INTRODUCTION

The study of xenoliths in volcanic pipes can enable the characterization of the magmatic and metamorphic rocks that constitute the crust at depth. This approach is particularly relevant where such rocks that have formed at deeper crustal levels are not exposed in a given basin or occur hundreds of kilometres away from the basin with no apparent relationship with it. The ascent of magma to its level of extrusion may involve the incorporation of fragments of solid rock material from the host rocks through which it has passed (e.g. Dostal et al., 2005; Puelles et al., 2019). Therefore, a volcanic pipe that cuts a sedimentary basin, whose basement rocks are unknown, may provide evidence of the deeper crustal rocks preserved as xenoliths, of which granites are a good example. This paper describes the field occurrence of two granitic xenoliths from the Papôa intrusive volcanic breccia in the Jurassic strata of the Lusitanian Basin (western Iberia).
In the present study, for the first time, zircon grains extracted from xenoliths of granite from a post-Sinemurian volcanic breccia are described using cathodoluminescence imaging and SHRIMP U-Pb geochronology. The findings are compared with the available literature and used to discuss the pre-Mesozoic basement of the Lusitanian Basin. We have considered not only the Paleozoic tectonic units from the Iberian Massif but also those located in other parts of the Appalachian-Variscan belt, such as Nova Scotia and southwest England. The analysis of the composition and age of the breccia matrix and other type of xenoliths are beyond the objective of the present study.

GEOLOGICAL SETTING

Lusitanian Basin

In western Iberia, the Mesozoic Lusitanian Basin extends landwards for about 250 kilometres along the coastline of Portugal from Setúbal to Porto (Fig. 1). In the Peniche region, a well-preserved Lower Jurassic section more than 450m thick can be observed (Duarte et al., 2004). This section includes the Toarcian Global Boundary Stratotype Section and Point (GSSSP) of the International Commission on Stratigraphy (Rocha et al., 2016) (Fig. 2A). The Triassic–Jurassic boundary is not recognised in Peniche, but marls with gypsum and dolomitic limestones of the Rhaetian–Lias include from bottom to top: bioclastic limestones with ammonites (Pliensbachian Vale das Fontes Formation), centimetre-thick layers of marls (Sinemurian–Pliensbachian Água de Madeiros formations, composed of bioclastic limestones and dolomitic limestones interbedded with marls (Duarte and Soares, 2002; Duarte, 2007). The carbonate sequence includes from bottom to top: bioclastic limestones with brachiopods and bivalves interbedded with centimetre-thick layers of marls (Sinemurian–Pliensbachian Água de Madeiros Formation), marls and limestones with crinoids (Pliensbachian Vale das Fontes Formation), centimetre-thick layers of limestones and marls containing belemnites and ammonites (Pliensbachian Lemedhe Formation), and marls and detrital limestones with crinoids (Toarcian Carvoeiro Formation) (Duarte and Soares, 2002; Duarte et al., 2004).

Magmatic activity in the Lusitanian Basin occurred in the Lower Jurassic (c. 200-180Ma) and Cretaceous (c. 147-141 and 94-72Ma) (Fig. 1B; Grange et al., 2008; Kullberg et al., 2013; Miranda et al., 2009). Given the absence of absolute ages, field relations provide the only basis for arguing that the Papôa volcanic breccia is younger than the Lower Jurassic strata. Some authors have pointed to the Cretaceous as the most probable age for this volcanic episode (Andrade, 1979; Romariz, 1963-1964).

Pre-Mesozoic basement

The structural complexity of the pre-Mesozoic basement of the Iberian Massif, that is unconformably overlain by the Mesozoic Lusitanian Basin, resulted from the progressive amalgamation of different tectonic units during the course of the Gondwana and Laurussia collision, forming the Appalachian-Variscan orogeny (Matte, 2001). In the Iberian Massif, an allochthonous nappé pile formed at c. 390-365Ma as a result of the continental subduction (Galicia-Trás-os-Montes Zone, GTMZ; Martínez Catalán et al., 2019). The following evolutionary stages of this collisional belt (c. 365-315Ma) led to the spatial reorganisation of
the different tectonic units derived from the margins of Gondwanan (Central Iberian Zone, CIZ, and Ossa Morena Zone, OMZ) and Laurussian (South Portuguese Zone, SPZ) involved in the Carboniferous collision (Azor et al., 2019).

The latest adjustments to this orogenic system configuration were caused by c. 315-300Ma strike-slip shearing and upright folding related to the waning stages of the continental collision (Diez Fernández and Pereira, 2017; Gutiérrez-Alonso et al., 2015; Martínez Catalán et al., 2007). Regarding the above-mentioned, we might expect that the basement of the Lusitanian Basin would present distinctive characteristics in accordance with the underlying Paleozoic tectonic zone (Pereira et al., 2016), but there is still debate on what this is. The pre-Mesozoic basement flanking the Lusitanian Basin is represented by four tectonic zones showing differences in terms of stratigraphy, deformation, metamorphism, and magmatism: the CIZ, GTMZ, OMZ and SPZ (Martínez-Catalán et al., 2007; Fig. 1A). Stratigraphy of the CIZ and OMZ includes Neooproterozoic to Permian rocks with some significant stratigraphic gaps (Fig. 3). These rocks include Ediacaran successions of greywackes and pelites interbedded with volcanic rocks formed in a magmatic arc (Cadomian orogeny; Pereira et al., 2012; Talavera et al., 2015). In the OMZ, Cambrian sedimentation is continuous and mostly siliciclastic, including two minor periods of carbonate production (Sánchez García et al., 2019) (Fig. 3). Cambrian siliciclastic rocks and minor carbonate rocks are only represented in the northern and central domains of the CIZ, reaching up to Cambrian Series 2, Stage 4 (Dias da Silva et al., 2014; Gutiérrez-Marco et al., 2019). In both tectonic zones, Ordovician to Lower Devonian marine siliciclastic sedimentation is relevant and include a few unconformities (Gutiérrez-Marco et al., 2019; Robardet and Gutiérrez-Maro, 2004), but Middle Devonian sedimentary rocks are almost absent (Robardet, 2003) (Fig. 3). In the interval c. 530-470Ma magmatism was significant in the OMZ (Diez Fernández et al., 2015; Sánchez-García et al., 2019), while in the CIZ, igneous rocks have yielded Upper Cambrian to Upper Ordovician ages (c. 498-450Ma; Colmenar et al., 2017; Dias da Silva et al., 2015; Montero et al., 2007; Rubio-Ordóñez et al., 2012) (Fig. 3). Furthermore, c. 460-455Ma volcanic rocks are found in the structurally higher GTMZ parautochthonous unit, which presents a stratigraphic record comparable with that of the CIZ (i.e. Autochthon) (Dias da Silva et al., 2016). Upper Devonian magmatic rocks and high-grade metamorphism are unknown in the CIZ and OMZ (Fig. 3). The only exception is a few gabbro-dioritic plutons in the OMZ that have yielded questionable ages of 362±13Ma and 376±22Ma (Rb-Sr, K-Ar and Sm-Nd ages; Ribeiro et al., 2019). Upper Devonian to Lower Carboniferous marine siliciclastic rocks are overlain by Upper Carboniferous terrestrial strata in the CIZ and OMZ (Martínez-Catalán et al., 2007; Quesada et al., 1990) and by Upper Carboniferous marine strata in the SPZ (Oliveira, 1990; Pereira et al., 2020). Carboniferous and Lower Permian magmatism (c. 350-290Ma) is widespread in the Iberian Massif (Castro et al., 2002; Dias da Silva et al., 2018; Fernández-Suarez et al., 2011; Pereira et al., 2015, 2017a) (Fig. 3). In the western Iberian Massif, the dextral movement of the Porto-Tomar fault zone at c. 310-308Ma (Gutiérrez-Alonso et al., 2015; Pereira et al., 2010) caused the lateral displacement of the adjacent CIZ, OMZ, and SPZ, resulting in a much greater tectonic complexity in the pre-Mesozoic basement of the Lusitanian Basin.

The discussion on the nature of the Paleozoic tectonic unit that constitutes the pre-Mesozoic basement of the Lusitanian Basin is still ongoing, with a number of authors pointing to a range of possibilities: i) the CIZ (Terrinha et al., 2019); ii) the OMZ and CIZ (Oliveira et al., 1992; Pereira et al., 2016); iii) the OMZ and SPZ (Alves, 2011; Capdevila and Mougenot, 1988; Kullberg et al., 2013); iv) the SPZ (Ribeiro et al., 2007); and v) the CIZ, OMZ and SPZ (Pimentel and Pena dos Reis, 2016). However, there is a clue that may help to solve the puzzle. Pre-Mesozoic basement rocks are described in the Berlengas and Fariñhões islands as being exposed along a horst on the continental shelf northwest of Peniche (Camarate França et
Granite from the Berlengas isles was first dated at 280±15 Ma (Rb-Sr whole-rock; Priem et al., 1965) and recently at 305±1 Ma (U-Pb on zircon and monazite fractions; Ribeiro et al., 2019; Valverde-Vaquero et al., 2011). A sample of anatectic granite/diatexite associated with paragneiss migmatites of the Farilhões isles (Bento dos Santos et al., 2010) yielded 376±3 Ma (U-Pb on monazite fractions; Valverde-Vaquero et al., 2011), interpreted as representing the best estimate for the age of high-grade metamorphism. Unfortunately, contact relationships between Berlengas granite and Farilhões high-grade metamorphic rocks have not yet been determined.

RATIONALE AND SAMPLING

In this study, two fresh xenoliths of granitic rocks were sampled from the Papôa volcanic breccia (Figs. 2B-C; 4A) for the characterization of the pre-Mesozoic basement of the Lusitanian Basin. The aim was to date granitic rocks derived from the underlying basement. Using the new U-Pb data, we aim to discuss: i) the source of the granitic xenoliths, and ii) the relation between the xenoliths of granite and quartz-feldspathic metamorphic rocks and the Paleozoic basement of the Berlengas and Farilhões isles. If a link between them can be established, as previously suggested by Andrade (1979) and Romariz (1963-1964), a comparative analysis with the pre-Mesozoic basements of the Appalachian-Variscan belt located both in the Iberian Massif and outside it (Meguma terrane in Nova Scotia and Rhenohercynian Zone in SW England) could be attempted.

In the study area of Papôa, the NE-SW-trending 30 metres-wide dyke of a matrix-supported mafic breccia cross-cutting the Lower Jurassic strata seems like a sub-vertical volcanic pipe (Fig. 2B). The matrix resembles coarse to fine-grained mafic tuff (Fig. 3A, B) with abundant rock fragments and great variation in size (Figs. 2C; 4A–D). In the dark tuffitic matrix which is commonly intensely altered and replaced by soft yellowish clay, rounded to subangular xenoliths ranging from millimetres to metres in size stand out, (Fig. 4A, B, D). Xenoliths occur in four main groups, each reflecting a different source: i) granitic rocks; ii) quartz-feldspathic foliated rocks (high-grade metamorphic rocks and/or deformed granitic rocks); iii) mafic (basaltic?), pumice, and fine-grained tuffitic rocks; and iv) quartzitic and carbonate hornfels derived from the host sedimentary rocks. Groups i) and ii) represent the deepest crustal sources and often are not easy to distinguish due to the intense weathering. Groups iii) and iv) represent the deeper crustal sources and are derived from sources formed during the subvolcanic process; The two xenoliths of granitic rocks selected for U-Pb geochronology had elongated shapes and were sub-angular to rounded, with a diameter of more than 20 centimetres. They were remarkably fresh, contrary
to what is often found due to intense weathering. Sample Xpp-2 coarse-grained biotite granite (Fig. 4E) was mainly composed of quartz, K-feldspar prevailing over plagioclase, and biotite, also including opaque minerals, muscovite, zircon, and apatite. This sample appeared to have been derived from a source with textural and compositional characteristics similar to those of Berlengas granite. Sample Xpp-3 coarse-grained two-mica granite (Fig. 4F), consisted of quartz, K-feldspar, plagioclase, muscovite, and biotite as their main components. It also included monazite, apatite, opaque minerals, and zircon as accessory minerals.

U-Pb GEOCHRONOLOGY

Zircon grains for U-Pb geochronology were selected using traditional techniques: density separation using a sieve with a mesh size of less than 500 microns, density (panning) separation procedures, and mineral identification using a binocular lens and preparation of epoxy resin mounts with zircon grains (Universidade de Évora, Portugal). Cathodoluminescence imaging and U-Pb measurements using SHRIMP were carried out at IBERSIMS (Universidad de Granada, Spain). Zircon grains were tracked by the primary beam during 120s prior to analysis and then analyzed over 6 scans following the isotope peak sequence $^{196}$Zr, $^{204}$Pb, 204.1 backgrounds, $^{208}$Pb, $^{207}$Pb, $^{208}$Pb, $^{238}$U, $^{248}$Th, $^{254}$U. Every peak of each scan was measured sequentially 10 times: 2s for mass 196; 5s for masses 238, 248, and 254; 15s for masses 204, 206, and 208, and 20s for mass 207. The primary beam, composed of $^{16}$O$^{16}$O$^{2+}$, was set to an intensity of 4-5nA, using a 120μm Kohler aperture, which generates 17x20μm elliptical spots on the zircon surface. The secondary beam exit slit was set at 80μm, reaching a resolution of about 5000 at 1% peak height. All calibration procedures were performed on the TEMORA zircon, SL13 zircon, and GAL zircon standards, and mass calibration was done using GAL zircon (ca. 480Ma; Montero et al., 2008). Analytical sessions used SL13 zircon (Claué-Long et al., 1995) as a concentration standard of U= 238ppm. TEMORA zircon (ca. 417Ma, Black et al., 2003) was used as isotope ratio standard having been subject to measurement every 4 unknowns. Data reduction was accomplished using SHRIMPTOOLS software (www.
Xenolith of biotite granite (sample Xpp-2)

Coarse-grained biotite granite (sample Xpp-2) contained stubby-to-elongated euhedral zircon grains (80-350μm in diameter). Zircon grains mostly showed well-developed crystal faces. Long prisms had a length-to-width ratio of up to 4:1. CL-imaging imaging showed simple grains with growth zoning typical of magmatic zircon (Corfu et al., 2003) varying from narrow to faint and showing broad zoning. Composite grains included unzoned cores or showed oscillatory growth zoning, banded zoning and sector zoning. The cores were mantled by narrow low-luminescence-to-broad rims with oscillatory zoning (Fig. 5). U and Th ranged from 202 to 3996ppm and from 42 to 1488ppm respectively, and Th/U ranged from 0.02 to 1.4. The average Th/U ratio of 0.4 is typical of igneous origin (Hoskin and Schaltegger, 2003).

Of a total of 22 U-Th-Pb isotopic analyses, 15 with a discordance of <5% yielded a weighted mean of 295±5Ma (MSWD= 4.2), indicating overdispersion or overestimated uncertainties (Fig. 5). By excluding six analyses (4.1, 9.1, 11.1, 16.1, 18.1 and 21.1), a weighted mean 206Pb/238U age of 298±4Ma (MSWD=1.7) was obtained. This age is interpreted as the best estimate for the crystallisation age of plutonic rock, which coincided within the error with the TuffZirc age of 299±3Ma (Fig. 5).
Xenolith of two-mica granite (sample Xpp-3)

Coarse-grained two-mica granite (sample Xpp-3) mostly included elongated zircon grains (70–420μm in diameter) with a few very long prisms and a 6:1 length-to-width ratio. Simple grains showed concentric oscillatory zoning with variable width or banded zoning. CL-imaging demonstrated the complex nature of composite grains, including cores of variable appearance with concentric zoning, banded zoning, and unzoned cores, which were surrounded by rims with concentric oscillatory zoning or were homogeneous and presented low level of luminescence (Fig. 6). A few cores showed the development of irregular domains of homogeneity, cutting discordantly across growth zoned domains, suggesting modifications during late and post-magmatic cooling (Corfu et al., 2003). U content, although variable, was quite high, ranging from 730 to 7847ppm, while Th values ranged from 99 to 1938ppm, resulting in Th/U ratios ranging from 0.04 to 1.24 (Table I), and an average Th/U ratio of 0.26. A total of 18 U-Th-U isotopic analyses, with a discordance of <5%, yielded a weighted mean of 293±4Ma (MSWD= 3.5) (Fig. 6).

The high MSWD value is associated with the scattering of data points. 207Pb/206Pb and 206Pb/238U ratios are well defined for a cluster of 10 analyses, yielding a weighted mean age of 292±2Ma (MSWD= 1.09) and a TuffZirc age of 291±3Ma, regarded as the crystallisation age of two-mica granite (Fig. 6). In this interpretation, the three older ages that provide a weighted mean of 305±5Ma (MSWD= 0.13; Fig. 6), probably represent inherited grains from a former zircon forming event. Alternatively, the scattering of data could be explained by the combination of Pb-loss, common Pb correction, and/or recrystallization (e.g. Kroner et al., 2014), and thus, the best estimation of the crystallisation age of two-mica granite could be c. 305Ma (i.e. similar to the Berlengas granite). A zircon grain showing two oscillatory zoning domains separated by a thin dark unzoned domain was analysed. The most internal oscillatory zoning domain yielded a concordant age of 307±3Ma, whereas a discordant age of c. 277±8Ma (Fig. 6), suggesting isotopic disturbance of grain structure. Thus, given the uncertainties of the data set is desirable to quote a maximum (c. 305Ma) and a minimum (c. 291Ma) crystallization age for this granitic rock.

FIGURE 6. Concordia diagram (blue ellipses- older ages), weighted mean of 207Pb/235U, and TuffZirc ages of analysed zircon grains of sample Xpp-3 two-mica granite. CL images of representative zircon grains.
DISCUSSION

The Papôa volcanic breccia, first described by Choffat (1880), has been traditionally interpreted as a fragment of a volcanic cone that was preserved from erosion due to the collapse of a crustal block between two subvertical normal faults (Andrade, 1979). However, our understanding is not the same, admitting that it is a subvertical dike cross-cutting the Lower Jurassic sedimentary host. The most abundant xenoliths in the Papôa volcanic breccia are of mafic tuff and of fine-grained material probably derived from previous crystallization in the dyke walls or representing lava fragments. Mafic xenoliths probably derived from the fragmentation of material from the walls of the subvolcanic pyroclastic dike, and/or from the falling back of pyroclastic material, pumice, welded-tuff, and lava fragments (Kano et al., 1997; Motoki et al., 2012; Winter et al., 2008). Xenoliths of metamorphic rocks mostly comprise carbonate rocks and also siliciclastic rocks derived from the host Mesozoic sedimentary sequence of the Lusitanian Basin. Granitic and quartz-feldspathic foliated rocks also occur as xenoliths, representing the pre-Mesozoic basement of the Peniche region (Fig. 7A). Xenoliths of granitic rocks present little compositional variation but show significant textural differences, including aplitic and pegmatitic textures. In some cases, they are altered and difficult to distinguish from quartz-feldspathic metamorphic rocks. These xenoliths of granitic rocks and quartz-feldspathic foliated metamorphic rocks, of probable upper and middle crust provenance, are comparable lithologically and geochemically to the pre-Mesozoic basement rocks forming the Berlengas and Farilhões isles (Camarate França et al., 1960; Freire de Andrade, 1937; Rosa et al., 2019) (Fig. 7B).

The new geochronology data obtained from the granitic xenoliths of the Papôa volcanic breccia show that the pre-Mesozoic basement of the Lusitanian Basin includes Permo-Carboniferous granites (c. 298 and 305-291Ma; this study). A number of provenance hypotheses may be advanced to explain the presence of xenoliths of Perman granitic rocks in the Papôa volcanic breccia. In the Iberian Massif, Permain magmatism is present throughout the southwest Iberian Massif. In the Meguma terrane, this study coincide within the margin of error with the age of crystallisation of the Permo-Carboniferous plutons of the SPZ and Rhenohercynian Zone. Permain igneous activity is not recognised in the Meguma terrane (Don Hermes and Murray, 1988). Meguma terrane stratigraphy includes Cambrian to Early Orдовician sedimentary rocks unconformably overlain by Silurian-Early Devonian sedimentary rocks dated at 446-434Ma (Keppie and Krogh 2000; White and Barr, 2012; White et al., 2018).

This Early Paleozoic succession may represent the covered, unknown oldest stratigraphic record of the SPZ in the southwest Iberian Massif. In the Meguma terrane, the Early Paleozoic sequence is intruded by c. 382-357Ma plutons (including the South Mountain and Musquodoboit batholiths, Clarke et al., 1997; Tate and Clarke, 1995). In Nova Scotia, the Late Devonian sequence comprises sedimentary rocks interbedded with volcanic rocks (Doig et al., 1996; Murphy et al., 2018; Pe-Piper et al., 2004). Zircon and monazite U-Pb ages obtained from granulate facies metasedimentary xenoliths from a Late Devonian mafic dyke that intrudes the Meguma terrane, yielded three age groups at c. 399, 377 and 354Ma, interpreted as representing metamorphic events (Greenough et al., 1999; Shellnutt et al., 2018). One of these three age groups recorded in the Meguma terrane was contemporaneous with the widespread plutonism (c. 378-368Ma) that involves the mafic stocks and large granitic plutons of the Musquodoboit and South Mountain
FIGURE 7. A) Sketch of the Lusitanian Basin stratigraphy overlying the pre-Mesozoic basement, showing the contact between the active feeder dyke and the host-rock sequences. B) Schematic sketch of the Papõa pyroclastic eruption showing entrapment of xenoliths, derived from distinct sources, in the ascending magmas.
batholiths (Moran et al., 2007). It deserves special mention because the metamorphic rocks of the Farilhões isles also experienced high-grade metamorphic conditions under granulite facies (Ribeiro et al., 2019) dated at 376±3Ma (Valverde Vaquero et al., 2011). By contrast, Late Devonian high-grade metamorphism has not been reported in the OMZ and CIZ (Fig. 3). Furthermore, U-Pb geochronology of magmatic rocks from the Iberian Pyrite Belt (Oliveira et al., 2013; Paşlawski et al., 2020; Rosa et al., 2008) and the Sierra Norte Batholith (Braid et al., 2012; Gladney et al., 2014; De la Rosa et al., 2002) in the SPZ, likewise provided Late Devonian-Early Carboniferous ages of c. 374-335Ma.

Whatever the case, as there are no dating for xenoliths of foliated quartz-feldspathic rocks, we cannot rule out the hypothesis that they may represent high degrees of partial melting of the basement entrapped in ascending magma (Dostal et al., 2005) contemporaneous with the formation of Papôa volcanic breccia. A detailed petrographic and geochronological study of the different sedimentary rocks and quartz-feldspathic xenoliths remains to be carried out.

CONCLUSIONS

The age of the granitic xenoliths from the Papôa volcanic breccia (c. 298 and c. 305-291Ma) corroborates the previous hypotheses, based on field, petrography, geochemical, and geochronology data, that the Lusitanian Basin rests on a pre-Mesozoic basement that includes Permo-Carboniferous intrusions like those described in the Berengas and Farilhões isles. This enable us to posit that: i) Permo-Carboniferous magmatism probably lasted at least 14Ma, from c. 305 to 291Ma, in this region, similarly to what happened in others regions of the Appalachian-Variscan belt; and ii) the Lusitanian Basin basement was affected by the Permo-Carboniferous granite intrusion and Upper Devonian high-grade metamorphism and magmatism, as evident in the Paleozoic terrain with Laurussian affinity including the SPZ, the Rhenohercynian Zone and the Meguma terrane. The present study thus confirms that the pre-Mesozoic basement represented by the OMZ and CIZ (Gondwana) was still connected to SPZ-Rhenohercynian Zone-Meguma Terrane (Laurussia) during the Mesozoic evolution of the Lusitanian Basin. Thus, the rifting that led to the opening of the Central Atlantic Ocean left behind large fragments of Paleozoic Laurussian continental material stranded in the western Iberian margin.

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### TABLE 1. Zircon U-Pb geochronological data (granitic xenoliths in the Papôa volcanic breccia; Xpp-2, 39º22'27.58''N; 9º22'36.78 ''W; Xpp-3, 39º22'25.60''N; 9º22'37.70''W)

| Sample | % isotope ratios | ages (Ma) |
|--------|-----------------|-----------|
| Xpp-3-1 | 28.00 | 28.00 |
| Xpp-3-2 | 30.00 | 30.00 |
| Xpp-3-3 | 28.00 | 28.00 |
| Xpp-3-4 | 30.00 | 30.00 |
| Xpp-3-5 | 28.00 | 28.00 |
| Xpp-3-6 | 30.00 | 30.00 |
| Xpp-3-7 | 28.00 | 28.00 |
| Xpp-3-8 | 30.00 | 30.00 |
| Xpp-3-9 | 28.00 | 28.00 |
| Xpp-3-10 | 30.00 | 30.00 |
| Xpp-3-11 | 28.00 | 28.00 |
| Xpp-3-12 | 30.00 | 30.00 |
| Xpp-3-13 | 28.00 | 28.00 |
| Xpp-3-14 | 30.00 | 30.00 |
| Xpp-3-15 | 28.00 | 28.00 |
| Xpp-3-16 | 30.00 | 30.00 |
| Xpp-3-17 | 28.00 | 28.00 |
| Xpp-3-18 | 30.00 | 30.00 |
| Xpp-3-19 | 28.00 | 28.00 |
| Xpp-3-20 | 30.00 | 30.00 |
| Xpp-3-21 | 28.00 | 28.00 |
| Xpp-3-22 | 30.00 | 30.00 |
| Xpp-3-23 | 28.00 | 28.00 |
| Xpp-3-24 | 30.00 | 30.00 |
| Xpp-3-25 | 28.00 | 28.00 |
| Xpp-3-26 | 30.00 | 30.00 |
| Xpp-3-27 | 28.00 | 28.00 |
| Xpp-3-28 | 30.00 | 30.00 |
| Xpp-3-29 | 28.00 | 28.00 |
| Xpp-3-30 | 30.00 | 30.00 |

**Errors at 1σ (1 sigma level)**

Pre-Kapre errors, calculated on replicates of the TEMORA standard at 9.0% confidence intervals, are 0.3% for 206Pb/204Pb and 0.3% for 207Pb/206Pb.
Continued

| Sample ID | Initial Isochron Ratio | Normalized Ratio | Initial Age (Ma) | Normalized Age (Ma) | Delta Age (Ma) | Delta % | Delta (Ma/a) |
|-----------|------------------------|------------------|------------------|---------------------|---------------|---------|--------------|
| Xpp-2-1   | 0.04733 0.0010 0.04694 | 0.03013 0.0005 | 0.30317 0.0011 | 0.2913 0.0008 | 0.1505 | 4.8 | -1.8 |
| Xpp-2-2   | 0.05184 0.0008 0.05184 | 0.03013 0.0005 | 0.30317 0.0011 | 0.2913 0.0008 | 0.1505 | 4.8 | -1.8 |
| Xpp-2-3   | 0.05127 0.0008 0.05127 | 0.03013 0.0005 | 0.30317 0.0011 | 0.2913 0.0008 | 0.1505 | 4.8 | -1.8 |
| Xpp-2-4   | 0.05184 0.0008 0.05184 | 0.03013 0.0005 | 0.30317 0.0011 | 0.2913 0.0008 | 0.1505 | 4.8 | -1.8 |
| Xpp-2-5   | 0.05184 0.0008 0.05184 | 0.03013 0.0005 | 0.30317 0.0011 | 0.2913 0.0008 | 0.1505 | 4.8 | -1.8 |
| Xpp-2-6   | 0.05184 0.0008 0.05184 | 0.03013 0.0005 | 0.30317 0.0011 | 0.2913 0.0008 | 0.1505 | 4.8 | -1.8 |
| Xpp-2-7   | 0.05184 0.0008 0.05184 | 0.03013 0.0005 | 0.30317 0.0011 | 0.2913 0.0008 | 0.1505 | 4.8 | -1.8 |
| Xpp-2-8   | 0.05184 0.0008 0.05184 | 0.03013 0.0005 | 0.30317 0.0011 | 0.2913 0.0008 | 0.1505 | 4.8 | -1.8 |
| Xpp-2-9   | 0.05184 0.0008 0.05184 | 0.03013 0.0005 | 0.30317 0.0011 | 0.2913 0.0008 | 0.1505 | 4.8 | -1.8 |

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