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# A U–Pb zircon age (479 ± 5 Ma) from the uppermost layers of the Ollo de Sapo Formation near Viveiro (NW Spain): implications for the duration of rifting-related Cambro-Ordovician volcanism in Iberia

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**Abstract** – The uppermost metavolcanic layer of the Cambro-Ordovician Ollo de Sapo Formation, the largest accumulation of pre-Variscan igneous rocks in the Iberian Peninsula, have been dated in its northernmost part using U–Pb SHRIMP-RG zircon age techniques at 479.0 ± 4.7 Ma. The age obtained is the youngest age found so far in the metavolcanic facies of Ollo de Sapo Formation and represents the cessation of the rifting-related Cambro-Ordovician Ollo de Sapo volcanism at the northernmost tip of the Iberian Peninsula. Our results show that the Cambro-Ordovician volcanism in the NW of the Iberian Peninsula is not as short-lived as previously thought and confirm the correlation between the Cambro-Ordovician volcanic sequences that crop out in the Central Iberian Zone and the French Southern Armorican Massif. Finally, our study suggests that the cessation of the Cambro-Ordovician volcanism along the Ibero-Armorican Arc was synchronic or, less probably, slightly diachronic with younger ages towards the north (in present-day geographical coordinates).

Keywords: Cambro-Ordovician volcanism, Ollo de Sapo Formation, Iberian Massif, northern Gondwana break-up, U–Pb SHRIMP-RG zircon dating.

## 1. Introduction

The Ollo de Sapo Formation (Parga-Pondal, Matte & Capdevila, 1964) is the largest accumulation of pre-Variscan igneous rocks in the Iberian Peninsula. It crops out at the core of an antiform, the Ollo the Sapo anticlinorium, which extends for 600 km from the Cantabrian coast near Viveiro southwards to Hiendelaencina following the trend of Variscan structures in the Iberian Peninsula (Fig. 1). Its stratigraphic position, until recently a matter of debate, was clearly established inside a siliciclastic sedimentary sequence (Capas de los Montes Formation; Riemer, 1963) located between the Arenigian Armorican-type Quartzite and the lower Cambrian Vegadeo Limestone (Arias, Farias & Marcos, 2002). The Ollo de Sapo Formation mostly consists of felsic metavolcanic rocks, ignimbrites and rhyodacitic tuffs (Navidad, 1978; Navidad, Peinado & Casillas, 1992; Díez-Montes, 2007; C. Talavera, unpub. PhD thesis, 2008) transformed into gneisses during the Variscan Orogeny. These metavolcanites are sometimes interbedded with immature feldspathic greywackes, pelites or sandstones (e.g. Ortega *et al.* 1996; Díez-Montes, 2007). Metagranites are also common but far less abundant than metavolcanic rocks (Montero *et al.* 2009).

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In the Ollo de Sapo metavolcanic sequence two main facies can be differentiated: coarse-grained in the lower part of the sequence and medium–fine-grained in the upper part. The main difference between them is the presence in the former of large (5–15 cm) K-feldspar megacrystals and oligoclase phenocrysts surrounded by a fine-grained and strongly foliated felsic peraluminous groundmass, which is common to both facies. Both facies show quartz phenocrysts with a noticeable blue colour resulting from sagenitic rutile inclusions (Parga-Pondal, Matte & Capdevila, 1964). Based on geological (lack of coeval regional metamorphism and deformation), chemical, isotopic and chronological evidence, this Cambro-Ordovician magmatic event has been recently associated with a rifting geodynamic environment (i.e. with the northern Gondwana break-up; Valverde-Vaquero & Dunning, 2000; Bea *et al.* 2007; Montero *et al.* 2009; Díez-Montes, Martínez-Catalán & Bellido-Mulas, 2010; Ballevrè *et al.* 2012).

Dating the Ollo de Sapo Formation protolith has historically been difficult because of the unusually elevated fraction of inherited zircon components, *c.* 70–80 % and in some cases nearly 100 % (Bea *et al.* 2007; Montero *et al.* 2007). Early attempts at dating the Ollo de Sapo Formation yielded ages ranging from Ediacaran to Ordovician (Lancelot, Allegret & Iglesias-Ponce-de-León, 1985; Viallete *et al.* 1986, 1987;

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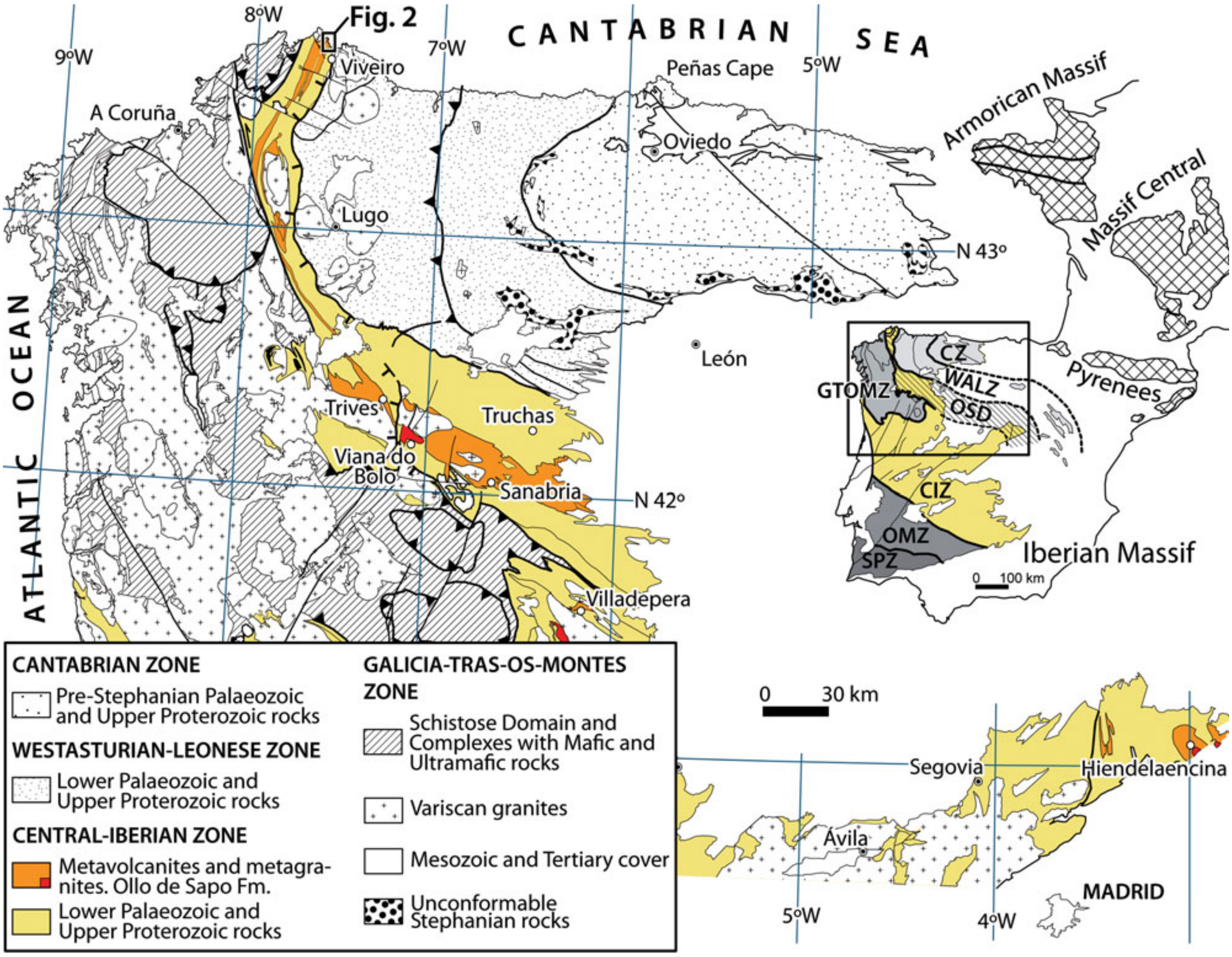


Figure 1. (Colour online) Simplified geological map of the NW Iberian Peninsula showing the outcrops of the Ollo de Sapo Formation and the location of the study area (partially based on Parga-Pondal *et al*. 1982). The inset in the top right shows the zones in which the Iberian Variscan belt is divided: CZ – Cantabrian Zone; WALZ – West Asturian-leonese Zone; GTOMZ – Galicia Tras-Os-Montes Zone; CZI – Central Iberian Zone (OSD, Ollo de Sapo Domain); OMZ – Ossa Morena Zone; and SPZ – South Portuguese Zone (based on Lotze, 1945; Julivert *et al*. 1972*a*; Farias *et al*. 1987).

Wildberg, Bischoff & Baumann, 1989; Fernández-Suárez *et al*. 1999, 2000) and it was not until the beginning of the last decade that Valverde-Vaquero & Dunning (2000) published the first reliable Early Ordovician U–Pb crystallization ages. The first comprehensive studies of crystallization age along and across the Ollo de Sapo Formation using a U–Pb high-resolution technique were performed by Bea *et al*. (2006) and Montero *et al*. (2007, 2009), who applied U–Pb spot analysis (ion microprobe and laser ablation inductively coupled plasma mass spectrometry; LA-ICP-MS) and Pb–Pb single-grain zircon analysis. Montero *et al*. (2009) constrained the age of the Ollo de Sapo Formation in the Iberian Peninsula as late Cambrian – Early Ordovician and revealed that the magmatism of the Ollo de Sapo Formation was synchronous and generally short-lived. Leaving aside the spatially associated San Sebastian metagranite that yielded an age of $470 \pm 3$ Ma, the ages obtained so far showed that the Ollo de Sapo Formation in the NW area of the Iberian Peninsula (Sanabria, Trives and Viveiro regions) spanned $492 \pm 4$ to $486 \pm 3$ Ma (less than 13 Ma of volcanic activity). Particularly remarkable is the case of the northernmost area, the Viveiro region, in which Montero *et al*. (2009) found the same age from top to bottom of the Ollo de Sapo Formation ($486 \pm 3$ Ma; 95 % confidence level). In contrast to those data, the SE area (Hiendelaencina region) showed that the metavolcanic rocks of the Ollo de Sapo Formation spanned $495 \pm 5$ to $483 \pm 3$ Ma ($12 \pm 8$ Ma of volcanic activity), with a punctual plutonic episode at $474 \pm 4$ Ma (Montero *et al*. 2007, 2009). Close to the Hiendelaencina region, Valverde-Vaquero & Dunning (2000) previously reported a zircon U–Pb (lower intercept) age of $480 \pm 2$ Ma in the metavolcanic facies of the Ollo de Sapo (Cardoso gneiss). This age would increase the time span of the Ollo de Sapo volcanic event in the SE area to $15 \pm 7$ Ma.

In the Viveiro region, published geological maps (see Arce-Duarte, Fernández-Tomás & Monteserín-López, 1977; Bastida *et al*. 1984; M.A. Lopez-Sanchez, unpub. Ph.D. thesis, 2013) show that the top of the Ollo de Sapo Formation and the bottom of the Capas de los Montes Formation are interdigitated; some Ollo-de-Sapo-type

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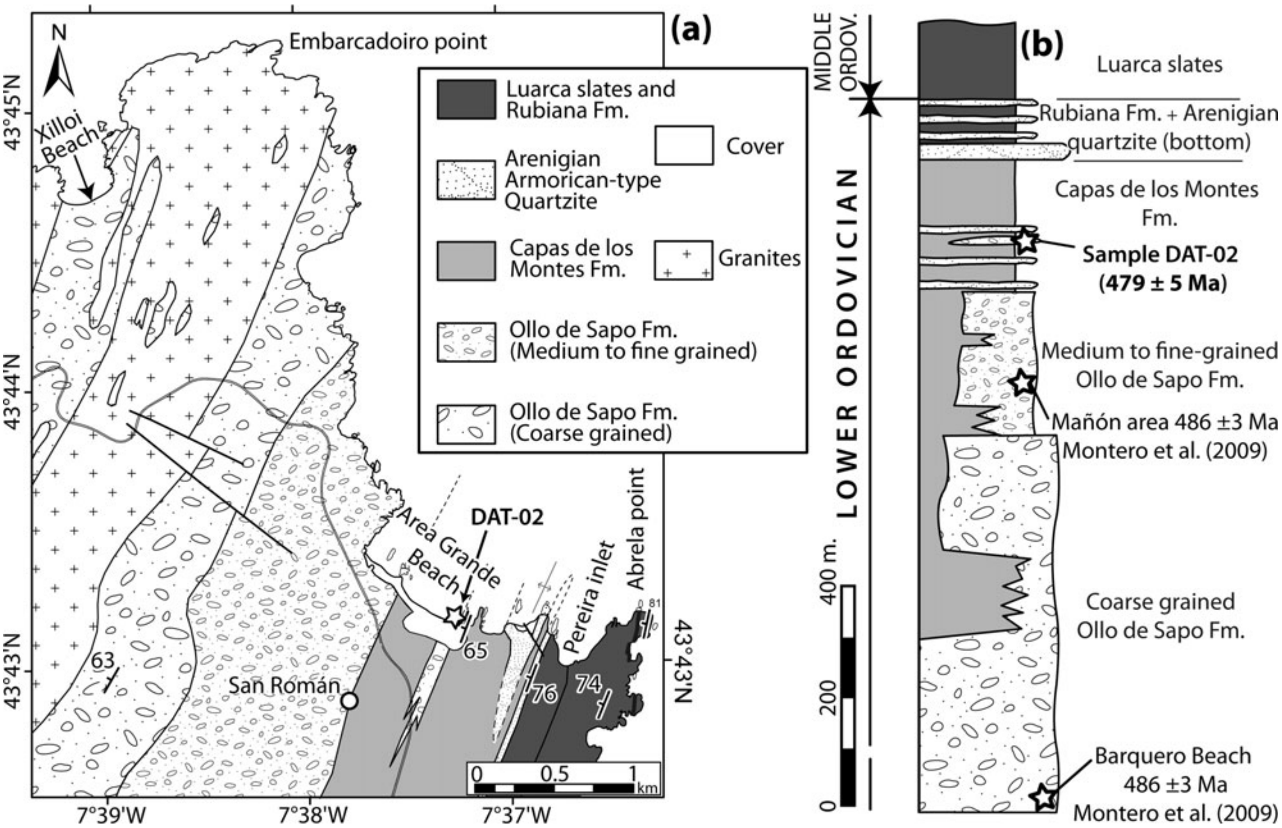


Figure 2. (a) Geological sketch map in the vicinity of Area Grande beach, NW of Viveiro (partially based on Arce-Duarte, Fernández-Tomás & Monteserín-López, 1977). (b) Synthetic stratigraphic column of the area.

lithosomes are found in the Capas de los Montes Formation and vice versa. It is also clear that in this area the top of the Ollo de Sapo Formation and the bottom of the Arenigian Armorican-type Quartzite are stratigraphically closer towards the north (Lopez-Sanchez, unpub. Ph.D. thesis, 2013). These stratigraphic features clearly indicate some degree of local diachronism during the deposition of the Ollo de Sapo Formation. At Area Grande beach, there is an isolated decametre-thick (≥ 35 m) level of fine-grained gneiss less than 200 m below the bottom of the Arenigian-Armorican-type quartzite that we consider as the top of the Ollo de Sapo Formation in Viveiro region.

Precise knowledge of crystallization ages along and across the Ollo de Sapo Formation is a key requirement to understand the duration and tectonic significance of the Cambro-Ordovician magmatism in the Iberian Peninsula. To test the inferences stated by Montero *et al.* (2009) – that is, the age span of Ollo de Sapo volcanic event in the NW area of the Iberian Massif is shorter (*c.* 492–486 Ma) and ceased before than that in the southern Hiendelaencia region (*c.* 495–483 Ma) – the aim of this study is to date the uppermost layer of the Ollo de Sapo Formation in its northernmost outcropping locality, the Viveiro region (Figs 1, 2). Since spatial resolution is in most cases a key issue to date this rock successfully, the U–Pb zircon analysis was performed using a sensitive high-resolution ion microprobe reverse geometry (SHRIMP-RG).

#### 1.a. Geological setting and previous ages in Viveiro region

The Viveiro region is the northernmost section across the Ollo de Sapo anticlinorium. The Ollo de Sapo Formation is exposed along the coast between the Xilloi and Area Grande beaches (Fig. 2). These rocks form part of the reverse limb of a great east-verging antiform, and show a pervasive and strongly developed tectonic foliation (Arce-Duarte, Fernández-Tomás & Monteserín-López, 1977; Bastida *et al.* 1984; M.A. Lopez-Sanchez, unpub. Ph.D. thesis, 2013). The metamorphic grade is greenschist facies conditions (chlorite to garnet zones; Bastida *et al.* 1984). The Ollo de Sapo in the coast section consists of coarse-grained and medium–fine-grained gneisses (Ortega *et al.* 1996). The coarse-grained gneiss (≥675 m thick) is the oldest rock cropping out in the region (Fig. 2a) and it is overlaid by *c.* 360 m of medium–fine-grained gneiss. Interbedded quartzites and mica-schist occur in some places (Fig. 2b).

Zircons from the lower and upper Ollo de Sapo metavolcanics on the Xilloi and Area Grande beaches (see Fig. 2 for location) were dated for the first time by Fernández-Suárez *et al.* (1999) using U–Pb LA-ICP-MS. They obtained ages ranging from 443 to 460 Ma (Late Ordovician), with late Neoproterozoic (*c.* 570–620 Ma), Mesoproterozoic (*c.* 1.0–1.2 Ga) and Palaeoproterozoic (*c.* 1.9–2.0 Ga) inheritances. Subsequently, Montero *et al.* (2009) dated three metavolcanic samples

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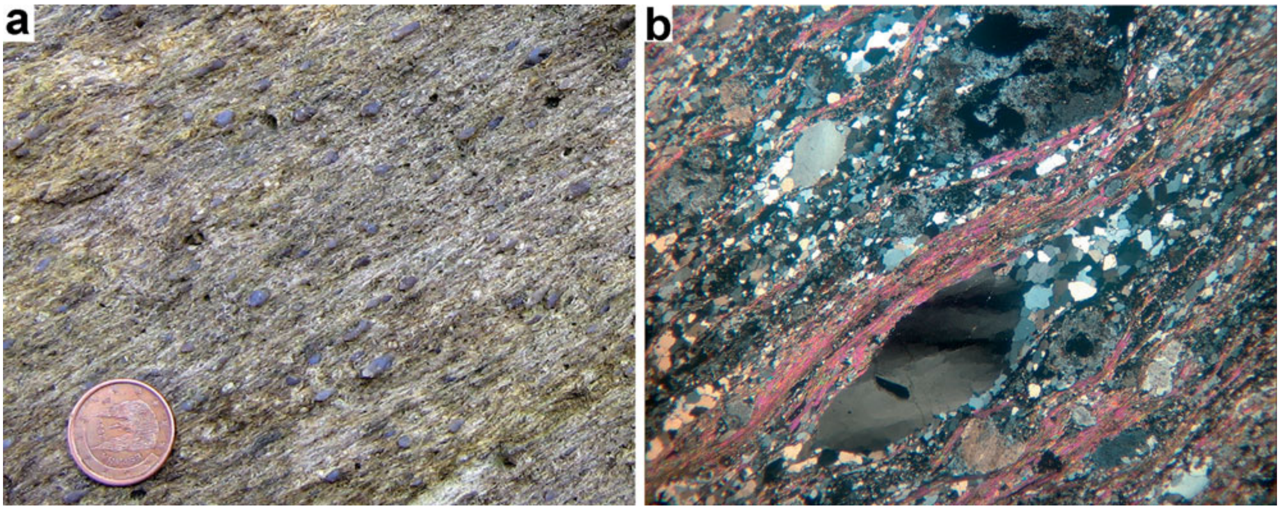


Figure 3. (Colour online) (a) Outcrop aspect of Ollo de Sapo Formation in the east of Area Grande Beach showing blue quartz phenocrysts. Coin is 21 mm in diameter. (b) Photomicrograph showing the mineralogy and texture of sample DAT-02. Field of view 8 mm.

in the same area, two fine-grained and one coarse-grained. All of them yielded a Pb–Pb plateau age at 486 ± 3 Ma (95 % confidence level), identical to the U–Pb age of the youngest concordant zircon population. These ages were considered as the crystallization age of the Ollo de Sapo Formation in Viveiro region, discarding the previous Late Ordovician ages obtained by Fernández-Suárez *et al*. (1999). Furthermore, the samples yielded inherited abundant concordant populations at 608 Ma, two concordant points at *c*. 1000 Ma, several concordant points at *c*. 2000 Ma and a discordia line with an upper intercept at *c*. 2600 Ma (Montero *et al*. 2009).

## 2. Materials and methods

### 2.a. Petrological description of the dated samples

About 10 kg of fresh meta-volcanic sample (DAT-02) was collected from the uppermost part of the Ollo de Sapo Formation at the Area Grande beach (NW of Viveiro, location 43° 43′ 04.96″ N; 7° 37′ 21.16″ W; Fig. 2).

Sample DAT-02 is a strongly foliated, quartz-feldspatic, acidic meta-igneous rock that can be described as a medium–fine-grained gneiss. Its porphyroclasts are mainly albitic plagioclase and quartz, with grain size ranging from hundreds of microns to 1.5 mm (Fig. 3a). Remnants of former K-feldspar (microcline) are still preserved in the cores of the albite porphyroclasts showing that the original microcline was replaced, most probably during deformation (Reche, Martínez & Arboleya, 1998). Some idiomorphic igneous zircons occur within the porphyroclasts. Quartz porphyroclasts (former igneous phenocrystals) are polycrystalline or show strong undulose extinction due to deformation (Fig. 3b). The matrix is strongly foliated; the foliation being mainly defined by muscovite and polycrystalline quartz ribbons. Most of the quartz in the matrix and some minor feldspar are recrystallized into fine-grained equant grains sandwiched between parallel-oriented muscovite grains. The matrix foliation wraps around the porphyroclasts. The absence of relic biotite and the relative abundance of secondary muscovite suggest a peraluminous protolith. The preceding petrographic features and the field structural relations enable the protolith to be considered as a fine-grained, porphyritic, leucocratic, quartz-feldspathic metavolcanic rock.

### 2.b. U(Th)–Pb SHRIMP-RG dating

Zircon crystals were separated after rock crushing using conventional heavy liquid and magnetic properties at Centro de Geociencias (UNAM). Zircon standard (R33) plus unknowns were mounted in epoxy resin and polished down to expose the near-equatorial section. The mounts were cleaned in distilled water and in 1 N HCl and gold-coated for maximum surface conductivity. Cathodoluminiscence (CL) imagery was performed with a JEOL 5600 instrument (SEM) at Stanford University.

The SHRIMP-RG U(Th)–Pb analyses were performed on individual zircon grains using the ion microprobe housed at Stanford University, California. The detailed procedures used in this study are similar to those reported in Williams (1998) and Nourse *et al*. (2005). Briefly, the primary oxygen ion beam ($O^{2-}$), operated at *c*. 2–4 nA, excavated an area of *c*. 20–40 μm in diameter (adjustable depending on grain size) to a depth of *c*. 1–2 μm; sensitivity was within the range 5–30 cps per ppm Pb. Data for each spot were collected in sets of five scans through the mass range. Nine peaks were measured sequentially for zircons with a single collector: $^{196}Zr_2O$, $^{204}Pb$, background (0.050 mass units above $^{204}Pb$), $^{206}Pb$, $^{207}Pb$, $^{208}Pb$, $^{238}U$, $^{248}ThO$ and $^{254}UO$. The reduced ratios were normalized to the zircon standard R33 (Black *et al*.

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2004). For the closest possible control of Pb/U ratios, one standard was analysed after every 4–5 unknown samples. Uranium concentrations were monitored by analysing a standard (MAD) with *c.* 4200 ppm U composition. The U and Th concentrations are accurate to *c.* 10–20 %.

Ion microprobe isotopic raw data were reduced using the software Squid (Ludwig, 2001). Data were projected to the Tera–Wasserburg concordia diagram along a model common Pb line based on Stacey & Kramers (1975). All the U(Th)–Pb age data presented in Table 1 were plotted using the software Isoplot/Ex 3.75 (Ludwig, 2012).

### 2.c. Zircon selection criteria

Previous attempts at dating the Ollo de Sapo Formation show that in general euhedral stubby zircon prisms yield inherited ages while euhedral needle-like zircon prisms yield crystallization ages (see Valverde-Vaquero & Dunning, 2000). Taking this into account, we discarded the stubby zircon prisms in order to avoid mixtures of inherited cores and magmatic overgrowths. Furthermore, the application of cathodoluminescence imaging combined with the SHRIMP-RG technique allowed us to analyse the zircons rims when the selected needle-like zircon grains show different features between the inner cores and the rims.

## 3. Results

### 3.a. Sample DAT-02

The selected zircon grains from the sample DAT-02 range between 130 and 250 μm (long axes) in size. They are mainly euhedral, with aspect ratios between 1:1.8 and 1:5.2, and display double-terminated prismatic shapes. With the exception of zircon number 10, all of them show brighter inner cores than the rims under CL (Fig. 4d). Some of the inner cores display a clear growth zoning. In the case of zircons 1 and 5 the inner core zoning is cut by the CL-dark rims, although this is not the general case. Most of the rims are unzoned or show a faint and broad zoning. In the case of zircons 5 and 8, rims display a weak sector zoning.

Results obtained from the zircon grains are listed in Table 1. Individual zircon data plotted in a Tera–Wasserburg concordia diagram (Fig. 4a) show that there is a group of concordant data points ($n = 10$) that form a coherent array yielding a weighted average $^{206}Pb/^{238}U$ age of 479.0 ± 4.7 Ma (95 % conf.; MSWD = 5.8; probability of fit = 0; Fig. 4c). Analysis number 8, which provides an older concordant age at 567 ± 5 Ma but presents no noticeable differences in SEM-CL compared to the younger grains, is a xenocryst (Fig. 4d).

The weighted average age yields a high and unsatisfactory MSWD value (> 2) (Wendt and Carl, 1991). This indicates that the scatter of the data exceeds the expected scatter from the analytical errors estimated.

Table 1. SHRIMP-RG U(Th)–Pb data for zircon from the uppermost levels of the Ollo de Sapo Formation in Viveiro region.

| | | | | | Corrected ratios* | | | | | | | | | | Corrected ages (Ma)‖ | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Zircon sample | Common $^{206}Pb$ (%) | U (ppm) | Th (ppm) | Th/U | $^{207}Pb/^{206}Pb^*$ | % error† | $^{207}Pb/^{235}U^*$ | % error† | $^{206}Pb/^{238}U^*$ | % error† | $^{208}Pb/^{232}Th^*$ | % error† | Rho‡ | % disc§ | $^{206}Pb/^{238}U$ | ±1σ | $^{207}Pb/^{235}U$ | ±1σ | $^{207}Pb/^{206}Pb$ | ±1σ | $^{208}Pb/^{232}Th$ | ±1σ | Best age (Ma) | ±1σ |
| DAT2-10 | 0.09 | 263 | 24 | 0.09 | 0.0565 | 2.1 | 0.5907 | 2.2 | 0.0758 | 0.7 | 0.0234 | 5.5 | 0.332 | 0 | 471.1 | 3.4 | 471.3 | 8.4 | 472.7 | 46.4 | 468.1 | 25.8 | 471.1 | 3.4 |
| DAT2-5 | 0.18 | 264 | 27 | 0.10 | 0.0574 | 2.0 | 0.6021 | 2.2 | 0.0761 | 0.8 | 0.0247 | 4.7 | 0.379 | 1 | 472.9 | 3.8 | 478.6 | 8.4 | 505.8 | 44.9 | 492.8 | 23.0 | 472.9 | 3.8 |
| DAT2-7 | 0.01 | 160 | 159 | 1.03 | 0.0567 | 2.4 | 0.5951 | 2.6 | 0.0762 | 0.9 | 0.0249 | 1.8 | 0.354 | 0 | 473.3 | 4.2 | 474.1 | 9.8 | 477.9 | 53.7 | 497.2 | 8.8 | 473.3 | 4.2 |
| DAT2-1 | 0.33 | 975 | 62 | 0.07 | 0.0555 | 1.8 | 0.5832 | 1.8 | 0.0761 | 0.4 | 0.0202 | 11.8 | 0.217 | −1 | 473.1 | 1.8 | 466.5 | 6.7 | 434.4 | 39.1 | 403.7 | 47.8 | 473.1 | 1.8 |
| DAT2-11 | 0.24 | 352 | 32 | 0.09 | 0.0586 | 1.6 | 0.6206 | 1.7 | 0.0768 | 0.6 | 0.0260 | 3.1 | 0.359 | 3 | 477.3 | 2.8 | 490.2 | 6.7 | 551.2 | 34.9 | 517.9 | 15.9 | 477.3 | 2.8 |
| DAT2-6 | −0.13 | 319 | 75 | 0.24 | 0.0556 | 1.7 | 0.5912 | 1.9 | 0.0771 | 0.7 | 0.0233 | 2.2 | 0.355 | −2 | 478.7 | 3.1 | 471.6 | 7.0 | 437.3 | 38.8 | 464.6 | 10.3 | 478.7 | 3.1 |
| DAT2-3 | 0.03 | 431 | 30 | 0.07 | 0.0564 | 1.6 | 0.6017 | 1.7 | 0.0774 | 0.6 | 0.0241 | 4.9 | 0.334 | −1 | 480.7 | 2.6 | 478.3 | 6.5 | 466.6 | 35.8 | 481.9 | 23.7 | 480.7 | 2.6 |
| DAT2-2 | −0.04 | 419 | 30 | 0.07 | 0.0565 | 1.5 | 0.6070 | 1.6 | 0.0779 | 0.6 | 0.0268 | 3.2 | 0.355 | 0 | 483.7 | 2.7 | 481.6 | 6.3 | 472.0 | 34.1 | 534.3 | 17.1 | 483.7 | 2.7 |
| DAT2-4 | 0.11 | 584 | 57 | 0.10 | 0.0577 | 1.3 | 0.6248 | 1.4 | 0.0785 | 0.5 | 0.0247 | 2.4 | 0.358 | 1 | 487.1 | 2.3 | 492.8 | 5.3 | 519.4 | 28.0 | 493.9 | 11.9 | 487.1 | 2.3 |
| DAT2-9 | −0.01 | 433 | 65 | 0.16 | 0.0560 | 1.7 | 0.6087 | 1.8 | 0.0789 | 0.6 | 0.0262 | 4.5 | 0.319 | −1 | 489.4 | 2.7 | 482.8 | 6.9 | 451.1 | 37.7 | 523.0 | 23.5 | 489.4 | 2.7 |
| DAT2-8 | 0.00 | 197 | 119 | 0.63 | 0.0590 | 2.1 | 0.7487 | 2.2 | 0.0920 | 0.8 | 0.0325 | 1.9 | 0.371 | 0 | 567.2 | 4.5 | 567.5 | 9.8 | 568.7 | 45.4 | 646.0 | 12.4 | 567.2 | 4.5 |

*Isotopic ratios are corrected relative to R33 standard zircon (418.9 ± 0.4 Ma; Black *et al.* 2004).
† All errors in isotopic ratios and ages are given at the 1σ level
‡Rho is the correlation error between errors in the $^{207}Pb/^{235}U$ and $^{206}Pb/^{238}U$ ratios
§Percentage of discordance obtained using the equation (100*[(age $^{207}Pb/^{235}U$)–(age $^{206}Pb/^{238}U$)]/age $^{207}Pb/^{235}U$). Positive and negative values indicate normal and inverse discordance, respectively.
‖Atomic ratios and ages corrected for initial Pb using the amount of $^{204}Pb$.

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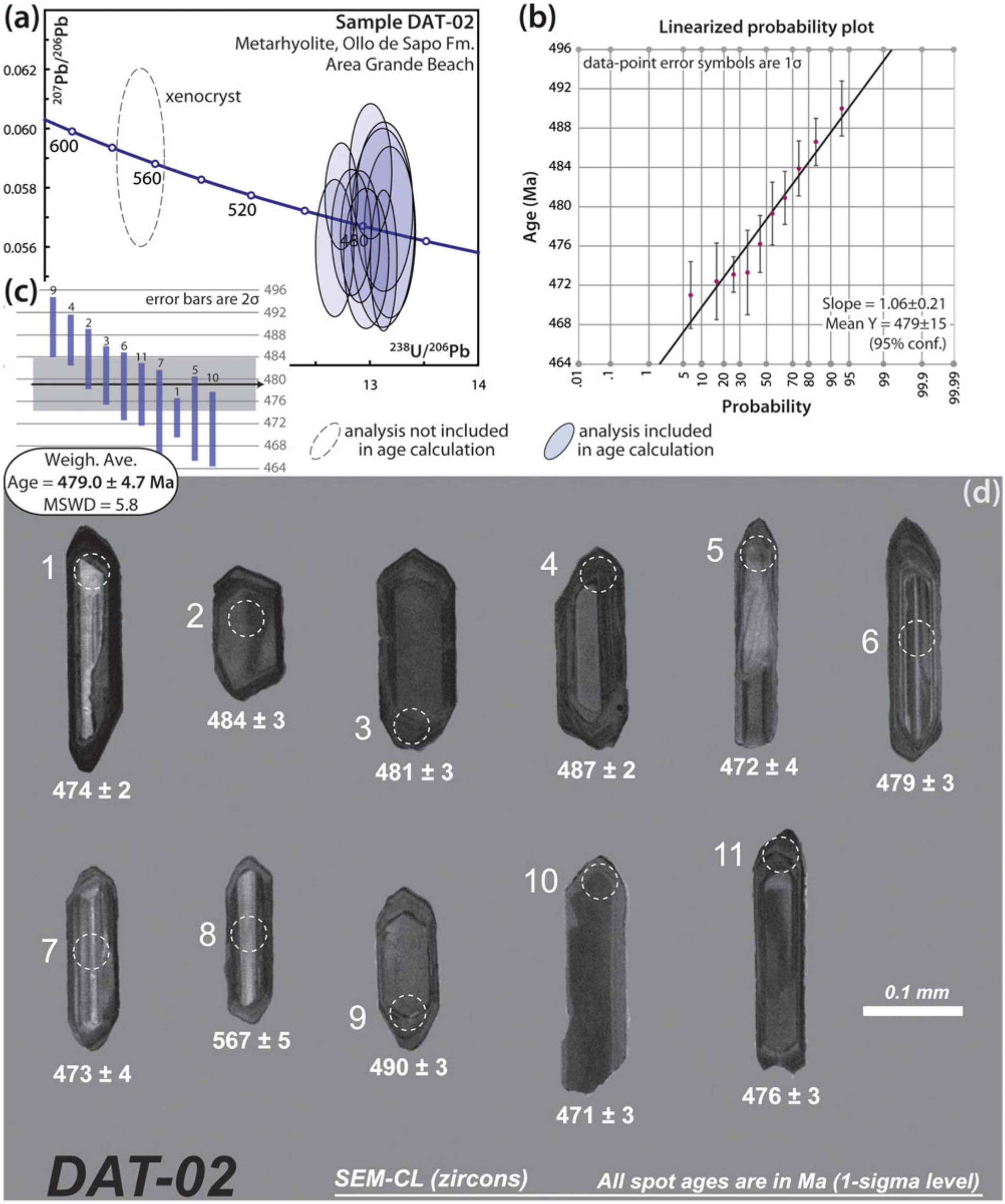


Figure 4. (Colour online) U–Pb SHRIMP zircon geochronology data for the volcanic sample DAT-02. (a) Tera–Wasserburg concordia plot. Solid-line filled ellipses represent data used to estimate the age. Dotted-line error ellipses represent data points excluded from the age calculation. Data-point error ellipses are 2σ. (b) Linearized probability plot of the data used to estimate (c) the age $^{206}Pb/^{238}U$ weighted mean plot. (d) SEM-CL image of dated zircons for sample DAT-02. Circles and the adjacent numbers represent the spot size (*c.* 30 μm) and the spot number, respectively. Zircons are sorted by spot number. These are $^{206}Pb/^{238}U$ ages reported in Ma at the 1σ level of precision.

If we assumed that the ‘internal’ assigned errors of the data points are well estimated, we think there are two possible causes that can lead to this high value of MSWD. The former may be due to the presence of an external source of error in addition to the assigned data point errors, which would produce an error underestimation. The alternative would arise from the possible presence of slightly older zircons within the population forming at different stages of the Ollo de Sapo Formation (i.e. antecryst in the sense of Charlier *et al.*

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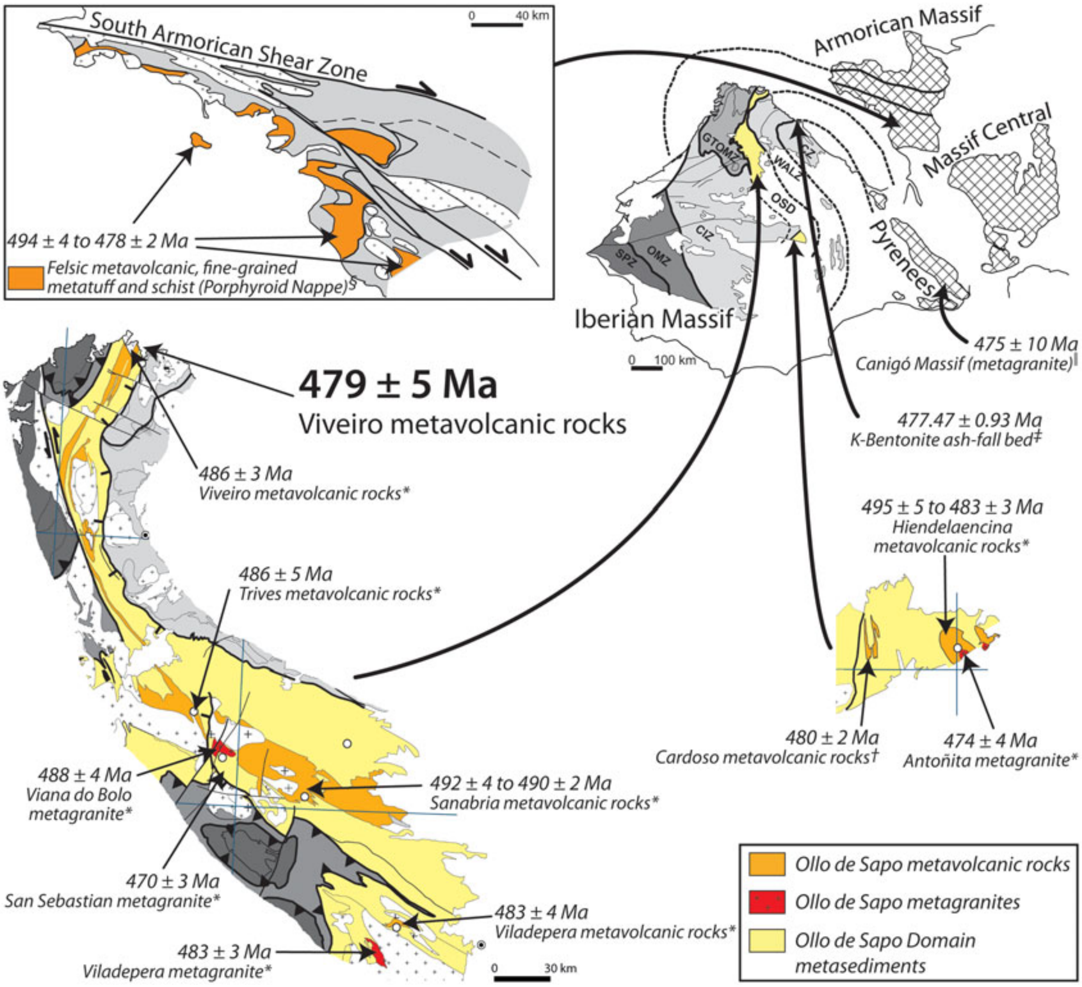


Figure 5. (Colour online) Zircon U-Pb crystallization ages of the Cambro-Ordovician magmas in the Central Iberian Zone and the Southern Armorican Massif. All ages in the Iberian Peninsula are in the Ollo de Sapo Formation, with the exception of the K-bentonite ash-fall bed in the Cantabrian Zone and the Canigó Massif in the Pyrenees. *Montero *et al*. 2009; †Valverde-Vaquero & Dunning, 2000; ‡Gutiérrez-Alonso *et al*. 2007; §Ballèvre *et al*. 2012; ‖Deloule *et al*. 2002.

2005). However, there is no evidence of the presence of antecryst based on zircon morphologies (Fig. 4d). We also test whether the age population obtained conform to a normal distribution. For this, we plotted the data in a normal probability plot (Ludwig, 2012) and performed a Shapiro–Wilk test (Shapiro and Wilk, 1965). As can be seen in the plot shown in Figure 4b, the data almost follow the same linear trend which means that the data approximately follow a normal distribution. Furthermore, the results of the Shapiro–Wilk test yield a $p$-value of 0.40 ($\gg$0.05), indicating that we cannot reject the possibility that the suite of concordant ages obtained came from a normally distributed population. Since we cannot derive a bimodal or multimodal population of ages from the dataset considered, this means that there is no convincing reason to establish with certainty that there are antecryst within the suite of zircons considered. Consequently, the high MSWD value has to be caused by the presence of an unknown external error not considered in the individual assigned errors of the data points or, less probably, by the presence of zircon antecrysts within the zircon population which could not be proved due to the precision of the technique used. Hence, the 479 ± 5 Ma age is the best estimation for the crystallization age.

## 4. Discussion and conclusions

The 479.0 ± 4.7 Ma age (Tremadocian, Early Ordovician) obtained is the youngest reliable crystallization age found so far for the Ollo de Sapo volcanic rocks. If we compare the zircon population ages from our sample DAT-02 to those obtained by Montero *et al*. (2007, 2009) in Viveiro (486 ± 3 Ma) and Hiendelaencina (483 ± 3 Ma, upper metavolcanics) regions performing a Welch's $t$-test (Welch, 1947), we determine that the probabilities of sample DAT-02 being younger are 99.47 % and 91.96 % respectively (Table 2). This means that the Cambro-Ordovician volcanism in the NW of the Iberian Peninsula is not as short-lived as previously thought and that the cessation of this volcanic event in Viveiro was younger than in the SE Hiendelaencia region, although similar to the age reported by Valverde-Vaquero & Dunning (2000) at 480 ± 2 Ma (zircon U–Pb, lower intercept) in the Cardoso gneiss (Fig. 5).

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Table 2. Welch $t$-test results for different samples compared to the sample DAT-02.

| Reference | Sample reference | Sample size[‡] | Age (weighted average, Ma) | $p$-value | Probability (%) of being older than sample DAT-02 |
|---|---|---|---|---|---|
| Montero et al. (2009)* | Viveiro metavolcanics | 15 | $486 \pm 2$ | 0.00535 | 99.47 |
| Montero et al. (2007)[†] | Hiendelaencina upper metavolcanics | 19 | $483 \pm 3$ | 0.08743 | 91.96 |
| Ballèvre *et al.* (2012) | Sample CG5 | 14 | $478 \pm 2$ | 0.74226 | 25.77[§] |
| | Sample FL31 | 16 | $472 \pm 4$ | 0.0278 | 97.22[§] |

∗Data from table 9: single-grain stepwise evaporation $^{207}Pb/^{206}Pb$ ages
[†]Data from table 3ESM (http://earthref.org/ERDA/724/)
[‡]Number of single age data considered to perform the Welch $t$-test
[§]Probability (%) of being *younger* than sample DAT-02

Recently, Ballevrè *et al.* (2012) found that the felsic volcanic layers that outcrop in the South Armorica domain (Variscan belt, France) yielded ages spanning from $494 \pm 4$ to $472 \pm 4$ Ma. If we compare the zircon population ages from sample DAT-02 to the youngest ages obtained by Ballèvre *et al.* (2012) in the Porphyroid Nappe ($478 \pm 2$ Ma) and the para-autochthon ($472 \pm 4$ Ma), the probabilities of sample DAT-02 being older are 25.77 % and 97.22 %, respectively (Table 2). Hence, the age obtained in the sample DAT-02 has a *c.* 75 % chance of being similar to the youngest age obtained in the Porphyroid Nappe. These results reinforce the interpretation made by Ballèvre *et al.* (2012), who correlated and compared the age, geochemistry and structural position of the metavolcanics of the Porphyroid Nappe in South Armorica domain to the Ollo de Sapo Formation in the Central Iberian zone.

The age obtained represents the cessation of Ollo de Sapo Cambro-Ordovician volcanism during the breakup of the northern Gondwanan margin in this part of the Ibero-Armorican Arc (i.e. at the northernmost tip of the Iberian Peninsula), but not the end of the rifting process itself; this continued, at least in this part of the Iberian Massif, until Late Ordovician time according to the important changes in thickness recorded in the Upper Ordovician stratigraphic sequence (Julivert, Marcos & Truyols, 1972*b*; Marcos, 1973; Pérez-Estaún *et al.* 1990; Martínez-Catalán *et al.* 1992, 2004; Marcos *et al.* 2004). Considering all the volcanic ages discussed here, they seem to suggest that the cessation of the Cambro-Ordovician volcanism along the Ibero-Armorican Arc was synchronic or perhaps slightly diachronic with younger ages towards the north (in present-day geographical coordinates; Fig. 5).

Additionally, the $479 \pm 5$ Ma age obtained is consistent with the age of the volcanic levels that crop out within the Arenigian Armorican-type quartzite in the Cantabrian Zone at $477 \pm 1$ Ma (Gutiérrez-Alonso *et al.* 2007) and with the Ordovician magmatism that built up the protoliths of the Variscan Pyrenean axial zone gneiss domes (Deloule *et al.* 2002; Castiñeiras *et al.* 2008; Denele *et al.* 2009; Martínez *et al.* 2011; Fig. 5).

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## 5. Declaration of interest

None

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