SHRIMP and LA-ICP-MS analyses carried out on zircons from the Río de los Sauces granite revealed their metamorphic and igneous nature. The metamorphic zircons yielded an age of 537±4.8 (2σ)Ma that probably predates the onset of the anatexis during the Pampean orogeny. By contrast, the igneous zircons yielded a younger age of 529±6 (2σ)Ma and reflected its crystallization age. These data point to a short time lag of ca. 8Myr between the High Temperature (HT) metamorphic peak and the subsequent crystallization age of the granite. Concordia age of 534±3.8 (2σ)Ma, for both types of zircon populations, can be considered as the mean age of the Pampean HT metamorphism in the Sierras de Córdoba.

**KEYWORDS** | Pampean Orogeny, Pampean Metamorphism. Río de los Sauces granite. U-Pb SHRIMP dating. LA-ICP-MS analyses.

**INTRODUCTION**

The Neoproterozoic to middle Cambrian Pampean orogen (Aceñolaza and Toselli, 2009), located at the north-central part of Argentina (Fig. 1), was part of the Terra Australis orogen at the western margin of Gondwana (Cawood, 2005). It was defined as a paired belt, with calc-alkaline magmatic rocks in the eastern belt and medium- to high-grade metamorphic rocks with peraluminous granites in the western belt (Rapela et al., 1998; Schwartz et al., 2008). This calc-alkaline magmatism is widely exposed in the Sierra Norte de Córdoba and is usually referred to as the Sierra Norte-Ambargasta batholith (Iannizzotto et al., 2013; Lira et al., 1997; Schwartz et al., 2008; Von Gosen and Prozzi, 2010). The U-Th-Pb dating in granitoids from the Sierra Norte-Ambargasta batholith gave crystallization ages between 530 and 537Ma and were linked to the development of a continental magmatic arc in an eastward subduction of oceanic lithosphere down to the western
FIGURE 1. Schematic geological map of NW Argentina showing the Sierras Pampeanas geological province, the main mountain ranges, the geological subdivisions, and the extent of the Pampean orogeny (from Weinberg et al., 2018). The location of Figure 2 is shown. Mountain ranges: Sierra de Aconquija (Ac), Sierra de Fiambala (Fi), Sierra de Ambato (Am), Sierra de Ancasti (An), Sierra de Cachi (Ca), Sierra de Velasco (Ve), Sierra de Famatina (Fa), Sierra de Maz-Espinal (SME), Sierra de Valle Fértil (VF), Sierra de Chepes (Ch), Sierra de Ulapes (SU), Sierra de Pie de Palo (PP), Sierras de Córdoba (SC), Sierra de Quilmes (SQ), Sierra Norte de Córdoba (SNC), Sierra de San Luis (SSL), Sierra de Toro Negro (STN), Sierra de Umango (Um), Western Sierras Pampeanas (WSP), Eastern Sierras Pampeanas (ESP).
The western belt of the Pampean orogen is formed mainly of sedimentary and metasedimentary rocks deposited during Neoproterozoic times. These sequences are represented by the low-grade metamorphosed turbidites of the Puncoviscana Formation, and their higher metamorphic grade counterparts (schists, gneisses and migmatites) located at the Sierras de Ancasti and Córdoba (Aceñolaza and Aceñolaza, 2007; Omarini, 1999; Rapela et al., 1998; Rapela et al., 2007; Schwartz and Gromet, 2004; Schwartz et al., 2008; Weinberg et al., 2018).

The Sierras de Córdoba are located at the southernmost part of the Eastern Sierras Pampeanas, central Argentina (Fig. 1). They are constituted by N-S trending mountain ranges mostly composed of Neoproterozoic to Devonian metasedimentary and igneous rocks. The main tectono-thermal event registered in the Sierras de Córdoba, during the Pampean orogeny, is the so called M2 (Casquet et al., 2018). This event was linked to an increase in the P-T conditions, due to crustal shortening with thrusts propagating into the fore-arc (Hauer et al., 2011; Rapela et al., 2007). The onset of subduction turned these sequences into an accretionary prism leading to medium to high-grade schists, gneisses, and migmatites (Weinberg et al., 2018 and references therein).

The temporal relationship between metamorphism and granite crystallization differ from orogen to orogen (Esteban et al., 2015; Keay et al., 2001; Vanderhaeghe et al., 1999; Whitney et al., 2003). Although the age of each of these processes can be obtained separately from migmatites and anatectic granites, Esteban et al. (2015) demonstrated that zircons extracted from one pluton may be enough to detect the time lag between the metamorphic peak and the magmatic activity in internal domains of orogenic belts. They found a time span of 7Myr between the metamorphic climax and the emplacement of the Lys-Caillaous pluton in the Axial Zone of the Pyrenees (southern France), by recognizing two populations of zircons, one with igneous and the other one with metamorphic affinities.

Here, we report a rather similar case in the Río de los Sauces granite, Sierra de Comechingones (Argentina), where two distinct zircon populations identified by LA-ICP-MS geochemistry and dated by U-Th-Pb SHRIMP analyses, allowed us to establish the time lag between the metamorphic climax and the crystallization of the granite.

FIELD RELATIONSHIPS AND PTEROGRAPHY OF THE RÍO DE LOS SAUCES GRANITE

In this work, we defined a new granitic body, the Río de los Sauces granite, located two kilometers to the north of Río de los Sauces village (Fig. 2A, B). The country rocks are garnet-rich gneisses and metatexite migmatites of the Calamuchita Metamorphic complex (Otamendi et al., 2004). These rocks show an ENE–WSW-trending foliation (Sf) with high angle dips (generally >70°) both to the south and the north. This foliation is parallel to the axial plane of the tight, isoclinal folds and is associated with constrictional non-coaxial strain (Martino and Guereschi, 2014 and references therein). The mineral assemblage of the gneisses is biotite+plagioclase+quartz z+garnet, whereas the metatexites are mainly stromatites
that contain leucosomes composed of quartz+plagioclase+K-feldspar+biotite+garnet and melanosomes composed of biotite+garnet+quartz+plagioclase+sillimanite. There are a few examples of leucosomes of stromatites that form phacoliths or patches within boudin necks, evidencing that melting took place during folding. Folded, layer-parallel leucosomes also merge continuously with leucosomes parallel to axial-plane foliation. Otamendi et al. (2004) described similar structures and interpreted them in the same way. Magma transfer through this fold-assisted leucosomes network has been described in other parts of the world (Weinberg and Mark, 2008). Finally, leucosomes coalesced giving rise to leucogranite and pegmatite sheets, and small plutons. Because of the continuous link from leucosomes to larger sheets and plutons (Fig. 2C), we interpret that leucogranites are autochthonous granites.

The Río de los Sauces granite was formed by the coalescence of numerous leucogranite sheets, which intruded concordantly the gneisses, close to the gneiss-metatexite boundary (Fig. 2A, B). The resulting granite body is 1.2km long and 100–150m wide. The regional metamorphic foliation trend deflects around the Río de los Sauces granite, implying that after solidification, it also behaved as a competent body during continued deformation. This is a hypidiomorphic coarse-grained leucogranite with a mineral association of microcline+quartz+plagioclase+biotite+muscovite+garnet+sillimanite, and accessories such as zircon, titanite and apatite. The modal proportion of main minerals is: microcline (35-50%) quartz (35-45%) and plagioclase (20-30%). It also shows late patches and pegmatite sheets. The Río de los Sauces granite is weakly foliated. The foliation is defined by oriented biotite and elongated quartz (Fig. 2D, E), and is parallel to the contacts and the gneiss structures.
U-Th-Pb SHRIMP GEOCHRONOLOGY

One sample from the Río de los Sauces granite (32°30′37.7″S/64°34′18.8″W) was processed according to routine zircon mineral separation (crushing, grinding, sieving under 250 μm, Wilfley table and methylene iodide) at the University of Río Cuarto (Argentina), in order to date the emplacement of the pluton. The selected zircons were mounted in epoxy resin together with the TEMORA 2 reference zircon (416.78Ma; Black et al., 2004), sectioned, polished and analyzed on a SHRIMP-IIe/MC at the Centro de Pesquisas Geocronológicas of the University of São Paulo (GeoLab-IGc-USP, Brazil). The obtained U-Pb ion microprobe data were processed with the SQUID and Isoplot/Ex 3.00 (Ludwig, 2003) software programs and are presented in Table 1. In order to select target areas, cathodoluminescence (CL) images were obtained by a FEI Quanta 250 Scanning Electron Microscope and XMAX CL detector, in the same laboratory. Further details on the sample preparation, analytical setup, acquisition and data processing are given in Sato et al. (2014).

According to CL images (Fig. 3) the zircons reveal a complex zoned pattern characterized by dark luminescent rims developed mainly over xenomorphic cores, and dark luminescent idiomorphic zircons with weak oscillatory zoning and extremely low Th/U ratios (<0.02) (Table 1: spots 9.1; 3.1; 4.1; 16.1; 11.1; 6.1; 8.1; 1.1; 2.1). These zircons contrast with those zircons showing euhedral morphology (prismatic or bipyramidal), concentric undisturbed oscillatory growth zoning, sometimes with progressively U-rich low luminescent rims, lack of inherited xenomorphic cores and high Th/U ratios (0.22-0.57) (Table 1: spots 1.2; 2.2; 5.1; 12.1; 7.1; 15.1; 14.1; 13.1; 10.1). All these different features point to two discrete zircon populations, ZP 1 and ZP 2.

Nine and eight spot analyses were carried out (Table 1) yielding 206Pb/238U Concordia ages of 537±4.8 (2σ) (Fig. 4A) and 529±6 (2σ)Ma (Fig. 4B) for ZP 1 and ZP 2, respectively. Although the Concordia age of ZP 2 displays a high Mean Squared Weighted Deviation (MSWD) and low probability of concordance (Fig. 4B), the less discordance analyses (<10 %) yield a weighted mean age of 530±8 (2σ)Ma. A high probability of age coherence in both zircon populations is indicated by the overlapping of the concordant data as they define a Concordia 206Pb/238U age of 534±3.8 (2σ)Ma (Fig. 4C).

TRACE AND RARE EARTH ELEMENT ANALYSES BY LA-ICP-MS

Trace and Rare Earth Elements (REE) on both zircon populations were analyzed by LA-Q-ICP-MS (Laser Ablation Quadrupole Inductively Coupled Plasma Mass Spectrometry) at the University of the Basque Country (SGIker) using a 213nm New Wave Nd:YAG laser, with a ~3.65J/cm² energy density at a repetition rate of 10Hz, coupled to a Thermo iCAP Qc quadrupole ICP-MS. The analytical spot size was 40µm in diameter, and in most cases, the zircons were completely pierced through during the 60 seconds of acquisition time. External calibration was performed to the NIST SRM 612 (Jochum et al., 2011). The internal standard was the stoichiometry-calculated 90Zr, and data reduction was carried out by the
Sixty-eight zircon crystals from sample GRS-215, after SHRIMP acquisition, were analyzed by LA-ICP-MS. Some of them (nine) were rejected due to the presence of inclusions of apatite, fractures or compositional zoning. The selected zircon data are presented in Table 2 and 3, according to their Th/U ratio. This ratio, with discrepancies (Harley et al., 2007), is used to discriminate between magmatic (Th/U>0.1) and metamorphic (Th/U<0.1) zircons (e.g. Hoskin and Schaltegger, 2003). However, examples of metamorphic zircon overgrowths with Th/U values higher than 0.1 (e.g. Carson et al., 2002; Harley et al., 2001; Hokada and Harley, 2004; Hu et al., 2004; Kelly and Harley, 2005; Kröner et al., 2000; Moller et al., 2003; Pidgeon, 1992; Schaltegger et al., 1999; Vavra et al., 1999) or even igneous zircons with values lower than 0.1 have been referenced (Ladenberger et al., 2014; Schärer, 1984; Sláma et al., 2008; Zeck and Whitehouse, 1999). The extremely low Th/U ratio (≈0.01) in zircons is still considered a common discriminator of metamorphic zircons but the work of López-Sánchez et al. (2016), which describes magmatic zircon overgrowths with Th/U ratios (<0.02), questions its validity.

In this work, the Th/U ratio and the observed compositional zircon zonings can be apparently correlated with ZP 1 (low Th/U ratios from 0.01 to 0.1) and ZP 2 (high Th/U ratios, from 0.1 to 1) (Fig. 5A; Table 1), allowing to link them to a metamorphic or an igneous origin, respectively. However, due to its questionable validity, another geochemical parameter should be taken into account to link the zircons with their origin.

The Nb and Ta can also be used as discriminating elements between these two zircon populations, as ZP 2 have almost invariably higher Nb/Ta ratios than ZP 1 (Table 2, 3; Fig. 5B). When plotted in the Nb+Ta vs. Th+U diagram

| Spot       | Spot 206Pbc (%) | U (ppm) | Th (ppm) | Th/U | 207Pb/206Pb | 207Pb/206U | ε (%) | ε (%) | err. corr. |
|------------|----------------|---------|----------|------|-------------|------------|------|------|-----------|
| GRS215-9.1 | 0.05           | 530     | 7        | 0.01 | 540 ± 8     | 533 ± 28   | -1   | 0.05809| 1.3       | 0.0874    | 1.5       | 0.8      |
| GRS215-8.1 | 0.27           | 319     | 4        | 0.01 | 537 ± 8     | 521 ± 45   | -3   | 0.05777| 2.1       | 0.0868    | 1.5       | 0.6      |
| GRS215-2.1 | 0.26           | 481     | 7        | 0.01 | 534 ± 8     | 534 ± 25   | +0   | 0.05811| 1.1       | 0.0864    | 1.6       | 0.8      |
| GRS215-4.1 | 0.13           | 535     | 8        | 0.02 | 528 ± 7     | 519 ± 19   | -2   | 0.05770| 0.9       | 0.0854    | 1.4       | 0.9      |
| GRS215-11.1| 0.06           | 535     | 8        | 0.02 | 538 ± 9     | 512 ± 18   | -5   | 0.05753| 0.8       | 0.0870    | 1.7       | 0.9      |
| GRS215-1.1 | 0.47           | 460     | 7        | 0.02 | 554 ± 8     | 553 ± 31   | -0   | 0.05862| 1.4       | 0.0897    | 1.6       | 0.7      |
| GRS215-6.1 | 0.04           | 684     | 12       | 0.02 | 541 ± 7     | 532 ± 14   | -2   | 0.05805| 0.7       | 0.0876    | 1.4       | 0.9      |
| GRS215-16.1| 0.11           | 598     | 10       | 0.02 | 536 ± 7     | 550 ± 18   | +3   | 0.05854| 0.8       | 0.0867    | 1.4       | 0.9      |
| GRS215-3.1 | 0.19           | 593     | 10       | 0.02 | 535 ± 7     | 546 ± 20   | +2   | 0.05843| 0.9       | 0.0865    | 1.4       | 0.8      |
| GRS215-7.1 | 2.43           | 359     | 39       | 0.11 | 500 ± 7     | 515 ± 7    | +3   | 0.05761| 3.4       | 0.0806    | 1.5       | 0.4      |
| GRS215-13.1| 0.99           | 245     | 53       | 0.22 | 521 ± 7     | 660 ± 61   | +22  | 0.06160| 2.9       | 0.0842    | 1.5       | 0.5      |
| GRS215-15.1| 0.41           | 238     | 52       | 0.22 | 530 ± 9     | 580 ± 41   | +9   | 0.05934| 1.9       | 0.0857    | 1.8       | 0.7      |
| GRS215-10.1| 0.89           | 212     | 59       | 0.28 | 527 ± 8     | 614 ± 61   | +15  | 0.06029| 2.8       | 0.0852    | 1.5       | 0.5      |
| GRS215-12.1| 0.07           | 178     | 58       | 0.33 | 527 ± 8     | 639 ± 46   | +18  | 0.06101| 2.1       | 0.0852    | 1.5       | 0.6      |
| GRS215-5.1 | 0.20           | 196     | 75       | 0.38 | 531 ± 8     | 585 ± 37   | +10  | 0.05948| 1.7       | 0.0859    | 1.5       | 0.7      |
| GRS215-1.2 | 0.94           | 62      | 31       | 0.50 | 542 ± 9     | 550 ± 186  | +1   | 0.05854| 8.5       | 0.0877    | 1.8       | 0.2      |
| GRS215-14.1| 0.60           | 247     | 133      | 0.54 | 509 ± 14    | 481 ± 49   | -6   | 0.05672| 2.2       | 0.0821    | 2.6       | 0.8      |
| GRS215-2.2 | 0.76           | 73      | 42       | 0.57 | 528 ± 9     | 512 ± 118  | -3   | 0.05753| 5.4       | 0.0854    | 1.7       | 0.3      |

Errors are 1-sigma; Pb and Pb* indicate the common and radiogenic portions, respectively. Error in Standard calibration was 0.46% (not included in above errors but required when comparing data from different mounts).

* Common Pb corrected using measured 204Pb.
(Fig. 5C), a clear differentiation can be observed between the two zircon groups. ZP₂ zircons tend to plot towards higher Nb+Ta and lower Th+U values compared to ZP₁.

In general, it is assumed that zircons with a metamorphic origin have higher U and Hf and lower Ce concentrations than the igneous ones (e.g. Hoskin and Schaltegger, 2003). Moreover, the metamorphically grown zircons usually have flatter HREE (Heavy Rare Earth Elements) patterns, relatively low contents of HREE (and Y), and MREE (Middle Rare Earth Elements), low (Lu/Gd)ₓ, Nb/Ta, Y/Ho and Ce/Ce* ratios and high Eu/Eu* ratios than the igneous ones (e.g. Chen et al., 2010).

The chondrite-normalized REE patterns (Sun and McDonough, 1989) of the analyzed zircons (Fig. 6) are strongly enriched in HREE compared to LREE (Light Rare Earth Elements) and show prominent anomalies in Ce (positive) and Eu (negative). The ranges of normalized REE patterns of both zircon populations overlap (Fig. 6), but differ from normalized trends for the HREE. The ZP₁ zircons have flat HREE patterns [(Lu/Gd)ₓ ratios up to 10] whereas ZP₂ shows steeper patterns [(Lu/Gd)ₓ ratios up to 46]. In the LREE, MREE or HREE vs. Th/U plots (Fig. 5D-F) the REE contents do not show significant variations but increases slightly as the Th/U increases in ZP₂.

In the Th/U vs. Hf plot (Fig. 5G), the Hf concentration decreases from ZP₁ to ZP₂. The U/Ce vs. Th/U or Th plots can be considered as potential indicators of metamorphic and igneous zircons since
the magmatic zircons show little variation in U/Ce while the metamorphic ones are enriched in U due to increase in water content during dehydration reactions (Castiñeiras et al., 2011). In our study, the analyzed zircons are again split into two main populations: ZP1 is characterized by high Th and little variation in the U/Ce content, and the ZP2 by low Th and highly variable U/Ce contents (Fig. 5H). The application of Grimes et al. (2007) diagrams agrees with zircons evolved from a continental crust (Fig. 7).

**DISCUSSION**

The zircons from the Rio de los Sauces granite yielded uncommon geochemical signatures and SHRIMP results. The following results stand out twofold as i) two zircon populations (ZP1 and ZP2) with different textures and geochemical signatures and ii) a bimodal age distribution in the geochemical two end-members, 537±4.8 and 529±6Ma, have been identified. According to presented geochemical features, zircon textural analysis and Ti-in-zircon geothermometry we proposed that the identified ZP1 and ZP2 can be correlated with metamorphic and igneous zircons populations respectively, and do confirm the utility of the Th/U, U/Ce and Nb/Ta as potential ratios for zircon nature indicator. These new data aid in the understanding of the metamorphic evolution of the Calamuchita complex migmatites and help to constrain the time gap between the Pampean HT metamorphism and the crystallization of anatectic granites.

Melting products forming leucosomes in migmatites are commonly related to the formation of sheets like the leucogranitic bodies (Barzola et al., 2021). Melting products forming leucosomes in migmatites are commonly related to the formation of sheets like the leucogranitic bodies (Barzola et al., 2021).
TABLE 3. Laser ablation trace, REE and calculated temperature data for analyzed igneous zircons (ZP) from Rio de los Sauces. In ppm. <dl:< under detection limit

| Sample | GRS 215 | Source file |
|--------|---------|-------------|
| P      | 32      | 29         | 9         | 19         | 26         | 50         | 68         | 120       | 78         | 19         | 43         |
| Sc     | 48      | 58        | 50         | 42         | 37         | 37         | 35         | 39         | 38         | 36         | 34         |
| Ti     | 64      | 56        | 68         | 50         | 42         | 41         | 38         | 39         | 36         | 34         | 32         |
| V      | 70      | 60        | 55         | 42         | 36         | 35         | 34         | 37         | 35         | 34         | 32         |
| Cr     | 80      | 72        | 66         | 58         | 52         | 51         | 49         | 50         | 48         | 47         | 46         |
| Mn     | 90      | 84        | 79         | 70         | 64         | 62         | 61         | 63         | 62         | 61         | 59         |
| Fe     | 100     | 95        | 90         | 82         | 78         | 76         | 75         | 77         | 75         | 74         | 72         |
| Co     | 200     | 180       | 170        | 160        | 150        | 140        | 130        | 130        | 120        | 110        | 100        |
| Ni     | 600     | 560       | 520        | 480        | 450        | 440        | 430        | 420        | 410        | 400        | 390        |
| Cu     | 1000    | 950       | 900        | 850        | 800        | 750        | 700        | 650        | 600        | 550        | 500        |
| Zn     | 2000    | 1900      | 1800       | 1700       | 1600       | 1500       | 1400       | 1300       | 1200       | 1100       | 1000       |
| Ga     | 6000    | 5500      | 5000       | 4500       | 4000       | 3500       | 3000       | 2500       | 2000       | 1500       | 1000       |
| Ge     | 15000   | 14000     | 13000      | 12000      | 11000      | 10000      | 9000       | 8000       | 7000       | 6000       | 5000       |

The presence of zircons with metamorphic affinity in the Río de los Sauces granite suggests the incomplete assimilation of the zircons derived from the metamorphic protoliths and opens an opportunity to study the timing of the metamorphism prior to the emplacement of the pluton. The mean temperatures of 735±17 and 750±22°C determined by Ti-in-zircon in metamorphic and igneous zircons, respectively, are, although slightly lower, coherent to the calculated metamorphic conditions in the Sierras de Córdoba (Fagiano, 2007; Otamendi et al., 2004; Rapela et al., 1998). Since the crystallization temperatures calculated for igneous and metamorphic zircons are within the uncertainty of each other, a further increase in the regional temperature during anatexis cannot be inferred. Finally, the age of the climax of the Pampamento metamorphism has been constrained between 537 and 529Ma, and the Concordia age of 334±4 (2σ)Ma,
obtained from both types of zircons, has been considered as the mean age of the HT Pampean metamorphism in the Sierras de Córdoba.

**CONCLUSIONS**

The presence of zircons with metamorphic and igneous affinities in the Río de los Sauces granite, identified by LA-ICP-MS analyses, suggest their incomplete assimilation during the anatexis and open the opportunity to date both processes from the same sample.

U-Pb SHRIMP analyses from both kind of zircons suggest that the Río de los Sauces granite crystallized at 529±6 (2σ) Ma, whereas the age of 537.1±4.8 (2σ) Ma refers to the Pampean metamorphism prior to the
emplacement of the pluton, and could be considered as a good approximation for the Pampean HT metamorphism of the study area.

A time gap of ≈8Myr is established between the metamorphism and the granite crystallization.

The Ti-in-zircon geothermometry yielded temperatures of ca. 750±22°C for the granite crystallization and thus constraint the minimum temperature reached during the anatexis.

According to obtained and previously published data, we suggest that LA-ICP-MS and U-Th-Pb SHRIMP analyses should be performed together in order to achieve a well geochemical zircon characterization, previous to the granitoid dating.

The Th/U, U/Ce and Nb/Ta ratios can be considered as very useful markers to infer the igneous or metamorphic nature of the zircons.

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