magmatic phases (Kloosterman, 1967; Priem *et al.*, 1971; Okida, 2001; CPRM, 2007). Four alkaline magmatic phases have been recognized (Fig. 1b): Massangana, Bom Jardim, São Domingos and Taboca, which are close in age (1096 – 993 Ma). The Massangana is the main and oldest phase composed of coarse rapakivi biotite-alkali-feldspar granite. The Bom Jardim and São Domingos phases are intrusive in the Massangana

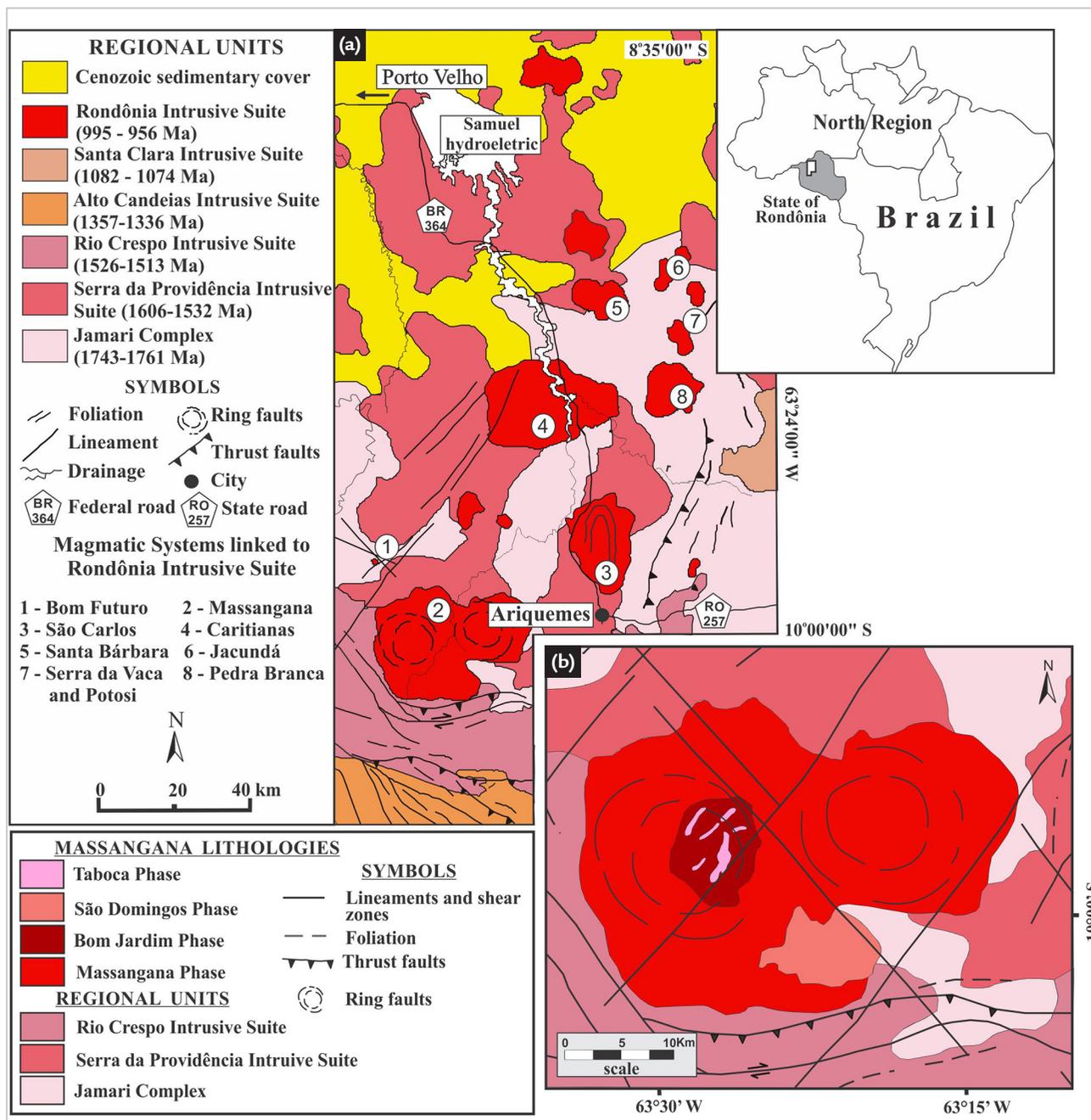
phase and composed of inequigranular biotite granites. Taboca is the youngest phase. It is composed of inequigranular syenite to quartz-syenite apophyses and intrudes the Bom Jardim phase (Priem *et al.*, 1966; Romanini, 1982; Bettencourt *et al.*, 1999; CPRM, 2007; Debowski, 2016).

Blue-gems, topaz and aquamarine, are located in feldspar-rich pegmatite granite bodies, related to the Bom Jardim and São Domingos phases. The pegmatites are often found within the contact zone between granite and Jamari Complex wall rocks, but may also occur in the granite. They are morphologically tabular to irregular shape, texturally simple or asymmetrically zoned, show medium to coarse grained, and their thickness and continuity are variable

(Fig. 2a). Generally, these pegmatites present the following zonal sequence: a cortex of elongated biotite crystals radially arranged or in comb texture, marking the wall- or border-zone. The intermediate zone is dominant and composed of K-feldspar (partially altered to

kaolin) quartz, mica (biotite) and Na-plagioclase (also argilized) inequigranular aggregates, whose crystals reach a size up to 1.5 cm. In this zone, there also occur disseminated crystals of fluorite, topaz, cassiterite, wolframite, columbite-tantalite and some sulfides. In the core-

or pocket-zone, which is lenticular, the blue-gems (topaz and aquamarine) occur normally associated with quartz, cassiterite, wolframite, columbite-tantalite and fluorite. In this place, the mineral assemblage may reach sizes above 5 cm, normally embedded in kaolin.



#### 4. Chemistry data

The aquamarine is represented by euhedral to subhedral short to long crystals (Fig. 2b), slight to moderate fractured, with concentric growth zones and color ranging from light- to medium-blue. Frequently, the growth zones host some micro-inclusions (mainly feldspars and mica), but opaque

and clay minerals also occur (Fig. 2c).

Representative EPMA analyses for 30 different spots are summarized in Table 1. However, in Figure 2c, only part of these analyses are indicated (15 in total), which can be used to illustrate and verify the chemical variations inside the aquamarine

crystal. The stoichiometric calculation results identified in Table 1 are applied due to the difficulty in obtaining EPMA accurate analytical results for Be and H<sub>2</sub>O (Groat *et al.*, 2002). Therefore, the sum of the oxides goes to below 100 wt.% (commonly between 97 and 99 wt.%).

Table 1 - Intervals of EPMA analysis of the 30 different spots obtained in aquamarine crystal. The number of ions in mineral formula were calculated on the basis of 3 Be and 18 O.

Elements	Average	Minimum	Maximum
SiO <sub>2</sub> (wt.%)	66.99	65.87	67.89
Al <sub>2</sub> O <sub>3</sub>	16.96	16.64	17.38
TiO <sub>2</sub>	0.02	0.00	0.07
FeOtot.	0.82	0.50	1.36
Cr <sub>2</sub> O <sub>3</sub>	0.02	0.00	0.13
MgO	0.01	0.00	0.03
MnO	0.02	0.00	0.09
CaO	0.01	0.00	0.03
Na <sub>2</sub> O	0.18	0.00	0.45
K <sub>2</sub> O	0.01	0.00	0.04
Cs <sub>2</sub> O	0.01	0.00	0.03
BeO*	13.72	13.55	13.90
H <sub>2</sub> O**	1.07	0.00	1.75
Total	98.74	97.51	99.94
Si (apfu)	6.09	6.06	6.13
Al	1.82	1.79	1.88
Ti	0.00	0.00	0.01
Fe	0.06	0.04	0.10
Cr	0.00	0.00	0.01
Mg	0.00	0.00	0.00
Mn	0.00	0.00	0.01
Ca	0.00	0.00	0.00
Na	0.03	0.00	0.08
K	0.00	0.00	0.01
Cs	0.00	0.00	0.01
Be*	3.00	3.00	3.00

The meaning of the symbols: \* = stoichiometric calculations, \*\* = stoichiometric calculated using the equation  $H_2O = (0.5401 \times \ln Na_2O) + 2.1867$  (Marshall *et al.*, 2016), and \*\*\* = ideal value for stoichiometric calculations.

The microanalyses were performed on a section perpendicular to the c-axis of the aquamarine crystal, following concentric growth zones (from edge to core) through profiles with spots approximately equidistant to each other (some of the analyses are shown in the Figures 2c and 2d). EPMA results revealed that the most important chromophore element for aquamarine is the FeO<sub>total</sub> (Fe<sup>3+</sup> and Fe<sup>2+</sup>), followed by some content trace of TiO<sub>2</sub>, MnO and Cr<sub>2</sub>O<sub>3</sub> (Fig. 2d), which are close or below to the detection limit. Furthermore, it is important to note the

irregular and low Na content, as well as the very low K, Ca and Mg contents (between 0 - 0.04 wt.%). A tendency for positive correlation between Fe+Mg and Na is observed (Fig. 2d and 2e), indicating the importance role of these compensating alkali ions. However, it is possible to note important variations on the Fe and Na contents: remarkable loss of Na at the edges, followed by progressive enrichment in the intermediate zone and ending with decreasing of Fe and Na in the central part of the aquamarine crystal. On the other hand,

there is a tendency for negative cationic correlation between Al and the sum of Fe + (Mg, Ti, Cr, Mn), which indicates that the major replacement process of these elements takes place within Al-octahedra site (Fig. 2f). The other trace elements with chromophore function (*i.e.*, Ti, Cr, Mn) have negligible participation (content below 0.10 wt.%), but it is probable that some content of these trace elements also was accommodated to the Be-tetrahedra site (*e.g.*, Aurisicchio *et al.*, 1988; Hawthorne and Huminicki, 2002).

## 5. Fluid inclusion data

The petrographic study, at room temperature ( $\pm 2.5^\circ$  C), identified only aqueous primary and secondary/pseudo-secondary fluid inclusions. The primary fluid inclu-

sions occur isolated or in small groups, frequently followed the concentric growth zone, with size between 10-75  $\mu$ m, composed of mono- and biphasic morphol-

ogy. On the other hand, the aquamarine crystals exhibit some microfractures or deformation features that contain fluid inclusions less than 10  $\mu$ m in size and

showed monophasic morphology. These fluid inclusion types were classified as

secondary or pseudo-secondary. This study addresses only the primary fluid

inclusions, which display adequate sizes for microthermometric observations.

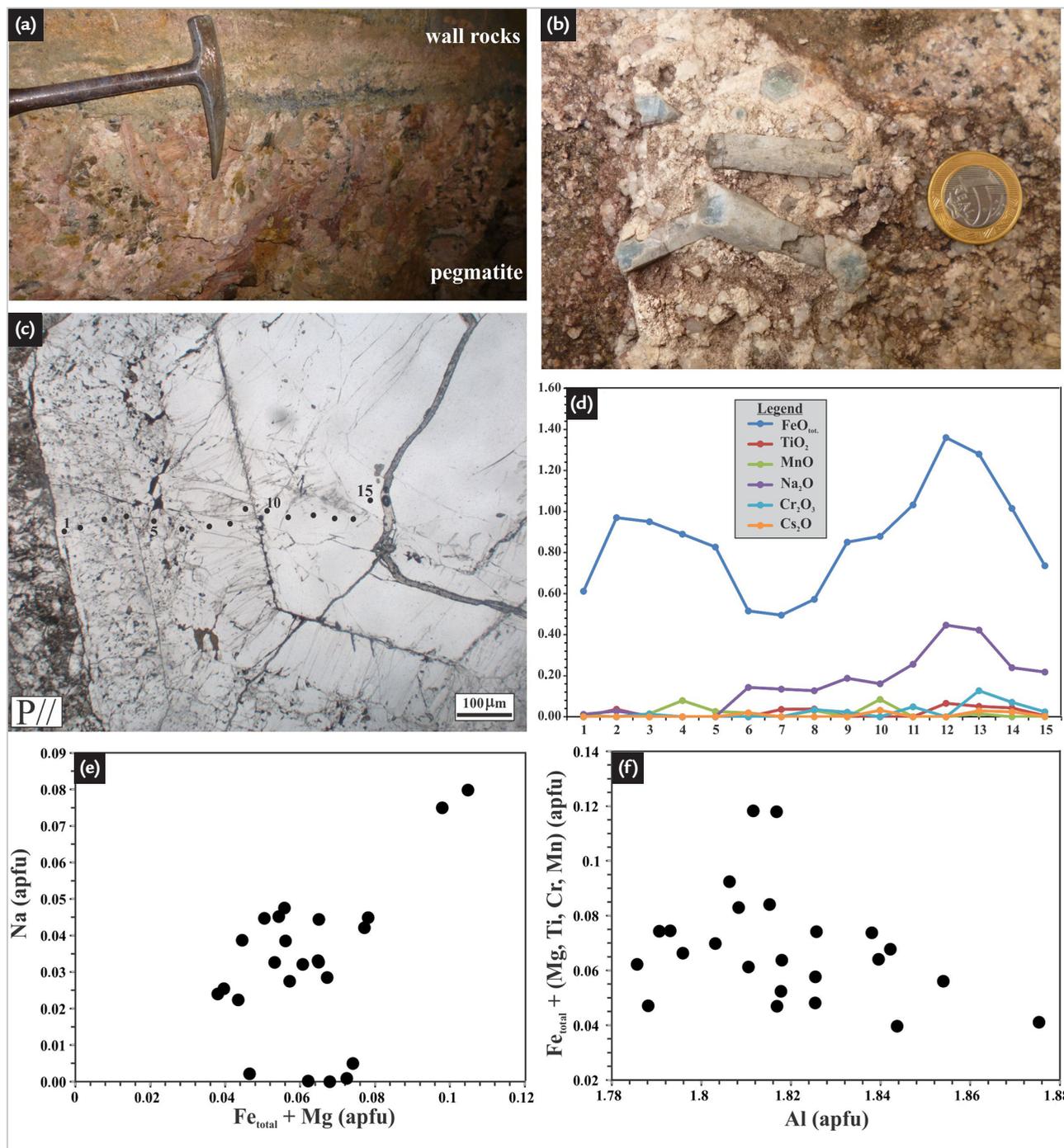


Figure 2 - a) Contact zone between tabular zoned pegmatite body and Jamari Complex wall-rocks; b) Euhedral short to long aquamarine crystals within granitic pegmatite, showing concentric growth zones and irregular coloration; c) photomicrography of a concentrically zoned aquamarine crystal. Note several micro-inclusions distributed mainly along the growth lines, as well as a set of electron-microprobe analyses along a crystal profile from edge to core (P// = parallel polars); d) Weight percent data for some selected oxides in aquamarine along a traverse. Note the tendency for positive correlation between  $\text{Fe}_2\text{O}_{3\text{total}}$  and  $\text{Na}_2\text{O}$ ; e) Cationic correlation between  $\text{Fe}_{\text{total}} + \text{Mg}$  vs  $\text{Na}$  (in *apfu*); and f) Cationic correlation between  $\text{Al}^{3+}$  vs sum of  $\text{Fe}_{\text{total}} + (\text{Mg}, \text{Ti}, \text{Cr}, \text{Mn})$  (in *apfu*).

## 5.1 Petrography

Two morphological types of primary aqueous fluid inclusions were identified and divided into: type 1 and type 2:

- Type 1 is most common and shows sizes between 40-75  $\mu\text{m}$ . It is composed of two immiscible phases: liquid-rich inclusions with a vapor

bubble (liquid + vapor). The liquid phase shows colorless to slightly gray color and low birefringence, while the vapor phase exhibits dark gray to black color. The vapor/liquid volumetric ratios (F factor) vary from 10 to 25%. However, morphological variations lead to

subdivision of type 1 into: 1a and type 1b. The type 1a shows sub-rounded to irregular shapes with  $F = 10\text{-}20\%$  (Fig. 3a). Occasionally, type 1a occurs hosting a solid phase, characterized by sub-rounded opaque micro-crystals with a size smaller than 5  $\mu\text{m}$  (Fig. 3b).

On the other hand, the type 1b presents elongated to cylindrical shape with F = 20-25%, usually parallel to the c-

axis (Fig. 3c).

• Type 2 are monophasic, composed by a liquid phase. It shows a sub-

rounded to elliptical shape, colorless to slightly gray color, low birefringence and size below 10 μm (Fig. 3d).

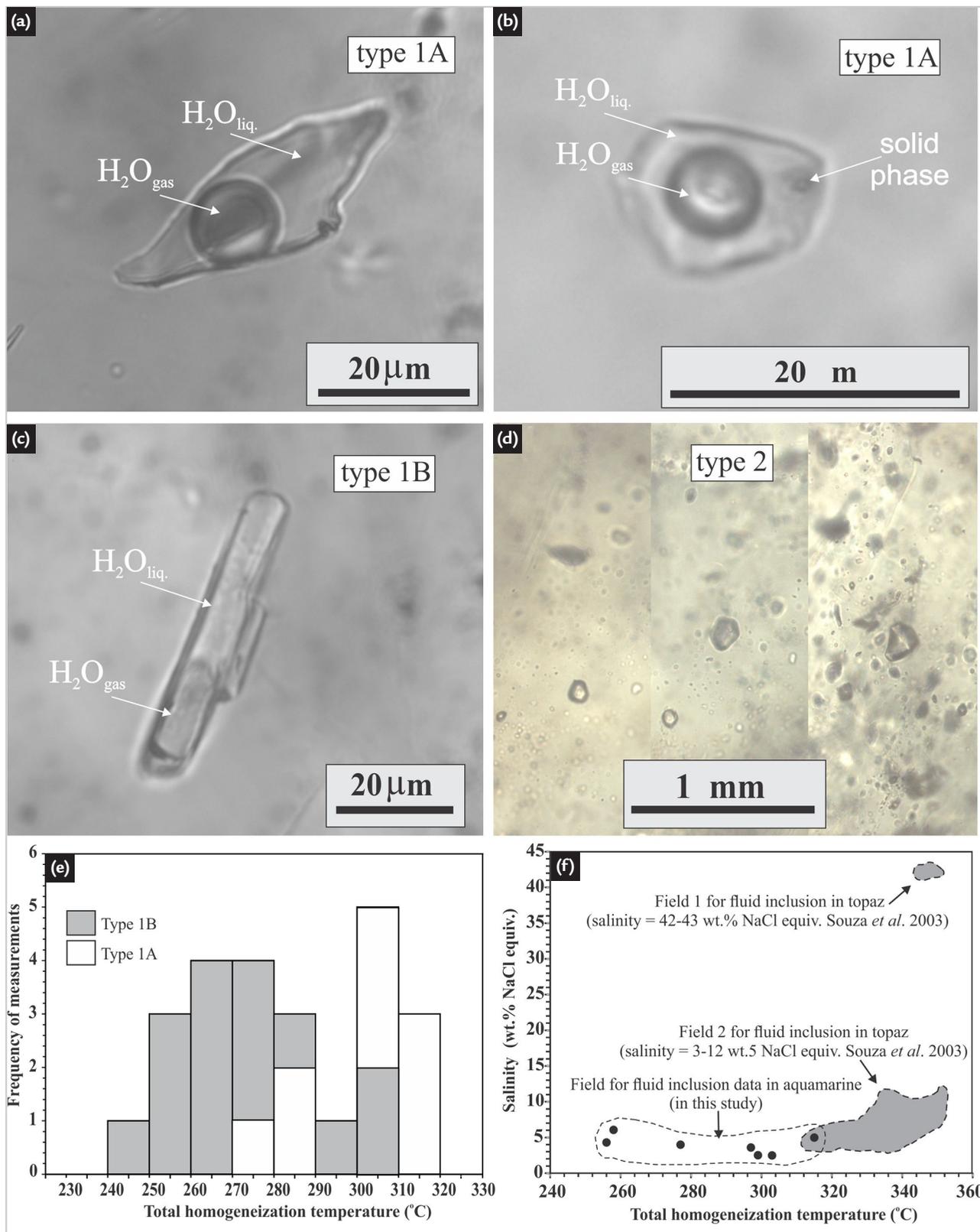


Figure 3 - Morphological features of main aqueous fluid inclusion types identified in the aquamarine crystals. a) and b) Morphological variation from type 1a aqueous fluid inclusions. Note in b, the type 1a sub-rounded shape and hosting an opaque micro-crystal (solid phase); c) Type 1B with cylindrical shape often found parallel to the c-axis; d) Type 3 fluid inclusion with sub-rounded to elliptical shape; e) Frequency histogram of total homogenization temperatures (T<sub>tot</sub>) of aqueous fluid inclusions at aquamarine (types 1a and 1b); and f) Total homogenization temperatures vs salinity diagram applied to fluid inclusions data obtained in aquamarine. For a simple comparative analysis on the fluid inclusion data in gems from Massangana pegmatites available in literature. We have also included in this diagram the fluid inclusion data obtained from topaz and reported by Souza *et al.* (2003).

## 5.2 Microthermometry

Approximately 20 fluid inclusions were analyzed. During the to-freezing stage (below  $-100^{\circ}\text{C}$ ), it was observed that the vapor + liquid phases make up an aqueous fluid system ( $\text{H}_2\text{O}_{(\text{vapor})} + \text{H}_2\text{O}_{(\text{liquid})}$ ). In this context, only a few type 1 and type 2 fluid in-

clusions showed appropriate size for observing the change phase for eutectic and ice final melt temperatures. The eutectic temperature was estimated between  $-22.2^{\circ}\text{C}$  and  $-20.1^{\circ}\text{C}$ , while the ice final melt ( $T_{\text{m ice}}$ ) varied from  $-2.2^{\circ}\text{C}$  to  $-1.1^{\circ}\text{C}$  (Tab. 2). These data

point for a  $\text{H}_2\text{O}$ -NaCl fluid system (e.g., Shepherd *et al.*, 1985; Bodnar and Vityk, 1994). The salinity was estimated from  $T_{\text{m ice}}$ , which varies from 6 to 2.5 wt.% NaCl equivalent, after applied the equation proposed by Bodnar (1993).

Table 2 - Microthermometric data summary on fluid inclusions obtained in aquamarine.

Fluid inclusion types	Microthermometric Intervals			
	T eut. ( $^{\circ}\text{C}$ )	T <sub>m</sub> . Ice ( $^{\circ}\text{C}$ )	T <sub>h</sub> total ( $^{\circ}\text{C}$ )	Salinity (% wt. NaCl equiv.)
Type 1A	-22.2 to -20.1	-2.2 to -1.1	258.4 - 210.4	2.5 to 6.0
Type 1B			243.2 - 314.8	
Type 2	-21.4 to -20.4		-	

During the heating stage, the contraction of the vapor phase occurred until blending into the liquid phase ( $\text{L} + \text{V} \rightarrow \text{L}$ ) at the total homogenization temperatures ( $T_{\text{htot.}}$ ) between  $243^{\circ}\text{C}$ - $315^{\circ}\text{C}$  (Fig. 3e). On the other

hand, the final melt temperatures of solid phase from type 1B fluid inclusion were not measured, due to crepitation of some fluid inclusions at temperatures close to  $400^{\circ}\text{C}$ . Probably this solid phase corresponds to minerals captured

together with liquid-gas mixtures from hydrothermal solutions during formation of cavities. The fluid density was estimated around  $0.95\text{ g/cm}^3$ , on base  $T_{\text{htot.}}$  vs salinity correlation (Shepherd *et al.*, 1985).

## 6. Discussion

The color changes in beryl crystals are directly connected to the entrance in the crystalline structure of chromophore elements in the Al-octahedra site (Hawthorne and Huminicki, 2002). However, the geological setting has an important role in this chemical-replacement mechanism, which can be linked to fractional crystallization and metasomatism processes, for example (e.g., Simmons and Webber, 2008; Groat *et al.*, 2008). The beryl-aquamarine variety linked to fractional crystallization of granitic-pegmatite phase is a result of nucleation and growth of crystals controlled by temperature decrease. In association, the entrance of Fe within the Al-bearing site is the main mechanism responsible for the blue color (Beal and Lentz, 2002; Viana *et al.*, 2002; Groat *et al.*, 2010).

Aquamarine from Massangana batholith has the Fe as the main chromophore element. The negative correlation between Al vs Fe  $\pm$  (Mg, Ti, Cr, Mn) associated to positive correlation between Fe + Mg vs Na, indicates that the major cationic replacement mechanism occurred within the octahedral Al-site. On the other hand, alkali ions (mainly Na, followed by some K and Ca content) and  $\text{H}_2\text{O}$  enter in the channel sites, positioning between the six-membered rings of Si tetrahedral of beryl structure, thus maintaining the

charge balance (Sampaio Filho *et al.*, 1973; Aurisicchio *et al.*, 1988; Sherriff *et al.*, 1991; Groat *et al.*, 2002).

The growth of aquamarine from Massangana batholith is marked by internal variations in color and chemical composition. The reasons for these variations are unclear. However, it is probable that some type of geochemical imbalance within the environment of growth leads mainly to lack of or irregular supply of Fe and Na. This imbalance then favored color (from light blue to blue) and chemistry oscillation, respectively. According to Aurisicchio *et al.*, (1988), zoning can occur because of chemical restrictions of the environment (bulk-rock chemistry and fluid-phase composition) or exchange reactions with other minerals, which can be influenced by changes in pressure, temperature and pH parameters. On the other hand, based on x-ray diffraction, Mössbauer, infrared and UV-visible spectra data, previous studies have shown that color change in beryl may be linked to charge imbalance created during the growth, relative to the proportion of  $\text{Fe}^{3+}$  in the octahedral sites and of  $\text{Fe}^{2+}$  in the channel sites (Viana *et al.*, 2002; Groat *et al.*, 2010).

$\text{H}_2\text{O}$ -rich fluid phases are common in fractionated granitic melts emplaced at the upper crust (Bodnar, 1995; Roedder

and Bodnar, 1997). Thermometry data indicate that the final temperatures in pegmatite pocket zones range from  $390^{\circ}\text{C}$  to  $240^{\circ}\text{C}$  (London, 1992; Johnson *et al.*, 2002). Previous studies on the fluid inclusion in topaz, reported by Souza *et al.* (2003), associated with fluid inclusion data of aquamarine presented in this study, corroborated with the essentially aqueous ( $\text{H}_2\text{O}$ -NaCl) nature of the paleofluids in the Massangana pegmatites. However, important microthermometric differences between these paleofluids can be observed. For example: paleofluids from topaz show different intervals of salinity (3-12 and 42-43 wt.% NaCl equiv.), density ( $0.65$ - $0.75$  and above  $1.2\text{ g/cm}^3$ ) and  $T_{\text{htotal}}$  between  $320^{\circ}\text{C}$ - $350^{\circ}\text{C}$ , indicating a fluid mixing process during evolution and cooling (Souza *et al.*, 2003). On the other hand, paleofluids from the aquamarine show low salinity (2.5 - 6 wt.% NaCl equiv.), density around  $0.95\text{ g/cm}^3$  and  $T_{\text{htotal}}$  between  $315^{\circ}\text{C}$ - $243^{\circ}\text{C}$ . These microthermometric data indicate that for topaz, this is linked to the fluids with variable salinity and higher temperatures, while for aquamarine, it is tied to fluids with lower salinity and temperature (Fig. 3f). Therefore, it is likely that the temperature decrease associated with the pressure drop and decrease in salinity

in pegmatites pocket zone (e.g., London, 2008), accompanied by mixing with oth-

ers aqueous fluids from the upper crust (e.g., Hedenquist 1995), were important

mechanisms in the crystallization history of the Massangana pegmatites.

## 7. Concluding remarks

The data presented in this study led us to the following conclusions:

- The aquamarine is linked to the fractional crystallization in the granitic-pegmatites phase. It is the result of nucleation and growth of crystals in the pocket zone, controlled by temperature decrease and marked by a concentric zoning and irregular color;

- The microthermometry data indi-

cate that the aquamarine growth under an aqueous (H<sub>2</sub>O-NaCl) fluid system with low salinity, low density e total homogenization temperature between 243°-315° C, is compatible to final temperatures in pegmatites pocket zone.

- Fe is the main chromophore element, which replaces the octahedral Al-site. This can be demonstrated by a negative correlation between Al and Fetotal.

On the other hand, the alkalis (mainly Na), occupy the channel sites together with H<sub>2</sub>O, and have an important role in the charge balance;

- The internal variations in color and chemical composition in aquamarine are mainly attributed to some type of geochemical imbalance that led to the lack or irregular supply of Fe and Na within the environment of growth.

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