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Comment on “Rheic Ocean ophiolitic remnants in southern Iberia questioned by SHRIMP U-Pb zircon ages on the Beja-Acebuches amphibolites” by A. Azor et al.

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1. Introduction

[1] In a recent contribution, *Azor et al.* [2008] present new U-Pb zircon age data obtained with the Sensitive High Resolution Ion Microprobe (SHRIMP) technique on four mafic rocks from the so-called Beja-Acebuches amphibolites (BAA) unit, believed to delineate a major suture between the Ossa-Morena Zone (OMZ) and the South Portuguese Zone (SPZ) of the Iberian Variscides. On the basis of their geochronological results, interpreted to reflect igneous crystallization of mid-ocean ridge basalt (MORB)-featured mafic protoliths from 332 ± 3 to 340 ± 4 Ma, Azor et al. (paragraph 1) conclude that the Beja-Acebuches amphibolite unit represents “a narrow and very ephemeral realm of oceanic-like crust that opened in Early Carboniferous times” as a result of the involvement of “a mantle plume underneath southern Iberia.”

[2] It is our opinion, however, that the dated samples were not appropriately characterized as to their geodynamic affinities and, more importantly, that the interpretation of the raw isotopic data is questionable. For these reasons, several assertions reported in this paper are not adequately supported by the data presented and should probably be reconsidered in the light of a more critical assessment of the data and other published lines of evidence available for this region.

2. Geological Setting and Characterization of the Studied Samples

[3] Azor et al. claim (paragraph 17) that their U-Pb data bring new pieces of information on the timing of MORB-featured mafic magmatism in this region. However, *Azor et al.* [2008] do not provide any evidence supporting their view that the four dated samples have an oceanic derivation,

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although they were clearly aware that the “geochemical data from the BAA are abundant, though their interpretation is far from being straightforward” (paragraph 9), as shown by earlier trace element and Nd isotope work [*Pin et al.*, 1999]. Indeed, geochemical and isotopic data documented conspicuous differences between the metagabbros and the metabasalts of the BAA unit (implying that these components do not belong to an ophiolitic suite) and showed that only few of the metabasalts are similar to NMORBs. Specifically, the samples dated by Azor et al. included, beside a gabbro (POR-5), two medium-grained amphibolites (POR-8 and POR-10) and a strongly foliated fine-grained amphibolite (POR-11) which might well correspond to variously deformed metagabbros. Only a brief petrographic description is given for these samples, without any attempt to further characterize the igneous affinities and the geodynamic significance of their protoliths. Considering that all the metagabbroic samples studied in this region are clearly not MORB-featured [*Pin et al.*, 1999, 2008] but instead display great geochemical and Sr-Nd isotopic similarities with samples from the postcollisional, crustally contaminated Beja Igneous Complex (BIC), it is inferred that *Azor et al.* [2008] actually did not study any rock of oceanic derivation. One might suggest that some of the strongly foliated amphibolites studied by *Azor et al.* [2008] really represent oceanic metabasalts. In this case, however, it is not clear why they should contain primary igneous zircons, bearing in mind the extreme rarity, not to say, the typical absence, of zircon crystallized from MORB-type magmas. For this reason, zircons found in ocean ridge metabasalts might be of metamorphic origin (if not xenocrystic), implying that the ages obtained could reflect the metamorphic event responsible for the transformation of basaltic protoliths into amphibolites. It is noticed however that well-preserved oscillatory zoning in cathodoluminescence images and high Th/U ratios, as typically observed in magmatic zircons, do not favor this interpretation.

[4] Instead, it seems most likely that the samples they dated belong to the deformed, southern margin of the BIC, close to the major tectonic boundary with the South Portuguese Zone, a possibility already evoked by *Figueiras et al.* [2002], following earlier suggestions by *Andrade* [1983, p. 73] and *Silva* [1996].

[5] In the absence of geochemical data for the samples dated by *Azor et al.* [2008], this alternative interpretation in terms of crustally contaminated mafic rocks is supported by the presence of ancient zircons in samples POR-5 (one grain at 533 ± 7 Ma) and POR-11 (four grains with $^{206}\text{Pb}/^{238}\text{U}$ ages ranging from 0.6 to 2.7 Ga). Bearing in mind the

solubility of zircons in hot, basaltic magmas, these ancient grains most likely reflect late stage crustal assimilation at the time of igneous emplacement.

[6] *Azor et al.* [2008, paragraph 11] correctly state, concerning the southern margin of the BIC, that “the relationships between the Beja Gabbros and the BAA unit cannot be discerned in the field, since they are separated by faults.” However, clearly intrusive contacts of the north-eastern margin of the BIC are beautifully exposed in the Guadiana river bed immediately downstream of Moinhos dos Doutorinhos, SW of Brinches [*Fonseca*, 1995]. There, a network of undeformed dioritic veins cuts across strongly foliated, banded amphibolites of the Moura Schists Unit, interpreted as a north verging accretionary complex [*Araljo et al.*, 2005]. Geochemical data obtained in the vicinity of this outcrop [*Pin et al.*, 2008] highlight the marked contrast between the posttectonic gabbro diorites of the Beja Igneous Complex (BIC) and the pre-tectonic amphibolites, which bear great similarities with broadly EMORB-like amphibolites occurring in the BAA unit, on the southern side of the Beja gabbro. The simplest geological interpretation of these observations is to consider that the BIC was emplaced as a late stage intrusion into an earlier deformed and metamorphosed accretionary complex containing metabasalts of broadly oceanic derivation. Subsequently, the BIC was reworked at its southern margin, close to the transpressional boundary between the Ossa-Morena and South Portuguese zones.

[7] In short, *Azor et al.* [2008, paragraph 23] failed to demonstrate that their samples were indeed “MORB-featured rocks,” as stated in their paper. In contrast, published geochemical and Sr-Nd evidence on similar rocks, along with the occurrence of xenocrystic zircons in two of their samples, strongly suggest that the dated rocks rather belong to the southern rim of the late stage Beja Igneous Complex, implying that all the further inferences on the “Rheic Ocean ophiolitic remnants in southern Iberia” discussed in the rest of their paper have no really sound basis.

3. U-Pb SHRIMP Data

3.1. Brief Theoretical Background

[8] The basic principles of U-Pb dating tell us that the three dates which can be derived from the measurements of $^{206}\text{Pb}/^{238}\text{U}$, $^{207}\text{Pb}/^{235}\text{U}$ ratios, and the $^{207}\text{Pb}/^{206}\text{Pb}$ ratio, merely represent apparent ages, which are not geologically significant unless they are identical (“concordant”), thereby documenting that the U-Pb system behaved as a closed system. In most zircons, however, the three measured ages disagree, with $T_{206/238} < T_{207/235} < T_{207/206}$, reflecting a partial opening of the radiometric clock, with preferential loss of daughter lead isotopes (not well accepted in the zircon lattice) relative to parent uranium isotopes (substituted to Zr at the time of igneous crystallization). By virtue of the existence of two independent, but perfectly coupled on geochemical grounds, radiometric clocks (i.e., ^{238}U - ^{206}Pb and ^{235}U - ^{207}Pb), a reliable crystallization age nevertheless can be computed from samples which were partially opened, a unique property of the U-Pb system. Following the

approach pioneered by *Wetherill* [1956], this can be achieved by combining the two chronometers on a suite of cogenetic zircons, that usually provide, in a $^{206}\text{Pb}/^{238}\text{U}$ versus $^{207}\text{Pb}/^{235}\text{U}$ diagram, data points defining a linear array whose “upper intercept” with the theoretical Concordia curve (defined as the locus of $^{207}\text{Pb}/^{235}\text{U}$ - $^{206}\text{Pb}/^{238}\text{U}$ points corresponding to a given age) gives the crystallization age. This requires the precise and accurate measurements of Pb/U elemental ratios, as can be achieved by the isotope dilution mass spectrometry (IDMS) method, and the determination of the $^{207}\text{Pb}/^{206}\text{Pb}$ isotopic ratio, which can be measured very precisely and accurately by thermal ionization mass spectrometry (TIMS), provided that contamination during sample preparation (that is, dissolution and chemical isolation of Pb) was kept to a negligible level. During the last decades, different mechanical [*Krogh*, 1982] or chemical [*Mattinson*, 2005] approaches have been developed to remove as much as possible, prior to U-Pb analyses, those zircon domains which suffered Pb loss, in an attempt to reduce the degree of discordance of $T_{206/238}$ and $T_{207/235}$ ages. This allows extremely precise and meaningful geological ages to be obtained by using the so-called “conventional” ID-TIMS method [e.g., *Schaltegger et al.*, 2008].

[9] With the advent and rapid development of in situ techniques (SHRIMP and laser ablation ICP-MS), an increasing number of U-Pb ages have become available, which generally proved to be reliable, albeit inherently less precise due to counting statistics constraints reflecting the much smaller amount of sample consumed. Because they allow U-Pb ages to be obtained in a relatively straightforward way, without demanding chemical procedures, microprobes based on ion sputtering or laser ablation are highly attractive, with the added benefit of ~ 20 - μm -scale spatial resolution which proves to be invaluable in the case of complex zircons with inherited cores or multistage growth. However, the use of microsampling techniques does not remove entirely the problem of radiogenic Pb loss, and therefore does not alleviate the need to consider $^{206}\text{Pb}/^{238}\text{U}$ ages as minimum estimates of the true crystallization ages. This is because the scale of the crystalline domains which behaved as closed systems for the U-Pb radiometric clock is in general far beyond the spatial resolution which can be achieved by ion or laser ablation microprobes.

[10] In addition, some potential pitfalls specific to these in situ techniques have been overlooked in certain circumstances, which may prove to be troublesome in the case of relatively recent zircons, such as those dealt with by *Azor et al.* [2008]. Specifically, the much shorter half-life of ^{235}U (0.704×10^9 years) compared to that of ^{238}U (4.468×10^9 years) has two important implications for Phanerozoic zircon dating. First, after rapid decay during Early Precambrian times, there is very little ^{235}U left and the amount of ^{207}Pb atoms produced by radioactive decay in relatively recent zircons is fairly small (e.g., a 300 Ma old zircon contains ~ 18 times less ^{207}Pb than a 2 Ga old zircon with the same U concentration). Therefore, the ^{207}Pb ion beams during in situ measurements of young zircons are weak, and plagued by poor precision due to counting statistics. More-

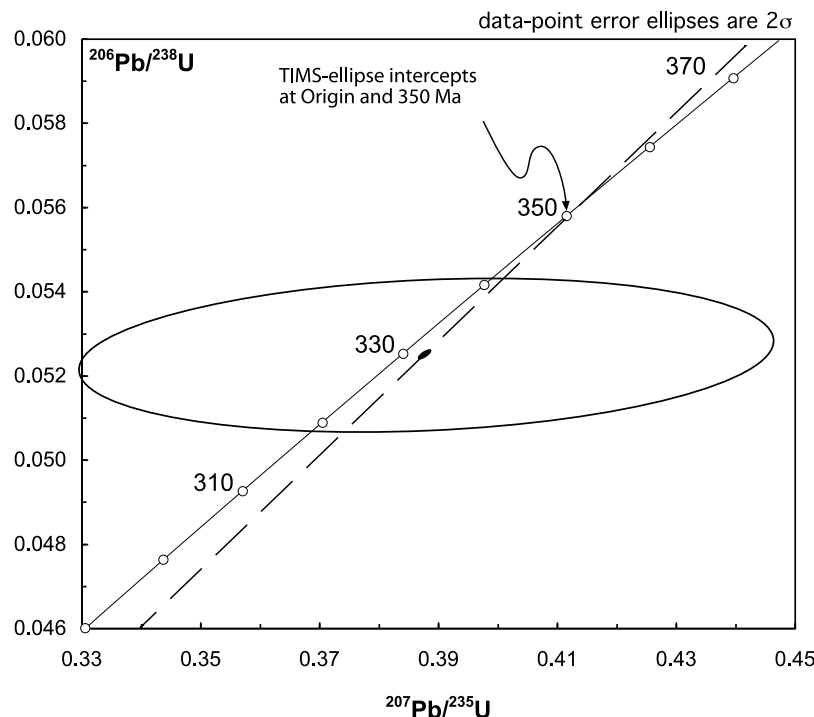


Figure 1. Concordia diagram illustrating the potential effects of contrasting ID-TIMS and SHRIMP analytical uncertainties on the interpretation of U-Pb data for Late Paleozoic zircons. For simple comparative purposes, the data point with error ellipses typical for both analytical methods is plotted for a 350 Ma old zircon which suffered some radiogenic lead loss in very recent times. This data point gives slightly discordant apparent $^{206}\text{Pb}/^{238}\text{U}$ and $^{207}\text{Pb}/^{235}\text{U}$ ages of 330.0 and 332.7 Ma, respectively, corresponding to a $^{207}\text{Pb}/^{206}\text{Pb}$ age of 350 Ma. The small error ellipse (in black) corresponds to the mean of the precisions quoted (at the 95% confidence level) for the 12 zircon fractions from mafic and intermediate samples of the Beja Igneous Complex analyzed by ID-TIMS [Pin *et al.*, 2008], specifically 0.17% for $^{206}\text{Pb}/^{238}\text{U}$ and 0.21% for $^{207}\text{Pb}/^{235}\text{U}$, with a correlation coefficient of 0.8. The larger error ellipse was drawn from the mean of the SHRIMP precisions listed by Azor *et al.* [2008] for their 69 spots free of inheritance, specifically 2.8% for $^{206}\text{Pb}/^{238}\text{U}$ and 12% for $^{207}\text{Pb}/^{235}\text{U}$ (also quoted at the 95% confidence level), with a correlation coefficient of 0.2. It can be seen that due to greater analytical uncertainty, the SHRIMP analysis overlaps the Concordia curve, thereby allowing consideration of the 330 Ma $^{206}\text{Pb}/^{238}\text{U}$ apparent age as geologically significant, although the true age is 350 Ma in this example. It is further noticed that pooling the data of some 10–20 spots with broadly similar characteristics, as usually done for SHRIMP measurements, would considerably improve the precision of the calculated age (e.g., to ± 5 Ma, or even better), without remedying the problem of accuracy (i.e., negative age bias of 20 Ma) inherent to the poor determination of ^{207}Pb , and the resulting impossibility to recognize that the U-Pb radiometric clock did not behave as a closed system. Error ellipses are drawn by using the ISOPLOT program [Ludwig, 2003].

over, at least occasionally, the relative contribution of non-radiogenic lead to the total ^{207}Pb signal cannot be neglected, and its correction is not a straightforward task. For these reasons, the $^{207}\text{Pb}/^{235}\text{U}$ and $^{207}\text{Pb}/^{206}\text{Pb}$ ratios have large analytical uncertainties for young zircons, and do not provide much valuable information, leaving the analyst with $^{206}\text{Pb}/^{238}\text{U}$ ages (that is, apparent, potentially too young ages) as the only usable data. Second, the Concordia curve displays almost no curvature in the 0–500 Ma age range, implying that discordant zircons, lying on a lead loss trajectory (or a two-component mixing line) very close to, and almost parallel to the Concordia curve, cannot be resolved from really concordant grains. In other words,

the “myopia” of SHRIMP or LA-ICP-MS (highlighted by large error ellipses) may easily lead to consider as concordant (that is, documenting a closed system behavior of U-Pb isotopes), and by inference, geologically significant, $^{206}\text{Pb}/^{238}\text{U}$ and $^{207}\text{Pb}/^{235}\text{U}$ ages which are in fact discordant, thereby biasing the quoted age toward spuriously young values (Figure 1).

3.2. Critical Assessment of U-Pb SHRIMP Results

[11] In their discussion of the timing of mafic magmatism, Azor *et al.* [2008] ignore these theoretical constraints and consider their ages in the 332–340 Ma range as geologically meaningful, and related to the crystallization

of the igneous protoliths. Only 8 spots (of a total of 69 spots free of inheritance) giving obviously too young ages (as low as 297 Ma) were “excluded from the age calculation because of suspected Pb loss” (Azor et al.’s Table 1), implying that all the 61 other spots were considered as concordant and thereby geologically significant, which requires a certain degree of faith.

[12] The significantly older ID-TIMS U-Pb dates previously reported for the Beja Igneous Complex at circa 352 Ma for the major pulse [Pin et al., 1999] are interpreted by Azor et al. [2008, paragraph 20] to reflect “the presence of some inheritance . . . not detected by conventional isotope dilution analysis.” However, no evidence for any inheritance or xenocrystic zircons has been found in the different samples dated by Pin et al. [1999, 2008], irrespective of the variable degree of crustal contamination they suffered (as monitored by their Sr-Nd isotope signature). Indeed, the same 352 ± 2 Ma age has been measured in a fairly primitive gabbro diorite (with $^{87}\text{Sr}/^{86}\text{Sr}_i = 0.7045$ and $\varepsilon\text{Nd}_i = +2.5 \pm 1$), a granodiorite (with $^{87}\text{Sr}/^{86}\text{Sr}_i = 0.7057$ and $\varepsilon\text{Nd}_i = -2.0$), and a strongly contaminated aplitic leucodiorite (with $^{87}\text{Sr}/^{86}\text{Sr}_i = 0.7093$ and $\varepsilon\text{Nd}_i = -6.1$). This absence of evidence for ancient zircons is in keeping with the high solubility of zircon in hot, metaluminous magmas, which should not allow inherited or xenocrystic grains to survive in the internal parts of a large, slowly cooling body. It is noteworthy that the zircons extracted from a felsic dike sampled in the BAA gave a slightly younger date of 345 ± 2 Ma, interpreted to reflect the waning stage of igneous activity in this area, in good agreement with the 342 ± 9 Ma SHRIMP zircon age obtained by Jesus et al. [2007] from an amphibole-bearing pegmatite dike, cutting the layered gabbros of the BIC, and interpreted as a late stage, fluid-rich residual melt. Therefore, it seems likely that the 332–340 Ma ages reported by Azor et al. [2008] are too young to reflect igneous crystallization. Indeed, these ages fit the range of $^{39}\text{Ar}/^{40}\text{Ar}$ ages (328–342 Ma) measured on amphiboles from both the Beja Igneous Complex and its relatively high-grade metamorphic country rocks [Dallmeyer et al., 1993; Castro et al., 1999], that are therefore best interpreted in terms of regional cooling below the $\sim 500^\circ\text{C}$ isotherm. However, Azor et al. [2008, paragraph 21] prefer to reject, without further discussion, the older $^{39}\text{Ar}/^{40}\text{Ar}$ ages as “unrealistic, since they overlap with (their) protolith ages,” a somewhat circular reasoning.

[13] There are two possible, nonmutually exclusive explanations for the probable bias toward the too young ages obtained by Azor et al. [2008].

[14] First, as emphasized in section 3.1, it is virtually impossible to assess whether the data points are really concordant, or whether they plot to the right of the Concordia curve (due to Pb loss) and merely overlap that curve by virtue of the large size of their analytical error ellipses (Figure 1). This is a major limitation of in situ dating methods in this age range. For this reason, it is reemphasized that the reported ages are basically $^{206}\text{Pb}/^{238}\text{U}$ ages, which should strictly be considered as minimum ages.

[15] Second, a cursory examination of the Concordia diagrams displayed in Figure 3 of Azor et al. [2008] shows

that several data points have been included in the age calculations in spite of the fact they plot above the Concordia curve. A closer examination of Azor et al.’s Table 1 confirms this observation. Indeed, the $T_{207/235}$ ages calculated from the $^{207}\text{Pb}/^{235}\text{U}$ ratios listed by Azor et al. for those spots free of inheritance show that even though the $T_{207/235}$ and $T_{206/238}$ ages may overlap within analytical uncertainty in many cases, only 8 spots give normally discordant data points (i.e., with $T_{207/235} > T_{206/238}$). If interpreted in terms of geochemical process, this largely predominant reverse discordance would imply a preferential loss of U relative to Pb, a very unlikely scenario for zircons. An analytical artifact may provide a better explanation for these anomalous data points. Whether the reversely discordant points reflect problems of calibration of Pb/U ratios during the analytical session, or an overcorrection for non-radiogenic lead (for example, due to an interference at m/z 204, in so far as the “204 method” was used to correct for common lead), or both, is beyond the scope of the present discussion.

[16] Surprisingly, the analytical methods used to obtain the radiometric data are not described, nor even evoked (e.g., as a reference to another, better documented paper) by Azor et al. [2008]. In particular, the standard(s) and the method used to calibrate the measurement of the Pb/U elemental ratios are not specified (even though the determination of Pb/U is a well-known Achilles’ heel of in situ dating techniques, due to major instrumental fractionation [e.g., Stern, 1997; Williams, 1998]), nor the method used to correct for nonradiogenic lead. The procedure used for data reduction and age calculation is not documented either. It also can be noticed that the heading of Azor et al.’s Table 1 appears to be incomplete, as inferred from the first column labeled “ ^{206}Pb %,” presumably referring to the relative abundance of “common” (i.e., nonradiogenic) lead in each spot analysis. Azor et al. (paragraph 13) also state that in sample POR-5, “three other analyses, one of them on a bright rim, yielded apparent younger ages down to 219 Ma, which are suspected of Pb loss, possibly related to metamorphism.” The analytical results corresponding to this 219 Ma spot are missing in Azor et al.’s Table 1, but this Triassic apparent age demonstrates that in spite of the absence of any metamorphic overprinting after the Variscan orogeny in this region, significant disturbances of the U-Pb system occurred in some of the studied zircons at a geologically recent (Mesozoic and/or Cenozoic) time. This should have incited the authors to exercise greater caution when interpreting their measured $^{206}\text{Pb}/^{238}\text{U}$ dates in terms of igneous crystallization ages.

4. Geological Interpretations and Conclusion

[17] On the basis of these questionable age data, Azor et al. [2008, paragraphs 22 and 23] claim that “the BAA unit cannot represent an oceanic realm related to the Rheic Ocean,” that “the Rheic suture is not represented at all in the BAA unit,” and that there are “some doubts on the tectonic meaning of the OMZ/SPZ boundary.” These assertions are not justified for two reasons: first, as discussed in

section 2, their data were most likely not obtained on oceanic rocks, but rather on postcollisional intrusives, and therefore are not directly relevant to the problem of the putative Rheic Ocean in that region; second, the interpretation of the broad OMZ/SPZ boundary zone in terms of an oceanic suture is well documented by the typical NMORBs occurring as tectonic slices south of Aracena [e.g., *Castro et al.*, 1996; *Pin et al.*, 2008], and as blocks in the Peramora Melange of the Pulo do Lobo unit [*Eden*, 1991; *Eden and Andrews*, 1990]. Further, the occurrence of HP/LT metamorphism in the southern part of the Ossa-Morena Zone in Portugal provides circumstantial evidence for an oceanic suture in this area [e.g., *De Jong et al.*, 1991; *Fonseca et al.*, 1999; *Booth-Rea et al.*, 2006]. The Middle to Late Devonian age of the Peramora Melange [*Eden and Andrews*, 1990] and the Late Devonian radiometric ages measured for the HP/LT metamorphism [*Moita et al.*, 2005] imply that the intervening oceanic lithosphere between the OMZ and SPZ was at least Mid-Devonian in age. This is indeed acknowledged by Azor et al. (paragraph 23) when they state that “the OMZ/SPZ boundary might still represent a cryptic suture.”

[18] *Azor et al.* [2008, paragraph 22] “favor the interpretation of the BAA unit as a narrow and very ephemeral realm of oceanic-like crust” generated in the 330–340 Ma time span. They further speculate (paragraph 26) that “the extensional/transensional event responsible for the formation of the BAA ophiolitic unit” occurred in the large-scale context of “a mantle plume affecting all of the European Variscan Belt” (paragraph 27). This interpretation reiterates, without further evidence, a model already proposed in two previous papers of the same group [*Simancas et al.*, 2003, 2006]. However, as discussed by *Pin et al.* [2008], this plume model is not supported by the geochemical data available for mafic igneous rocks. Alternative interpretations based on purely plate tectonic processes (e.g., slab break-off following consumption of oceanic lithosphere and continent-continent collision in Late Devonian times) may

probably account in a more realistic way for the observed geochemical features and the inferred sequence of events [*Pin et al.*, 2008].

[19] Nevertheless, the data reported by *Azor et al.* [2008] bring a valuable contribution to the problem of the so-called Beja-Acebuches Ophiolitic Complex (BAOC). They indeed confirm the suspicion, based on geochemical and Sr-Nd isotope data [*Pin et al.*, 1999, 2008], that the BAOC is a composite assemblage gathering minor slices of truly oceanic metabasalts of unknown (albeit probably pre-Mid-Devonian) age, and predominant mafic intrusives of Early Carboniferous age. These metagabbros are best interpreted as the deformed southern margin of the crustally contaminated, circa 352 Ma old Beja Igneous Complex. It is inferred that this large, elongated intrusive body was emplaced, following the Late Devonian collision between the Ossa-Morena and South Portuguese Zones, in a broad accretionary prism containing slices of NMORBs. This domain documents a real, albeit somewhat cryptic, oceanic suture, which later evolved into a transpressive strike-slip boundary.

[20] On general grounds, this discussion offers an opportunity to emphasize that the accuracy of U-Pb ages measured by using in situ techniques in relatively young zircons might not always be as good as the quoted precisions suggest, due to the great difficulties in assessing whether or not the U-Pb system remained closed in the microsample analyzed. For this reason, it is suggested that $^{206}\text{Pb}/^{238}\text{U}$ ages should conservatively be viewed as minimum estimates of the crystallization ages, in accordance with the theoretical requirements of U-Pb dating.

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