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Reply to comment by C. Pin and J. Rodríguez on “Rheic Ocean ophiolitic remnants in southern Iberia questioned by SHRIMP U-Pb zircon ages on the Beja-Acebuches amphibolites”

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1. Introduction

[1] We acknowledge the invaluable opportunity that the comment by *Pin and Rodríguez* [2009] offers us in order to corroborate the conclusions of our paper and clarify our analytical methods. Our response will be focused on aspects concerning (1) the accuracy of our SHRIMP analyses, (2) sample distribution, (3) sample geochemistry, and (4) geological implications of the ages.

2. Accuracy of the SHRIMP Ages

[2] Pin and Rodríguez lament the lack of analytical details in the work by *Azor et al.* [2008]. We welcome the opportunity to include in this reply the details on sample preparation and data treatment that were omitted in the final version of our paper.

2.1. Analytical Procedure

[3] Zircons for U-Pb analysis were prepared as mineral separates, mounted in epoxy and polished down to expose the grain centers. Cathodoluminescence (CL) images were obtained at the Electron Microscope Unit of the Australian National University with a HITACHI S2250-N scanning electron microscope, operating at 15 kV, $\sim 60 \mu\text{A}$ and ~ 20 mm working distance.

[4] U-Pb analyses were performed using sensitive, high-resolution ion microprobes (SHRIMP II and SHRIMP RG) at the Research School of Earth Sciences in Australia. Instrumental conditions and data acquisition were generally

those described by *Williams* [1998]. The data were collected in sets of six scans throughout the masses. The measured $^{206}\text{Pb}/^{238}\text{U}$ ratio was corrected using reference zircon TEM (417 Ma [*Black et al.*, 2003]), while the U content in the target was referred to SL13 zircon of known composition. Isotopic ratios were corrected for common Pb on the basis of the measured ^{204}Pb as in *Williams* [1998] and adopting a Pb composition according to the model of *Stacey and Kramers* [1975]. Common Pb correction based on $^{207}\text{Pb}/^{206}\text{Pb}$ and $^{208}\text{Pb}/^{206}\text{Pb}$ ratios [*Williams*, 1998] was also performed, yielding ages, within error, identical to those of ^{204}Pb -based correction. The U-Pb data were collected over a number of analytical sessions using the same standard, with different sessions yielding comparable calibration errors between 2.1 and 2.7% (2σ). Age calculation was performed with the software *Isoplot/Ex* [*Ludwig*, 2003]. Average ages are quoted at 95% confidence level, while single ages and ratios are quoted at 1σ .

2.2. Pb Loss and Age Concordance

[5] The theoretical background given by Pin and Rodríguez on U-Pb dating is correct and helpful to the reader. We certainly agree that radiogenic Pb loss from zircon is possible, as documented in a number of cases. However, we would like to add that microsampling can help in avoiding Pb loss during analysis. Pb loss may occur by diffusion, generally at higher T than those considered here, or by a number of processes affecting the crystal structure, such as deformation, leaching by fluids, metamictization and recrystallization. These processes can be identified by CL investigation because they produce dislocations, fractures, porosity and/or changes in the zoning (ghost or patchy zoning, crosscutting CL domains [see *Pidgeon et al.*, 1998; *Rubatto and Gebauer*, 2000; *Reddy et al.*, 2006; *Rubatto et al.*, 2008]). Thus, microsampling guided by CL imaging provides some assistance in avoiding zones with signs of Pb loss.

[6] According to Pin and Rodríguez, the 332 ± 3 to 340 ± 4 Ma U-Pb zircon ages presented by *Azor et al.* [2008] for the Beja-Acebuches amphibolite (BAA) protoliths are biased toward young ages due to Pb loss, and should thus be reinterpreted as “minimum ages” only. Pin and Rodríguez argue that the precision of the SHRIMP analyses presented by *Azor et al.* [2008] would not allow detecting a small amount of Pb loss, which would have slightly rejuvenated the age from 352 Ma, their preferred ages for the samples in question. Nevertheless, a statistically

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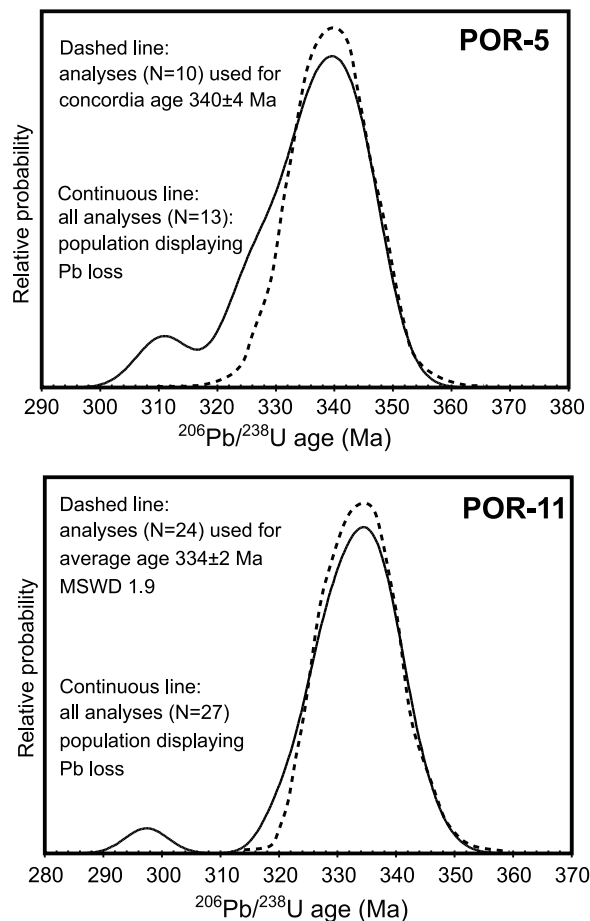


Figure 1. Relative probability diagram of $^{206}\text{Pb}/^{238}\text{U}$ ages for two of the samples dated by Azor *et al.* [2008]. Note the Gaussian distribution after removal of analyses with Pb loss. The removed analyses are ~ 13 and 11 Ma younger than the main population and demonstrate that such degree of Pb loss can be identified at this level of precision.

homogeneous age cluster must show a Gaussian distribution, while Pb loss would skew such distribution producing a tail of younger ages. Each of the age clusters, on which Azor *et al.* [2008] based their mean ages, have indeed a Gaussian distribution, which cannot be reconciled with Pb loss (Figure 1).

[7] The data in Azor *et al.* [2008] can be used to demonstrate that at this level of precision, the Pb loss necessary to shift the ages of 10–20 Ma, as suggested by Pin and Rodríguez, would have been detected. In three of the samples dated, a few analyses were excluded from the average age calculation because of suspected Pb loss. Their exclusions were based on statistical grounds using the F test to identify outliers of a statistically consistent population of data. The age of the oldest outlier excluded from each sample is between 11 and 17 Ma younger than the average of the main population for each sample (analysis POR5–5.1 at 326.8 ± 4.3 Ma, excluded from a population averaging at 340 ± 4 Ma, difference 13.2 ± 4.7 Ma; analysis POR10–11 at 315.4 ± 6.4 Ma, excluded from a population at $332 \pm$

3 Ma, difference 16.6 ± 6.6 Ma; analysis POR11A–2.2 at 322.8 ± 3.3 Ma excluded, from a population at 334 ± 2 Ma, difference 11.2 ± 1.9 Ma). All the ages quoted are $^{206}\text{Pb}/^{238}\text{U}$ ages, with uncertainties of 1σ for single analyses and 95% confidence level for average ages. Removing such analyses returned a homogeneous Gaussian population from which the average age was obtained. This is illustrated in Figure 1 for samples POR-5 and POR-11, demonstrating that, at the level of uncertainty of the SHRIMP data, our statistical treatment would have detected Pb loss in the order of 10–20 Ma.

[8] Pin and Rodríguez are correct in presenting the problem of apparent concordance for analyses of relatively young (<500 Ma) zircons due to the lack of appreciable curvature of the Concordia. We do agree that the relative poor precision of single U-Pb SHRIMP analyses makes this problem particularly relevant. This is why age calculation by ion microprobe protocols relies on cluster of analyses rather than on single data points. Pin and Rodríguez comment that “the reported ages are basically $^{206}\text{Pb}/^{238}\text{U}$.” However, three of the average ages (POR-5, POR-8, and POR-10) are “Concordia ages” as defined by Ludwig [2003]. This calculation makes use of all the three isotopic ratios obtained from the U-Pb decay system in order to calculate the most precise age given the data set. For sample POR-11, a Concordia age was not possible to calculate because the data are slightly overconcordant; that is, they plot above the Concordia curve. This is due to an analytical artifact in that ion microprobe session, which by no means compromises the $^{206}\text{Pb}/^{238}\text{U}$ ratios. The age quoted for this sample is an average $^{206}\text{Pb}/^{238}\text{U}$ age, which remains identical independently of the common Pb correction adopted (^{204}Pb -, ^{207}Pb -, or ^{208}Pb -based correction [see Williams, 1998]).

[9] To conclude, the Gaussian distribution of the data for each sample, as well as the fact that Pb loss is detectable at this level of precision, enable us to plausibly conclude that the ages reported by Azor *et al.* [2008] are robust and within error of the intrusion age of the BAA protoliths.

3. Geographical and Geological Location of the Dated Samples

[10] Pin and Rodríguez question the geological interpretation of our ages by assuming that we sampled the deformed southern part of the Beja Igneous Complex (BIC) instead of the BAA unit. However, the geographical and geological location of our samples [see Azor *et al.*, 2008, Figure 2b] is decidedly clear. The BAA unit constitutes a WNW-ESE oriented 200 km long and 500–2000 m thick strip, which extends from Spain to Portugal, while the BIC crops out in Portugal, north of the BAA unit. Samples POR-5 and POR-8 were taken in Portugal, close to the southern contact of the BIC, but clearly inside the BAA unit. Samples POR-10 and POR-11 were collected in Spain, close to Almonaster la Real and south of Aracena (road to Campofrío), respectively, in two classical transects of the BAA unit. Therefore, the geographical location of our samples is unambiguously inside the BAA unit.

Table 1. Whole-Rock Major and Trace Element Compositions of Studied Samples From the BAA Unit^a

	Lithology			
	Gabbro	Amphibolite	Amphibolite	Amphibolite
Sample	POR-5	POR-8	POR-10	POR-11
SiO ₂	50.93	49.17	50.28	48.06
TiO ₂	1.82	1.39	0.93	1.53
Al ₂ O ₃	19.21	19.24	17.34	15.00
Fe ₂ O ₃	7.89	9.41	7.88	11.70
MnO	0.13	0.11	0.12	0.18
MgO	6.10	4.91	7.25	6.60
CaO	10.08	9.59	12.51	11.88
Na ₂ O	2.71	3.54	2.48	2.08
K ₂ O	0.25	0.60	0.12	0.39
P ₂ O ₅	0.02	0.18	0.10	0.14
LOI	0.00	1.60	0.66	1.57
Total	99.15	99.74	99.67	99.13
Mg #	60	51	65	53
Zr	24	102	75	96
Sr	324	327	203	176
Nb	4.0	5.8	1.9	2.4
La	4.0	14	4.6	4.3
Ce	7.5	38	12	12
Pr	1.1	5.6	1.8	2.0
Nd	4.6	24	8.6	11
Sm	1.3	6.4	2.7	3.6
Eu	0.96	1.7	1.0	1.4
Gd	1.4	6.7	3.4	4.7
Tb	0.24	1.1	0.59	0.88
Dy	1.6	7.1	3.7	5.6
Ho	0.36	1.5	0.82	1.2
Er	0.99	4.0	2.3	3.3
Tm	0.15	0.60	0.35	0.50
Yb	0.97	3.7	2.2	3.0
Lu	0.15	0.53	0.32	0.40
Ta	0.41	0.50	0.33	0.28
Th	0.073	0.031	0.70	0.32

^aMajor elements are in units of wt % and trace elements are in units of ppm. LOI, loss on ignition; Mg #, 100 × molar MgO/(MgO + FeO).

[11] Pin and Rodríguez suppose a composite nature for the BAA unit, which would include dominant Lower Carboniferous metagabbros/metabasalts attributable to the deformed southern rim of the BIC, as well as very subordinate pre-Mid-Devonian truly oceanic metabasalts. Thus, Pin and Rodríguez base all their reasoning on the assumption that we have only sampled lithologies belonging to the deformed southern rim of the BIC, but not the MORB-featured ones. Nevertheless, the geochemical data presented below will unequivocally show that we have sampled both rock types. In this respect, sample POR-11 was taken at a locality where *Quesada et al.* [1994] describe volcanic textures preserved from the deformation, while *Pin et al.* [2008] recognize an NMORB signature according to the highly positive ϵ Nd values.

4. Whole-Rock Geochemistry of the Dated Samples

[12] The geochemical characterization of the BAA unit, amply reported in previous works [e.g., *Bard and Moine*, 1979; *Quesada et al.*, 1994, *Castro et al.*, 1996], was

beyond the scope of our paper and thus the geochemical data were not included by *Azor et al.* [2008]. However, these data are available and now included in this section.

4.1. Analytical Techniques

[13] Whole-rock powders were made by crushing and powdering large amounts of each sample (5–10 kg). Major elements and Zr were analyzed by XRF at the Centro de Instrumentación Científica (CIC) at the Universidad de Granada (Spain) in a PHILIPS PW-2440 instrument using standard sample preparation (i.e., fusion with lithium tetraborate) and analytical procedures. Typical precision is better than $\pm 1.5\%$ for an analyte concentration of 10 wt% and $\pm 2.5\%$ for 100 ppm Zr. Trace elements (REE, Th, Nb, Ta, and Sr) were analyzed by a Perkin Elmer Elan-5000 ICP-MS at the CIC. Trace element concentrations were determined by external calibration after HNO₃+HF digestion of 0.1 g of sample powder in a Teflon[®] lined vessel at $\sim 180^\circ\text{C}$ and ~ 200 psi, evaporation to dryness and subsequent dissolution in 100 ml of 4 vol.% HNO₃; only extra clean Teflon[®] beakers were used to minimize contamination during sample dissolution and lower chemical blank corrections. Precision, estimated by the analyses of 10 replicates of one sample, is better than $\pm 2\%$ and $\pm 5\%$ for analyte concentrations of 50 and 5 ppm, respectively. The compositions of the reference materials (PMS, WSE, and AGV), analyzed as unknowns during the same analytical runs of the BAA samples, show good agreement with working values for these international standards [*Govindaraju*, 1994].

4.2. Major and Trace Elements

[14] Table 1 presents the whole-rock major and trace element compositions of the samples dated. They have a subalkaline basaltic-gabbroic major element composition (SiO₂ = 48–51 wt%, TiO₂ = 0.93–1.82 wt%, Na₂O + K₂O = 2.47–4.14 wt%, Mg# = 51–65), which indicates a rather primitive signature of the parental magmas. Their chondrite-normalized REE concentrations span from 5.7 to 61.4 and show variable patterns (Figure 2): POR-5 and POR-8 have LREE-enriched (La/Yb_(N) = 2.6–2.9; La/Sm_(N) = 1.4–1.9) and HREE-depleted (Sm/Yb_(N) = 1.5–1.9) compositions and their patterns display evident positive and negative Eu anomaly, respectively; POR-10 and POR-11 have flatter REE patterns (La/Yb_(N) = 1.0–1.5) with unfractionated LREE (La/Sm_(N) = 0.8–1.1) and slightly depleted HREE (Sm/Yb_(N) = 1.3–1.4) segments. LREE enrichment in POR-5 and POR-8 can be ascribed to crustal contamination or reflect the composition of the mantle source, since common enriched mid-oceanic ridge basalts (EMORB) have similar patterns (Figure 2a). The relatively low REE contents in POR-5 and the prominent positive Eu anomaly reveal conspicuous plagioclase accumulation in the parental magma [e.g., *Ross and Elthon*, 1997]. The negative Eu anomaly displayed by POR-8 is indicative of plagioclase fractionation as usually observed in oceanic tholeiitic lavas. The relatively flat REE patterns of POR-10 and POR-11 coincide with those of representative normal (N) to transi-

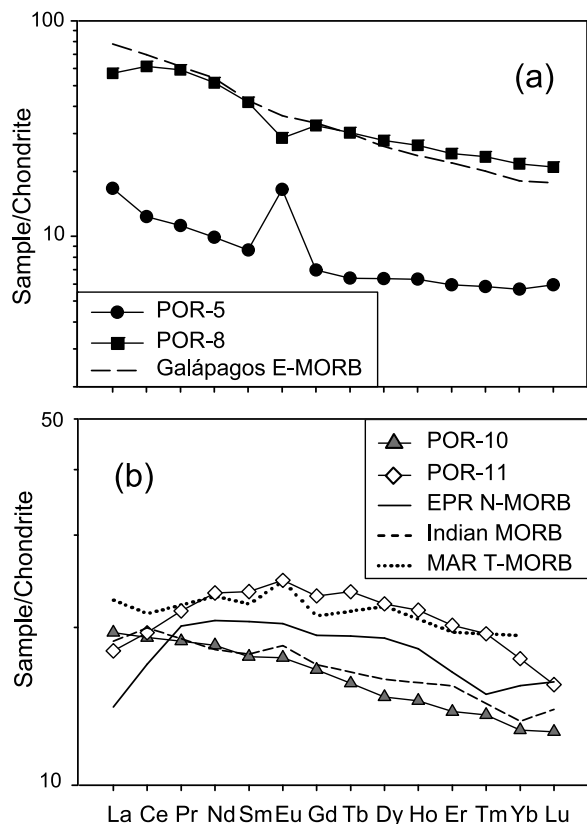


Figure 2. Chondrite-normalized abundances of REE in the BAA dated samples. Black circle, POR-5; black square, POR-8; gray triangle, POR-10; white diamond, POR-11. Normalizing values are from *Sun and McDonough* [1989]. (a) Representative composition of EMORB lavas from the Galápagos Islands (long-dashed line) from *Geist et al.* [2008]; (b) compositions of East Pacific Rise (EPR) NMORB (solid line), Indian MORB (short-dashed line) and Mid-Atlantic Ridge (MAR) TMORB (dotted line) are from *Klein* [2003, and references therein].

tional (T) MORB from different ocean floors worldwide (Figure 2b).

[15] The samples dated are highly deformed metamorphic mafic rocks with evident foliation, at times accompanied by stretching lineation, being difficult to recognize the intrusive (gabbroic) or extrusive (basaltic) origin of their protoliths. We remark however that *Pin et al.* [2008] distinguished gabbroic and metabasaltic rocks in the BAA unit and concluded that these rocks have clearly different geochemical signatures: metabasalts have higher HREE than gabbros (Figure 3) and their Sr-Nd isotopic composition indicates a broadly oceanic origin that is much less influenced by crustal contamination than in gabbros. The whole-rock composition of POR-5 (and in particular its relatively low HREE abundances) is strongly similar to that of the BAA gabbros and the BIC upper gabbros (Figure 3) studied by *Pin et al.* [2008]. On the other hand, the trace element patterns of POR-8, POR-10, and POR-11 coincide with

those of the broadly oceanic BAA metabasalts analyzed by *Pin et al.* [2008] (Figure 3). Contrary to the supposition of *Pin and Rodríguez*, this fact indicates that only POR-5 is geochemically akin to the crustally contaminated gabbros from the BAA unit and the BIC studied by *Pin et al.* [2008], while POR-8, POR-10, and POR-11 are strongly similar to rocks classified by these authors as metabasalts of broadly oceanic origin. Therefore, the U-Pb zircon ages of samples POR-8, POR-10, and POR-11 were correctly interpreted by *Azor et al.* [2008] as the crystallization ages of oceanic rocks cropping out in the BAA unit.

4.3. Assessment of Crustal Contamination in the Dated Samples

[16] LREE enrichment and Zr negative anomaly in the trace element normalized patterns have been interpreted by *Pin et al.* [2008] as possible evidences of crustal contamination in the mafic rocks of the BAA unit; the Sr-Nd isotopic signatures of these rocks reinforce their conclusion especially for the variably deformed intrusive gabbros [*Pin et al.*, 2008]. The chondrite-normalized patterns of POR-5 and POR-8 have these compositional characteristics (Figure 2a and 3) that may suggest assimilation of crustal material during the ascent of their parental magmas. *Pin et al.* [2008] concluded that possible crustal contamination does not support the model of an oceanic derivation for the BAA gabbros, and *Pin and Rodríguez* argue that the presence of rare xenocrystic or inherited zircon crystals (POR-5) or cores (POR-11) is an additional argument to cast doubts on the oceanic origin of the samples investigated by *Azor et al.* [2008]. We stress here that the inclusion of host continental rocks during magma ascent was already proposed in section 5.1 of *Azor et al.* [2008] to explain the occurrence of these old zircon grains, whose presence is not inconsistent with an oceanic setting as inherited zircons

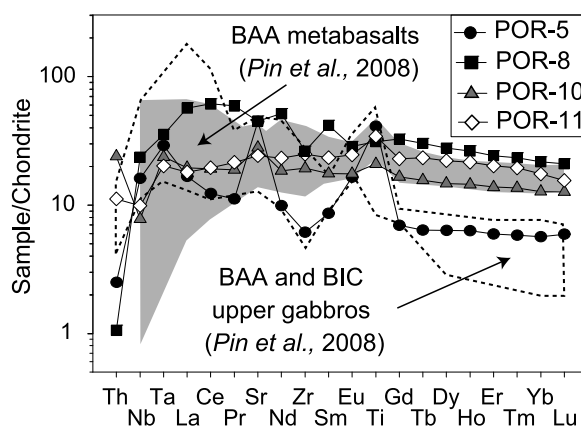


Figure 3. Chondrite-normalized trace element patterns of the BAA dated samples. Symbols are as in Figure 2. Normalizing values are from *Sun and McDonough* [1989]. The short-dashed area encloses the compositional range of the BAA and BIC upper gabbros, while the shaded area depicts the one of the BAA metabasalts from *Pin et al.* [2008].

have been detected in gabbros from the Mid-Atlantic Ridge [Pilot *et al.*, 1998]. Moreover, crustal contamination in mantle-derived magmas is highly coherent with our preferred tectonic scenario for the BAA unit [Azor *et al.*, 2008, section 5.1, paragraph 20]: “the general tectonic regime in southern Iberia at Early Carboniferous times seems to be an extensional/transensional one, where volcanism, gabbroic magma formation (Beja Gabbros), ephemeral oceanic crust generation (BAA unit) ... would have occurred on a thinned continental crust”. Therefore, any evidence for possible crustal contamination in these samples does not invalidate at all our conclusions relating these rocks to continental rifting and short-lived oceanization. Finally, Pin *et al.* [2008] considered the absence of Nb positive anomaly in the trace element patterns of the BIC gabbros to be a piece of evidence against the mantle plume model that we proposed. However, these gabbros are clearly contaminated by the continental crust, thus being not suitable to infer the geochemical signature of their mantle source. Instead, more analyses of uncontaminated BAA rocks should be performed to this end. ICP-MS analyses are recommended as Nb measurement by XRF as performed by Pin *et al.* [2008] is highly imprecise for concentrations lower than 10 ppm [e.g., Münker, 1998].

5. Geological Implications

[17] Three main points constraining the geological interpretation of the BAA unit have been discussed: the accuracy of our geochronological data, their geographical/geological significance and their geochemistry. We conclude, reinforcing the conclusions of Azor *et al.* [2008] that the Early Carboniferous ages reported are robust and within error of the intrusion age of the BAA protoliths. A significant Pb loss and discordance can be discarded due to the Gaussian distribution shown by the population data used for age calculation in each sample. The assumption by Pin and Rodríguez that we did not collect truly oceanic rocks, but only non-MORB metagabbros representing the deformed southern rim of the BIC, can be ruled out drawing on the geographical/geological location of our samples and the geochemical signature of three of them. Therefore, both metagabbros and MORB-featured metabasites with variable degrees of crustal contamination are Lower Carboniferous in age, thus excluding the previous interpretation of the BAA unit as an ophiolite of the Rheic Ocean. Instead, the BAA unit represents ocean-like crust generated in an ephemeral rift corridor between the Ossa-Morena and the South Portuguese zones.

[18] In their comment, Pin and Rodríguez mistakenly consider the mafic rocks included in the so-called Moura phyllonitic complex as equivalent to the BAA rocks. The

BAA unit and the Moura phyllonitic complex are in fact two units displaying many differences in outcrop, lithology, geochemistry and tectonometamorphic evolution [e.g., Araújo *et al.*, 2005]. Nevertheless, we concur with Pin and Rodríguez that the MORB-featured rocks included in the Moura phyllonitic complex, together with the basalts of the Pulo do Lobo unit, are still potential remnants of the Rheic Ocean in Southern Iberia, although absolute ages are needed to prove it. Azor *et al.* [2008] state that the Ossa-Morena/South Portuguese Zone boundary might still be viewed as a cryptic suture of the Rheic Ocean, though the BAA unit itself has to be completely discarded as a Rheic ophiolite.

[19] Our BAA ages and the ages available for the BIC indicate that both belong to the same magmatic suite. Our data, however, inextricably lead to a discussion on the time span between the ages of the BIC and those of the BAA unit. Pin and Rodríguez invoke an age of ca. 352 Ma for the BAA rocks that we have investigated, on the basis of U-Pb geochronological work done in the neighboring BIC [Pin *et al.*, 1999, 2008; Jesus *et al.*, 2007]. On the contrary, we believe that there is no essential disagreement between the 340–332 Ma old mafic magmatism in the BAA unit and a slightly older magmatism in the BIC, i.e., the BAA unit and the BIC may well be part of the same magmatic suite, extended in time over several million years. In this respect, the stronger contamination by the continental crust deduced for BAA and BIC gabbros compared with other metabasites in the BAA unit may indicate that the gabbros record the very earlier stages of ephemeral oceanization in southern Iberia.

[20] Pin and Rodríguez also discuss the large-scale tectonic scenario for southern Iberia in Early Carboniferous times by confronting a mantle plume hypothesis [Azor *et al.*, 2008] to a slab break-off one [Pin *et al.*, 2008]. We will not elaborate this point because it has little to do with both our main conclusions and the main issues raised by Pin and Rodríguez. Nevertheless, we would like to emphasize that the extent of Early Carboniferous magmatism and coeval sedimentation in southern Iberia, affecting the South Portuguese and Ossa-Morena zones, as well as the southernmost part of the Central Iberian Zone and neighboring regions, in pre-Mesozoic reconstructions, of the Canadian Maritimes and Morocco, makes the slab break-off hypothesis highly implausible.

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