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Isotopic geochronology of granitic rocks from the Central Iberian Zone: comparison of methodologies

Geocronología isotópica de rocas graníticas de la Zona Centro-Ibérica: comparación de metodologías

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ABSTRACT

Five granitic rocks, concentrically disposed from core to rim, were distinguished in the Castelo Branco pluton. U-Pb-Th electron microprobe monazite ages from granitic rocks are similar and ranging between 297-303 Ma. The granitic rocks from Castelo Branco pluton are 310 ± 1 Ma old, obtained by U-Pb (ID-TIMS) in separated zircon and monazite crystals, indicating a similar emplacement age for all granitic rocks of the pluton. Initial $^{87}Sr/^{86}Sr$ isotopic ratios and εNd_{310} and $\delta^{18}O$ values suggest three distinct pulses of granitic magma and that they are derived from partial melting of heterogeneous metasedimentary materials. The other granitic rocks are related by magmatic differentiation and show small variations in $(^{87}Sr/^{86}Sr)_{310}$, εNd_{310} and $\delta^{18}O$. The granitic pluton of Castelo Branco shows a rare reverse zoning.

Key words: geochronology, zircon, monazite, U-Pb, U-Pb-Th, Sr-Nd, $\delta^{18}O$, Castelo Branco pluton, Portugal.

RESUMEN

En el plutón de Castelo Branco, se distinguen cinco granitoides, dispuestos concéntricamente de núcleo a borde del plutón. Las edades U-Pb-Th obtenidas en cristales de monacita por microsonda electrónica en estos granitoides son similares entre sí y varían entre 297 y 303 Ma. Los resultados de datación por U-Pb (ID-TIMS) en cristales de circón y de monacita de los tres granitos seleccionados, indican una edad de implantación de 310 ± 1 Ma y que son rocas emplazadas simultáneamente. Las relaciones isotópicas iniciales de $^{87}Sr/^{86}Sr$ y los valores de εNd_{310} y $\delta^{18}O$ de los tres pulsos magmáticos son característicos de granitos resultantes de anatexia cortical a partir de rocas metasedimentarias heterogéneas. En la secuencia de diferenciación magmática, las rocas graníticas presentan pequeñas variaciones en $(^{87}Sr/^{86}Sr)_{310}$, εNd_{310} y $\delta^{18}O$. El plutón de Castelo Branco presenta un zonado inverso poco frecuente.

Palabras clave: geocronología, circón, monazita, U-Pb, U-Pb-Th, Sr-Nd, $\delta^{18}O$, Castelo Branco plutón, Portugal.

Introduction

The emplacement age of granitic rocks can be obtained using recent analytical techniques of high precision. This allows the identification of important geologic episodes and constitutes an important component in the study of the evolution of orogenic processes and crustal materials. U-Pb-Th age determination from electron microprobe geochemical analyses of monazites is a relatively recent technique with relevant application on the geochronology of magmatic episodes (Montel *et al.*, 1996). This is a fast and cost-effective methodology, with good resolution, being able to date crystals with 5 µm diameter and allowing the study of heterogeneities inside of the same crystal. Minerals are not destructed and the textural relationship of the sample is preserved. The main disadvantage can be related to the lesser precision and minor sensitivity presented by this methodology. Therefore, U-Pb-Th monazite

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datation can be considered as a first approach to relative information on the age of a rock.

Associated to the necessity of a more accurate and precise analytical techniques, the U-Pb isotopic data of zircon and monazite crystals by ID-TIMS (Isotope Dilution Thermal Ionization Mass Spectrometry) became more usual. In this methodology, the radiogenic Pb (^{207}Pb and ^{206}Pb), produced by U isotopic decay, allows to get two independent ages and a third one, not independent, from the isotopic ^{207}Pb/^{235}U and ^{206}Pb/^{238}U ratios, and corresponding to the ^{207}Pb/^{206}Pb ratio (Rollinson, 1993). These three different ages obtained using the same mineral, confers a high validity to the U-Pb system. In U-enriched and Pb-poor minerals, like zircon and monazite, the amount of initial common Pb is insignificant relatively to the radiogenic Pb, which supports the application to these minerals.

Strontium and Nd occur in different amounts in the rocks, occurring as trace elements, and dispersed in SiO_2-enriched rocks. Rb-Sr datation method is applied in the geochronology of granitic rocks. Rb-Sr and Sm-Nd isotopic data are important geochronological indicators. Initial ^{87}Sr/^{86}Sr and εNd_T isotopic magma ratios are characteristic of the magma sources and will remain constant during fractional crystallization processes (Rollinson, 1993).

Whole-rock oxygen isotopic data (δ^{18}O) provides important information relative to the conditions of origin and emplacement of rocks. These data have been particularly used together with geochemical and petrogenetic studies on the magmatic identification and characterization processes.

The Castelo Branco pluton consists of five concentrically zoned peraluminous granitic rocks. The aim of this work is to present and to interpret the different isotopic data (U-Pb-Th; U-Pb; Rb-Sr, Sm-Nd and δ^{18}O) obtained in the granitic rocks of Castelo Branco pluton. These data will be used to infer the emplacement age of the pluton as well as information on its source.

Geological setting

The Central Iberian Zone (CIZ) is located at the central part of the Iberian Massif. This zone contains Pre-Cambrian to Lower Palaeozoic rocks and two different domains can be identified: The Ollo de Sapo formation and the Schist-Metagraywacke Complex (CXG) (Martínez Catalán *et al.*, 2004). The Castelo Branco pluton intruded the Cambrian schist-metagraywacke complex, which consists of alternating metapelites and metagraywackes with metaconglomerate and marble intercalations.

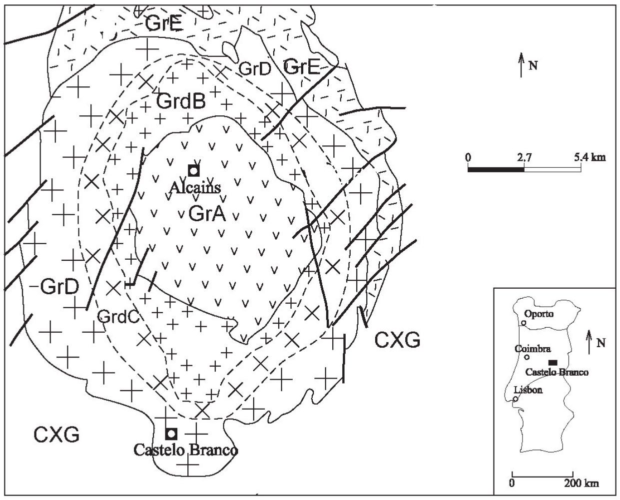

Fig. 1.—Geological map of the Castelo Branco pluton (adaptated from Antunes *et al.*, 2008). CXG-Schist-metagraywacke complex; GrA: medium- to fine-grained muscovite>biotite granite; GrdB: medium- to fine-grained slightly porphyritic biotite>muscovite granodiorite; GrdC: medium- to coarse-grained porphyritic biotite>muscovite granodiorite; GrD: medium- to coarse-grained porphyritic biotite-muscovite granite; GrE: coarse-grained muscovite>biotite granite.

Several classifications have been presented for the granitoid rocks of the CIZ. The CIZ granitoids have been emplaced mainly during and after the ductile deformation phase D3 of Namurian-Westphalian age (Ferreira *et al.*, 1987). They are classified according to their emplacement ages as syn-D3, late-D3, late- to post-D3 and post-D3 (e.g., Neiva & Gomes, 2001; Dias *et al.*, 2002; Azevedo *et al.*, 2005).

The Castelo Branco pluton is exposed over an area of 390 km^2 and consists of five concentric late-tectonic Variscan granitic rocks, with an age of 310 ± 1 Ma and presenting synchronism of installation (Fig. 1).

A medium- to fine-grained muscovite > biotite granite (GrA) occupies the pluton's core and is the oldest one. Granite GrA is surrounded by a medium- to fine-grained slightly porphyritic biotite > muscovite granodiorite (GrdB). The contact between granite GrA and granodiorite GrdB is sharp. The amount of feldspar phenocrysts increases towards the porphyritic biotite > muscovite granodiorite (GrdC). The medium- to coarse-grained granodiorite GrdC encircles gradually the granodiorite GrdB, grading to the medium- to coarse-grained porphyritic biotite-muscovite granite (GrD), which contains equal amounts of biotite and muscovite (Fig. 1). A coarse-grained muscovite > biotite granite (GrE) occupies the periphery of the pluton in the sector from N to E (Fig. 1). The contact between granites GrD and GrE is also sharp, but the two granites are usually altered at this contact. The pluton produced mica schists in the outer zone and a pelitic hornfels in the inner zone of the contact metamorphic aureole, particularly on the southern and eastern zones of the pluton.

Analytical methods

The U-Pb-Th monazite individual ages are calculated from the U, Pb and Th monazite concentrations obtained by electron microprobe on thin sections. This age can be considered valid if, at the time of crystallization, the Pb is practically inexistent and losses of Pb, Th and U had not occurred. The U-Pb-Th determinations on monazites were performed on a Cameca SX100 electron microprobe at the Laboratoire Magmas et Volcans of the University of Blaise Pascal, Clermont-Ferrand (France). Analytical conditions included an accelerating voltage of 15 kV and a beam current of 150 nA. Standards used included UO_2 (U Mα), ThO_2 (Th Mα), apatite (Ca Kα, P Kα), zircon (Si Kα) and synthetic phosphates (Y Lα, La Lα, Ce Lα, Pr Lα, Nd Lα, Sm Lα, Gd Lα). The theoretical basis and associated statistical treatment of data follow the analytical procedure detailed by Montel *et al.* (1996). A least-square modelling approach was applied to the ages of the crystals and its quality is assessed from the mean square of weighted deviates (MSWD).

U-Pb isotopic data were determined using zircon and monazite separated crystals from selected representative granitic rocks of Castelo Branco pluton. Zircon and monazite separation was carried out by a combination of magnetic separation and flotation in heavy liquids. The U-Pb isotopic results for zircon and monazite were obtained by isotope dilution thermal ionization mass spectrometry (TIMS) using a Finnigan-Mat 262 spectrometer at the Department of Geosciences, University of Oslo, Norway, following the standard procedure of Krogh (1973) with the adaptations described by Corfu (2004).

The Sr and Nd isotope analyses were obtained at the Centro de Instrumentación Científica of the University of Granada, Spain. Samples were digested using ultra-clean reagents and analyzed by TIMS in a Finnigan Mat 262 spectrometer, after chromatographic separation with ion-exchange resins. Normalization values were $^{86}Sr/^{88}Sr$ = 0.1194 and $^{146}Nd/^{144}Nd$ = 0.7219 and the blanks were 0.6 and 0.09 nanograms for Sr and Nd, respectively. The standards yields values of $^{87}Sr/^{86}Sr$ = 0.710252 ± 0.000021 (2σ) NBS 987 and $^{143}Nd/^{144}Nd$ = 0.511844 ± 0.000010 (2σ) La Jolla. $^{87}Rb/^{86}Sr$ and $^{147}Sm/^{144}Nd$ were determined by ICP-MS following the method developed by Montero & Bea (1998), with a precision better than ± 1.2% and 0.9% (2σ), respectively.

Oxygen isotope analyses of whole-rock samples were carried out at the Department of Earth Sciences, University of Western Ontario, Canada, using a conventional extraction line and employing chlorine trifluoride as reagent. This methodology has a precision of ±0.2‰ and the used standards include quartz and laboratory CO_2.

Isotope geochemistry

To compare different isotopic data and associated emplacement ages for the granitic rocks of Castelo Branco pluton, three different geochronological techniques were used. It will be also presented the whole-rock Rb-Sr, Sm-Nd and $\delta^{18}O$ isotopic ratios from granitic rocks to characterize the magmatic sources.

U-Th-Pb on monazite

Electron microprobe U-Th-Pb isotopic data on monazite, considers that monazite contains small amounts of common Pb, resulting from Th and U isotopic decay. Th and U enrichment of monazite promotes a radiogenic Pb accumulation on the mineral structure, remaining in precise amounts during less than 100 Ma (Montel *et al.*, 1996).

Monazites in all granitic rocks of the Castelo Branco pluton were dated by EPMA (Table 1). Those in samples GrA, GrdB, GrdC and GrE yield average U-Th-Pb ages of 303 Ma to 301 Ma. U-Pb-Th monazite ages from granite GrD is somewhat lower at 297 ± 3 Ma (Table 1). However, the granitic rocks obtained ages are similar and consistently within the analytical error (Table 1).

The highest MSWD values obtained for GrD and GrE granites can be associated with the number of

Table 1.—**U-Pb-Th monazite ages of granitic rocks from Castelo Branco pluton (central Portugal)**

	T (Ma)	MSWD	N
GrA	303 ± 3 Ma	9	31
GrdB	301 ± 4 Ma	2	38
GrdC	301 ± 4 Ma	3	32
GrD	297 ± 3 Ma	35	45
GrE	301 ± 3 Ma	14	49

GrA: muscovite > biotite granite.
GrdB: slightly porphyritic biotite > muscovite granodiorite.
GrdC: porphyritic biotite > muscovite granodiorite.
GrD: biotite-muscovite granite.
GrE: muscovite > biotite granite.
N: number of analyses.

points and some scatter, which may reflect some initial Pb associated with unresolved geological complexity or due to alteration processes.

U-Pb on zircon and monazite

U-Pb isotopic analyses were carried out on separated zircon and monazite crystals from three samples representing the GrA, GrdB and GrE of Castelo Branco pluton, using the ID-TIMS method (Table 2). Granodiorite GrdC and granite GrD were not considered because field relationships and geochemical characteristics suggest that they are related to granodiorite GrdB by a fractional crystallization process (Antunes *et al.*, 2008). U-Pb zircon and monazite ages inside the granitic rocks are consistent and similar (Table 2).

The zircon crystals from granite GrA are concordant, defining a good alignment (MSWD = 1.11) and yielding an emplacement age of 309.9 ± 1.1 Ma. The more concordant zircon yields a $^{207}Pb/^{206}Pb$ age of 309.9 ± 1 Ma with a discordance of 1.1% (Table 2).

The monazite crystals plot slightly reversely discordant, a fact commonly linked to Palaeozoic geological processes. The $^{207}Pb/^{235}U$ ratio is not affected by this disequilibrium effect and can be used as the closest estimate for the age of the monazite (Table 2).

The zircon analyses for a sample of granodiorite GrdB plot on or close to the Concordia curve. However, one zircon crystal suggests minor inheritance and the age of 310.1 ± 0.8 Ma from the more concordant zircon should be considered (Table 2). The monazite and zircon crystals yields a similar age (Table 2).

The zircon analyses from granite GrE defines a discordia line which yields a lower intersect age of 309.1 ± 0.6 Ma and an upper intersect age of about 1,000 Ma. The emplacement age is similar to the $^{207}Pb/^{206}Pb$ age for the more concordant zircon crystal (Table 2). The monazite analyses scatter towards the Concordia curve, however the more concordant monazite yields and age of 309.7 ± 0.4 Ma, similar to the concordant zircon ages (Table 2).

Considering the U-Pb zircon and monazite isotopic data of the granitic rocks from Castelo Branco pluton and according to the age uncertainties associated to the method, it could indicate a 310 Ma emplacement for these granitic rocks. This suggests that the granitic rocks from Castelo Branco pluton are contemporaneous (Table 2).

Table 2.—**U-Pb zircon and monazite ages of granitic rocks from Castelo Branco pluton (central Portugal)**

	Zircon age (Ma)[1] (MSWD)	Zircon age (Ma)[2] $^{207}Pb/^{206}Pb$	Monazite age (Ma)[3] $^{207}Pb/^{235}U$	Zircon + monazite age (Ma)[4] (MSWD)
GrA	309.9 ± 1.1 Ma (MSWD = 1.11)	309.9 ± 1.0 Ma	309.5 ± 0.9 Ma	Not defined
GrdB	Not defined	310.1 ± 0.8 Ma	310.6 ± 1.5 Ma	307 ± 28 Ma (MSWD = 1.20)
GrE	309.1 ± 0.6 (MSWD = 0.11)	309.7 ± 0.4 Ma	309.7 ± 0.4 Ma	Not defined

GrA: muscovite > biotite granite.
GrdB: slightly porphyritic biotite > muscovite granodiorite.
GrE: muscovite > biotite granite.

[1] Zircon concordia ages.
[2] $^{207}Pb/^{206}Pb$ age of concordant zircon crystal.
[3] $^{207}Pb/^{235}U$ age of concordant monazite crystal.
[4] Zircon and monazite concordia ages.

Whole rock Rb-Sr, Sm-Nd and $\delta^{18}O$

The average values of initial $^{87}Sr/^{86}Sr$ and εNd_T isotopic ratios were calculated using the U-Pb zircon and monazite age of 310 Ma (Table 3). The isotopic ratios and whole-rock granitic $\delta^{18}O$ values are characteristic of rocks resulting by crustal anatexis from metasedimentary materials. The muscovite>biotite granite GrA, slightly porphyritic biotite>muscovite granodiorite GrdB and muscovite>biotite granite GrE (Fig. 1) correspond to three distinct pulses of granite magma. The porphyritic biotite>muscovite granodiorite GrdC and biotite-muscovite granite GrD are the products of in situ fractional crystallization of granodiorite magma GrdB controlled by fractionation of plagioclase, quartz, biotite and ilmenite (Antunes *et al.*, 2008).

The granitic rocks GrA, GrdB and GrE have distinct initial $^{87}Sr/^{86}Sr$ ratios, εNd_{310} and $\delta^{18}O$ values (Table 3), indicating that they are related to distinct pulses of magma, associated with the heterogeneous Lower Palaeozoic pelitic country rock.

The $(^{87}Sr/^{86}Sr)_{310}$ and εNd_{310} variations on the magmatic sequence between GrdB, GrdC and GrD are small (Table 3) and whole-rock $\delta^{18}O$ values increase from granodiorite GrdB (12.27‰) to granite GrD (12.75‰), which can be associated with the fractional crystallization process (e.g., White, 2003).

The Castelo Branco pluton shows an uncommon reverse zoning. The oldest muscovite>biotite GrA intruded the core of the pluton. Granodiorite GrdB magma intruded and originated the biotite>muscovite granodiorite GrdC and biotite-muscovite granite GrD towards the pluton outer zone. Muscovite>biotite granite GrE correspond to another magmatic pulse, which forms some external parts of the pluton.

Table 3.—**Average values of $(^{87}Sr/^{86}Sr)_{310}$, εNd_{310} and $\delta^{18}O$ of granitic rocks from Castelo Branco pluton (central Portugal)**

	$(^{87}Sr/^{86}Sr)_{310}$	εNd_{310}	$\delta^{18}O$ (‰)
GrA	0.7090	–3.8	13.53
GrdB	0.7108	–1.7	12.27
GrdC	0.7104	–0.8	12.50
GrD	0.7086	–2.8	12.75
GrE	0.7120	–3.0	12.91

GrA: muscovite > biotite granite.
GrdB: slightly porphyritic biotite > muscovite granodiorite.
GrdC: porphyritic biotite > muscovite granodiorite.
GrD: biotite-muscovite granite.
GrE: muscovite>biotite granite.

Conclusions

1. U-Pb ID-TIMS isotopic zircon and monazite ages are the more precise. However, U-Pb-Th monazite ages obtained by electron microprobe provide similar ages.
2. The granitic rocks GrA, GrdB and GrE, which are related to three distinct magma pulses, have a similar U-Pb ID-TIMS zircon and monazite age of 310 ± 1 Ma.
3. These granitic rocks have distinct values of $(^{87}Sr/^{86}Sr)_{310}$, εNd_{310} and $\delta^{18}O$.
4. The granitic rocks GrdB, GrdC and GrD are related by a fractional crystallization process and present small variations in the $(^{87}Sr/^{86}Sr)_{310}$, εNd_{310} and $\delta^{18}O$ isotopic values.
5. Castelo Branco pluton shows an uncommon reverse zoning and the first one known in Portugal.

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