


# Age and petrogenesis of the beryl-bearing granitic magmatism of the Velasco Pegmatite District, Pampeana Province, NW Argentina

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## ABSTRACT

Two porphyritic granitoids (Huaco and Sanagasta) in the Velasco district of the Pampeana Pegmatite Province in Northwestern Argentina are recognized. They are considered the fertile granites of the beryl-bearing pegmatites and can be described as post-orogenic and peraluminous A-type granites formed in an intraplate tectonic setting during an extensive regime, whose magma source is predominantly of cortical origin and to a lesser extent, mantle-derived. The pegmatites are classified as Rare Elements of the beryl type and beryl-columbite-phosphate subtype, while the entire district shows characteristics related to the NYF (Nb-Y-F) petrogenetic family. From rocks and cogenetic minerals of an individual 'Ismiango' pegmatite of the Velasco district, two Rb/Sr isochrons have been constructed. They define an age of  $330.3 \pm 8.3$  and  $331.7 \pm 2.3$  Ma and fall in the Lower Carboniferous period, consistent with the age of the parental and host-rock, the Huaco granite. As the Ismiango pegmatite has a similar composition and structure to the other beryl mineralized pegmatites of the Velasco district, the obtained age is attributed extensively for the entire district. According to the initial  $^{87}\text{Sr}/^{86}\text{Sr}$  value obtained of 0.713, the pegmatite-magmatism of the Velasco District might be mainly derived from the crust with some minor participation of mantle materials.

**KEYWORDS:** Rb-Sr isochron; petrogenesis; pegmatite; Velasco District; Pampean Pegmatite Province.

## INTRODUCTION

Granitic pegmatites are igneous rocks characterized by large crystals with mineralogical and textural variations between different sectors of these bodies, as well as a chemical heterogeneity that can be manifested even within the same grain (London 1992). In addition, in many cases, these rocks are of great economic importance since they are sources of various rare metals and minerals for industrial use, jewelers, and collectibles. All of these reasons have, for a long time, increased the interest of numerous researchers in the study of pegmatites, who despite having a rich and abundant knowledge on their mineralogy and geochemistry, still have to deal with the scarcity of studies on isotopic composition in pegmatites. The rock-forming minerals of pegmatites commonly contain variable amounts of potassium, such as K-feldspars and micas. Geochemical behaviors, such as ionic size and charge, allows the tracing of radiogenic element Rb in order to substitute the major element potassium in these minerals. Due to this reason, the isotopic Rb-Sr method

can be properly used for geochronology studies on pegmatites (e.g., Lima *et al.* 2009, Barros *et al.* 2017).

The Pampeana Pegmatite Province (PPP) is located in Central and Northwestern Argentina and was defined by Galliski (1994). It is composed of pegmatites of different mineralogical nature, geochemical signature, and age. In the PPP, the Muscovite and Rare Elements classes after classification of Černý and Ercit (2005) are predominant. Among the Rare Elements, the LCT (lithium-cesium-tantalum) family are more abundant than the NYF (niobium-yttrium-fluorine) family according to the Černý and Ercit (2005) classification. Based especially on the availability of isotopic and geologic data in parental granitic rocks, Galliski (2009) groups four districts of the muscovite class and 15 districts of the Rare Elements class in the PPP, hosted in metamorphic rocks of different degrees of metamorphism as well as granitic rocks. Furthermore, the author introduces the designation of 'orogenic pegmatitic fields or districts' for those developed during the Lower Paleozoic, but mainly during Early and Middle Ordovician age — Famatinian orogenic cycle — and 'post-orogenic pegmatitic fields or districts' to those genetically related to parental granites of Upper Devonian/Lower Carboniferous age — generically called 'Achalán' magmatic event. Recently, Galliski *et al.* (2021) determined columbite U/Pb ages from several pegmatites of the PPP. The obtained ages define two clear periods: one between 490 and 440 Ma ( $T_1$ ), corresponding to 75% of the LCT Rare-Elements pegmatites formed during the Famatinian orogeny, and the other

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between 370 and 340 Ma ( $T_2$ ), comprising post-orogenic LCT and NYF (or mixed) pegmatitic signatures.

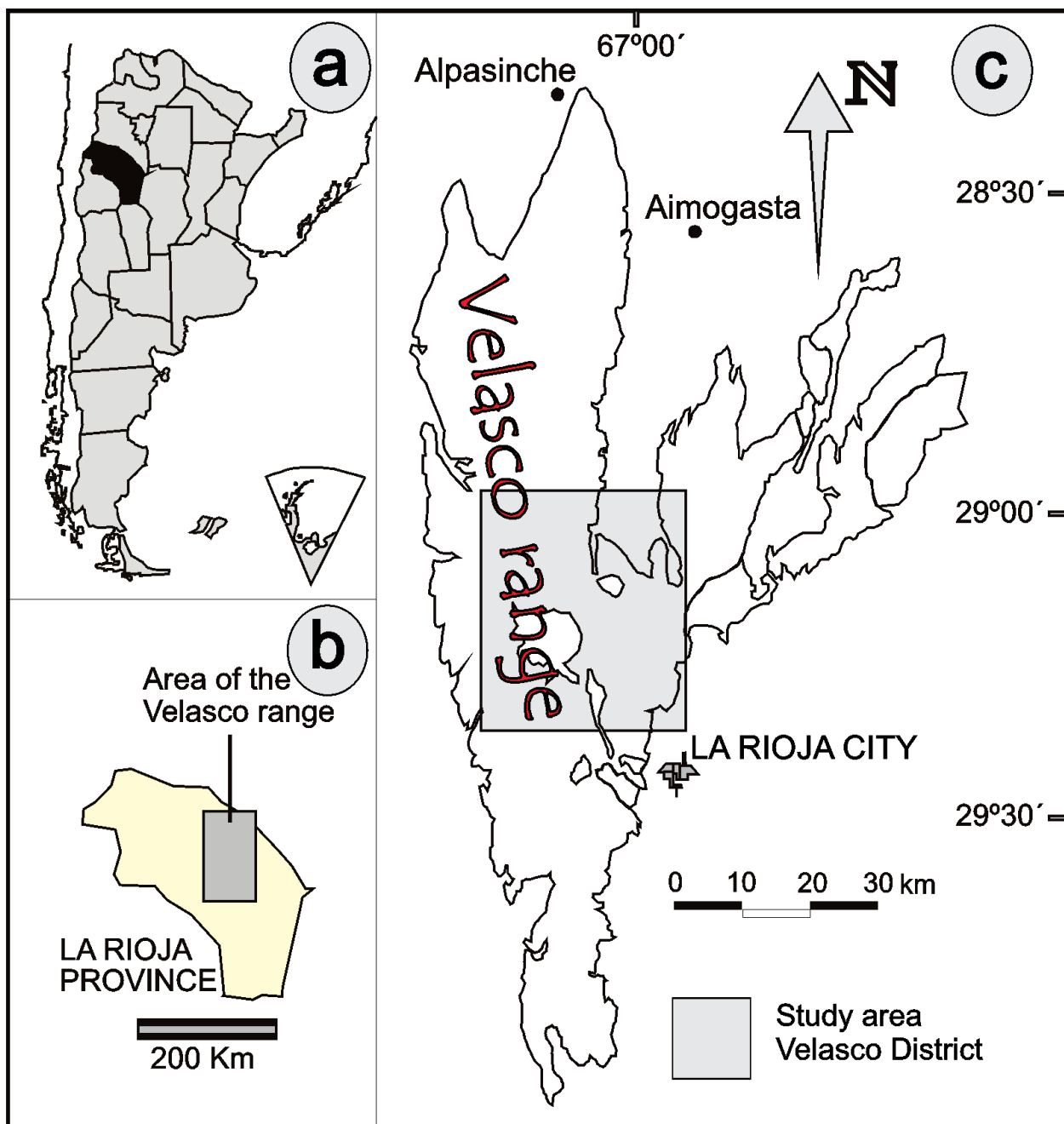
The Velasco pegmatite district forms part of the PPP (Fig. 1). This post-orogenic pegmatite district contains features of the NYF petrogenetic family (Galliski 1994, Sardi *et al.* 2015). The goal of this study was to determine the pegmatitic Be-mineralization age and discuss the source of the ore-forming magma in the Ismiango pegmatite, located in the Velasco district.

## GEOLOGICAL SETTING

The PPP is located in the ‘Sierras Pampeanas’ geotectonic province. The crystalline basement of the Sierras Pampeanas is essentially composed of metamorphic rocks of varying degrees of metamorphism and igneous bodies, which are predominantly

granitoid in composition (Toselli *et al.* 1986, Pankhurst *et al.* 2000). The dominant lithology of the metamorphic rocks consists of a succession of metapelites and metapsamites (regional metamorphism of low to very low grade) that outcrops in wide sectors in Northwest Argentina, although in some sectors rocks of medium and high degree of metamorphism are also recognized (Rossi *et al.* 2002, Larrovere *et al.* 2011). The protolith is mainly composed of clastic material deposited in a marine basin, with retro-arc characteristics, developed in the pro-to-western margin of Gondwana temporarily between Late Precambrian and Early Paleozoic, which has been recognized as the Pampean cycle (e.g., Aceñolaza and Toselli 1981, Willner *et al.* 1990, Rapela *et al.* 1998, Rapela *et al.* 2001).

The igneous rocks of the crystalline basement of Sierras Pampeanas present two ages that are highly contrasting



**Figure 1.** Schematic geographic location of the Velasco District in the homonymous mountain-range, Northwestern Argentina. (a) Republic of Argentina. (b) La Rioja province. (c) Velasco mountain range.

geochemical and petrogenetic characteristics: Lower-Middle Ordovician (Famatinian cycle; Aceñolaza *et al.* 1996, Pankhurst *et al.* 1998, Pankhurst *et al.* 2000, Miller and Söllner 2005, Dahlquist *et al.* 2008, Dahlquist *et al.* 2016) and Middle-Upper Devonian to Lower Carboniferous (generically called Achaian event; Sims *et al.* 1998, Dahlquist *et al.* 2018). The magmatism of the Famatinian cycle was developed in an active arc framework towards the west of the Gondwana continent (e.g., Rapela *et al.* 2001, Miller and Söllner 2005, Dahlquist *et al.* 2006, Dahlquist *et al.* 2013, Toselli *et al.* 2008). Pankhurst *et al.* (2000) recognize three types of granitoids in the Famatinian belt: trondhjemite-tonalite-granodiorite (TTG) originated by the fusion of an asthenospheric mantle source depleted in gabbroids at depths of 10-12 kbar, metaluminous I-type granitoids, and peraluminous S-type granites. Meanwhile, the Achaian magmatic event was developed in an intraplate extensional setting (e.g., Grosse *et al.* 2009).

### Velasco district

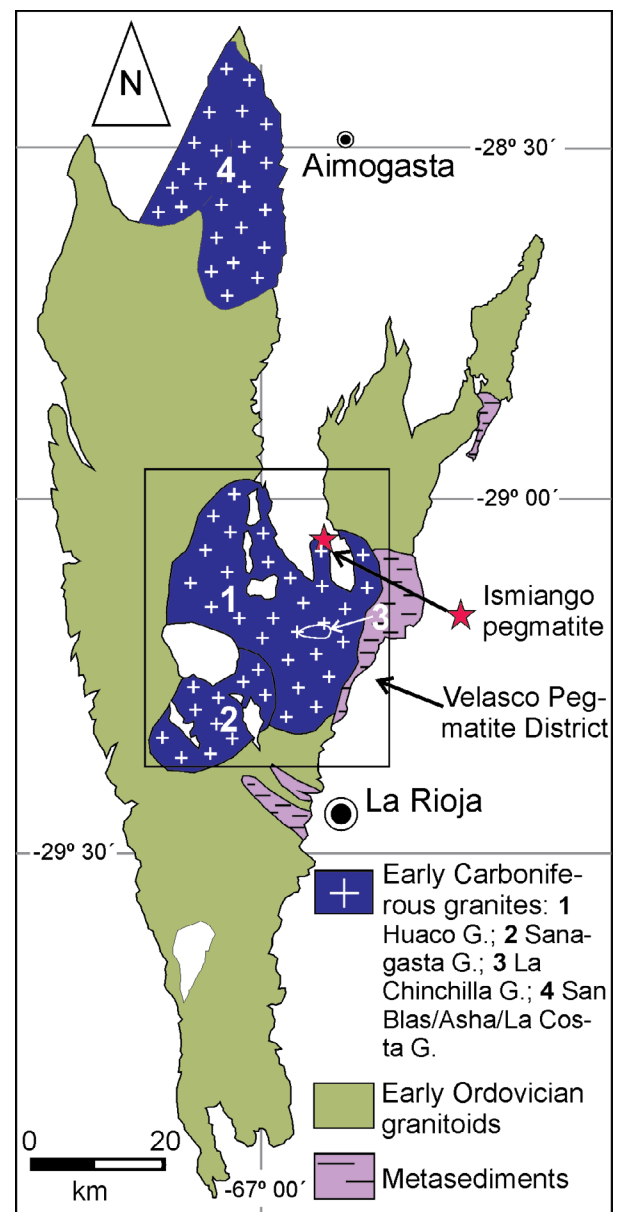
The Velasco pegmatitic district is located in the Central and Central-Eastern region of the Velasco mountain range in the La Rioja province, Northwestern Argentina. Velasco mountain range has a N-S direction and is one of the largest mountain ranges in the Sierras Pampeanas with about 175 km in length and about 50 km at its greatest width (Fig. 1).

Geologically, the Velasco mountain range is constituted of a batholith composed of several granitic plutons of different ages, evolution, and petrogenesis (Toselli *et al.* 2005, 2008). In the area where the pegmatites form the district of the same name, two important granite bodies are recognized (Fig. 2). They are called the Huaco and Sanagasta granites of approximately 40 km x 30 km and 25 km x 15 km in size, respectively (Toselli *et al.* 2000, Grosse and Sardi 2005, Grosse *et al.* 2009, Dahlquist *et al.* 2010). The Lower Carboniferous geochemically evolved La Chinchilla granite (Grosse *et al.* 2005, 2009) is a very small body that intruded the Huaco granite. It is characterized by having beryl as an accessory mineral (Sardi *et al.* 2016). The Huaco and the Sanagasta granites are not deformed nor intruded into older metagranitoids and mylonites (Grosse *et al.* 2009), as the contact between both granitoid bodies is transitional.

The Huaco granite is leucocratic, porphyritic, and mainly constituted of microcline megacrysts, whereas the Sanagasta granite is also porphyritic but pink-colored and characterized by the incipient replacement of microcline by plagioclase (Grosse *et al.* 2009). The lithological composition of both granites is similar and corresponds to monzogranite and syenogranite with accessory minerals, such as biotite, muscovite, apatite, zircon, monazite, and ilmenite. Chemically, the granitoids are peraluminous, rich in SiO<sub>2</sub> and alkalis, especially K<sub>2</sub>O, and derived from a predominantly crustal source (primarily derived from the Ordovician granitoids) and a minor mantle component (Grosse *et al.* 2009). Furthermore, Dahlquist *et al.* (2010) classified both granites as A-type (as well as the La Chinchilla granite).

The beryl-bearing pegmatites that are part of the Velasco district have a spatial and genetic relationship with the

Huaco and Sanagasta granites (Sardi *et al.* 2010, 2015, 2018). According to the nomenclature given by Černý and Ercit (2005), they belong to the Rare Elements class, the beryl type and the beryl-columbite-phosphate subtype. In return, the district gathers outstanding trends towards the NYF petrogenetic family, such as parental granites of post-orogenic character and A-type affinity and minerals that carry rare-earth elements and fluorine (F), both in the granites and the pegmatite border zone (Sardi *et al.* 2015). The pegmatites show ellipsoidal, elongated, and semi-circular morphologies, with major and minor axis ratios between 1 and 4.7. The direction of the major axis is variable in each pegmatite. The zoning is simple. From the outside inwards, the following zones could be recognized in the pegmatites: border and intermediate zones and a quartz core. The graphic and perthitic microcline of the intermediate zone is incipiently substituted by plagioclase (Sardi *et al.* 2015).



G: granites.

**Figure 2.** Simplified geological map of the Velasco mountain range, Northwestern Argentina.

*Ismiango pegmatite*

The Ismiango pegmatite is an intragranitic pegmatite hosted in the Huaco granite located at the 29°02'42"S – 66°51'15"W coordinate. It has been exploited for beryl as well as wolframite based on the testimony given by residents of the Ismiango village in the middle of the last century. Due to the exploitation process, the body exhibits a NE-SW direction front of about 12 m and an underground labor. According to Sardi *et al.* (2015), the body includes, from the contact to the center, a thin border zone (6.6% of the total body volume) and an intermediate zone composed mainly of block K-feldspar (78.2%) and plagioclase to a lesser extent (15.2%), as in Figure 3A. A small body of felsic porphyritic granite is located in the intermediate microcline zone, which can be described as an enclave attributed to the remnants of the previously emplaced aplite (Sardi *et al.* 2018).

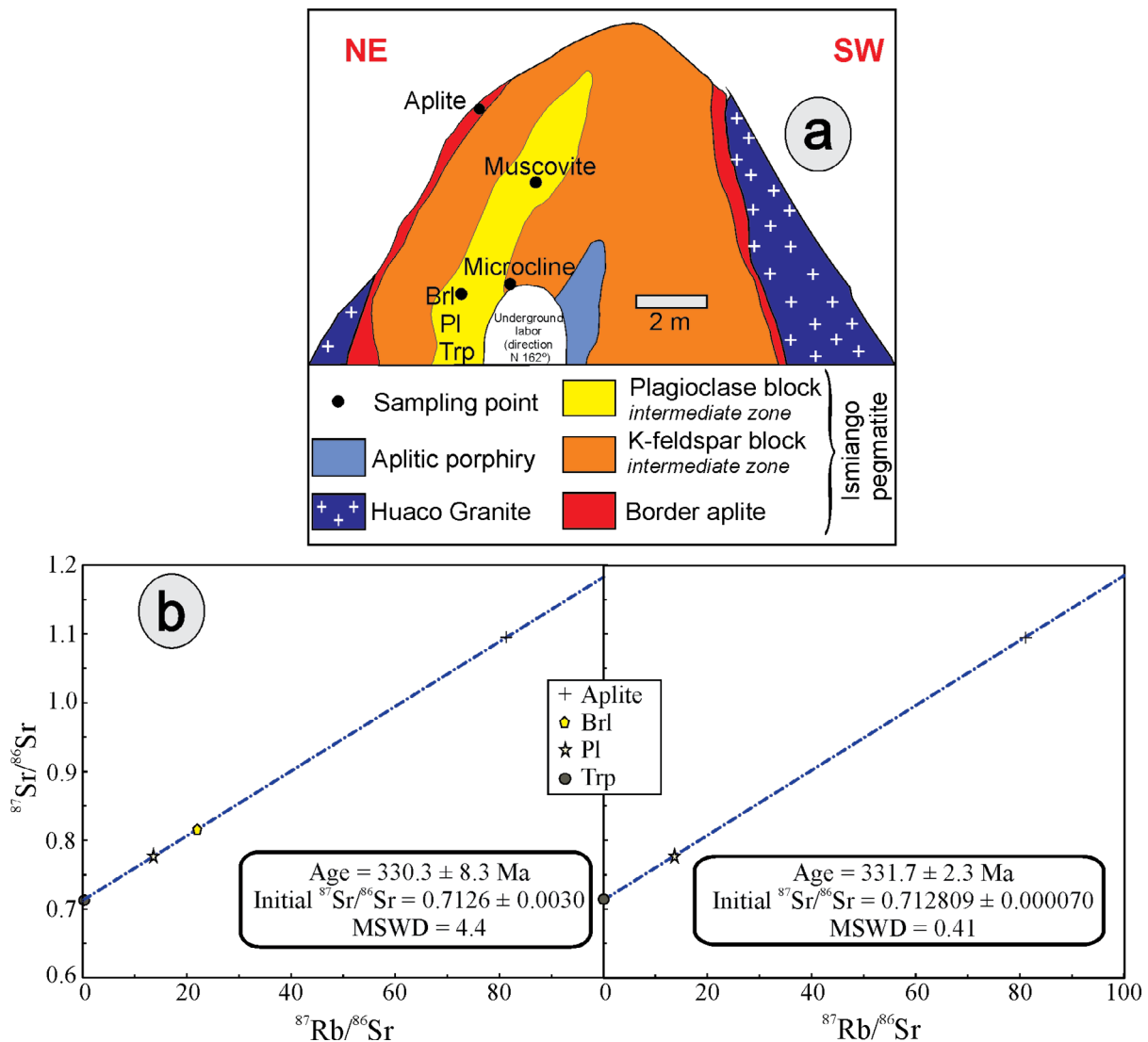
The border zone is an aplite of granodioritic to monzogranitic composition containing two micas with the biotite/muscovite ratio of 0.3 (Sardi *et al.* 2010) and subordinate amounts of apatite and fluorite (Sardi *et al.* 2015). Geochemically, the

rock has a high and very restricted amount of silica (73-76%),  $Fe_2O_3 + MgO + TiO_2$  of approximately 1.5% and an Alumina Saturation Index (ASI) of 1.11 (Sardi *et al.* 2010). The mineralogy of the intermediate zone is mainly composed of perthitic microcline, graphic in certain sectors (77% of the volume of the intermediate zone), quartz (7.4%), plagioclase (7%), occasional muscovite in radial arrangement (5.7%), yellow dominant beryl (2.1%), apatite (0.3%), triplite (0.1%) and scarce amounts of tourmaline (Sardi *et al.* 2015).

**RESULTS**

**Materials and methods**

The Rb-Sr geochronology study presented in this paper has been performed using cogenetic ‘material’ sampled from the Ismiango pegmatite: aplite from the border section of the pegmatite and minerals from the intermediate zone, such as microcline, plagioclase, muscovite, beryl and triplite (Fig. 3A).



Brl: beryl; Pl: plagioclase; Trp: triplite.

**Figure 3.** (a) Mineralogical map of the Ismiango pegmatite showing the sampling point (vertical scale is the same as the horizontal scale). (b)  $^{87}Sr/^{86}Sr$  vs  $^{87}Rb/^{86}Sr$  diagram (isochron) for the Ismiango Be-pegmatite from the Velasco District, PPP.

According to Sardi *et al.* (2015), feldspars have light colors (white, pink), vitreous luster, subidiomorphic texture and cleavage in two directions that are sometimes not clearly manifested. Potassium feldspar is predominantly microcline, often showing a perthitic texture and less frequently, a graphitic one. Plagioclase commonly presents polysynthetic twinning according to the albite law. Muscovite is the dominant mica present in Ismiango pegmatite, occurring in laminar packages that can reach up to a few tens of cm and a thickness of a few cm. It forms idiomorphic crystals with individual transparent and colorless to somewhat brownish leaves. Muscovite appears associated with feldspars and in areas with significant beryl-bearing mineralization. Beryl has an erratic distribution in the intermediate zone of the pegmatitic body. It is associated with quartz as well as muscovite, while less frequently with feldspars. Beryl crystals are idiomorphic and small (a few cm long). The dominant beryl color in the Ismiango pegmatite is yellow, which would have formed later than the green color that occurs in the pegmatites of the Velasco district (Sardi and Heimann 2014). The triplite is black with a reddish tinge, a non-metallic luster and a predominant in masses that can reach about 12 cm in diameter. It is associated with muscovite and feldspar and rarely with quartz.

After checking the purity of each of the preceding minerals using a binocular lens, fine tweezers and needles, and prior to the analytical processing in the laboratory, each of the minerals was grounded to a fine power using a carbon tungsten ring mill in the Instituto Superior de Correlación Geológica's Laboratory of Petrology (INSUGEO; Tucumán, Argentina).

## Isotopic composition

The Rb-Sr isotope analysis of six samples previously described from Ismiango pegmatite was performed at the Geochronology Service (Universidad Complutense de Madrid, Spain), and the results can be seen in Table 1. For mineral Sr isotopic analysis by isotope dilution-thermal ionization mass spectrometry (ID-TIMS), the samples were first dissolved in ultra-pure reagents in order to separate the isotopes via exchange chromatography (Strelow 1960), and they were subsequently analysed using a Sector 54 VG — Micromass multi-collector spectrometer. Resulting  $^{87}\text{Sr}/^{86}\text{Sr}$  isotopic ratios (Tab. 1) were

corrected for possible isobaric interferences from the  $^{87}\text{Rb}$  and normalized to  $^{88}\text{Sr}/^{86}\text{Sr} = 0.1194$  in order to correct mass fractionation. The NBS 987 international isotopic standard was analysed along with the samples, yielding an average value of  $^{87}\text{Sr}/^{86}\text{Sr} = 0.710262$  for seven replicas, with an internal precision of  $\pm 0.00007$  ( $2\sigma$ ). These values were used to correct the measured ratios for possible sample drift. The analytical errors estimated for the  $^{87}\text{Rb}/^{86}\text{Sr}$  and  $^{87}\text{Sr}/^{86}\text{Sr}$  are of 1% and 0.01%, respectively. The Isoplot/Ex 3.00 software (Ludwig 2003) was used for the isotopic data plotting (Fig. 3B) and age calculations.

Figure 3B contains two Rb-Sr isochrones, one of which has been constructed with the isotopic values of aplite, beryl, triplite and plagioclase, and the other with all values mentioned above, with the exception of beryl. Muscovite Rb-Sr values have not been used for this purpose due to their unacceptably high instrumental error, likely related to their low Sr content (3.6 ppm). This may be the result of a post-crystallization open-system remobilization of the Sr, which could be captured by replacing K in the potassium feldspar. Both minerals present high Rb contents, which produce highly radiogenic  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios and unrealistic values of the initial Sr ratios that prevent their use in petrogenetic interpretations.

The Rb/Sr ratio was always greater than 1 in all analyzed materials, except for the triplite. This relationship is very significant in perthitic microcline and especially in the muscovite sample, which has very high  $K_d$  values (always greater than 1) in both minerals found in highly siliceous magmas (e.g., Rapela and Shaw 1979). Regarding the ages obtained from both isochrons, they are very similar ( $330.3 \pm 8.3$  Ma and  $331.7 \pm 2.3$  Ma) and are clearly included in the Lower Carboniferous period and corresponding to the Achaian magmatic event. On the other hand, the initial  $^{87}\text{Sr}/^{86}\text{Sr}$  relation of petrogenetic interest coincides in both isochrons, giving an approximate value of 0.713.

It should also be mentioned that the high mobility of the elements involved in this report in late magmatic and/or hydrothermal processes can cause a disturbance in the Rb-Sr geochronological system (e.g., Černý 1991). In both feldspars and micas, the system is subjected to the gain or loss of radioactive and radiogenic elements (London 2008 and

**Table 1.** Analytical isotope Rb-Sr data for aplite and cogenetic minerals of the Ismiango zoned pegmatite, Velasco District, Northwestern Argentina.

Sample	Rb (ppm)	Sr (ppm)	Rb/Sr	$^{87}\text{Rb}/^{86}\text{Sr}$	$^{87}\text{Sr}/^{86}\text{Sr}$	error abs.	$^{87}\text{Sr}/^{86}\text{Sr}$ init
Perthitic microcline	1,408.4	30.4	46.33	142.333	1.339993	8	0.671457
Muscovite	2,522.3	3.628*	695.23	5579.728	18.841060	1017	-7.366903
Beryl (Brl)	46.4	6.1	7.61	22.241	0.815951	5	0.711486
Plagioclase (Pl)	32.2	6.9	4.67	13.594	0.777125	5	0.713276
Triplite (Trp)	1.321*	57.741*	0.02	0.066	0.713120	6	0.712809
Aplite	361.7	13.4	26.99	81.052	1.094558	8	0.713858

\*Values of Rb and Sr concentrations were obtained in the Geochronological Service (UCM) using ID-TIMS. The remaining parts were obtained in the geochemistry laboratory of the Geological and Mining Institute of Spain (IGME) with XRF methodology.



references therein). In the Ismiango pegmatite as well as in others in the Velasco District, a post-crystallization activity of sodium metasomatism has been observed, which affected the K-feldspar very lightly (Sardi *et al.* 2015) and could have caused disturbances in the Rb-Sr system.

## DISCUSSION

The two Rb-Sr isochrons define an absolute age of  $330.3 \pm 8.3$  and  $331.7 \pm 2.3$  Ma for the Ismiango pegmatite and are included in the Lower Carboniferous period, belonging to the Achalian magmatism of the Sierras Pampeanas. The Rb-Sr dating is interpreted as the best estimation for the crystallization age of the Ismiango pegmatite. Since this body has a similar composition and structure to the other beryl mineralized pegmatites of the Velasco district, the age obtained may be proposed for the whole district. The results are very close to the 'T<sub>2</sub> ages' (370-340 Ma) obtained by Galliski *et al.* (2021) for post-orogenic pegmatites of the PPP with lithium-cesium-tantalum (LCT), niobium-yttrium-fluorine (NYF) or mixed NYF-LCT signatures. According to these authors, these post-orogenic pegmatites show chemical and geochronological features compatible with the A-type parental monzogranites, a similar framework defined for the Early Carboniferous intrusive rocks of the Velasco mountain range. In it, the granitic magmatism of the Upper Devonian-Lower Carboniferous is widely represented (Fig. 2; Toselli and Rossi de Toselli 2018) and it has been grouped as the Aimogasta batholith by Toselli *et al.* (2006). All these bodies whose crystallization ages would have occurred predominantly in the Lower Carboniferous would belong to the so-called Early Gondwana event by Dahlquist *et al.* (2010), although in this study we will continue to use the generic nomenclature for the event: 'Achalian'.

The Achalian magmatic event occurred in an extensional tectonic framework, giving rise to essentially post-orogenic peraluminous granitoids with A-type affinity, usually rich in F and crustal-derived with minor contributions from the mantle material (e.g., Grosse *et al.* 2009, Dahlquist *et al.* 2010, Colombo *et al.* 2011). From north to south of the mountain range, the 'Achalian' granites of San Blas (Báez *et al.* 2004), Asha (Toselli *et al.* 2011), La Costa (Alasino *et al.* 2006), Huaco and Sanagasta (Grosse and Sardi 2005) and La Chinchilla (Grosse *et al.* 2005) can be recognized. These last three are part of the Velasco pegmatite district, although the Huaco and Sanagasta units are recognized as parental granites of the beryl pegmatites (Sardi *et al.* 2010, 2015, 2018). McBride *et al.* (1976) recorded absolute ages in several granitoids of the Velasco mountain range that would belong to the Achalian event using K-Ar methodology in minerals as well as in whole rock. More recently, the absolute ages using U/Pb methodology (conventional, SHRIMP, and LA-ICP-MS) in zircons and/or monazite of the Huaco and Sanagasta granites are in the 378-334 Ma range (Báez *et al.* 2004, Dahlquist *et al.* 2006, Grosse *et al.* 2009, Dahlquist *et al.* 2013, Dahlquist *et al.* 2018, Söllner *et al.* 2007, Toselli *et al.* 2011, Macchioli Grande *et al.* 2020).

Particularly, the Huaco granite (host-rock of the Ismiango beryl-bearing pegmatite and several others from the Velasco district) has been dated yielding the following absolute ages:

- $354 \pm 4$  Ma, U-Pb LA-ICP-MS on zircons (Söllner *et al.* 2007);
- $350 \pm 5$  Ma y  $358 \pm 5$ , U-Pb on conventional monazite (Grosse *et al.* 2009);
- $357 \pm 3$  Ma, U-Pb LA-ICP-MS on zircons (Dahlquist *et al.* 2013).

In general, the age obtained in this paper can be considered in agreement to the crystallization age of the parental and host-rock, the Huaco granite, separating a lapse around 22-23 Ma between the granitic-magmatism episode and a pegmatitic episode. It is related to the experimental and observational measurement made a long time ago by Webber *et al.* (1999) for pegmatite body crystallization. A probable reason could be that frequent <sup>87</sup>Sr loss from the Rb-Sr system during the post-consolidation stage which would usually amount to an unrealistically younger age (Černý 1991), as well as the different closure temperatures of each of the minerals and rocks involved in the isotopic system used in this report (e.g., Cliff 1985, Fletcher *et al.* 1997). The initial <sup>87</sup>Sr/<sup>86</sup>Sr ratio of 0.713 for the Ismiango pegmatite is therefore attributed to a pegmatitic magma derived essentially from the continental crust. The low value of the <sup>87</sup>Sr/<sup>86</sup>Sr initial ratio in the microcline could be attributed to the loss of Sr due to Na-metasomatism post-crystallization alteration. The obtained value can be considered lower compared to the values between 0.730 and 0.752 (average of 0.741) that were found for the parental and host-rock, the Huaco granite by Grosse *et al.* (2009). These values would support the hypothesis that granite-pegmatite evolution in the Velasco district would have occurred from a common magma source as it was geochemically supported previously by Sardi *et al.* (2010).

Based on these <sup>87</sup>Sr/<sup>86</sup>Sr initial values as well as other observations such as field and petrographic and other analytical results, especially isotopic (εNd between -2.1 and -4.3), Grosse *et al.* (2009) suggest that the Huaco granite (coupled with the adjacent Sanagasta granite) would have a mainly crustal source with some participation of a more primitive, possibly mantle-derived source, which is also similar to the conclusions of Dahlquist *et al.* (2010) and Macchioli Grande *et al.* (2020) for these granites.

## CONCLUSIONS

The results obtained in this research represent the first isotopic study carried out on the pegmatites of the Velasco District of the PPP. Based on the linear array of the two isochrons constructed with data points from the border aplite and cogenetic minerals from the intermediate zone of the Ismiango pegmatite, the emplacement may have taken place at c.a. 330-332 Ma, belonging to the Lower Carboniferous period of the Achalian event. This age is extended to the entire Velasco district due to the similarity of its composition and structure. However, the absolute age obtained could result in some rejuvenation due to

disturbances in the Rb-Sr system. The absolute age difference of around 20 Ma with the parental Huaco granite is attributed to this inherent cause in the methodology used. The formation of the beryl-bearing pegmatites would represent the last magmatic activity of the Paleozoic in the Velasco mountain range of the Sierras Pampeanas.

According to the initial  $^{87}\text{Sr}/^{86}\text{Sr}$  value obtained of 0.713 in the Ismiango pegmatite and combined with the other isotopic data available for the host rock Huaco granite, the pegmatite-magmatism of the Velasco District may be mainly derived from the crust with some minor participation of mantle materials.

## ARTICLE INFORMATION

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F.S. has written the draft manuscript and prepared Figure 2. He then made the corrections and observed the suggestions of the reviewers of the manuscript. As for S.M., he has contributed with the knowledge of regional and local geology, also making contributions to the introductory chapter of the article. S.M. drew up Figure 1. J.F. performed the analysis task of the paper in his laboratory and has mainly written the isotopic composition section of the Results chapter. Table 1 has been prepared by J.F. The discussion and conclusions of the paper were a consensus of all the authors.

Competing interests: The authors declare no competing interests.

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